SAMPLING AND ANALYSIS PLAN
For
LONG-TERM MONITORING OF
SURFACE WATER AND GROUNDWATER

BALLARD, HENRY, AND ENOCH VALLEY MINES

FINAL REVISION 1

Henry Mine: Little Blackfoot River, MST045

April 2015

P4 PRODUCTION, LLC

Prepared by: Prepared by:
MWH Americas, Inc.

TABLE OF CONTENTS

TABLE OF CONTENTS ..... **i**

LIST OF TABLES ..... **i**

LIST OF FIGURES ..... **ii**

LIST OF APPENDICES ..... **ii**

ACRONYOMS AND ABBREVIATIONS ..... **iii**

1.0 INTRODUCTION **1-1**

2.0 BACKGROUND **2-1**

3.0 DATA QUALITY OBJECTIVES FOR LONG-TERM MONITORING OF SURFACE WATER AND GROUNDWATER **3-1**

4.0 LONG-TERM MONITORING LOCATIONS AND ANALYTES **4-1**

4.1 LTM SAMPLE LOCATIONS - SURFACE WATER 4-1

4.2 LTM ANALYTICAL LIST - SURFACE WATER 4-1

4.3 SURFACE WATER - SEASONAL SAMPLING EVALUATION 4-2

4.4 LTM SAMPLE LOCATIONS - GROUNDWATER 4-2

4.5 LTM ANALYTICAL LIST - GROUNDWATER 4-2

4.6 GROUNDWATER - SEASONAL SAMPLING EVALUATION 4-3

5.0 REPORTING **5-1**

6.0 REFERENCES **6-1**

TABLES **6-1**

FIGURES **6-2**

APPENDICES **6-3**

LIST OF TABLES

Table 3-1 Surface Water and Groundwater Monitoring Data Quality Objectives

Table 4-1 LTM Surface Water Monitoring Locations, Frequency, and Schedule

Table 4-2 LTM Surface Water and Groundwater Analyte List

Table 4-3 Spring LTM Sample Tracker for Surface Water Stations

Table 4-4 LTM Groundwater Monitoring Well and Borehole Well Locations, Frequency, and Schedule

Table 4-5 LTM Sample Tracker for Monitoring Wells and Borehole Wells

LIST OF FIGURES

Figure 1-1 P4 Mines Vicinity Map

Figure 3-1 LTM Surface Water and Groundwater Sample Locations at P4 Sites

Figure 4-1 LTM Sample Locations Ballard Mine Site

Figure 4-2 LTM Sample Locations Enoch Valley Mine Site

Figure 4-3 LTM Sample Locations Henry Mine Site

LIST OF APPENDICES

Appendix A Long-Term Monitoring Surface Water and Ground Water Monitoring Field Sampling Plan

Appendix B Long-Term Monitoring Surface Water and Groundwater Quality Assurance Project Plan

Appendix C Ballard, Henry, and Enoch Valley Mines Remedial Investigation and Feasibility Study Health and Safety Plan

ACRONYOMS AND ABBREVIATIONS

A/Ts	Agencies and Tribes
BRA	Baseline Risk Assessment
CO/AOC	Consent Order/Administrative Order on Consent
COPC	Contaminant of Potential Concern
COPEC	Contaminant of Potential Ecological Concern
DQO	Data Quality Objective
DSRs	Data Summary Reports
e.g.	exempli gratia
FSP	Field Sampling Plan
GW	Groundwater
HSP	Health and Safety Plan
LTM	Long-Term Monitoring
P4	P4 Production L.L.C.
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation and Feasibility Study
SAP	Sampling and Analysis Plan
SOW	Statement of Work
SW	Surface Water
TDS	Total Dissolved Solids
Tribes	Shoshone-Bannock Tribes
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

This document presents the Sampling and Analysis Plan (SAP) for Long-Term Monitoring (LTM) of surface water (SW) and groundwater (GW) at the P4 Production, L.L.C. (P4) mines near Soda Springs, Idaho. This SAP has been prepared, in accordance with the requirements of the Administrative Settlement Agreement and Order on Consent/Consent Order for Remedial Investigation/Feasibility Study (CO/AOC) (2009 CO/AOC; USEPA, 2009). The mines include the inactive properties of Ballard, Henry, and Enoch Valley Mines, collectively referred to as the Sites (refer to Figure 1-1).

This SAP defines the LTM network of relevant SW and GW stations and analytical parameters that will provide an annual assessment of SW and GW conditions downstream of potential sources associated with the Sites. The components of this SAP include the field sampling plan (FSP), Quality Assurance Project Plan (QAPP), and Health and Safety Plan (HSP). The FSP, QAPP, and HSP are included in Appendix A, B, and C, respectively.

A brief background on monitoring and characterization efforts at the Sites is provided in Section 2.0. The data quality objectives (DQOs) for the LTM program are presented in Section 3.0. Proposed monitoring locations, analyte lists, and supporting rationale are provided in Section 4.0. Reporting schedules are provided in Section 5.0.

2.0 BACKGROUND

Monitoring and characterization of SW and GW in association with the Remedial Investigation/Feasibility Study (RI/FS) at the Sites has been conducted since 2004. The intent of SW and GW monitoring has been to characterize SW/GW impacts, evaluate SW/GW interaction, and refine the Sites hydrogeologic conceptual models to be utilized in the individual Site RI/FS evaluations. The specific objectives of SW and GW monitoring have evolved over time as detailed below.

Surface Water Monitoring. From 2004 through 2008, SW monitoring supported characterization of nature and extent of impacts to water quality associated with releases of contaminants of potential concern (COPCs) and contaminants of potential ecological concern (COPECs) from Site sources. In 2009, the SW monitoring program transitioned to an interim monitoring program that provided an annual/bi-annual assessment of SW conditions downstream of potential source areas and supported remedy selection in the FS for both SW and source area cleanup.

Groundwater Monitoring. From 2004 to 2012, GW monitoring supported characterization of nature and extent of COPCs/COPECs in GW. Starting in 2013, sampling and monitoring of COPC/COPEC concentrations in GW supported the evaluation of potential future Site remedial actions, similar to the SW sampling objective.

Review of the 2014 Proposed P4 Long-Term Surface Water and Groundwater Monitoring Plan Memo (*2014 LTM Memo*; MWH, 2014a) resulted in the following comment from the Agencies and Tribes (A/Ts):

> *"EPA's QA Office has reviewed the proposed monitoring plan, which is tiered to the approved 2009/2010 SAP and QAPP. It appears that methods and reporting limits are current and adequate to meet project requirements. In addition, data validation and QA/QC procedures have not changed and are adequate to meet project requirements.*
>
> *As a note for future reference, EPA's QA office expects QA plans to be closed out after a period of five years, and then re-issued with any necessary changes. This ensures that plans remain up-to-date. For long-term sampling in 2015, please plan to re-issue the SAP."*

This stand-alone SAP fulfills the United States Environmental Protection Agency's (USEPA) expectation by providing an updated FSP, QAPP, and HSP that will guide the LTM program through the next five year period, unless superseded by another document.

3.0 DATA QUALITY OBJECTIVES FOR LONG-TERM MONITORING OF SURFACE WATER AND GROUNDWATER

The DQOs discussed in this section identify the objectives and quality of data that must be obtained during continued LTM of SW and GW at the Sites that guide development of the LTM plan and support the decision making process of the RI/FS program.

The conceptual site model (CSM) used to develop the DQOs is detailed in Table 3-1 (Step 1). The DQOs specifically have been developed to guide the screening and selection of monitoring stations and analytes for LTM of SW and GW. The proposed SW and GW sample locations identified through this process are presented on Figure 3-1.

Conceptually, DQOs are established through a seven-step process, as outlined by USEPA guidance (USEPA, 2006). Those steps include:

1. State the problem
2. Identify the goals of the study
3. Identify information inputs
4. Define the boundaries of the study
5. Develop the analytic approach
6. Specify performance or acceptance criteria
7. Develop the plan for obtaining data

Each step of the DQO process defines criteria that will be used to establish the final data collection design. The first five steps presented above are primarily focused on identifying qualitative criteria, such as:

- The nature of the problem that has initiated the study and a conceptual model of the environmental hazard to be investigated;
- The decisions or estimates that need to be made and the order of priority for resolving them;
- The type of data needed; and
- An analytic approach or decision rule that defines the logic for how the data will be used to draw conclusions from the study findings (USEPA, 2006).

The sixth step establishes acceptable quantitative criteria on the quality and quantity of the data to be collected, relative to the ultimate use of the data. For the SW and GW LTM program, the data are primarily collected for the measurement of COPC/COPEC concentration downstream and downgradient of potential P4 source areas to evaluate long-term trends, and as such, the uncertainty in the data will be estimated and evaluated once collected. Current USEPA-approved methods will be used for estimation of the level of uncertainty associated with the data.

In the seventh step of the DQO process, a data collection design is developed that will generate data meeting the quantitative and qualitative criteria specified at the end of Step 6. The output from this step is largely contained in the FSP.

4.0 LONG-TERM MONITORING LOCATIONS AND ANALYTES

The purpose of this SAP is to:

- Identify a network of SW and GW LTM stations, whose water quality is influenced by potential P4 sources,
- Identify an appropriate analyte list based on past sampling, and
- Monitor and further establish long-term data trends

Therefore, the locations and analytes used during past monitoring events were evaluated in the DQO process for relevance in this LTM program (refer to Section 3.0).

4.1 LTM SAMPLE LOCATIONS - SURFACE WATER

Selection of LTM locations for SW sampling utilized rationale presented in previous SW SAPs for the Sites in which the number and locations of SW monitoring sites evolved over time. With this in mind, the LTM program for SW will follow the 2013/2014 monitoring programs with no changes made to the SW sampling program and station selection. Sample collection and analysis will be performed according to the methods and procedures outlined in the FSP (see Appendix A). Table 4-1 presents the list of LTM SW locations. Figure 3-1 presents the LTM SW locations for all three Sites, 36 total, on a topographic map. Figures 4-1 through 4-3 present the SW locations on an aerial, photographic background for each individual Site.

4.2 LTM ANALYTICAL LIST - SURFACE WATER

Selection of the LTM SW analyte list utilized detailed analyte screening and analyte selection rationale presented in past SW SAPs for the Sites, and the Ballard RI Report Baseline Risk Assessment (*Ballard Mine RI Report BRA*, MWH 2014b). The LTM analyte list consists of analytes that are pervasively elevated above promulgated surface water standards/background (e.g., cadmium) and indicate potential impacts from the Site (e.g., sulfate and total dissolved solids [TDS]).

The LTM SW program will follow the 2013/2014 monitoring programs with one exception: the removal of vanadium from the analyte list. Vanadium was removed from the LTM analyte list based on the COPC/COPEC screening performed in the *Ballard Mine RI BRA*, which eliminated this constituent from further evaluation in the risk assessment. Initial comparison of vanadium concentrations in SW samples at Henry and Enoch Valley Sites also show that vanadium will not be a primary risk driver in their respective risk assessments.

The sample collection and analysis will be performed according to the methods and procedures outlined in the FSP included as Appendix A. The recommended LTM SW analyte and water quality list are presented in Tables 4-1, 4-2, and 4-3.

4.3 SURFACE WATER - SEASONAL SAMPLING EVALUATION

As shown on Table 4-1, a subset of the SW monitoring locations will be evaluated after the spring LTM event to determine whether they will be sampled in the fall LTM event. Those sampling locations with consistently higher selenium and sulfate concentrations in the fall, than observed in the spring, will be retained for fall sampling. The fall sampling decision will use both the current sampling year's spring LTM data and historical data to determine which stations, if any, should be sampled in the fall. A LTM Fall Sample Tracker table will be prepared and provided to the A/Ts at least three weeks prior to the fall sample event. On dry or drought years many of the selected locations may be dry during the fall round of sampling and no sample will be collected.

4.4 LTM SAMPLE LOCATIONS - GROUNDWATER

Selection of LTM GW sampling locations utilized location selection rationale presented in previous GW SAPs for the Sites. With this in mind, the LTM GW program will follow the 2014 monitoring program with one change made to the GW station location selection: locations MBW011 and SB-07 will not be sampled for the data gap specific analytes identified in the *2014 LTM Memo*.

The sample collection and analysis will be performed according to the methods and procedures outlined in the FSP (see Appendix A). Table 4-4 presents the list of LTM GW locations. Figure 3-1 shows the LTM GW locations for all three Sites on a topographic map. Figures 4-1 through 4-3 present the GW locations on an aerial, photographic background for each individual Site.

4.5 LTM ANALYTICAL LIST - GROUNDWATER

Selection of the LTM GW analyte list utilized detailed analyte screening and analyte selection rationale presented in previous GW SAPs for the Sites and the *Ballard Mine RI Report BRA*. The LTM analyte list consists of analytes that are elevated above promulgated groundwater standards/background (e.g., cadmium, selenium) at the Sites.

The sample collection and analysis will be performed according to the methods and procedures outlined in the FSP (see Appendix A). The recommended LTM GW analyte and physical water quality parameter lists are presented in Tables 4-2, 4-4, and 4-5.

4.6 GROUNDWATER - SEASONAL SAMPLING EVALUATION

The *2009 GW SAP* (MWH, 2009) included a seasonal evaluation of the spring and fall selenium and sulfate data collected in 2004, 2007, and 2008 at spatially relevant GW sample locations. This evaluation supported the decision to sample some stations in the fall as well as the spring. The 2009 evaluation included the retention and fall sampling of two GW well locations (MMW010 and MMW013). The 2010 evaluation included the retention and fall sampling of three GW well locations (MMW010, MMW013, and MMW029). The 2012 evaluation concluded that no additional GW sampling events in the fall were necessary based on the historical selenium and sulfate data review for MMW010, MMW013, and MMW029. Therefore the LTM program for GW does not include fall sampling.

5.0 REPORTING

P4 will continue to submit all validated data and data validation summaries for each sampling effort within 120 days of sample collection as required by Task B Data Management in the 2009 AOC/CO Scope of Work (SOW). In compliance with the 2009 CO/AOC, Data Summary Reports (DSRs) will be submitted each year to the A/Ts within 120 days of completion of the season's field work or within 90 days of the receipt of the final laboratory data, whichever is earlier.

6.0 REFERENCES

MHW, 2009. 2009 Groundwater Monitoring Sampling and Analysis Plan. August 2009.

MWH, 2014a. 2014 Proposed P4 Long-Term Surface Water and Groundwater Monitoring Plan Memo. April 2014.

MWH, 2014b. Remedial Investigation Report for P4's Ballard Mine Final Revision 2. November 2014.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4. U.S. Environmental Protection Agency, Washington DC, EPA/240/B-06/001.

USEPA, 2009. Administrative Settlement Agreement and Order on Consent/Consent Order for Performance of Remedial Investigation and Feasibility Study at the Enoch, Henry, and Ballard Mine Sites in Southeastern Idaho. United States Environmental Protection Agency, U.S. EPA Region 10, Idaho Department of Environmental Quality, United States Department of Agriculture, Forest Service Region 4, United States Department of the Interior, Bureau of Land Management, Shoshone-Bannock Tribes, in the Matter of Enoch Valley Mine, Henry Mine, Ballard Mine, P4 Production, L.L.C., Respondent. Effective Date of November 30, 2009

TABLES

TABLE 3-1 SURFACE WATER AND GROUNDWATER MONITORING DATA QAULITY OBJECTIVES	
Step 1 - State the Problem	Historical mining operations have resulted in mine waste materials in mine dumps throughout the Sites being potential sources of selenium and other COPCs/COPECs in the environment. In general, the predominant mobilization mechanism for COPCs/ COPECs from these potential source areas occur during the spring runoff when melting snow percolates into and through the mine dumps materials and dissolved constituents move from the Sites in SW and shallow GW. P4 and the A/T's believe there is adequate SW and GW data to support the development of RI/FS reports and preparation of human and ecological risk assessments for each of the Sites. Continued SW and GW monitoring downgradient of t h e P4 sources is needed to provide an annual baseline record of conditions, both spatially and temporally, in order to more effectively evaluate long-term trends and the fate and transport of dissolved constituents in the shallow alluvial GW and SW. These efforts will assist in refinement of the conceptual hydrogeologic models of the Sites and the identification of potential future remedial measures. Following implementation of individual Site remedies, the long-term SW/GW data will help managers evaluate the effectiveness/performance of the Selected Remedy. The primary components of the conceptual model that support the DQOs are summarized as follows for SW and GW: • Source – COPCs/COECs present in interburden and overburden rocks deposited in waste rock dumps, but possibly present in mine pits and other facilities that are able to enter SW and GW due to runoff and infiltration. • Release mechanisms – Exposure to air and water results in mobilization of COPCs/COECs from increased surface area; precipitation may leach and mobilize COPCs/COECs primarily during spring runoff to receiving SW and GW. • Exposure pathways – COPC/COPEC exposure through ingestion, dermal contact (for SW) irrigation and uptake by plants • Receptors – Livestock, wildlife, or human The planning team, decision makers and principal data users include P4 and the A/T.

TABLE 3-1 SURFACE WATER AND GROUNDWATER MONITORING DATA QAULITY OBJECTIVES	
Step 2 – Identify the Goals of the Study	Principal Study Question #1 Are temporal trends in SW and GW quality associated with potential P4 sources being adequately monitored by the monitoring station network? Alternative actions: 1. Evaluation indicates that the monitoring network can be optimized and the number of downgradient stations and/or analytes adjusted for future long-term SW and GW monitoring. Data will be forwarded to the respective Site RI and FS report. 2. No Action - station data and analyte list are of adequate quality, quantity, and in proper locations to characterize potential P4 source areas. Data will be forwarded to the respective Site RI and FS report. Decision statement: Determine whether monitoring stations and analytes allow for sufficient characterization of changes in upgradient P4 sources and will allow development of potential future remedies. If not, modify the number, analyte list, and/or locations of the monitoring stations sampled; otherwise, the existing stations and analyte list will continue to be sampled.
Step 3 – Identify Information Inputs	The information inputs for the decision process include the following items that may exist or will need to be created. • List of analytes • Current conceptual Site models (i.e., for each of the Sites) • Existing facilities investigation information to be updated as needed (inventory and spatial delineation of potential source areas)

TABLE 3-1 SURFACE WATER AND GROUNDWATER MONITORING DATA QAULITY OBJECTIVES	
	• Sample location maps (contained in FSP) • Historic SW and GW data will be analyzed using the appropriate methods for determining inorganic concentrations with detection limits suitable for comparison to risk-based screening levels • Hydrogeologic information such as flow directions and flow rates. • Previous SW and GW quality sampling and analysis plans and procedures • risk-based screening benchmarks for analytes
Step 4 – Define the Boundaries of the Study	<u>Population of Interest:</u> Metals/metalloids and general water quality parameters in SW and GW <u>Spatial boundaries:</u> SW and GW monitoring locations downgradient of potential source areas at Ballard, Henry, and Enoch Valley Sites <u>Vertical boundary:</u> Mid-depth of the river or stream at SW monitoring locations; screen interval depth for GW monitoring locations <u>Temporal boundary:</u> LTM SW and GW sample collection is planned for spring during the peak flow (i.e., peak potential loading) for all proposed stations and in the fall for those SW stations that have higher historical selenium and sulfate concentrations at that time of year. <u>Scale of Decision Making:</u> Spatially-relevant SW and GW monitoring locations from each sampling event <u>Practical Constraints</u> Sampling to occur during peak flow typically in late April or May

TABLE 3-1 SURFACE WATER AND GROUNDWATER MONITORING DATA QAULITY OBJECTIVES	
Step 5 – Develop the Analytic Approach	<u>Principal Study Question #1</u> If a monitoring station and/or analyte are not required to evaluate potential impacts to SW and GW from potential upgradient P4 sources, then the need for the monitoring points and/or analytical parameters will be evaluated. Otherwise, the proposed locations and analytes will be considered adequate for the intended purpose of establishing effective monitoring for potential future remedies.
Step 6 – Specify Performance or Acceptance Criteria	A SW and GW sampling station located downstream or downgradient of a potential P4 source area solely characterizing potential P4 SW and GW quality impacts will be retained for effectiveness monitoring. Analytical parameters will be compared to the applicable SW and GW quality risk-based screening level in data summary reports prepared annually. The precision, accuracy, representativeness, comparability, and completeness criteria and the minimum detection limits will be used to evaluate the usability of analytical data in making decisions about potential impacts to SW and GW downstream or downgradient of potential source areas from P4 mine related activities and at a minimum will help ensure that data collected for the interim monitoring will be of comparable quality when compared to the Site characterization data. Measurement errors will be minimized by implementing standard operating procedures for the sample collection, handling, preparation and analysis methods. All data must meet approved usability as defined in the QAPP in Appendix B.
Step 7 – Develop the Plan for Obtaining Data	The existing SW and GW data were reviewed as part of the preparation of this SAP. The sampling rationale and design, based on existing data, is presented in Section 3.0 of this SAP. The sampling design will be further evaluated if SW and GW monitoring suggests that the proposed locations and analytes are not sufficient to determine the effectiveness of LTM for potential future remedies. The field methods and quality assurance requirements are presented in the FSP located in Appendix A.

TABLE 4-1
LTM SURFACE WATER MONITORING LOCATIONS, FREQUENCY, AND SCHEDULE

Mine	Station ID[a]	Feature Name[b]	Location[c]		Sample Schedule	
			Latitude	Longitude	Spring	Fall[d]
Ballard Mine	MST019	Blackfoot River below Ballard Creek	42 48 49.28	111 30 21.52	X	TBD
	MST020	Blackfoot River below State Land Creek	42 48 31.97	111 30 06.28	X	TBD
	MST050	*Long Valley Creek* below Ballard Mine	42 50 54.00	111 29 49.00	X	
	MST066	*Ballard Creek* above Blackfoot River	42 48 56.30	111 30 07.32	X	
	MST067	*Ballard Creek* headwaters	42 49 23.79	111 29 36.31	X	
	MST069	*Short Creek* below Ballard Mine	42 49 11.23	111 29 19.75	X	TBD
	MST089	*Wooley Valley Creek*, below *North Fork Wooley Valley Creek*	42 49 28.70	111 26 19.40	X	
	MST090	*Wooley Valley Creek*, above *North Fork Wooley Valley Creek*	42 49 28.00	111 26 49.00	X	
	MST092	*North Fork Wooley Valley Creek* above *Wooley Valley Creek*	42 49 40.00	111 27 04.30	X	
	MST094	*Spring-fed trib. #1 of N. Fork Wooley Valley Cr.,* below Ballard Mine	42 49 56.00	111 28 10.00	X	
	MST095	*Spring-fed trib. #2 of N. Fork Wooley Valley Cr.,* below Ballard Mine	42 49 39.00	111 28 05.00	X	
	MST096	Tributary of North Fork Wooley Valley Creek, below Ballard Mine	42 49 30.00	111 27 45.00	X	
	MDS030	*Pit #2 Upper Dump Seep*	42 49 44.25	111 29 20.95	X	TBD
	MSG004	*Holmgren Spring*	42 49 16.75	111 28 16.04	X	TBD
	MSG005[f]	*Cattle Spring*	42 49 19.07	111 28 01.90	X	
	MSG006	*Southeast Spring*	42 49 41.43	111 27 51.34	X	
	MSG007	*South of Southeast Spring*	42 49 38.19	111 27 42.68	X	
Henry Mine	MST044	Immediately below Henry Mine (1997 #24)	42 53 50.60	111 29 24.80	X	TBD
	MST045	Little Blackfoot River above Henry Creek	42 54 10.70	111 29 30.10	X	TBD
	MST051	East Fork Long Valley Creek below Henry Mine	42 52 18.23	111 28 58.49	X	
	MST057	West Fork Lone Pine Creek above Lone Pine Creek	42 51 59.49	111 26 21.78	X	
	MST063	Strip Mine Creek below Henry Mine	42 52 01.70	111 27 03.40	X	
	MST226	Tributary to Lone Pine Creek	42 51 42.27	111 25 55.16	X	
	MST275	Tributary to Lone Pine Creek	42 51 56.33	111 25 04.24	X	TBD
	MDS034	Henry Mine, Dump Seep #3	111 29 23.87	42 53 46.90	X	TBD
Enoch Valley Mine	MST128	*Angus Creek* above Rasmussen Creek	42 51 08.00	111 22 32.00	X	
	MST131	*Rasmussen Creek* above Angus Creek	42 51 08.00	111 22 31.00	X	
	MST132	Angus Creek Above *No Name Creek* and below *Rasmussen Creek*	42 51 07.00	111 22 29.00	X	
	MST133	*Rasmussen Creek* below Enoch Valley Mine	42 51 48.00	111 23 50.00	X	
	MST136	*Rasmussen Creek* headwaters near *Enoch Valley Mine Shop Pond*	42 52 34.00	111 25 03.00	X	

TABLE 4-1
LTM SURFACE WATER MONITORING LOCATIONS, FREQUENCY, AND SCHEDULE

Mine	Station ID[a]	Feature Name[b]	Location[c]		Sample Schedule	
			Latitude	Longitude	Spring	Fall[d]
	MST143	*East Fork Rasmussen Creek* above *Rasmussen Creek*	42 51 31.70	111 23 01.40	X	
	MST144	*West Pond Creek headwaters*, below West Pond	42 52 16.07	111 24 18.66	X	TBD
	MST269	*East Fork Rasmussen Creek* headwaters	42 52 16.57	111 23 41.69	X	
	MST274	West Fork of Rasmussen Creek	42 51 31.00	111 23 34.00	X	TBD
	MDS025	West Dump Seep	42 52 13.00	111 24 11.00	X	TBD
	MDS026	South Dump Seep	42 51 54.20	111 23 36.27	X	TBD

ID - Identification
LTM - Long Term Monitoring
MDS - Dump Seep
MSG - Spring
MST – Stream
TBD - To be determined

[a]Table does not include all available surface water monitoring locations at the P4 Sites, only those locations identified for continued LTM monitoring

[b] Stream names in *italics* were assigned by either IMA or P4 Production as these streams are unnamed on USGS maps or, as far as is known, are unnamed by common usage of local inhabitants. The reason for this is that most such streams are small with intermittent or ephemeral flows.

[c]Location (GPS) coordinates use the NAD27 datum and are presented in ddd mm ss.ss format (degrees minutes seconds).

[d]Fall sampling program will be evaluated based on the recent year historical results and will follow the protocol set forth in the 2015 SAP.

TABLE 4-2
LTM SURFACE WATER AND GROUNDWATER
ANALYTE LIST

Category	Fraction	Analytes (Analytical Method)
Surface Water		
Streams , Seeps, and Springs,	Unfiltered Filtered Filtered Filtered Unfiltered Unfiltered	Se (EPA 6020A) Cd, Se (EPA 6020A) Ca, Mg (EPA 6010C)[a] SO_4 (EPA 300.0) TDS (EPA 160.1) Field Parameters[b]
Groundwater		
Monitoring Wells and Direct Push Borehole Wells[c]	Unfiltered[d] Filtered Filtered Unfiltered Unfiltered	Cd, Mn, Se (EPA 6020A) Se (EPA 6020A) SO_4 (EPA 300.0) TDS (EPA 160.1) Field Parameters[b]

Notes:

[a]Hardness is calculated on filtered fractions of Ca and Mg

[b]Field Parameters are listed on Table 4-3 and Table 4-5.

[c]Borehole Monitoring Wells (direct push) are differentiated from “regular or standard” monitoring wells based on their methods and means of completion. In general, they have a smaller diameter and a shorter pre-packed screened interval than the regular monitoring wells.

[d]Direct-push monitoring wells may be sampled for dissolved or dissolved and total fractions depending on turbidity (see *2010 GW SAP*).

Ca - Calcium
Cd - Cadmium
LTM - Long Term Monitoring
Mg – Magnesium
Mn - Manganese
Se - Selenium
SO_4 - Sulfate
TDS - Total Dissolved Solids

Table 4-3
Spring LTM Sample Tracker for Surface Water Stations
(Page 1 of 4)

					Lab Parameters - Analyte List							Water Quality and Field Parameters									
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Selenium (SW6020A)	Dissolved Cd, Se (SW6020A)	Dissolved Ca, Mg (SW6010C)	Dissolved SO4 (E300.0)	TDS (E160.1)	Dissolved Hardness (SM2340B-Calc)		Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (ºC)	Air Temperature (ºC)	Discharge (ft^3/sec)
SWMDS025-U	MDS025	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMDS025-F	MDS025	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMDS026-U	MDS026	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMDS026-F	MDS026	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMDS030-1-U	MDS030	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMDS030-1-F	MDS030	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMDS030-2-U	MDS030	Water	Unfiltered	Duplicate	X				X			X	X	X	X	X	X	X	X	X	X
SWMDS030-2-F	MDS030	Water	Filtered	Duplicate		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMDS034-U	MDS034	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMDS034-F	MDS034	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG004-U	MSG004	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG004-F	MSG004	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG004-U-MS	MSG004	Water	Unfiltered	MS	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG004-F-MS	MSG004	Water	Filtered	MS		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG004-U-MSD	MSG004	Water	Unfiltered	MSD	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG004-F-MSD	MSG004	Water	Filtered	MSD		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG005-U	MSG005	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG005-F	MSG005	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG006-U	MSG006	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG006-F	MSG006	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMSG007-U	MSG007	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMSG007-F	MSG007	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST019-U	MST019	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST019-F	MST019	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST020-U	MST020	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST020-F	MST020	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST044-U	MST044	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST044-F	MST044	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST045-1-U	MST045	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST045-1-F	MST045	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST045-2-U	MST045	Water	Unfiltered	Duplicate	X				X			X	X	X	X	X	X	X	X	X	X
SWMST045-2-F	MST045	Water	Filtered	Duplicate		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST050-U	MST050	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST050-F	MST050	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST051-U	MST051	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST051-F	MST051	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X

Table 4-3
Spring LTM Sample Tracker for Surface Water Stations
(Page 2 of 4)

					Lab Parameters - Analyte List							Water Quality and Field Parameters									
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Selenium (SW6020A)	Dissolved Cd, Se (SW6020A)	Dissolved Ca, Mg (SW6010C)	Dissolved SO4 (E300.0)	TDS (E160.1)	Dissolved Hardness (SM2340B-Calc)		Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (ºC)	Air Temperature (ºC)	Discharge (ft^3/sec)
SWMST057-U	MST057	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST057-F	MST057	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST063-U	MST063	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST063-F	MST063	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST063-U-MS	MST063	Water	Unfiltered	MS	X				X			X	X	X	X	X	X	X	X	X	X
SWMST063-F-MS	MST063	Water	Filtered	MS		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST063-U-MSD	MST063	Water	Unfiltered	MSD	X				X			X	X	X	X	X	X	X	X	X	X
SWMST063-F-MSD	MST063	Water	Filtered	MSD		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST066-U	MST066	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST066-F	MST066	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST067-U	MST067	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST067-F	MST067	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST069-U	MST069	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST069-F	MST069	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST089-U	MST089	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST089-F	MST089	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST089-U-MS	MST089	Water	Unfiltered	MS	X				X			X	X	X	X	X	X	X	X	X	X
SWMST089-F-MS	MST089	Water	Filtered	MS		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST089-U-MSD	MST089	Water	Unfiltered	MSD	X				X			X	X	X	X	X	X	X	X	X	X
SWMST089-F-MSD	MST089	Water	Filtered	MSD		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST090-U	MST090	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST090-F	MST090	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST092-U	MST092	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST092-F	MST092	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST094-U	MST094	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST094-F	MST094	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST095-U	MST095	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST095-F	MST095	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST096-U	MST096	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST096-F	MST096	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST128-U	MST128	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST128-F	MST128	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST131-U	MST131	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST131-F	MST131	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST132-1-U	MST132	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST132-1-F	MST132	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST132-2-U	MST132	Water	Unfiltered	Duplicate	X				X			X	X	X	X	X	X	X	X	X	X

Table 4-3
Spring LTM Sample Tracker for Surface Water Stations
(Page 3 of 4)

					Lab Parameters - Analyte List							Water Quality and Field Parameters									
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Selenium (SW6020A)	Dissolved Cd, Se (SW6020A)	Dissolved Ca, Mg (SW6010C)	Dissolved SO4 (E300.0)	TDS (E160.1)	Dissolved Hardness (SM2340B-Calc)		Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (ºC)	Air Temperature (ºC)	Discharge (ft^3/sec)
SWMST132-2-F	MST132	Water	Filtered	Duplicate		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST133-U	MST133	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST133-F	MST133	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST136-U	MST136	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST136-F	MST136	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST143-U	MST143	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST143-F	MST143	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST144-U	MST144	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST144-F	MST144	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST226-U	MST226	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST226-F	MST226	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST269-U	MST269	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST269-F	MST269	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST274-U	MST274	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST274-F	MST274	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
SWMST275-U	MST275	Water	Unfiltered	Primary	X				X			X	X	X	X	X	X	X	X	X	X
SWMST275-F	MST275	Water	Filtered	Primary		X	X	X		X		X	X	X	X	X	X	X	X	X	X
ER-SW-01-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-01-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-02-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-02-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-03-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-03-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-04-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-04-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-05-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-05-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-06-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-06-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-07-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-07-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-08-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-08-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-09-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-09-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-10-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-10-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											

Table 4-3
Spring LTM Sample Tracker for Surface Water Stations
(Page 4 of 4)

					Lab Parameters - Analyte List							Water Quality and Field Parameters									
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Selenium (SW6020A)	Dissolved Cd, Se (SW6020A)	Dissolved Ca, Mg (SW6010C)	Dissolved SO4 (E300.0)	TDS (E160.1)	Dissolved Hardness (SM2340B-Calc)		Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (ºC)	Air Temperature (ºC)	Discharge (ft^3/sec)
ER-SW-11-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-11-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-12-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-12-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-13-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-13-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
ER-SW-14-U	na	Water	Unfiltered	Equip Rinsate	X				X												
ER-SW-14-F	na	Water	Filtered	Equip Rinsate		X	X	X		X											
B-SW-01-U	na	Water	Unfiltered	Source Water	X				X												
B-SW-01-F	na	Water	Filtered	Source Water		X	X	X		X											

Ca - Calcium	µS/cm	microsiemens per centimeter	°C Degrees celsius
Cd - Cadmium	% sat	percent saturation	ft^3/sec cubic feet per second
Mg - Magnesium	mg/L	Milligrams per liter	
Se - Selenium	mV	Millivolts	
SO4 - Sulfate	ftu	Formazin Turbidity Unit	
TDS - Total Dissolved Solids			

B – source water blank sample, to be taken once per field effort
ER - equipment rinsate blank sample, to be taken once per field team per day, total ERs taken may not add up to what is accounted for here
GPS - global positioning system
ID - identification
QC - quality control
UTM - universal transverse mercator
na - not applicable
[a] Sample Identification will also include a date prefix reflecting the year and month the sample was taken. For example, a sample taken in June of 2013 would have a prefix of (1306) followed by the normal sample ID.

TABLE 4-4

LTM GROUNDWATER MONITORING WELL AND BOREHOLE WELL LOCATIONS, FREQUENCY, AND SCHEDULE

Mine	Station ID	Station Description	Location		Well Install Year	Groundwater System Monitored Screened Interval (ft bgs)	Sample Schedule
			Latitude	Longitude			
Ballard Mine	MBW006	Short Creek	42 49 04.15	111 29 25.80	2008	Alluvial 14-9	Spring
	MBW009	Blackfoot River Road @ Monsanto Haul Road	42 49 12.96	111 29 48.71	2008	Alluvial 11-6	Spring
	MBW011	Ballard Creek	42 49 23.46	111 29 38.93	2008	Alluvial 15-10	Spring
	MBW027	Ballard, Western Deeper	42 50 02.23	111 29 54.34	2008	Alluvial 16-11	Spring
	MBW028	Torgesen	42 49 34.44	111 29 42.93	2008	Alluvial 21-16	Spring
	MBW032	Holmgren	42 49 33.92	111 28 11.14	2008	Alluvial 15-10	Spring
	MBW048	Wooley Valley Creek	42 50 03.76	111 27 56.21	2008	Alluvial 9-4	Spring
	MBW130	East of Ballard Mine, along Wooley Valley Creek	42 49 40.00	111 27 10.00	2009	Alluvial 25-20	Spring
	MBW131	Northeast of Ballard Mine, near MST093	42 50 24.00	111 28 33.00	2009	Alluvial 8-3	Spring
	MBW135	Southeast of Ballard Mine, across Blackfoot River Road	42 48 40.00	111 29 43.00	2009	Alluvial 20-15	Spring
	MMW006	South of West Ballard Pit; south of waste rock dumps	42 49 20.00	111 29 03.00	2007	Wells Formation 330-310	Spring
	MMW017	Northwest of Ballard Mine into Long Valley Creek alluvial flow field	42 49 59.60	111 29 47.40	2007	Alluvial 56-36	Spring
	MMW018	East of Ballard Mine in Wooley Valley alluvial flow field	42 49 39.40	111 28 04.50	2007	Alluvial/ Dinwoody Formation 33-18	Spring
	MMW020	East side of West Ballard Pit (MMP035); replacement of MMW001	42 49 36.40	111 29 03.30	2007	Wells Formation 408-388	Spring
	MMW021	West side of West Ballard Pit (MMP035); replacement of MMW002	42 49 35.60	111 29 23.90	2007	Wells Formation 250-230	Spring
	MMW029	East Ballard mine area in the vicinity of MMW018	42 49 37.78	111 28 08.74	2008	Dinwoody Formation 60-45	Spring
	MMW030	Along the southwestern portion of Ballard Mine in the vicinity of MMW016A	42 49 10.72	111 29 17.03	2008	Wells Formation 155-135	Spring
	MMW031	Along the western perimeter of Ballard Mine in the vicinity and north of MMW017	42 50 11.87	111 29 43.05	2008	Wells Formation 200-180	Spring
	MMW032	Adjacent to MWD084	42 50 09.30	111 28 21.80	2009	Dinwoody Formation65-55	Spring

TABLE 4-4

LTM GROUNDWATER MONITORING WELL AND BOREHOLE WELL LOCATIONS, FREQUENCY, AND SCHEDULE

Mine	Station ID	Station Description	Location		Well Install Year	Groundwater System Monitored Screened Interval (ft bgs)	Sample Schedule
			Latitude	Longitude			
	MMW033	**Deeper well nested with MMW029**	42 49 35.30	111 2811.90	2009	Dinwoody Formation 150-130	Spring
	MW-15A	West Ballard Mine near MST068	42 49 35.99	111 29 36.55	2006	Alluvial 40-30	Spring
	MW-16A	Southwest Ballard Mine near MST069	42 49 08.83	111 29 16.53	2006	Alluvial 30-20	Spring
Henry Mine	MBW152	North Henry Mine, along Little Blackfoot River	42 54 14.00	111 29 23.00	2009	Alluvial 15-10	Spring
	MMW010	Southeast of Center Henry Pit; near MPW023	42 52 22.30	111 27 51.30	2007	Alluvial 32-12	Spring
	MMW011	Northwest of Center Henry Pit; south of Little Blackfoot River	42 53 48.30	111 29 30.00	2007	Wells Formation 115-95	Spring
	MMW022	Northeast lobe of Henry Mine waste rock dump MWD086	42 53 09.20	111 28 18.30	2007	Dinwoody Formation 326-306	Spring
	MMW023	Henry Mine North Pit	42 54 20.50	111 30 27.50	2007	Wells Formation 357-352	Spring
	MMW028	Near the Little Blackfoot River northwest of MMW019	42 53 50.24	111 28 60.00	2008	Dinwoody Formation 96-76	Spring
Enoch Valley Mine	MBW085	Rasmussen Creek	42 51 55.52	111 24 04.12	2008	Alluvial 12.25-7.25	Spring
	MBW087	Rasmussen Road and Agrium Haul Road intersection	42 51 38.34	111 23 44.32	2008	Alluvial 12-7	Spring
	MBW099	Agrium Haul Road North	42 51 57.08	111 23 28.14	2008	Alluvial 29-24	Spring
	MBW107	Western Enoch Valley, East of fence	42 53 28.32	111 25 55.86	2008	Alluvial 40-35	Spring
	MBW112	Western Enoch Valley, west of fence	42 53 34.67	111 26 13.72	2008	Alluvial 18-13	Spring
	MMW007	South of EVM South Dump; near edge of dump footprint	42 51 48.50	111 23 34.40	2007	Alluvial/ Dinwoody Formation 90-70	Spring
	MMW009	Central North Dump (MWD091)	42 53 34.60	111 25 33.80	2007	Wells Formation 554-549	Spring
	MMW012	Northwest of EVM North Dump in Lone Pine Creek alluvial flow field	42 53 28.75	111 25 53.34	2007	Alluvial/ Dinwoody Formation 52-28	Spring
	MMW013	Southwest of EVM in Rasmussen Creek alluvial flow field	42 52 05.70	111 24 12.00	2007	Dinwoody Formation 35-25	Spring
	MMW024	Along the south end of Enoch Valley Mine, near MMW013	42 52 11.73	111 24 11.86	2008	Dinwoody Formation 200-180	Spring

TABLE 4-4

LTM GROUNDWATER MONITORING WELL AND BOREHOLE WELL LOCATIONS, FREQUENCY, AND SCHEDULE

Mine	Station ID	Station Description	Location		Well Install Year	Groundwater System Monitored Screened Interval (ft bgs)	Sample Schedule
			Latitude	Longitude			
	MMW025	Along the south end of Enoch Valley Mine, near MMW007	42 51 49.16	111 23 31.95	2008	Dinwoody Formation 200-180	Spring
	MMW026	Northeast of MPW006/MMW008	42 51 56.48	111 23 25.75	2008	Wells Formation 355-335	Spring
	MMW027	Near MMW012	42 53 28.97	111 25 53.50	2008	Dinwoody Formation 120-100	Spring
	MMW034	Deeper well nested with MMW013	42 52 09.10	111 24 14.80	2009	Dinwoody Formation 156-136	Spring
	MMW035	Deeper well nested with MMW027	42 53 29.50	111 25 54.30	2009	Dinwoody Formation 199 – 179	Spring
	MMW036	Well west of MMW027	42 53 26.70	111 26 05.60	2009	Basalt 135 – 115	Spring
	MMW037	Nested with MMW036	42 53 26.14	111 26 06.82	2010	Dinwoody 302 – 292	Spring

Notes:

ID - Identification
LTM - Long Term Monitoring
MBW - Borehole Monitoring Well (direct-push pre-packed screen monitoring well)
MMW - Monitoring Well

Borehole Monitoring Wells (direct push) are differentiated from “regular or standard” monitoring wells based on their methods and means of completion. In general, the direct-push borehole monitoring wells have a smaller diameter and a shorter pre-packed screened interval than the regular monitoring wells.

Table 4-5
LTM Sample Tracker for Monitoring Wells and Borehole Wells
(Page 1 of 4)

					Lab Parameters					Water Quality and Field Parameters							
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Cd, Mn, and Se (SW6020A)	Dissolved Se (SW6020A)	TDS (E 160.1)	S04 (EPA 300.0)		Specific Conductivity (µS/cm @ 25°C)	pH	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	Water Elevation (ft amsl)
GWMBW006-U	MBW006	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW006-F	MBW006	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW009-U	MBW009	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW009-F	MBW009	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW011-U	MBW011	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW011-F	MBW011	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW027-U	MBW027	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW027-F	MBW027	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW028-U	MBW028	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW028-F	MBW028	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW032-1-U	MBW032	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW032-1-F	MBW032	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW032-2-U	MBW032	Water		Duplicate	X		X			X	X	X	X	X	X	X	X
GWMBW032-2-F	MBW032	Water	X	Duplicate	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW048-U	MBW048	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW048-F	MBW048	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW085-U	MBW085	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW085-F	MBW085	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW087-1-U	MBW087	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW087-1-F	MBW087	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW099-U	MBW099	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW099-F	MBW099	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW107-U	MBW107	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW107-F	MBW107	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW112-U	MBW112	Water		Primary	TBD		TBD			X	X	X	X	X	X	X	X
GWMBW112-F	MBW112	Water	X	Primary	TBD	TBD	TBD	TBD		X	X	X	X	X	X	X	X
GWMBW130-U	MBW130	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW130-F	MBW130	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW131-U	MBW131	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW131-F	MBW131	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW135-U	MBW135	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW135-F	MBW135	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMBW152-U	MBW152	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMBW152-F	MBW152	Water	X	Primary	O	X	O	X		X	X	X	X	X	X	X	X
GWMMW006-U	MMW006	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW006-F	MMW006	Water	X	Primary		X		X		X	X	X	X	X	X	X	X

Table 4-5
LTM Sample Tracker for Monitoring Wells and Borehole Wells
(Page 2 of 4)

					Lab Parameters					Water Quality and Field Parameters							
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Cd, Mn, and Se (SW6020A)	Dissolved Se (SW6020A)	TDS (E 160.1)	S04 (EPA 300.0)		Specific Conductivity (µS/cm @ 25°C)	pH	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	Water Elevation (ft amsl)
GWMMW007-U	MMW007	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW007-F	MMW007	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW007-U-MS	MMW007	Water		MS	X		X			X	X	X	X	X	X	X	X
GWMMW007-F-MS	MMW007	Water	X	MS		X		X		X	X	X	X	X	X	X	X
GWMMW007-U-MSD	MMW007	Water		MSD	X		X			X	X	X	X	X	X	X	X
GWMMW007-F-MSD	MMW007	Water	X	MSD		X		X		X	X	X	X	X	X	X	X
GWMMW009-U	MMW009	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW009-F	MMW009	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW010-U	MMW010	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW010-F	MMW010	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW011-U	MMW011	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW011-F	MMW011	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW012-U	MMW012	Water		Primary	TBD		TBD			X	X	X	X	X	X	X	X
GWMMW012-F	MMW012	Water	X	Primary		TBD		TBD		X	X	X	X	X	X	X	X
GWMMW013-U	MMW013	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW013-F	MMW013	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW017-U	MMW017	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW017-F	MMW017	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW018-U	MMW018	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW018-F	MMW018	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW018-U-MS	MMW018	Water		MS	X		X			X	X	X	X	X	X	X	X
GWMMW018-F-MS	MMW018	Water	X	MS		X		X		X	X	X	X	X	X	X	X
GWMMW018-U-MSD	MMW018	Water		MSD	X		X			X	X	X	X	X	X	X	X
GWMMW018-F-MSD	MMW018	Water	X	MSD		X		X		X	X	X	X	X	X	X	X
GWMMW020-U	MMW020	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW020-F	MMW020	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW021-U	MMW021	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW021-F	MMW021	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW022-U	MMW022	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW022-F	MMW022	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW023-U	MMW023	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW023-F	MMW023	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW024-U	MMW024	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW024-F	MMW024	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW025-U	MMW025	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW025-F	MMW025	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW026-U	MMW026	Water		Primary	X		X			X	X	X	X	X	X	X	X

Table 4-5
LTM Sample Tracker for Monitoring Wells and Borehole Wells
(Page 3 of 4)

					Lab Parameters					Water Quality and Field Parameters							
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Cd, Mn, and Se (SW6020A)	Dissolved Se (SW6020A)	TDS (E 160.1)	S04 (EPA 300.0)		Specific Conductivity (µS/cm @ 25°C)	pH	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	Water Elevation (ft amsl)
GWMMW026-F	MMW026	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW027-U	MMW027	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW027-F	MMW027	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW028-1-U	MMW028	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW028-1-F	MMW028	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW028-2-U	MMW028	Water		Duplicate	X		X			X	X	X	X	X	X	X	X
GWMMW028-2-F	MMW028	Water	X	Duplicate		X		X		X	X	X	X	X	X	X	X
GWMMW029-U	MMW029	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW029-F	MMW029	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW030-U	MMW030	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW030-F	MMW030	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW031-U	MMW031	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW031-F	MMW031	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW032-U	MMW032	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW032-F	MMW032	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW033-U	MMW033	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW033-F	MMW033	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW034-1-U	MMW034	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW034-1-F	MMW034	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW034-2-U	MMW034	Water		Duplicate	X		X			X	X	X	X	X	X	X	X
GWMMW034-2-F	MMW034	Water	X	Duplicate		X		X		X	X	X	X	X	X	X	X
GWMMW035-U	MMW035	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW035-F	MMW035	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW036-U	MMW036	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW036-F	MMW036	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW036-U-MS	MMW036	Water		MS	X		X			X	X	X	X	X	X	X	X
GWMMW036-F-MS	MMW036	Water	X	MS		X		X		X	X	X	X	X	X	X	X
GWMMW036-U-MSD	MMW036	Water		MSD	X		X			X	X	X	X	X	X	X	X
GWMMW036-F-MSD	MMW036	Water	X	MSD		X		X		X	X	X	X	X	X	X	X
GWMMW037-1-U	MMW037	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMMW037-1-F	MMW037	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMMW037-2-U	MMW037	Water		Duplicate	X		X			X	X	X	X	X	X	X	X
GWMMW037-2-F	MMW037	Water	X	Duplicate		X		X		X	X	X	X	X	X	X	X
GWMW-15A-U	MW-15A	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMW-15A-F	MW-15A	Water	X	Primary		X		X		X	X	X	X	X	X	X	X
GWMW-16A-U	MW-16A	Water		Primary	X		X			X	X	X	X	X	X	X	X
GWMW-16A-F	MW-16A	Water	X	Primary		X		X		X	X	X	X	X	X	X	X

Table 4-5
LTM Sample Tracker for Monitoring Wells and Borehole Wells
(Page 4 of 4)

					Lab Parameters					Water Quality and Field Parameters							
Field Sample Identification [a]	Location	Matrix	Filtered (check for yes)	QC Sample Type	Total Cd, Mn, and Se (SW6020A)	Dissolved Se (SW6020A)	TDS (E 160.1)	S04 (EPA 300.0)		Specific Conductivity (µS/cm @ 25°C)	pH	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	Water Elevation (ft amsl)
B-GW-01-U	na	Water		B	X		X										
B-GW-01-F	na	Water	X	B		X		X									

Cd - Cadmium	µS/cm	microsiemens per centimeter			
Mn - Manganese	% sat	percent saturation	°C	Degrees celsius	
Se - Selenium	mg/L	Milligrams per liter	ft^3/sec	cubic feet per second	
SO_4 - Sulfate	mV	Millivolts	ftu	Formazin Turbidity Unit	
TDS - Total Dissolved Solids					

O - Direct-push monitoring wells will be sampled for total and dissolved fraction when the turbidity is less than 5 NTU.
If the turbidity is greater than 5 NTU, then the sample will be analyzed for the dissolved fraction only.
B – source water blank sample, to be taken once at the beginning of each sampling event and whenever new source water is used.
ID - identification
LTM - Long Term Monitoring
MBW - Borehole Monitoring Well (direct-push pre-packed screen monitoring well)
MMW - Monitoring Well
na - not applicable
QC - quality control
TBD - to be determined, analyte list for MBW112 and MMW012 will be determined in consult with A/Ts if water is encountered and the well recovers following purging
UTM - universal transverse mercator
Primary QC sample indicates that it is the first samples collected.
[a] Sample Identification will also include a date prefix reflecting the year and month the sample was taken. For example, a sample taken in June of 2013 would have a prefix of (1306) followed by the normal sample ID.
Borehole Monitoring Wells (direct push) are differentiated from “regular or standard” monitoring wells based on their methods and means of completion. In general, they have a smaller diameter and a shorter pre-packed screened interval than the regular wells.

FIGURES

DRAWN BY J. Kester

Date: 3/16/2015

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 1-1_MONSANTO_Vicinity Map_Mar2015.mxd

BLACKFOOT RESERVOIR

Little Blackfoot River

Blackfoot River

HENRY MINE

ENOCH VALLEY MINE

RASMUSSEN RIDGE MINE

BALLARD MINE

WOOLEY VALLEY MINE

Sheep Creek

Angus Creek

Blackfoot River

34

Soda Springs

FIGURE 1-1 AREA

0 3 6

Miles

ENOCH VALLEY MINE

HENRY MINE

RASMUSSEN RIDGE MINE

LANES CREEK MINE

BALLARD MINE

WOOLEY VALLEY MINE

NORTH MAYBE MINE

DRY VALLEY MINE

SOUTH MAYBE MINE

CONDA MINE

Soda Springs

EXPLANATION

Mine pit location (approximate)

Waste rock pile location (approx.)

National Forest

Bureau of Land Management

State of Idaho

Private Land

MAP AREA

IDAHO

Boise

Pocatello

15

84

86

84

15

N

W

E

S

0 2500 5000

Feet

MWH

P4 Production, LLC

P4 MINES VICINITY MAP

FIGURE 1-1

DRAWN BY J. Kester

Date: 3/25/2015

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 3-1_MONSANTO_Vicinity Map_Mar2015.mxd

EXPLANATION

- Mine pit location (approximate)
- Waste rock pile location (approximate)
- Waste rock dump location or pit backfill (approximate)
- 7000 Post-mine contour (approximate) (Contour interval 40 feet)
- Lake/Pond
- Natural Drainage - Perennial
- Natural Drainage - Intermittent
- Highway
- Road
- Monsanto haul road (active-inactive)
- Railroads

Groundwater Sample Location

- Direct push alluvial aquifer well
- Local aquifer monitoring well (generally alluvial system)
- Intermediate aquifer monitoring well (generally Dinwoody Formation)
- Regional aquifer monitoring well (Wells Formation)

Surface Water Sample Location

- MDS Dump seep sample location
- MSG Spring sample location
- MST Stream sample location

Sample Label

Red - Locations are LTM groundwater monitroing well locations that will be sampled

Blue - Locations are LTM surface water locations that will be sampled

Black - Locations are legacy groundwater monitoring well or surface water locations that will not be sampled

Key

MMP - Mine pit

MWD - Waste rock dump

LTM - Long-term monitoring

N W E S

0 2000 4000

Feet

ENOCH VALLEY MINE

HENRY MINE

BALLARD MINE

MWD085 BACKFILLED MMW023 MWP041 BACKFILLED MBW152 MST045 MDS034 MST044 MMW028 MWD088 MMW011 MWP043 BACKFILLED MMW022 MWD086 MWD087 MMW010 MST051 MWP042 BACKFILLED BACKFILLED MST063 MST057 MST226 MWP044 NOT BACKFILLED MWD090

MWD091 MMW035 MBW112 MMW009 MWP045 PARTIALLY BACKFILLED MMW036 MMW037 MMW027 MBW107 MMW012 MWD091 MST136 MST144 MDS025 MMW024 MMW034 MMW013 MST275 MBW085 MST133 MDS026 MBW087 MST269 MWD092 MBW099 MMW026 MMW025 MMW007 MST143 MST274 MST128 MST131 MST132

MWP035 PARTIALLY BACKFILLED MMP036 PARTIALLY BACKFILLED MST050 MWD093 MMP040 NOT BACKFILLED MWP035 IN-PIT WASTE ROCK MBW131 MWD084 MWP039 NOT BACKFILLED MBW027 MMW031 MMW017 MMW032 MBW048 MDS030 MST094 MMW018 MMW020 MST095 MMW021 MSG006 MW-15A MBW028 MSG007 MST092 MWP035 NOT BACKFILLED MBW130 MST096 MST089 MST067 MMW033 MBW011 MBW032 MWD081 MMW029 MST090 MBW009 MWD082 MST069 MSG004 MSG005 MST019 MST066 MMW006 MMP036 IN-PIT WASTE ROCK MWD083 MST020 MBW135 MMW030 MW-16A MBW006 MMP038 NOT BACKFILLED

Little Blackfoot River

Sheep Creek

Argus Creek

Blackfoot River

MWH

P4 Production, LLC

LTM SURFACE WATER AND GROUNDWATER SAMPLE LOCATIONS

FIGURE 3-1

Date: 3/25/2015 DRAWN BY J. Kester

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 4-1_BallardMine_Mar2015.mxd

N
W E
S

0 750 1500
Feet

MST050
MBW131
MMW031
MMW032
BALLARD MINE
MBW027
MMW017
MBW048
MST094
MDS030
MMW018
MST095
MSG006
MSG007
MBW130
MST092
MW15A
MBW028
MMW021
MMW020
MMW033
MMW029
MBW032
MST096
MST090
MST089
MST067
MBW011
MMW006
MSG005
MSG004
MBW009
MMW030
MST069
MW16A
MBW006
MST066
MST019
MBW135
MST020

MAP AREA

0 1 2
Miles

HENRY MINE
ENOCH VALLEY MINE
RASMUSSEN RIDGE MINE
BALLARD MINE
WOOLEY VALLEY MINE

EXPLANATION

Mine pit location (approximate)

Waste rock pile location (approximate)

LTM Long-term monitoring

Groundwater Sample Location

- Direct push alluvial aquifer well
- Local aquifer monitoring well (generally alluvial system)
- Intermediate aquifer monitoring well (generally Dinwoody Fm.)
- Regional aquifer monitoring well (Wells Fm.)

Surface Water Sample Location

- MDS Dump seep sample location
- MSG Spring sample location
- MST Stream sample location

MWH

P4 Production, LLC

LTM SAMPLE LOCATIONS
Ballard Mine Site

FIGURE 4-1

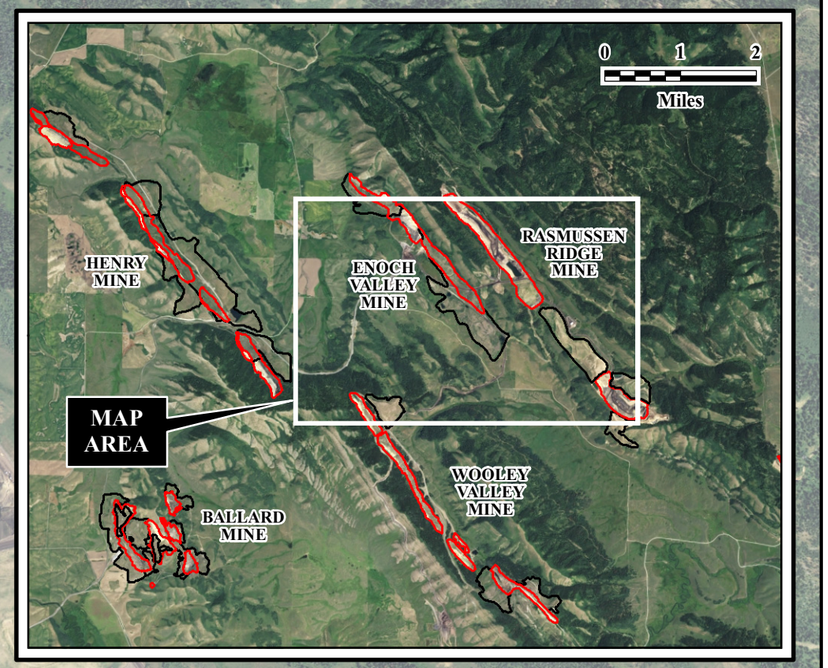
Date: 3/25/2015 DRAWN BY J. Kester

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 4-2_EnochValleyMine_Mar2015.mxd

MBW112
MMW009
MMW035
MMW027
MMW036
MMW012
MBW107
MMW037

ENOCH VALLEY MINE

MST136
MST144
MDS025
MST269
MMW024
MMW034
MMW013
MBW085
MBW099
MMW026
MDS026
MMW025
MST133
MMW007
MBW087
MST274
MST143
MST131
MST128
MST132



N
W E
S

0 750 1500
Feet

EXPLANATION

Mine pit location (approximate)

Waste rock pile location (approximate)

LTM Long-term monitoring

Groundwater Sample Location

- Direct push alluvial aquifer well
- Local aquifer monitoring well (generally alluvial system)
- Intermediate aquifer monitoring well (generally Dinwoody Fm.)
- Regional aquifer monitoring well (Wells Fm.)

Surface Water Sample Location

- MDS Dump seep sample location
- MSG Spring sample location
- MST Stream sample location

MWH

P4 Production, LLC

LTM SAMPLE LOCATIONS
Enoch Valley Mine Site

FIGURE 4-2

Date: 3/25/2015 DRAWN BY J. Kester

EXPLANATION

Mine pit location (approximate)

Waste rock pile location (approximate)

LTM Long-term monitoring

Groundwater Sample Location

- Direct push alluvial aquifer well
- Local aquifer monitoring well (generally alluvial system)
- Intermediate aquifer monitoring well (generally Dinwoody Fm.)
- Regional aquifer monitoring well (Wells Fm.)

Surface Water Sample Location

- MDS Dump seep sample location
- MSG Spring sample location
- MST Stream sample location

MAP AREA

ENOCH VALLEY MINE

HENRY MINE

RASMUSSEN RIDGE MINE

WOOLEY VALLEY MINE

BALLARD MINE

0 1 2 Miles

MMW023

MBW152

MST045

Little Blackfoot River

MST044

MDS034

MMW011

MMW028

HENRY MINE

MMW022

MMW010

MST051

MST063

MST057

MST275

MST226

N W E S

0 1200 2400 Feet

MWH

P4 Production, LLC

LTM SAMPLE LOCATIONS
Henry Mine Site

FIGURE 4-3

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 4-3_HenryMine_Mar2015.mxd

APPENDICES

APPENDIX A

FIELD SAMPLING PLAN

FOR

LONG-TERM-MONITORING OF

SURFACE WATER AND GROUNDWATER

DRAFT

Revision 0

MARCH 2015

TABLE OF CONTENTS

TABLE OF CONTENTS i

1.0 INTRODUCTION 1-1

2.0 PROGRAM BACKGROUND AND OBJECTIVES 2-1

2.1 SURFACE WATER BACKGROUND AND OBJECTIVES 2-1

2.2 GROUNDWATER BACKGROUND AND OBJECTIVES 2-1

2.3 SURFACE WATER AND GROUNDWATER LTM OBJECTIVES 2-2

3.0 SAMPLE LOCATIONS AND FREQUENCY 3-3

4.0 SAMPLE COLLECTION AND ANALYSIS 4-1

4.1 SITE ACCESS, LOGISTICS, AND SAFETY 4-1

4.2 SURFACE WATER SAMPLE COLLECTION 4-1

4.3 GROUNDWATER SAMPLE COLLECTION 4-2

4.4 SURFACE WATER AND GROUNDWATER FIELD PARAMETER MEASUREMENTS 4-1

4.5 SURFACE WATER FLOW MEASUREMENTS 4-2

4.6 WATER LEVEL DATA LOGGER RETRIEVAL 4-2

4.7 SAMPLE DESIGNATION 4-3

4.8 SAMPLE HANDLING AND SHIPPING 4-4

4.9 SAMPLE ANALYSIS 4-4

4.10 SAMPLING QUALITY ASSURANCE 4-5

4.11 SAMPLE EQUIPMENT DECONTAMINATION 4-6

5.0 DOCUMENTATION AND RECORDS REQUIREMENTS 5-1

5.1 FIELD LOGBOOKS 5-1

5.2 Field Forms 5-2

5.3 Chain-of-Custody Records 5-2

5.4 Documents and Records 5-3

5.5 Field Change Request Forms 5-3

5.6 Reports to Management 5-4

5.7 Training Requirements 5-4

6.0 PROJECT ORGANIZATION 6-1

7.0 REFERENCES 7-1

ATTACHMENT A 7-1

LIST OF TABLES

Table 4-1 Groundwater Well Build Specifications and Recommended Sampling Order

Table 4-2 Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Table 4-3 Calibration and Maintenance Requirements for Field Equipment

Table 4-4 Surface Water and Groundwater Analytes

Table 6-1 Project Contacts

LIST OF FIGURES

Figure 4-1 Recommended Groundwater Sampling Order

ATTACHMENTS

Attachment A Standard Operating Procedures

ACRONYMS AND ABBREVIATIONS

A/Ts	Agencies and Tribes
Ca	Calcium
Cd	Cadmium
COPC	Contaminant of Potential Concern
DQOs	Data Quality Objectives
e.g.	*exempli gratia* (Latin, for example)
EDD	Electronic Data Deliverable
FCR	Field Change Request
FS	Feasibility Study
FSP	Field Sampling Plan
FTL	Field Team Leader
ft	Foot or feet
GPS	Global Positioning System
GW	Groundwater
HSP	Health and Safety Plan
i.e.	*id est* (Latin, that is to say; in other words)
LTM	Long-Term Monitoring
mL/min	milliliters per minute
Mg	Magnesium
Mn	Manganese
MWH	MWH, Inc. (formerly Montgomery Watson Harza, Inc.)
P4	P4 Production, L.L.C.
PM	Program Manager
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RI/FS	Remedial Investigation / Feasibility Study
SAP	Sampling and Analysis Plan
Se	Selenium
Sites	Collectively the Ballard, Henry and Enoch Valley mine sites
SO4	Sulfate
SOP	Standard Operating Procedure
SW	Surface Water
TDS	Total Dissolved Solids
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

This Field Sampling Plan (FSP) describes the scope of work for Long-Term Monitoring (LTM) of surface water (SW) and groundwater (GW) stations throughout P4 Production, L.L.C.(P4's) inactive properties of Ballard, Henry, and Enoch Valley Mines (collectively known as the Sites). The Sampling and Analysis Plan for LTM of SW and GW *(2015 LTM SAP)* presents the Data Quality Objectives (DQOs) that have been developed to guide the monitoring program presented in this FSP. The complementary documents to the FSP are the Quality Assurance Program Plan (QAPP) and Health and Safety Plan (HSP). The FSP, QAPP, and HSP are included as Appendices A through C of the *2015 LTM SAP*, respectively.

The FSP is organized as follows:

- Section 1.0 – Introduction
- Section 2.0 – Program Background and Objectives: discusses the need and objectives for LTM of SW and GW at the Sites, presents supporting historic data, and specific sampling objectives and rationale
- Section 3.0 – Sample Locations and Frequency: presents the the SW and GW locations to be sampled and the frequency of sample collection for the LTM activities
- Section 4.0 – Sample Collection and Analysis: summarizes protocols for SW and GW sample collection and handling procedures and sample analyses and laboratory methods
- Section 5.0 – Documentation and Records Requirements: details the various field forms, field change requests, and chain-of-custody records to be completed during each field event.
- Section 6.0 – Project Organization: presents the project team, schedule, and deliverables
- Section 7.0 – References

2.0 PROGRAM BACKGROUND AND OBJECTIVES

This section provides brief background information related to LTM SW and GW monitoring for the Sites. Additional program background details may be obtained from the following Site Work Plans and SAPs:

- *2009 Groundwater Monitoring Sampling and Analysis Plan* (MWH, 2009)
- *2009 and 2010 Surface Water Monitoring Sampling and Analysis Plan* (MWH, 2009)
- *2010 Groundwater Monitoring Memorandum* (MWH, 2010)
- *2011 Remedial Investigation / Feasibility Study Work Plan for P4's Ballard, Henry, and Enoch Valley Mines – Revision 2 Final* (MWH, 2011)
- *2012 Surface Water and Groundwater Memo* (MWH, 2012)
- *2013 Proposed P4 LTM Surface Water and Groundwater Monitoring Plan Memo* (MWH, 2013)
- *2014 Proposed P4 Long-Term Surface Water and Groundwater Monitoring Plan Memo* (MWH, 2014)

From 2010 through 2014, P4 performed SW and GW monitoring under plans provided as brief letter memoranda relying on the FSP and SOPs provided in the 2009 SAPs. In 2014, the United States Environmental Protection Agency (USEPA) requested that P4 prepare a single SW and GW LTM SAP including a FSP, QAPP, and HSP for future collection of SW and GW at the Sites.

2.1 SURFACE WATER BACKGROUND AND OBJECTIVES

The objective of SW monitoring conducted at the Sites from 2004 through 2008 was to support nature and extent characterization of impacts to water quality associated with releases of contaminants of potential concern (COPCs) and contaminants of potential ecological concern (COPECs) to SW from potential Site sources. In 2009, the SW monitoring program transitioned from a characterization to an interim monitoring program that provided an annual/bi-annual assessment of SW conditions downstream of potential P4 sources associated with each Site. The objective of the sampling conducted from 2009 to present and beyond is to establish long-term data trends for SW and GW at each of the Sites and assist in the development of potential future Site-specific remedies.

2.2 GROUNDWATER BACKGROUND AND OBJECTIVES

The objective of GW monitoring conducted at the Sites from 2004 to 2012 was to provide ongoing monitoring of concentrations and piezometric conditions in monitoring wells to characterize the nature and

extent of COPCs/COPECs in GW. Starting in 2013, the monitoring of COPC/COPEC concentrations in monitoring wells began supporting the evaluation of potential future Site remedial actions. This *2015 LTM SAP*, and specifically this *FSP*, will continue that effort.

2.3 SURFACE WATER AND GROUNDWATER LTM OBJECTIVES

Ongoing LTM of SW and GW COPC/COPEC concentrations and piezometric conditions in monitoring wells is needed to support the evaluation of potential future remedies in the respective Site FS reports and the implementation of any potential future corrective action that may be necessary. Long-term sampling of these SW and GW locations also allows for understanding of potential long-term SW/GW trends in COPC/COPECs following implementation of potential future selected remedies the Sites.

3.0 SAMPLE LOCATIONS AND FREQUENCY

This section describes the LTM SW and GW monitoring program sampling locations and frequencies. The SW and GW sampling locations and frequencies are summarized programmatically in Table 4-1 and Table 4-4, of the *2015 LTM SAP*.

The locations of the 36 SW sampling stations are shown in Figure 3-1 and Figures 4-1 through 4-3 of the *2015 LTM SAP*. These sampling locations were selected according to the rationale in Section 4.1 of the *2015 LTM SAP*.

The locations of the 45 GW sampling stations are shown in Figure 3-1 and Figures 4-1 through 4-3 of the *2015 LTM SAP*. These sampling locations were selected according to the rationale in Section 4.4 of the *2015 LTM SAP*.

4.0 SAMPLE COLLECTION AND ANALYSIS

This section presents the site access requirements, equipment, and procedures for the collection, handling, and analysis of each SW and GW sample. Where applicable, references to Standard Operating Procedures (SOPs) in Attachment A are provided.

4.1 SITE ACCESS, LOGISTICS, AND SAFETY

P4 has access to all SW and GW monitoring locations. The Agencies and Tribes (A/Ts) will be notified, at minimum, five business days prior to commencement of field activities. The MWH Field Team Leader (FTL) will notify the P4 Project Manager (P4 PM, Rachel Roskelley) at minimum three days prior to working at a mine area. Such notification is necessary to allow time for site-specific safety training by P4, and if necessary, to arrange for a P4 representative to accompany the crew to provide access through locked gates. Relevant P4, agency contacts, and other field contacts are provided in Section 6.0.

Field equipment and samples will be stored at the Fox Hills Machine Shed, owned by P4 (or in another location designated by the P4 PM in the future should a new location be necessary). Equipment, supplies, and samples will be received and shipped from the Monsanto plant, in Soda Springs, in care of the P4 PM. Additional sample handling and shipping information is presented in Section 4.8.

Safety procedures for the site investigation are described in the *HSP* (in *Appendix C* of the *2015 LTM SAP*). The mine-specific safety requirements involve a short training orientation for hazard recognition and avoidance provided by P4. In the event that any of P4's corporate safety policies are stricter than the requirements of the HSP, those safety requirements will take precedence.

4.2 SURFACE WATER SAMPLE COLLECTION

SW samples will be collected using the protocols outlined in SOP-NW-9.1, Collection of Surface Water Samples. This SOP is located in Attachment A.

Water samples will be collected from streams using a Kemmerer horizontal water bottle sampler by taking a composite of three grabs across the channel. If the stream is too narrow or shallow to accommodate three grabs across the channel, three grabs will be taken along the channel, beginning downstream. Samples collected from small streams, seeps, and springs (stations not suitable for sampling by Kemmerer) will be taken using a suitable polyethylene container, i.e., the 1-Liter bottle from the swing sampler or a suitable pitcher. Water samples will be transferred to appropriate sample containers after collection and any required filtering.

The following acidified/unacidified and filtered/unfiltered SW samples will be collected:

- Unfiltered, acidified samples will be collected and analyzed for selenium [Se].
- Filtered, acidified samples will be collected and analyzed for "dissolved" metals (cadmium [Cd], calcium [Ca], magnesium [Mg], and Se).
- Filtered, un-acidified samples will be collected for sulfate (SO4) anion
- Unfiltered, un-acidified samples will be collected for total dissolved solids (TDS).

The necessary filtering for the analyses above will be conducted in the field using a new 0.45-micron disposable filter at each sample location. Parameters to be analyzed for are described below in Section 4.4. Refer to Table 4-1 for analyte sample container requirements. Water samples will be collected upon arrival at the station, prior to field water quality and flow measurements, to ensure high-quality, undisturbed samples. For sample documentation, digital photos will be taken at each sample location.

4.3 GROUNDWATER SAMPLE COLLECTION

GW samples will be collected using the protocols outlined in SOP-NW-5.3, Collection of Groundwater Quality Samples and the SOP Low Stress Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells. This SOP is located in Attachment A.

Upon arrival at a monitoring well, static water level will be measured prior to sampling. Measurement of the static water level is performed using an electronic water level indicator. The same measuring point on the well casing is used to ensure the measurements are consistent. The wellhead reference point is on the top of well casing - north side. Refer to SOP-5 Groundwater Level Data Collection, which is located in Attachment A for specific procedures.

In accordance with USEPA protocol, low-stress sampling techniques will be used for sampling wells where possible. Low-stress sampling may not be possible in existing wells if dedicated, high-flow pumps are already installed, or if the required sampling equipment cannot access the well. Detailed sampling protocol is included in SOP-NW-5.3. The sampling protocol states requirements for water-level stabilization and parameter stabilization. Bladder pumps for low-stress sampling will be set at least 20 feet (if possible) below the air-water interface in the well, and pumped through dedicated tubing to the surface. The wells will be purged in accordance with USEPA protocols, such that water from the formation will be transported from the aquifer to the surface with minimal agitation. Groundwater samples will be collected only after water level and parameter stabilization has occurred.

In the event that a well produces water at an extremely slow rate and excessive drawdown is occurring during purging (i.e., > 0.30 feet at a low pumping rate of 100 milliliters per minute (mL/min)), then a "purge and sample" method shall be employed. Under these circumstances, the well will be purged dry one time and allowed to recharge to a minimum of 80% of the original water level, and then the sample will be drawn. Monitoring wells and boreholes that may need this method are MMW007, MMW025, MMW030, MMW032, MMW033, MBW011, MBW028, MBW085, MBW099, MBW130, MBW135, and MBW152.

Table 4-2 provides GW well build and pump specifications, general comments on past sampling efforts that may affect sample collection, and recommenced sampling order of GW locations to optimized sampling time efficiency. This sampling order is based on an optimal 8 day field sampling effort with the understanding that the sampling duration can be affected by numerous factors including weather, station accessibility, equipment breakdowns, etc. Previous spring SW/GW sampling events have typically occurred in a 10 day span. This sampling order also is presented on Figure 4-1 of this FSP.

Groundwater samples will be transferred to appropriate sample containers following initial collection and any required filtering. The following acidified/unacidified and filtered/unfiltered GW samples will be collected:

- Filtered, acidified samples will be collected and analyzed for "dissolved" metals (Se).
- Unfiltered, acidified samples will be collected and analyzed for total metals (Cd, Mn, and Se).
- Filtered, un-acidified samples will be collected for SO4
- Unfiltered, un-acidified samples will be collected for TDS.

The necessary filtering for the analyses above will be conducted in the field using a new 0.45-micron disposable filter at each sample location. Parameters to be analyzed for are described below in Section 4.4. Refer to Table 4-1 for the sample container, required preservation, sample preparation method(s) and maximum holding times for each of the analyses to be performed during the LTM program.

Table 4-1

REQUIREMENTS FOR CONTAINERS, PRESERVATION TECHNIQUES, SAMPLE VOLUMES, AND HOLDING TIMES

Method	Sample Container—Water Matrices	Preservative	Parameter*	Sample Preparation Method	Maximum Holding Time (Days)
EPA 6010C and 6020A	250 mL HDPE (minimum of 20 mL for dissolved and 20 mL for total)	Field filter; HNO_3 pH < 2 (dissolved) HNO_3 pH <2 (total)	Dissolved and total metals	microwave-assisted acid digestion (3015 ICP-MS) or hot plate acid digestion (3005A ICP) - dissolved and total	180
300	250 mL HDPE (minimum of 50 mL)	Field filter; ≤ 6 °C	anions	dissolved (field filtered)	28
160.1	250 mL HDPE (minimum of 50 mL)	≤ 6 °C	total dissolved solids	total (raw)	7

Notes:

*Refer to Table 4-2 of *2015 LTM SAP* for parameters and methods.

HDPE – high-density polyethylene

mL - Milliliter

Table 4-2
Groundwater Well Build Specifications and Recommended Sampling Order
(Page 1 of 2)

Well Sampling Information and General Comments										Well Build Specifications																
Recommended Sampling Order (Field Effort Day)	Well ID	Mine	Well Location	Pump Type	Sample Technique	Tubing Length (ft.)	Minimum Purge Volume in Liters[2x(tube + pump volume)]	Estimated Purge Time (hours)	Sample Notes	Date Installed	Formation Screened	Ground Surface Elevation (ft-AMSL)	Boring TD (ft bgs)	Depth Water Encountered when Drilling (ft bgs)	Depth to Formation Contacts (ft bgs)	Well Completion TD (ft bgs)	Borehole Diameter (in.)	Casing Diameter (in.)	Casing Material	Pump Depth (ft bgs)	Screened Interval [Length] (ft bgs)	Screen Slot Size (in.)	Sand Size at Screen	Primary Filter Interval (ft bgs)	Bentonite Seal (Pellet) Interval (ft bgs)	Bentonite Seal (Slurry) Interval (ft bgs)	Backfill Below Screen (ft bgs)	Permanent Steel Casing (ft bgs)
1	MBW006	Ballard	South of MST069 on South Side of Road	Peristaltic	Low Flow	15	--	--	-	5/13/2008	Alluvial	6319.31	--	8	--	14	1.5	1.5	sch 80 PVC	--	9-14	0.011	20x40	7-14	0-7	--	--	--
1	MBW135	Ballard	Southeast of Ballard Mine , Across Blackfoot River Road	Peristaltic	Purge/Sample	23	--	--	Drawdown in an issue. Typically pumps dry after 5 Liters and 9 minutes and takes minimum of 2 days to recharge.	5/14/2009	Alluvial	6290.56	--	15	--	20	1.5	1.5	sch 80 PVC	--	15-20	0.011	20x40	13-20	0-13	--	--	--
1	MBW152	Henry	North Henry Mine, Along Little Blackfoot River	Peristaltic	Purge/Sample	18	--	--	Drawdown in an issue. Typically pumps dry after 5 L and 9 minutes and takes minimum of 2 days to recharge.	5/12/2009	Alluvial	6280	--	7.5	--	15	1.5	1.5	sch 80 PVC	--	10-15	0.011	20x40	8-15	0-8	--	--	--
1	MMW025	Enoch Valley	Along the south end of Enoch Valley Mine, near MMW007	Bladder Pump	Standard Purge	195	--	--	Drawdown is an issue, low flow will not stabilize due to drawdown. Sample standard purge once field parameters stabilize.	7/28/2008	Dinwoody	6612.87	201	58,160	0-Alluvium 115-Dinwoody	200	8	4	shc 40 PVC	192	200-180 [20]	0.02	10x20	201-175	175-surface	NA	NA	16
1	MMW030	Ballard	Along the southwestern portion of Ballard Mine in the vicinity of MW-16A	Bladder Pump / compressor	Purge/Sample	155	--	--	Drawdown an issue. Well typically pumped dry after 8 hours of purging, takes up to 4 days to recover.	7/10/2008	Wells	6355.25	221	155	0-Alluvium 75-Wells 170-Phosphoria	155	8	4	sch 40 PVC	150	155-135 [20]	0.01	20X40	155-130	130-surface	NA	221-155 Bentonite (Chips)	16
1	MW16A	Ballard	Southwest Ballard Mine near MST069	Peristaltic	Low Flow	30	1.8	0.43	--	11/8/2006	Alluvium	6346.71	40	10.25	0-Alluvium	35	6	2	sch 40 PVC	NA	30-20 [10] blank PVC sump from 35-30	0.02	10x20	40-15	15-5	5-surface	40-35 sand	12
2	MBW009	Ballard	Blackfoot River Road at Monsanto Haul Road	Peristaltic	Purge/Sample	13	--	--	Drawdown an issue, can not do low flow. Once the water level reached the water table well can be purged indefinitely with very little drawdown.	5/14/2008	Alluvial	6310.69	--	6	--	11	1.5	1.5	sch 80 PVC	--	6-11	0.011	20x40	4-11	1.5-4	--	--	--
2	MBW011	Ballard	Near MST067, Off East Side of Haul Road	Peristaltic	Purge/Sample	18	--	--	Drawdown, could not low flow. Purged dry and let recover. Well typically recovered at a rate of 1.0 feet per 1.5 minutes.	5/14/2008	Alluvial	6339.8	--	9	--	15	1.5	1.5	sch 80 PVC	--	10-15	0.011	20x40	8-15	1.5-8	--	--	--
2	MMW006	Ballard	South of West Ballard Pit; south of waste rock dumps	Bladder Pump/Nitrogen	Low Flow	327	20.62	1.23	--	7/23/2007	Wells	6485.46	335	315-335	0-Wells	330	9.5	4	sch 80 PVC	327	330-310 [20]	0.02	10x20	335-305 20x40 sand 305-300	300-290	290-surface	NA	NA
2	MMW020	Ballard	East side of West Ballard Pit (MMP035); replacement of MMW001	Bladder Pump/Nitrogen	Low Flow	403	25.18	0.70	--	10/5/2007	Wells	6525.71	416	225, 250, 315, 370	0-Rex Chert 370-Wells	408	9.5	4	sch 80 PVC	320	408-388 [20]	0.01	20x40	408-378	378-368	368-surface	NA	NA
2	MMW021	Ballard	West side of West Ballard Pit (MMP035); replacement of MMW002	Bladder Pump / Nitrogen / or compressor	Low Flow	242	15.52	0.65	--	9/24/2007	Wells	6436.3	260	229, 238	0-Waste Rock 25-Wells	250	9.5	4	shc 80 PVC	242	250-230 [20]	0.01	20x40	250-219	219-210	210-surface	NA	NA
3	MBW032	Ballard	Holmgren Ranch	Peristaltic	Low Flow	17	--	--	--	5/28/2008	Alluvial	6499.13	--	8	--	15	1.5	1.5	sch 80 PVC	--	10-15	0.011	20x40	8-15	0-8	--	--	--
3	MBW130	Ballard	East of Ballard Mine, Along Wooley Valley Creek	Peristaltic	Purge/Sample	28	--	--	Drawdown and issue. Typically pumps dry after 11.4 Liters and 11 minutes. Took 1 day to recharge.	5/11/2009	Alluvial	6416.29	--	18	--	25	1.5	1.5	sch 80 PVC	--	20-25	0.011	20x40	17-25	0-17	--	--	--
3	MMW018	Ballard	East of Ballard Mine in Wooley Valley alluvial flow field	Bladder Pump	Low Flow	30	2.8	0.31	--	8/12/2007	Alluvium/ Dinwoody	6459.52	33	31	0-Alluvium 30-Dinwoody	33	9.5	4	sch 80 PVC	30	33-18 [15]	0.01	20x40	33-15	15-10	10-surface	NA	NA
3	MMW029	Ballard	East Ballard mine area upgradient of MMW018	Bladder Pump	Low Flow	55	4.3	0.18	--	7/17/2008	Dinwoody	6498.67	61	60	0-Alluvium 25-Dinwoody	60	8	4	sch 40 PVC	55	60-45 [15]	0.02	10x20	60-40	40-surface	NA	NA	16
3	MMW032	Ballard	Adjacent to MWD084	Bladder Pump / compressor	Purge/Sample		4.8	1.8	Drawdown and issue. Typically pumps dry after 63 gallons and 106 minutes. Takes 3 days to recharge.	8/16/2009	Dinwoody	6446.39	160	35	0-Alluvium 55-Dinwoody 65-Phosphoria	65	8.5	4	sch 40 PVC	61	65-55 [10]	0.01	20x40	69-24 (sand and native)	24-surface	NA	160-69 Bentonite & Native	17
3	MMW033	Ballard	Deeper Well Nested with MMW029	Bladder Pump	Standard Purge	0	--	--	Drawdown an issue sample standard purge	8/28/2009	Dinwoody	6489.84	160	17	0-Alluvium 18-Dinwoody	150	8.5	4	sch 40 PVC	141	150-130 [20]	0.01	20x40	155-124	124-surface	NA	155-150 Sand	16.5
4	MBW027	Ballard	West Side of Haul Road in Ballard Close To MBW026, Deeper of Two	Peristaltic	Low Flow	20	--	--	--	5/21/2008	Alluvial	6313.33	--	6	--	25	1.5	1.5	sch 80 PVC	--	11-16	0.011	20x40	7.5-20	1.5-7.5	--	--	--
4	MBW028	Ballard	Torgesen Ranch	Peristaltic	Purge/Sample	23	--	--	Drawdown, and issue, typically purged well dry and recharged in 22 minutes.	5/21/2008	Alluvial	6339.99	--	8	--	21	1.5	1.5	sch 80 PVC	--	16-21	0.011	20x40	14-21	1.5-14	--	--	--
4	MBW048	Ballard	Wooley Valley Creek	Peristaltic	Low Flow	12	--	--	Drawdown an issue, can not do low flow. Once the water level reached the water table well can be purged indefinitely with very little drawdown.	5/30/2008	Alluvial	6421.72	--	2, 4	--	10	1.5	1.5	sch 80 PVC	--	4-9	0.011	20x40	2-9	0-2	--	--	--
4	MBW131	Ballard	Northeast of Ballard Mine, Near MST093	Peristaltic	Low Flow	11	--	--	--	5/11/2009	Alluvial	6468.52	--	1.5	--	8	1.5	1.5	sch 80 PVC	--	3-8	0.011	20x40	2-8	0-2	--	--	--
4	MMW017	Ballard	Northwest of Ballard Mine into Long Valley Creek alluvial flow field	Bladder Pump	Low Flow	56	4.36	0.91	--	8/27/2007	Alluvium	6313.86	62	35	0-Alluvium	57	9.5	4	shc 40 PVC	56	56-36 [20]	0.01	20x40	56-31	31-20	20-surface	Pellets 32-60 20x40 sand 60-56	NA
4	MMW031	Ballard	Along the western perimeter of Ballard Mine north of MMW017 and on the east side of the haul road.	Bladder Pump	Low Flow	195	12.7	0.53	--	7/12/2008	Wells	6346.42	201	120, 185	0-Alluvium 160-Wells	200	8	4	shc 40 PVC	196	200-180 [20]	0.01	20x40	200-175	175-surface	NA	NA	16
4	MW15A	Ballard	West Ballard Mine near MST068	Peristaltic	Low Flow	45	2.7	0.56	--	11/9/2006	Alluvium	6364.37	48	22.25	0-Alluvium	45	6	2	sch 40 PVC	NA	40-30 [10] blank PVC sump from 45-40	0.02	10x20	48-28	28-5	5-surface	NA	25
5	MBW085	Enoch Valley	Rasmussen Creek	Peristaltic	Purge/Sample	15	--	--	Drawdown an issue, typically pumps dry after 11.6 L and 19 min with a quick recharge.	6/11/2008	Alluvial	6639.57	--	6.9	--	12.25	1.5	1.5	sch 80 PVC	--	7.25-12.25	0.011	20x40	7-12.25	0-7	--	--	--
5	MBW087	Enoch Valley	Rasmussen Road and Agrium Haul Road Intersection	Peristaltic	Low Flow	13	--	--	--	6/11/2008	Alluvial	6587.3	--	NR	--	12	1.5	1.5	sch 80 PVC	--	7-12	0.011	20x40	7-12	0-7	--	--	--
5	MBW099	Enoch Valley	Agrium Haul Road North	Peristaltic	Purge/Sample	20	--	--	Drawdown an issue, pump dry and typically takes 3 days to recharge.	6/13/2008	Alluvial	6599.25	--	NR	--	29	1.5	1.5	sch 80 PVC	--	24-29	0.011	20x40	22-29	0-22	--	--	--
5	MMW007	Enoch Valley	South of EVM South Dump; near edge of dump footprint	Bladder Pump	Purge/Sample	87	--	--	Drawdown and issue, once water level stabilizes at approximately 79 ft. bgs well can be low flow sampled once field parameters stabilize.	8/23/2007	Alluvium/ Weathered Dinwoody	6619.89	90	88	0-Alluvium	89.5	9.5	4	sch 80 PVC	87	90-70 [20]	0.01	16x30	90-64	64-61	61-surface	NA	NA
5	MMW026	Enoch Valley	Northeast of MPW006 and MMW008	Bladder Pump/Nitrogen	Low Flow	345	21.7	0.60	--	8/11/2008	Wells	6599.21	363	6, 65, 100, 200, 300	0-Alluvium 218-Wells	355	8	4	shc 80 PVC	350	355-335 [20]	0.01	20x40	355-330	330-surface	NA	Boring Caved 363-355	322
6	MMW009	Enoch Valley	Central North Dump (MWD091)	Bladder Pump/Nitrogen	Low Flow	552	34.12	1.42	--	10/26/2007	Wells	6789.2	563	150 (Wells Fm contact 530)	0-Waste Rock 90-Phosphoria 530-Wells	554	9.5	4	sch 80 PVC	360	554-549 [5]	0.01	20x40	554-546	546-536	536-surface	NA	360
6	MMW013	Enoch Valley	Southwest of EVM in Rasmussen Creek alluvial flow field	Bladder Pump / Nitrogen / or compressor	Low Flow	31	2.86	0.10	--	8/13/2007	Dinwoody	6634.46	35	29	0-Alluvium 6-Dinwoody	35	9.5	4	sch 40 PVC	31	35-25 [10]	0.02	10x20	35-21	21-16	16-surface	NA	NA
6	MMW024	Enoch Valley	Along the south end of Enoch Valley Mine, upgradient of MMW013	Bladder Pump/Nitrogen	Low Flow	195	12.7	2.12	--	7/25/2008	Dinwoody	6704.05	201	15, 60, 100, 140	0-Alluvium 10-Dinwoody	200	8	4	shc 40 PVC	195	200-180 [20]	0.02	10x20	201-175	175-surface	NA	NA	16
6	MMW034	Enoch Valley	Deeper Well Nested with MMW013	Bladder Pump/Nitrogen	Low Flow	167	10	0.42	--	9/2/2009	Dinwoody	6640.31	160	156	0-Alluvium 19-Dinwoody	156	8.5	4	sch 40 PVC	146	156-136 [20]	0.01	20x40	160-126	126-surface	NA	160-156 Sand	18
7	MBW107	Enoch Valley	Western Enoch Valley, East of Fence	Peristaltic	Historically dry	43	--	--	Historically dry.	6/16/2008	Alluvial	6486.78	--	30	--	40	1.5	1.5	sch 80 PVC	--	35-40	0.011	20x40	33-40	0-33	--	--	--

Table 4-2
Groundwater Well Build Specifications and Recommended Sampling Order
(Page 2 of 2)

Well Sampling Information and General Comments										Well Build Specifications																		
Recommended Sampling Order (Field Effort Day)	Well ID	Mine	Well Location	Pump Type	Sample Technique	Tubing Length (ft.)	Minimum Purge Volume in Liters[2x(tube + pump volume)]	Estimated Purge Time (hours)	Sample Notes	Date Installed	Formation Screened	Ground Surface Elevation (ft-AMSL)	Boring TD (ft bgs)	Depth Water Encountered when Drilling (ft bgs)	Depth to Formation Contacts (ft bgs)	Well Completion TD (ft bgs)	Borehole Diameter (in.)	Casing Diameter (in.)	Casing Material	Pump Depth (ft bgs)	Screened Interval [Length] (ft bgs)	Screen Slot Size (in.)	Sand Size at Screen	Primary Filter Interval (ft bgs)	Bentonite Seal (Pellet) Interval (ft bgs)	Bentonite Seal (Slurry) Interval (ft bgs)	Backfill Below Screen (ft bgs)	Permanent Steel Casing (ft bgs)
7	MBW112	Enoch Valley	Western Enoch Valley, Two Small Drainages North of MBW107 Near Tilled Field	Peristaltic	Historically dry	21	--	--	Historically dry.	6/17/2008	Alluvial	6404	--	NR	--	20	1.5	1.5	sch 80 PVC	--	13-18	0.011	20x40	12-20	0-12	--	--	--
7	MMW012	Enoch Valley	Northwest of EVM North Dump in Lone Pine Creek alluvial flow field	Bladder	Historically dry	55	--	--	Historically dry.	8/28/2007	Alluvium/ Dinwoody	6488.72	58	Dinwoody contact @ 60 ft-BGL	0-Alluvium 37-Dinwoody	58	9.5	4	sch 40 PVC	55	58-28 [30]	0.01	20x40	58-23	23-13	13-surface	NA	NA
7	MMW027	Enoch Valley	Near MMW012	Bladder Pump / compressor	Low Flow	115	7.9	0.26	--	8/15/2008	Dinwoody	6491.07	123	88	0-Alluvium 20-Basalt 45-Alluvium 53-Dinwoody	120	8	4	sch 40 PVC	115	120-100 [20]	0.01	20x40	120-95	95-Surface	NA	NA	16
7	MMW035	Enoch Valley	Deeper Well Nested with MMW027	Bladder Pump / Nitrogen / or compressor	Low Flow	205	12.32	0.41	--	8/30/2009	Dinwoody	6500.34	200	199	0-Alluvium 22-Basalt 33-Dinwoody	179	8.5	4	sch 40 PVC	146	199-179 [20]	0.01	20x40	200-176	176-surface	NA	200-199 Sand	31
7	MMW036	Enoch Valley	Well West of MMW028	Bladder Pump / compressor	Low Flow	147	8.8	0.37	--	9/1/2009	Basalt	6425.18	160	135	0-Alluvium 88-Basalt	135	8.5	4	sch 40 PVC	126	135-115 [20]	0.01	20x40	137-111	160-137	NA	160-137 Bentonite	16
7	MMW037	Enoch Valley	Paired with MMW036 and downgradient of MMW027 and MMW035	Bladder Pump / Nitrogen / or compressor	Low Flow	318	19.06	0.45	--	9/20/2010	Dinwoody	6419	302.5	114.3	0-Alluvium 80-Basalt 263- Dinwoody	302	9.25	4	sch 40 PVC	297	302-292 [10]	0.01	20x40	302-286	286-surface	NA	NA	15.5
8	MMW010	Henry	Southeast of Center Henry Pit; near MPW023	Bladder Pump	Low Flow	29	2.74	0.15	--	9/9/2007	Alluvium	6462.62	38	17	0-Alluvium	32	9.5	4	sch 40 PVC	29	32-12 [20]	0.01	20x40	32-8	NA	8-surface	20x40 sand 38-32	NA
8	MMW011	Henry	Northwest of Center Henry Pit; south of Little Blackfoot River	Bladder Pump	Low Flow	112	7.72	0.64	--	9/8/2007	Wells	6268.31	120	101	0-Wells	115	9.5	4	sch 80 PVC	112	115-95 [20]	0.01	20x40	115-85	85-75	75-surface	20x40 sand 120-115	NA
8	MMW022	Henry	Northeast lobe of Henry Mine waste rock dump MWD086	Bladder Pump / compressor	Low Flow		20.38	2.26	--	7/28/2007	Dinwoody	6635.85	360	320, 340	0-Waste Rock 5-Dinwoody	326	9.5	4	shc 80 PVC	323	326-306 [10]	0.02	10x20	326-286 20x40 sand 286 281	281-271	271-surface	Boring caved 360-326	NA
8	MMW023	Henry	Henry Mine North Pit (MMP041)	Bladder Pump / compressor	Low Flow	352	22.12	0.37	--	9/11/2007	Wells	6230.92	362	128, 188	0-Phosphoria 350-Wells	357	9.5	4	shc 80 PVC	352	357-352 [5]	0.01	20x40	357-350	350-340	340-surface	NA	NA
8	MMW028	Henry	Near the Little Blackfoot River northwest of MMW019	Bladder Pump	Low Flow	90	6.4	0.12	--	7/15/2008	Dinwoody	6316.91	100	80, 100	0-Alluvium 40-Basalt 63-Alluvium 70-Dinwoody	96	8	4	sch 40 PVC	90	96-76 [20]	0.01	20x40	96-70	70-surface	NA	Boring Caved 100-96	16

-- not applicable
Ft-AMSL: Feet above mean sea level
Ft-bgs: Feet below ground surface
in. : inch
NA: Information not known
NR: Water was not reached during borehole well installation.

DRAWN BY J. Kester

Date: 3/17/2015

J:\Industrial Projects\P4 Monsanto\GIS\MXDs\2015 LTM SAP_QAPP\Fig 4-1_MONSANTO_SampleOrder_Mar2015.mxd

BACKFILLED
MWD085
MMW023
MWP041
BACKFILLED
Little Blackfoot River
MBW152
MST045
MDS034
MST044
MMW028
MWD088
MMW011
MWP043
BACKFILLED
MMW022
HENRY
MINE
MWD086
MWD087
MMW010
MST051
MWP042
BACKFILLED
MST063
MST057
BACKFILLED
MST226
MWP044
NOT BACKFILLED
MWD090
BALLARD
MINE
MWP035
PARTIALLY BACKFILLED
MST050
MWD093
MMP036
PARTIALLY BACKFILLED
MMP040
NOT BACKFILLED
MWP035
IN-PIT WASTE ROCK
MBW131
MWD084
MWP039
NOT BACKFILLED
MBW027
MMW031
MMW017
MMW032
MBW048
MDS030
MMW020
MMW021
MST094
MMW018
MST095
MW-15A
MBW028
MSG006
MSG007
MST092
MWP035
NOT BACKFILLED
MBW130
MST096
MST089
MST067
MMW033
MBW011
MBW032
MWD081
MMW029
MST090
MWD082
Blackfoot River
MBW009
MSG004
MSG005
MST069
MST019
MST066
MMW006
MMP036
IN-PIT WASTE ROCK
MWD083
MST020
MBW135
MMW030
MW-16A
MBW006
MMP038
NOT BACKFILLED
MWH
ENOCH
VALLEY
MINE
MWD091
MMW035
MMW009
MBW112
MMW036
MWP045
PARTIALLY BACKFILLED
MMW037
MMW027
MMW012
MBW107
MWD091
MST136
MST269
Sheep Creek
MST144
MWD092
MDS025
MBW099
MMW024
MMW034
MMW026
MST275
MMW013
MBW085
MMW025
MST133
MMW007
MDS026
MBW087
MST143
MST274
MST128
MST131
MST132
Angus Creek
Blackfoot River

EXPLANATION

- Mine pit location (approximate)
- Waste rock pile location (approximate)
- Waste rock dump location or pit backfill (approximate)
- 7000 Post-mine contour (approximate) (Contour interval 40 feet)
- Lake/Pond
- Natural Drainage - Perennial
- Natural Drainage - Intermittent
- Highway
- Road
- Monsanto haul road (active-inactive)
- Railroads

Groundwater Sample Location

- Direct push alluvial aquifer well
- Local aquifer monitoring well (generally alluvial system)
- Intermediate aquifer monitoring well (generally Dinwoody Formation)
- Regional aquifer monitoring well (Wells Formation)

Surface Water Sample Location

- MDS Dump seep sample location
- MSG Spring sample location
- MST Stream sample location

Sample Label

Red - Locations are LTM groundwater monitroing well locations that will be sampled
Blue - Locations are LTM surface water locations that will be sampled
Black - Locations are legacy groundwater monitoring well or surface water locations that will not be sampled

Key

MMP - Mine pit
MWD - Waste rock dump
LTM - Long-term monitoring

Groundwater Location Sampling Order

- Day 1
- Day 2
- Day 3
- Day 4
- Day 5
- Day 6
- Day 7
- Day 8

N
W
E
S

0 2000 4000
Feet

P_4 Production, LLC

LTM RECOMMENDED GROUNDWATER SAMPLING ORDER

FIGURE 4-1

4.4 SURFACE WATER AND GROUNDWATER FIELD PARAMETER MEASUREMENTS

Field measurements of SW parameters will be made in-situ whenever possible before the collection of SW samples. If an in-situ measurement is not possible, then the measurement will be made streamside from a 5-Liter pitcher.

Monitoring well (GW) parameter measurements will be made using a flow-through cell, and readings will be recorded after parameter stabilization has occurred. Refer to the *Low Stress Purging* SOP in Attachment A for stabilization requirements. Field parameter values will be recorded on field data forms and in the field notebooks.

The following field water quality parameters will be measured at all SW and GW locations:

- pH;
- Conductivity (SW only);
- Specific conductivity;
- Water Temperature;
- Dissolved oxygen;
- Turbidity; and,
- Oxidation-reduction potential (ORP).

Field meters will be used in accordance with the manufacturer's instructions and calibration performed in accordance with Table 4-3. Conductivity (SW only) and specific conductivity, dissolved oxygen, turbidity, and pH meter performance will be calibrated and turbidity meters will be checked each morning prior to field sampling as well as at the end of each day. At the quality assurance/quality control (QA/QC) monitoring stations, all the parameter measurements will be taken three times to confirm consistency in the measurement of these parameters. Refer to Table 4-3 and Table 4-5 of the *2015 LTM SAP* for QA/QC sample locations.

TABLE 4-3 CALIBRATION AND MAINTENANCE REQUIREMENTS FOR FIELD EQUIPMENT*			
Field Parameter	**Required Procedure**	**Minimum Frequency**	**Required Equipment or Calibration Fluids**
pH	2-point calibration	Each day prior to sampling and end of day	One pH buffer (7 pH), Reference Standard
Temperature	N/A	N/A	N/A
Dissolved Oxygen	Atmospheric calibration	Each day prior to sampling and end of day	Atmospheric oxygen with elevation
Conductivity and Specific Conductivity	1-point reference	Each day prior to sampling and end of day	Reference Standard
ORP	1-point reference	Each day prior to sampling and end of day	Reference Standard
Turbidity	1-point calibration	Each day prior to sampling and end of day	Reference Standard
Flow	Velocity equivalence and spin test (Price meters) or equivalent	When the rotor is changed	Stopwatch, measuring tape
Notes: *In the event of a discrepancy, the manufacturer's instruction manual shall take precedence.			

4.5 SURFACE WATER FLOW MEASUREMENTS

SW flow rate measurements or estimates, if applicable, will be collected at all SW monitoring locations listed programmatically in Table 4-1 of the *2015 LTM SAP* in conjunction with the collection of the water quality sample. Flow measurements will always be recorded after the stream/seep water quality sample and field parameters have been collected to prevent potential contamination. Flow measurements typically will be measured with current meters using the velocity-area method (Rantz, 1982). For streams less than 0.2 feet deep, flow will be measured using a volumetric method, if possible. If a stream is too fast or deep to safely measure, then the flow will be estimated using an estimate of the stream cross-sectional area and the stream surface velocity by means of the "float method." Details of SW flow measurement methods are presented in SOP-NW-9.2a, *Surface Water Flow Measurements Using Man-Portable Devices or Estimation Techniques* (refer to in Attachment A).

4.6 WATER LEVEL DATA LOGGER RETRIEVAL

A Solinst Levelogger® data logger was installed in each well upon well completion. The data loggers record the GW water level, GW temperature, and time once each day. Field teams will download the data logger readings upon arrival at the monitoring well and prior to the start of sampling.

4.7 SAMPLE DESIGNATION

Samples will be labeled with all necessary information on laboratory supplied labels using waterproof ink. At a minimum, each sample label shall contain the following information:

- Station identification;
- Sample identification;
- Date and time of sample collection, with sampler's initials;
- Analyses required;
- Filtered or unfiltered;
- Method of preservation, if used; and,
- Sample matrix.

Each sample shall be assigned a unique identification number. This number shall be coded based on the sample location in the following format:

AABBSW or **GWMYYaaa-b-c**

- **AA** indicates the year (two digits) the sampling event started
- **BB** indicates the month (two digits) the sampling event started
- **SW** or **GW** denotes that surface water or groundwater is sampled.
- **M** designates "M" for Monsanto and is used to differentiate from other sample stations identified by MWH for Idaho Mine Association (IMA) mine-specific investigations.
- **YY** denotes the station type; station type which is ST for streams, DS for dump seeps, SG for spring, BW for borehole well, and MW for monitoring well.
- **aaa** denotes the specific station number/location.
- **b** denotes the replicate number (blank shall indicate no replicate samples) if there are QA/QC replicate samples, then 1, 2 and 3 represent the replicate samples.
- **c** denotes whether the sample involved special field handling or is to be handled in a specific manner; handling codes are as follows:

  F: Filtered

- U: Unfiltered

A SW sample example is, sample number **1505SWMST020-F** describes a non-replicated, filtered SW sample collected at stream monitoring station MST020 collected in May 2015. A GW sample example is, sample number **1505GWMMW020-1-F** describes the first field duplicate of a filtered groundwater sample collected at monitoring well MMW020 in May 2015.

4.8 SAMPLE HANDLING AND SHIPPING

Upon sample collection, the sample containers will be placed in Ziploc plastic bags, sealed, and immediately placed on ice in an insulated cooler to ≤ 6 °C. Insulated coolers will be provided by Microbac (or other approved laboratory) or purchased locally. All samples will be stored in the coolers and handled as specified in Section 2.0 of the QAPP. All samples will remain in the coolers until the end of the day when they will be transferred to a locked refrigerator at the Fox Hills Machine Shed.

Samples will be shipped to Microbac with double-bagged wet ice in coolers secured with packing tape, via overnight Federal Express service to Microbac. MWH will fill out appropriate chain-of-custody forms; the chain-of-custody will be included with the sample shipment, and copies of all chains-of- custody along with Federal Express waybills will be kept by MWH field personnel.

All samples will be sent to Microbac or other approved laboratory. Microbac's address and contact person are listed below:

Microbac
158 Starlite Drive
Marietta, OH 45750
(740) 373-4071
Attn: Kathy Albertson

Supplies including sample containers and coolers will be sent to the Monsanto Plant:

Monsanto Company
1853 HWY 34
Soda Springs, ID 83276 (208) 547-1439
Attn: Rachel Roskelley

4.9 SAMPLE ANALYSIS

SW and GW samples will be analyzed for the analytes listed in Table 4-4. The analyte list was developed based on the screening in Section 3.2 and 3.5, respectively, of the *2015 LTM SAP*. Microbac of Marietta, Ohio will analyze for the analytes listed.

TABLE 4-4 SURFACE WATER AND GROUNDWATER ANALYTES						
Parameter	**Matrix**	**Basis**	**Method**	**RL**	**Reporting Units**	**Holding Times (days)**
cadmium	SW	dissolved	6020A	0.0006	mg/L	180
cadmium	GW	total	6020A	0.0006	mg/L	180
calcium	SW	dissolved	6010C	0.5	mg/L	180
hardness	SW	dissolved	2340B-Calculation	5	mg/L	n/a
magnesium	SW	dissolved	6010C	0.5	mg/L	180
manganese	GW	total	6020A	0.002	mg/L	180
selenium	SW, GW	dissolved	6020A	0.08	mg/L	180
selenium	SW, GW	total	6020A	0.08	mg/L	180
sulfate	SW, GW	dissolved	300.0	1	mg/L	28
total dissolved solids	SW, GW	total	160.1	20	mg/L	7

Notes:
Method—Method to be utilized by Microbac.
GW-Groundwater
RL-Reporting Limit of Microbac
SW-Surface Water.

4.10 SAMPLING QUALITY ASSURANCE

The following QA/QC procedures will be followed during SW and GW sampling:

- Collection of field duplicate samples will occur at a minimum rate of 1 in 10 samples or at least 10 percent of total stations.
- The equipment rinsate sample will be collected by rinsing decontaminated sampling equipment with distilled water in a manner similar to actual sample collection.
- The water blank will be a sample of the distilled water used for the equipment rinsate sample.
- Equipment rinsate and water blank samples will be collected on a daily basis, and, if more than one team collects samples on a given day, also by each sampling team.
- For matrix spike analysis, field teams will collect additional volume and designate on the chain-of-custody forms that the samples are for matrix spike analysis. Matrix spike samples will be collected at a minimum rate of 1 in 20 samples or at least 20 percent of total stations.
- All sampling equipment will be decontaminated prior to sample collection.

Refer to the QAPP for further details regarding SW and GW sampling QA/QC procedures. The QA/QC samples as well as a summary of all samples are provided on Table 4-3 or Table 4-5, respectively, of the *2015 LTM SAP*.

4.11 SAMPLE EQUIPMENT DECONTAMINATION

Sampling equipment decontamination will be performed in accordance with methodology presented in the attached SOPs for SW and GW sample collection (SOP-NW-9.1 and SOP-NW-5.3).

5.0 DOCUMENTATION AND RECORDS REQUIREMENTS

5.1 FIELD LOGBOOKS

SW and GW sample collection activities will be documented in permanently bound, page-numbered, weather-resistant field logbooks assigned to the FTL, or, if multiple sampling teams are used, to a designee in charge of each team. Each notebook will be identified to the project, task, and to the individual assigned custody of the logbook. For all sampling to be performed, the appropriate SOP, appended to this FSP, will also be employed. If logbook custody is transferred to another individual, such transfer will be noted in the logbook and signed and dated by both parties. All entries will be made in indelible ink; errors will be corrected by one single line through the text being revised, and all such corrections will be initialed and dated.

With the exception of the information contained in the appropriate SOP, governing the media to be sampled, bound field logbooks will be used to record the following information, as appropriate for the type of sampling being performed:

- Date, time, subjects, and attendees of daily tailgate training sessions
- Sample date, time, types, numbers, and quantities
- Sample container preservation steps performed
- Sample locations, including Global Positioning System (GPS) coordinates
- Numbers of associated photographs, with appropriate cross-references to the affected camera
- Sampling equipment used
- Decontamination steps performed
- Acknowledgements that chain-of-custody forms and express shipment information were properly completed

In addition, other ancillary information will be recorded, including:

- Time of arrivals/departures of MWH personnel and/or other visitors to the sampling site(s)
- Weather conditions
- Presence of livestock or wild game

- Time and subject of any incoming or outgoing telephone/radio contacts
- Any unusual events

The logbooks will be kept up to date on a daily basis; backup copies of each day's entries will be made on a weekly basis and forwarded to the project quality records.

5.2 FIELD FORMS

In addition to the field logbooks, field forms will be required to be filled out by the sampling team conducting the sampling. All efforts will be made to fill out the information at the sampling location. Field forms for the sampling activities are used to supplement the field logbooks. The appropriate forms are located in the applicable SOPs provided in Attachment A of this FSP.

5.3 CHAIN-OF-CUSTODY RECORDS

Documentation of sample custody must be maintained from the time the samples are collected through: receipt at the destination laboratory; preparation, and analysis; data recording and reduction; data validation; and final release of laboratory analytical data. Initial information concerning sample collection will be recorded in the field logbook as described in Section 5.1. Information on the custody, transfer, handling, and shipping of samples will be recorded by field personnel on a project-specific chain-of-custody form for the laboratory(ies) being utilized for the analyses. A chain-of-custody form will be completed for each set of samples collected daily and will contain the following information:

- Sampler's signature and affiliation
- Project name and identification number
- Date and time of collection
- Sample identification number and matrix
- Analyses requested
- Number of containers
- Signature of persons relinquishing custody, dates, and times
- Signature of persons accepting custody, dates, and times
- Method of shipment

- Shipping papers/waybill identification number (e.g., Federal Express tracking number as identified on pre-printed packing labels)

A copy of each as-transmitted chain-of-custody form will be made on a weekly basis and forwarded to the project quality records. An example chain-of-custody form is provided as Figure 1-2 of the *QAPP*.

5.4 DOCUMENTS AND RECORDS

Documents and records are defined as completed, legible documents which furnish objective evidence of the items or services, activities affecting quality or the completeness of data, and which are maintained for the specific project. These records will be organized and managed in MWH's program office and will include, at a minimum, the following:

- Original and backup copies of all bound field logbooks
- Field copies and original (laboratory) copies of all chain-of-custody forms
- Personnel training records (except that any medical monitoring program will be maintained in MWH's personnel files)
- Incoming and outgoing project correspondence (letters, telephone conversation records, faxes, and hard copies of e-mail messages)
- Copies of all laboratory agreements and amendments thereto
- Purchasing records for project supplies
- As-received laboratory data packages (hard copy and electronic data deliverable [EDDs])
- Validated laboratory data packages
- All approved Field Change Request (FCR) forms
- Draft and final versions of all reports and any associated presentation materials associated with these field efforts.

5.5 FIELD CHANGE REQUEST FORMS

Due to the conditions associated with field sampling activities, unexpected situations may occur that will require deviations or modifications to the requirements of the *2015 LTM SAP*. Other changes may be required by P4 during the course of this project. In such situations, the MWH Project Manager (PM) may authorize the FTL or designee to undertake SAP modifications, provided that the scope of such

modifications is discussed with the P4 PM and the USEPA PM and approved beforehand by both these individuals. For significant changes, the USEPA PM will consult with the A/T before providing approval.

Any modification to the SAP, FSP, or QAPP will be documented on a FCR form, which is discussed in greater detail in the QAPP. Each FCR will be uniquely numbered and will identify the project and task, the affected sections of the SAP or its supporting procedures, the scope of the requested variation, and the justification for its acceptance. At the MWH PM discretion, the FCR may be forwarded to appropriate P4 representatives for review purposes prior to implementation. The FTL will update field personnel of any changes.

5.6 REPORTS TO MANAGEMENT

The FTL will summarize the daily sampling activities in a Daily Team Leader Progress Report form. This form requires the input of the following information:

- Date activities occurred
- Identification of the field team leader and all other field sampling personnel
- Identification of subcontractors and visitors
- Summary of the work accomplished
- Identification of work planned or expected but not accomplished
- Description of activities planned for the next day of sampling

The daily progress report form is due to the P4 and MWH Project Managers at the end of day.

5.7 TRAINING REQUIREMENTS

MWH field personnel will be trained in the requirements of the *2015 LTM SAP* in a project meeting prior to the initiation of field activity. All personnel will read the *2015 LTM SAP* documents prior to the start of field work, and will acknowledge completion of training at the time of the project meeting. Meeting notes and attendance sheets will be kept and forwarded to the project records. In addition, prior to conducting each day's sampling activities, the FTL, or designee will conduct a "tailgate" meeting with field staff to review field procedures and sampling requirements, in order to better ensure that samples are collected and handled according to FSP and QAPP requirements. Tailgate meeting discussion subjects and attendees will be documented in the Field Logbook. The FTL will maintain a hard copy of the current approved version

of the entire *2015 LTM SAP* for ready-reference in the field vehicle or field office. Additionally, each field team will have a hard copy of the *2015 LTM SAP*.

6.0 PROJECT ORGANIZATION

6.1 PROJECT TEAM

The project team member information is presented below. Contact information for each member of the project team is presented in Table 6-1. The FTL will perform a thorough review of the day's field sampling forms and update the tracker table according to the day's activities (i.e., stations sampled with sample ID's and field parameters taken). This process will add an extra step in the QA/QC process to help ensure every sample is collected as discussed in the *2015 LTM SAP* and that nothing is over looked.

TABLE 6-1
PROJECT CONTACTS

Company or Agency	Contact	Title	Telephone
P4 Production	Rachel Roskelley	Program Manager	208-547-1248
USEPA	Dave Tomten	Remedial Project Manager	208-378-5763
MWH	Vance Drain	Project Manager	801-617-3250
	Emily Yeager	MWH Program Safety Officer/Field Team Lead	801-349-6276
	Ken Moosman	Database Manager	801-617-3238
	Betty VanPelt	Project Chemist	801-617-3243
Microbac Laboratories, Inc.	Kathy Albertson	Project Manager (laboratory)	800-373-4071 x179
Laboratory Data Consultants, Inc	Stella Cuenco	Project Manager (data validation subcontractor)	760-634-0437

6.2 PROJECT SCHEDULE

- Sampling –: May and September annually (SW), May annually (GW). These dates will vary depending on the precipitation during the year. The MWH/P4 PMs and EPA RPM will agree to the exact dates each year prior to beginning both the spring and the fall field programs.
- Data validation – within 60 days of receipt of laboratory data
- Data Summary Report (DSR) – within 60 days of final, validated data set approval by the A/T or as agreed to for overall annual DSR.

6.3 PROJECT DELIVERABLES

The raw data and data validation reports will be submitted to the A/T when available. Once the validated sampling data are approved by the A/T, a DSR will be submitted. This report will include:

- Field activity summary
- Data summary and detailed tables
- Figures presenting spatial distribution of data

.

7.0 REFERENCES

MHW, 2009. 2009 and 2010 Surface Water Monitoring Sampling and Analysis Plan. May 2009

MWH, 2010. 2010 Groundwater Monitoring Memorandum. May 2010.

MWH, 2011. 2011 RI/FS Work Plan – Revision 2 Final. Prepared for P4 Production. May 2011.

MWH, 2012. 2012 Surface Water and Groundwater Memo. May 2012.

MWH, 2013b. 2013 Proposed P4 LTM Surface Water and Groundwater Monitoring Plan Memo.

April 2013.

MWH, 2014. 2014 Proposed P4 Long-Term Surface Water and Groundwater Monitoring Plan Memo. April 2014.

MWH, 2015. Long Term Monitoring Surface Water and Groundwater Sampling and Analysis Plan. March 2015.

Rantz, S.E., et al. 1982. Measurement and Computation of Streamflow: Volume 1; Measurement of Stage and Discharge. Geological Survey Water-Supply Paper 2175. U.S. Government Printing Office. Washington, D.C.

ATTACHMENT A

STANDARD OPERATING PROCEDURES

SOP-NW-9.1

Collection of Surface Water Samples

Note: This document is proprietary, revision controlled, and is intended strictly for use by MWH and its teaming partners or subcontractors in support of specific contractual responsibilities. Copying and further dissemination in any manner is not permitted without written authorization by the responsible MWH Project Manager, except as may be agreed upon by MWH and its clients in the terms and conditions of applicable contracts.

1.0 SCOPE

The purpose of this document is to define the standard operating procedures (SOP) for the collection and handling of surface water samples. This SOP applies to any work performed by MWH or subcontractor personnel for any portion of surface water sampling and is intended for use in conjunction with site-specific workplans or sampling and analysis plans (SAPs). Modifications to this SOP may be made with approval of the Project Manager or Task Leader and the Quality Assurance (QA) Manager. Sampling locations shall be as specified in the governing workplan or SAP.

2.0 RESPONSIBILITIES

2.1 Field Sampling Engineer

The Field Sampling Engineer is responsible for sample collection, sample custody in the field, sample preservation, field testing, total and accurate completion of data sheets, sample shipment and delivery of data to the Project Manager, as described in this technical procedure. All staff are responsible for reporting deviations or nonconformance of the procedure to the Field Team Leader, Project Manager, or Quality Assurance (QA) Manager, in compliance with the governing workplan or SAP requirements.

2.2 Field Team Leader

- The Field Team Leader is responsible for supervising the Field Sampling Engineers. Supervision includes ensuring that samples are collected, documented, preserved, field analyzed, handled and shipped to the appropriate laboratory as specified in project work documents and this technical procedure.

2.3 Project Manager

The Project Manager has overall management responsibilities for the project, is responsible for designing the sampling program, for arranging the logistics of the program, and for providing any required clarifications in the use of this procedure. The Project Manager may assume the responsibilities of the Field Team Leader on smaller projects. The Project Manager is also responsible for maintaining project files and filing project documents, project correspondence, chain of custody forms, sediment sampling forms, generated data, and other associated and pertinent project information.

2.4 QA Manager

The QA Manager is responsible for developing and managing procedures outlined in the SOPs and in site specific SAPs, QA plans, and/or work plans.

ATTACHMENT A

STANDARD OPERATING PROCEDURES

SOP-NW-9.1

Collection of Surface Water Samples

Note: This document is proprietary, revision controlled, and is intended strictly for use by MWH and its teaming partners or subcontractors in support of specific contractual responsibilities. Copying and further dissemination in any manner is not permitted without written authorization by the responsible MWH Project Manager, except as may be agreed upon by MWH and its clients in the terms and conditions of applicable contracts.

1.0 SCOPE

The purpose of this document is to define the standard operating procedures (SOP) for the collection and handling of surface water samples. This SOP applies to any work performed by MWH or subcontractor personnel for any portion of surface water sampling and is intended for use in conjunction with site-specific workplans or sampling and analysis plans (SAPs). Modifications to this SOP may be made with approval of the Project Manager or Task Leader and the Quality Assurance (QA) Manager. Sampling locations shall be as specified in the governing workplan or SAP.

2.0 RESPONSIBILITIES

2.1 Field Sampling Engineer

The Field Sampling Engineer is responsible for sample collection, sample custody in the field, sample preservation, field testing, total and accurate completion of data sheets, sample shipment and delivery of data to the Project Manager, as described in this technical procedure. All staff are responsible for reporting deviations or nonconformance of the procedure to the Field Team Leader, Project Manager, or Quality Assurance (QA) Manager, in compliance with the governing workplan or SAP requirements.

2.2 Field Team Leader

- The Field Team Leader is responsible for supervising the Field Sampling Engineers. Supervision includes ensuring that samples are collected, documented, preserved, field analyzed, handled and shipped to the appropriate laboratory as specified in project work documents and this technical procedure.

2.3 Project Manager

The Project Manager has overall management responsibilities for the project, is responsible for designing the sampling program, for arranging the logistics of the program, and for providing any required clarifications in the use of this procedure. The Project Manager may assume the responsibilities of the Field Team Leader on smaller projects. The Project Manager is also responsible for maintaining project files and filing project documents, project correspondence, chain of custody forms, sediment sampling forms, generated data, and other associated and pertinent project information.

2.4 QA Manager

The QA Manager is responsible for developing and managing procedures outlined in the SOPs and in site specific SAPs, QA plans, and/or work plans.

3.0 DISCUSSION

The methods described by this procedure may be used to acquire water samples for chemical or radiological analysis. Methods should be selected at the discretion of the Field Team Leader or Project Manager in accordance with any specific provisions of governing SAPs, QA plans, and/or work plans.

4.0 PROCEDURES

The sampling methods described in this SOP are suitable for collecting water samples that are located at or near active and historic phosphate mines in the southeast Idaho project area. This SOP describes collecting surface water samples at four different types of water bodies, streams, seeps, reservoir or lakes, and standing water locations (ponds). Sample documentation and labeling, as well as, sampling frequency, locations, volumes and analyses shall be as specified in the governing workplan or SAP.

4.1 Decontamination

Before sampling at a new location, all water sample collection equipment will be decontaminated by rinsing the water collection equipment three times with source water, or as specified in the governing workplan or SAP.

4.2 Instrument Calibration

Electronic equipment used during sampling to obtain field parameters will include, but is not limited to, a pH meter with automatic temperature compensation, a specific conductivity meter, a dissolved oxygen meter, a turbidity meter, and an oxidation-reduction potential meter (ORP or eH). Before going into the field, the field team leader will verify that all equipment is operating properly. Calibration, times and appropriate readings will be recorded in the field notebook and as specified in the governing workplan or SAP. Meters will be calibrated according to manufacturer's instructions.

4.3 Filtering

Samples to be analyzed for dissolved state will be filtered during the field sampling event by using a disposable 45-micron filter apparatus and peristaltic pump, vacuum pump, syringe, or equivalent equipment. Other samples for a particular analysis may require filtering as specified in the governing workplan or SAP. Filtered samples will be collected according to the following procedure:

- Assemble filter device according to manufacturer's instructions.
- Filter sample either by pouring sample in the top portion of filter unit or pumping it through an in-line filter using the pump or syringe.

- Transfer filtered sample to appropriate sample bottle with required preservative.
- Dispose of the used filter and tubing.
- Decontaminate any reusable filtering equipment. Place decontaminated equipment in a clean plastic bag or container for transportation between sampling locations.

4.4 Obtaining Water Samples

The following general procedures will be used before collecting surface water samples:

- Describe physical characteristics of the water source to be sampled, including type (seep, pond, stream, reservoir, lake, etc.), any visual discoloration of water, odor, clarity, or any other notable characteristic of the water source.
- Collect field measurements of water quality parameters.
- Assemble all necessary sample collection and filtering equipment.
- Make sure that the sample labels have been correctly filled out for the sampling location. Assemble bottles for filling.
- Decontaminate sample collection equipment by rinsing the equipment three times with source water.
- Collect water sample. Ideally, the sample will be taken from a point away from where the rinsing of collection equipment occurred when sampling standing water or upstream of the rinsing point when collecting flowing water. The water collection container will be lowered into the water, taking care to avoid collecting items floating in the water and disturbing any sediment during sample collection. After the container is filled, carefully lift it out of the water and empty it into a clean sample composite container. Repeat the process at the sampling location until a sufficient amount of sample has been placed into the composite container as described below. The amount of sample to be composited shall be as specified in the governing workplan or SAP.
- Transfer to appropriately labeled containers

4.4.1 Seeps

Water samples will be collected by immersing the sample transfer container as described above, or for a raw sample, the sample container in the water source. This collection method results in a grab sample that characterizes the medium at a point in space and time, and assumes that the water body sampled (seep) is

homogeneously mixed and has no stratification. Water sample collection from seeps will be done closest to the point of discharge as possible, to minimize contamination from surficial contact.

4.4.2 Ponds

Water samples will be collected by immersing the sample transfer container as described above, or for a raw sample, the sample container in the water source. This collection method results in a grab sample that characterizes the medium at a point in space and time, and assumes that the water body sampled (pond) is homogeneously mixed and has no stratification due to it's small size. Collecting water samples from standing water will have the emphasis placed upon the sampler not to disturb the water to be sampled, and the use of a swing sampler to collect as far from the shoreline as possible. This method will be especially important when collecting samples from ponds that have loose material and high gradient slopes that define the pond shoreline, which is typical of mine pit ponds. This will also be important to minimize the immersion risk of sampling personnel. Water samples for ponds may be collected from any point along the shoreline.

4.4.3 Streams and Rivers

Samples will be collected from the same cross section of the stream as that which is used for the discharge (flow) measurement, if discharge is measured. Samples will always be collected prior to making discharge measurements. The sampler will stand downstream of the water to be sampled if safe to do so. If sampling multiple stations on the same stream, downstream samples will always be collected first. Prior to sampling, the stream will be observed for any upstream activities or events that may affect the sample quality. If such events are occurring, the sample will not be collected until the stream clears and the occurrence will be recorded in the field notes.

Selection of sampling methods and equipment based on flow conditions is as follows:

- If the stream is less than 10 feet (ft) wide, the sample will be collected from the center of the flow at mid-depth.
- If the stream is greater than 10 ft wide, one composite, consisting of three samples will be collected. The water samples will be taken at ¼, ½, and ¾ of the distance across the river or stream cross-section. These samples should be taken at mid-depth in the water column at each location across the stream channel. All three samples will be composited into one larger container, and apportioned as necessary, into the appropriate sample containers.

Water will be collected from smaller streams and rivers using a standard Kemmerer or Van Dorn horizontal water bottle sampler with the field technician standing downstream and to the side of the bottle. The sampler is a plastic cylinder with rubber stoppers that leave the ends open while being lowered to allow free passage of water through the cylinder. The Kemmerer horizontal water bottle sampler may need to be lowered on a line to reach the desired depth. Once the sampler reaches the desired depth, a 'messenger' is sent down the line, causing the stoppers to close the cylinder, which is then raised. In shallow and faster moving streams and rivers, operation of the Kemmerer horizontal water bottle sampler will be done by hand, and not with a 'messenger'. The water inside the sampler will then be transferred to a clean composite container. The process will be repeated at the sampling location until a sufficient amount of sample has been placed into the composite container. When a sufficient amount has been collected, transfer the water from the composite container into the appropriate sample containers. If unsafe wadding conditions exist (i.e., deep water, fast flowing water, slippery surfaces, etc.) a swing sampler will be utilized instead of the horizontal water bottle sampler to collect sample water for compositing. The method of sample collection shall be recorded in the field notebook.

4.4.4 Reservoirs and Lakes

Each lake and/or reservoir sample consists of collecting and compositing samples at several depths along a vertical profile using a standard Kemmerer or Van Dorn horizontal water bottle sampler. This collection method results in a vertically composited sample that characterizes the medium at a point in space and time, and takes into account the possibility of the water body sampled (i.e., reservoir or lake) is not homogeneously mixed and may be stratified. The sampler is a plastic cylinder with rubber stoppers that leave the ends open while being lowered to allow free passage of water through the cylinder. Once the sampler reaches the desired depth, a 'messenger' is sent down the line to cause the stoppers to close the cylinder, which is then raised. The water inside the sampler will then be transferred to a clean composite container. Repeat the process at the sampling location until a sufficient amount of sample has been placed into the composite container. When a sufficient amount has been collected, transfer the water from the composite container into the appropriate sample containers. A boat shall be utilized to collect samples from reservoirs and lakes.

4.5 Sampling Documentation

A field sampling data sheet will be completed at each sample location. Items not applicable to the sampling will be labeled as not applicable (NA). Sampling information will also be recorded in a bound field notebook. The information recorded on the data sheet and in the field notebook will include the following:

- Sampling location

- Date and time of sampling
- Persons performing the sampling
- Field water quality parameter measurements (specific conductivity, temperature, dissolved oxygen, pH, oxidation-reduction potential, etc.)
- Physical description of the water body to be sampled (color, odor, etc.)
- Sample identification numbers
- Number of samples taken- note containers, analytes, filtering and preservation methods
- Identification numbers of any QC samples from the site
- Any irregularities or problems which may have a bearing on sample quality

4.6 Equipment List

Sample bottles and preservatives (ultra-pure or metal grade nitric acid) will be obtained from the laboratory selected to perform the chemical analyses of the samples. Extra sample bottles with appropriate preservatives will be obtained in case of breakage or other problems.

Equipment used during surface water sample collection includes:

- Camera
- Chain of custody
- Coolers with ice/blue ice
- Copy of this SOP, SOP-NW-9.2 and -9.2a (if applicable) and the governing workplan or SAP
- Dissolved oxygen meter
- Filtering apparatus with disposable 0.45 micron filters
- GPS unit
- Hand pump, peristaltic pump, vacuum pump, or equivalent equipment
- Oxidation-reduction potential meter

- Plastic HDPE sample pitcher
- Plastic squeeze bottle filled with distilled water
- Plastic trash bags
- pH/eH meter (with automatic temperature compensation)
- Rope if obtaining deep samples
- Rite-in-the-Rain™ field notebook
- Sample containers that are certified clean
- Sample labels
- Specific conductivity meter
- Strapping and clear tape
- Swing sampler
- Kemmerer or Van Dorn horizontal water bottle sampler
- Suitable HDPE 5 L container
- Turbidity meter
- Silicon tubing
- Wooden stakes
- Writing instruments
- Measuring tape

The governing workplan or SAP may require the use of other equipment based on the scope and objective of an individual project. Project personnel will review the workplan or SAP for any equipment not listed in this SOP.

4.7 Field Quality Assurance/Quality Control Samples

Field QA/QC sample requirements shall be as specified in the governing workplan or SAP.

4.8 Sample Containers

All sample bottles must be properly cleaned and prepared. Coordinate with selected analytical laboratory for appropriate sample bottle types and preparation requirements. Plastic, such as PVC, polyethylene, polypropylene, and Tygon, is an acceptable material for contacting samples when the analyses are for inorganic analytes (metals, radionuclides, anions, and cations). Stainless steel and fluorocarbon resin (Teflon, PTFE, FEP, HDPE, or PFA) are acceptable materials that may contact surface water samples. Glass is an acceptable material for contacting samples except when silica or fluoride analyses are to be performed.

4.9 Sample Handling

Sample handling procedures and chain of custody requirements shall be as specified in the governing workplan or SAP. Typical handling procedures for surface water samples are as follows:

- Store sample containers in coolers for transportation in compliance with the sample handling and chain of custody requirements specified in the workplan or SAP.
- Sample documentation and labeling requirements shall be as specified in the governing workplan or SAP.

All surface water samples shall be labeled and sealed and immediately placed in ice-filled coolers with securely closed lids for storage and transport. The analytical laboratory must receive samples in sufficient time to conduct the requested analyses within the specified holding time.

During surface water sampling operations, the proper personal protective equipment will be worn, as described in the applicable workplan or site safety and health plan, to minimize cross-contamination and to ensure the safety of the field sampling personnel.

SURFACE WATER SAMPLE COLLECTION FORM

Project P4 Production SE Idaho Mine-Specific Program –SW Sampling

Date ___/___/___ **Arrival Time** ___

Field Personnel

_____ **Signatures** _____

_____ _____

SITE DESCRIPTION

Station Name _____ **Station Number** _____

Latitude N ___° ___′ ___″ **Longitude** W ___° ___′ ___″

Elevation _____ ft **Datum** NAD 27 **Photo Number** _____

Site & Stream Description _____

_____

Surface Water Characteristics (color, odor, appearance): _____

_____

SAMPLE COLLECTION

Collection Method: 1L bottle, Horizontal-bottle, Swing-sampler, Other(_____). Up-stream / Across-stream

Sample ID: _____ **Sample Time**:_____

Parameter	Sample 1	Sample 2	Sample 3
Time			
Water Temperature (°C)			
Specific Conductivity (µS/cm) @ 25° C			
Conductivity (µS/cm)			
TDS (g/L)			
Dissolved Oxygen (% sat.)			
Dissolved Oxygen (mg/L)			
pH			
ORP (mV)			
Turbidity (FTU)			
Air Temperature (°C)			

MWH
MONTGOMERY WATSON HARZA

SOP-NW-5.3

Collection of Groundwater Quality Samples

Note: This document is proprietary, revision controlled, and is intended strictly for use by MWH and its teaming partners or subcontractors in support of specific contractual responsibilities. Copying and further dissemination in any manner is not permitted without written authorization by the responsible MWH Project Manager, except as may be agreed upon by MWH and its clients in the terms and conditions of applicable contracts.

1.0 SCOPE

This technical procedure establishes methods for collecting groundwater samples for chemical analysis representative of aquifer water quality.

This technical procedure is designed to cover general techniques for obtaining valid, representative samples from groundwater monitoring wells. The scope is intended to provide guidance while preparing for and during actual field sampling activities. The specific methods to be used at each monitoring well are described in the applicable workplan or sampling analysis plan (SAP).

This technical procedure is applicable to all MWH personnel engaged in the collection of groundwater samples from wells for purposes of chemical analysis and are intended to be used in conjunction with a project-specific workplan or SAP.

2.0 DEFINITIONS

2.1 Dedicated Pump System

A dedicated pump system is a permanently installed device for removing water from a well. The system is not removed from the well and does not have the potential to become contaminated between uses.

2.2 Well Storage Volume

Well storage volume is defined as the volume of water enclosed by the well casing and screen gravel/sand pack under equilibrium conditions.

2.3 Bailer

A bailer is a tubular device, with a check-valve at the top and/or bottom, used for collecting and removing groundwater from wells. The three most common types of bailers are: Single check valve; double check valve; and "thief" or "messenger" samplers.

- A single check valve bailer will expose part of the sample to the atmosphere. The typical sample transfer occurs by inserting a tube into the bottom of the bailer, pushing up the check valve, which allows the water to be released into the sample container. If

used for VOC sampling the bailer should have a sample cock or draft valve at or near the bottom of the sampler allowing withdrawal of the sample from the well below the exposed surface water. Otherwise, discard the first few inches of sample at the bottom of the bailer.

- The double check valve sampler allows for point source sampling at a specific depth. The liquid will pass through the sampler while it is descending until the desired depth is reached. Once retrieval of the sample commences, both check valves close simultaneously, thereby allowing the specific depth to be sampled.

- The thief or messenger sampler also allows for point source sampling at a specific depth. A weighted messenger is dropped down the suspension line and closes the sampling vessel thus obtaining a sample at the desired depth.

To avoid potential sources of contamination, keep the bailer suspension lines off the ground and away from any other potentially contaminating sources to minimize the possibility of contaminants being carried down into the well. If needed, a decontaminated tarp or plastic bag is recommended for storage of the bailer suspension line.

2.4 Non-Dedicated Sampling Apparatus

A non-dedicated sampling apparatus is sampling equipment that is not used solely for collection of groundwater samples from a single well. This term is also used to describe equipment that is only used for sampling a single well, but is removed from the well and could potentially become contaminated.

2.5 Groundwater Sample

A groundwater sample is defined as water acquired from a well for chemical analyses that is representative of groundwater within the aquifer or the portion of the aquifer being sampled.

2.6 Positive Pressure Pump

A positive pressure pump is a device for removing water from a well by forcing water to the surface through positive pressure when operated below the water level in a well. A positive pressure pump may be operated electrically, mechanically, or by air/nitrogen pressure. Submersible impeller, bladder, and check valve pumps are common types of positive pressure pumps.

2.7 Suction Lift Pump

A suction lift pump is a device for removing water from well by negative pressure (suction). Direct line, peristaltic and centrifugal pumps are the three types of suction lift pumps. The limitation for lifting water by suction is usually 20 to 25 feet. These pumps are only acceptable for non-volatile analytes and analytes that are not affected by aeration or changes in pH. They are useful as purging devices for shallow groundwater wells.

Peristaltic pumps are preferred when a suction lift pump is required. This is the only suction lift pump, of the three mentioned above, in which the liquid being sampled moves entirely within the sample tubing. The groundwater sample never contacts the actual pump apparatus during sampling thus eliminating a possible source of sample contamination from the actual pump, lubricants, or parts.

Standard silicon tubing is the most commonly used sample tubing. Most pump manufacturers and rental companies offer acceptable tubing lined with TFE-fluorocarbons or Viton. The National Council of the Paper Industry for Air and Stream Improvement recommends medical grade silicon tubing for organic sampling;

however, it is limited to use over a restricted range of ambient temperatures. Standard silicon tubing uses an organic vulcanizing agent, which has been shown to leach into samples.

2.7 Sample Bottles

Sample bottles are containers specifically designed and prepared for storing liquid samples. Sample bottle type, material, size, and type of lid shall be as specified in the governing workplan or SAP. Sample bottles must be properly cleaned and prepared by a laboratory or the manufacturer in accordance with standard laboratory methods and procedures. Coordinate with selected analytical laboratory for bottle type and preparation requirements.

2.8 Acceptable Material

Acceptable materials are defined as pump systems that have minimal effect on water quality when used to obtain groundwater samples from wells. The use of pumps is dependent on the analyses being conducted on the acquired samples. The parts of pumps that will contact the groundwater sample contain only acceptable materials.

3.0 RESPONSIBILITIES

3.1 Field Sampling Engineer

The Field Sampling Engineer is responsible for sample collection, sample custody in the field, sample preservation, field testing, total and accurate completion of data sheets, sample shipment and delivery of data to the Project Manager and designated project secretary, all as described in this technical procedure.

3.2 Field Team Leader

The Field Team Leader is responsible for supervising the Field Sampling Engineers. Supervision includes ensuring that samples are collected, documented, preserved, field analyzed, handled and shipped to the appropriate laboratory as specified in project work documents and this technical procedure.

3.3 Project Manager

The Project Manager has overall management responsibilities for the project, is responsible for designing the sampling program, for arranging the logistics of the program, and for providing any required clarifications in the use of this procedure. The Project Manager may assume the responsibilities of the Task Leader on smaller projects.

The Project Manager is also responsible for maintaining project files and filing project documents, project correspondence, chain of custody forms, groundwater purging and sampling forms, generated data, and other associated and pertinent project information.

4.1 DISCUSSION

The following is a listing of general procedures that should be followed during every sampling event.

- Groundwater samples shall be collected in quantities and types as described in the governing workplan or SAP.
- Water-level data collection (see MWH SOP-5 for appropriate procedure).
- Determination of well storage volume (see Section 5.2.1, below).

- All instruments used for field analyses should be calibrated in accordance with the manufacturer's instructions.

- All non-dedicated sampling equipment shall be decontaminated before and after each use.

- If so directed by the governing workplan or SAP, purge water and decontamination fluids shall be captured and contained for disposal.

- Samples shall be collected in properly prepared containers of the appropriate size and type, and all samples shall be appropriately labeled and sealed, as required by the governing workplan or SAP.

- Samples shall be stored and transported in coolers chilled to 4° Celsius or less. Sample handling and chain of custody shall be as specified in the governing workplan or SAP.

- The Groundwater Purging and Sampling Form (see Appendix A) and the field log book shall be used to document daily site activities and sample collection.

- All variations from established procedure shall be documented on the Groundwater Purging and Sampling Form (see Appendix A) and shall be approved by the Project Manager prior to proceeding with the work variation.

5.1 General Considerations

5.1.1 Decontamination

All non-dedicated sampling equipment that may contact the sample must be decontaminated before and after each use unless it is disposable and being used for only a single well. Non-dedicated pumps or bailers require decontamination of internal and external parts prior to being lowered into the well. Non-dedicated equipment shall first be washed with clean tap water (whose chemistry is known and acceptable), non-phosphate detergent, and rinsed with clean tap water. For inorganic analytes a weak hydrochloric acid (HCl) or nitric acid (HNO_3) solution shall be used for the second wash. For

organic analytes, reagent-grade methanol shall be used for the second wash. A final rinse with organic-free distilled/deionized water shall complete the decontamination. At a minimum, all acid and methanol wash solutions must be captured (see Section 5.5.1, below).

5.1.2 Sample Quantities, Types, and Documentation

Samples shall be collected in quantities and types as specified in the governing workplan or SAP.

The Groundwater Purging and Sampling Form (Appendix A) and the field logbook shall be used to document daily site activities and sample collection (see Section 5.6, below). Samples shall be transferred to the analytical laboratory under formal chain of custody, which shall be documented and maintained in accordance with procedure.

5.1.3 Sample Containers

All sample bottles must be properly cleaned and prepared. Coordinate with selected analytical laboratory for appropriate sample bottle types and preparation requirements. All groundwater samples shall be labeled and sealed and immediately placed in coolers chilled to 4º Celsius or less with securely closed lids and custody seals on the outside of the cooler for storage and transport. Samples must be received by the analytical laboratory in sufficient time to conduct the requested analyses within the specified holding time.

5.1.4 Acceptable Materials

Stainless steel and fluorocarbon resin (Teflon, PTFE, FEP, or PFA) are acceptable materials that may contact groundwater samples. Glass is an acceptable material for contacting samples except when silica or fluoride analyses are to be performed. Plastics (PVC, polyethylene**,** polypropylene, tygon) are an acceptable material for contacting samples when the analyses are for inorganic analytes (metals, radionuclides, anions, cations).

5.1.5 Sample Acquisition

Groundwater samples shall be removed from the well with the use of a permissible pump or bailer. Electric positive-pressure pumps, as defined in Section 3.6, made of acceptable materials are permissible to use for acquiring any groundwater sample. Air/nitrogen pressure activated positive-pressure pumps made of acceptable materials are permissible to use for acquiring any groundwater sample if the air/nitrogen does not contact the sample. Positive-pressure pumps operated by mechanically forcing water through check valves are permissible for acquiring groundwater samples. Bailers made of acceptable materials are permissible for acquiring groundwater samples.

Peristaltic pumps and airlift pumps are not preferred for acquiring groundwater samples but are permissible when samples are to be analyzed for analytes that are not volatile, are not affected by aeration, and are not affected by changes in pH.

Other types of pumps (peristaltic, centrifugal, air lift, recirculation, etc.) may be used for purging groundwater from wells prior to sample acquisition, if: (1) pump materials contacting well water are acceptable; (2) pumping does not aerate or change the pH of the remaining well water; and (3)

pumped water does not mix with remaining well water during pumping or after the pumping is stopped.

5.2 Equipment And Materials

If wells are equipped with dedicated pump systems, equipment to operate the dedicated pump systems (i.e., air compressor, compressed air or nitrogen cylinders, electric generator, etc.) as well as non-dedicated sampling apparatus, such as surface discharge tubing and valves, may be necessary.

If wells do not have permissible and dedicated pump systems, permissible pump systems or bailers and accessories of small enough diameter to enter the wells will be necessary. All equipment that could contact the sample shall be made of acceptable materials.

Sample bottles and preservatives appropriate for the parameters to be samples will be necessary. Coordinate with selected analytical laboratory for bottle type and preparation requirements.

Field test equipment necessary for groundwater sampling:

- Thermometer
- pH meter and standards
- Conductivity meter and standards
- dissolved oxygen meter (optional)
- turbidity meter and standards
- Filtration apparatus (0.45 micron), if necessary
- Depth to water measuring device
- Coolers and ice packs
- Organic-Free Distilled or deionized water
- Cleaning equipment (scrub brushes) and solutions (non-phosphate detergent)
- Indelible ink pens and felt-tip markers
- Sample labels and seals
- Container(s) for capturing, containing, treating and measuring waste decontamination solutions, if necessary.
- Well specifications
- Groundwater Purging and Sampling Forms (Appendix A)
- Field logbook
- Chain of Custody Forms (provided by selected laboratory)
- As required, 55 gallon steel drums fitted with bung holes, or suitable tank(s) for containing purged groundwater.
- Additional preservatives (i.e., HNO_3)
- Copy of this SOP, SOP-5 and the governing workplan or SAP.

5.3 Groundwater Sample Acquisition

5.3.1 Purging the Well

The pump or bailer shall be used and operated in accordance with the manufacturer's operational manual. Before collecting the actual groundwater sample, a minimum of three (3) well bore storage volumes of water shall be purged from the well by pumping unless low-flow sampling techniques are being employed. Calculate this volume by measuring the depth to water and subtracting this depth from the total depth of the well. If a gravel/sand pack surrounds the screen, the pore volume of the gravel/sand pack (assume a porosity of 25 percent if unknown) shall be added to the total well volume. While purging water from the well, the conductivity, pH, temperature, dissolved oxygen (optional) and turbidity of the water shall be periodically measured. If the conductivity (within 10%), pH (within 0.1 pH units), temperature (within 0.5 degrees Celsius), dissolved oxygen (within 10%) or turbidity (within 10% and less than 5 NTU) of the water has not stabilized when a minimum of three (3) well volumes have been purged, then continue to purge water until these parameters stabilize as specified above. If the parameters of interest in the investigation include VOCs, care must be taken to ensure that purging does not induce degassing within the well. Where the well screen and sand pack are completely below the water table, the rate of purging should be controlled such that it does not draw the water level in the well below the top of the well screen. Where the well screen and sand pack are intersected by the groundwater level, the rate of purging should correspond with the rate of sampling, if continuous sampling methods are used. Large drawdowns in water table wells should be avoided. Purged groundwater that has a reasonable potential of containing hazardous substances shall be captured and characterized prior to discharge or disposal (see Section 5.5.1, below). Record the time after each well volume has been purged in order to keep track of actual pumping rate.

5.3.2 Samples for Major Cation, Metal, and Metallic Radionuclide Analyses

Samples for major cation, metal, and metallic radionuclide analyses (including Se) shall be immediately filtered after acquisition. Filtration is best accomplished with the use of an in-line filter system in which the sample is directly fed from the discharge port of a positive-pressure pump through the filter and into the appropriate sample bottle. A less preferred but acceptable method is the collection of an adequate amount of sample from a permissible positive-pressure pump or bailer into a properly cleaned and prepared high-density linear polyethylene or glass bottle. Samples shall be immediately fed through the filter and collected directly into the appropriate sample bottle. The final filter pore size should be 0.45 micron. (Note: New filters shall be used for each sample, and the filter system must be decontaminated before and after each sample.) These groundwater samples shall be preserved after filtration with nitric acid ($HN0_3$) to a pH less than 2.

In addition, if toxic metal or priority pollutant metal analyses are to be performed, an unfiltered sample will also be obtained (in addition to the filtered sample) directly from a permissible positive-pressure pump discharge port or from the bailer into appropriate sample bottles and preserved with nitric acid ($HN0_3$) to a pH less than 2. The only exception is analysis of chromium VI, in which case preservatives shall not be added to the sample.

5.3.3 Samples for Cyanide Analyses

Samples for cyanide analyses shall be collected directly into appropriate sample bottles from the bailer or the port of a positive-pressure pump. Samples shall not be filtered nor shall they be allowed to overflow the sample bottle. Samples shall be immediately preserved with sodium hydroxide (NaOH) to a pH greater than 12.

5.3.4 Samples for Major Anion and Biological Oxygen Demand (BOD) Analyses

Samples for major anions (chloride, fluoride, sulfate, alkalinity, acidity, total silica, bromfide) and for biological oxygen demand shall be collected directly into appropriate sample bottles from the port of the pump or from the bailer. These samples do not require filtration, but may be filtered, if desired. Preservatives shall not be added.

5.3.5 Samples for Total Phosphate and Orthophosphate Analyses

Groundwater samples for total phosphate and orthophosphate analyses shall be immediately filtered after initial sample acquisition. Filtration is best accomplished with an in-line system in which a positive-pressure pump discharge port feeds groundwater directly through the filter system into an appropriate sample bottle. A less preferred but acceptable method is to collect a liter of sample from a positive-pressure pump or bailer in a properly cleaned and prepared high-density polyethylene or glass bottle, and then immediately feed the sample through a filter system which discharges into the appropriate sample bottle. The final filter pore size shall be 0.45 micron. (Note: New filters must be used for each sample and the filter system must be decontaminated before and after each use.) Samples shall be immediately preserved with sulfuric acid (H_2SO_4) to pH less than 2.

5.3.6 Samples for Nitrogen Compound, Chemical Oxygen Demand, Oil and Grease, and Total Organic Carbon Analyses

Groundwater samples for nitrogen compound, chemical oxygen demand, oil and grease, and total organic carbon analyses shall be collected directly into appropriate sample bottles from a permissible positive pressure pump discharge port or from the bailer. These samples shall not be filtered and shall be preserved with sulfuric acid (H_2SO_4) to a pH less than 2.

5.3.7 Samples for Analysis of Total Dissolved Solids

Groundwater samples for analyses of total dissolved solids shall be immediately filtered in the field by methods discussed in Section 5.3.3 above, and collected directly into an appropriate sample bottle. Samples shall not be preserved with additives.

5.4 Field Analyses

5.4.1 Calibration of Instruments

All instruments used for field analyses shall be calibrated prior to use and daily during use. Only equipment with a calibration tag showing a recall date later than the anticipated date of use shall be taken to the field. Each instrument should be accompanied by a copy of the manufacturer's operation manual.

5.4.2 Water Temperature

A pocket thermometer or other measuring device shall be used to measure the temperature of the water on an aliquot of purged water obtained just before or after sampling. The thermometer reading shall be allowed to stabilize and shall be recorded to the nearest 0.5 degree centigrade. The instrument shall be rinsed with distilled or deionized water before and after each use.

5.4.3 pH Measurement

A pH meter shall be used to measure the pH of the sample on sample of purged water that was obtained just before or after sampling. Measurements shall be made immediately on the obtained sample (note: if possible, measure pH continuously on the purged water in a closed flow-through system). Calibration shall be in accordance with the manufacturer's procedures (provided with the instrument). Calibration shall be performed with standardized buffered pH solutions bracketing the range of expected pH and conducted at the beginning and end of each day. Before and after each reading, the probe shall be thoroughly rinsed with distilled or deionized water. The pH shall be recorded to one-tenth (or one-hundredth if the meter is stable enough) of a pH unit.

5.4.4 Conductivity Measurement

A conductivity probe shall be used for conductivity measurement on an aliquot of purged water obtained just before or after sampling. Measurements shall be made as soon as possible on the obtained aliquot. The meter shall be calibrated in accordance with manufacturer's procedures (provided with the instrument) with standardized KCl solutions. At a minimum calibration shall be performed at the beginning and ending of each days use. The conductivity shall be recorded to two significant figures. The temperature of the sample at the time of conductivity measurement shall also be recorded. The probe must be thoroughly rinsed with distilled/deionized water before and after each use.

5.4.5 Dissolved Oxygen Measurement

A dissolved oxygen meter is used to measure dissolved oxygen (DO) in water samples. Measurements shall be made immediately on aliquots obtained just before or after sample acquisition. (Note: If possible, measure DO continuously on the purge water in a closed flow-through system). The meter shall be calibrated in accordance with the manufacturer's procedures (provided with the instrument) using distilled/deionized water that has been allowed to equilibrate with the atmosphere at a given elevation. The salinity adjust shall be adjusted to the appropriate salinity of the water. Measure the temperature and concentration of dissolved oxygen in the sample while the salinity is on the fresh setting. The probe must be thoroughly rinsed with distilled or deionized water before and after each use. Measurements shall be recorded to the nearest 0.1 ppm concentration.

5.4.6 Turbidity Measurements

A turbidity meter shall be used to make turbidity measurements on aliquots of water samples obtained just before or after sample acquisition. Measurements shall be made as soon as possible on the obtained aliquot. Operation and calibration shall be in accordance with the manufacturer's procedures (provided with the instrument). Standardized solutions shall be used for calibration. The instrument shall be calibrated at least once during the purging and sampling of each well. The outside of the glass vials used for containing the aliquot for measurement must be wiped thoroughly dry before and after each use. Measurements shall be recorded to the nearest 0.1 NTU when less than 1 NTU; the nearest 1 NTU when between 1 and 10 NTU; and the nearest 10 NTU when between 10 and 100 NTU.

5.5 Capture And Disposal Of Purge Water And Decontamination Solutions

5.5.1 Purge Water

If specified in the applicable workplan or SAP, purged groundwater shall be captured and contained in 55 gallon steel drums or suitable tank(s). If so required, each drum or tank containing captured purge water shall be properly labeled with a weatherproof label as to the contents, the well(s) from which the contained purge water originated and the date in which the contents were generated. Storage of the drums or tanks shall be as specified in the project work documents or as directed by the Project Manager.

Captured and contained purge water shall be characterized for discharge, treatment and/or disposal. Characterization of the captured and contained purge water should be specified in the project work documents or by the Project Manager, but could rely on the analytical results of groundwater samples associated with each drum or tank, or could involve direct sampling and analyses of the contained water.

The requirements and options available for discharge, treatment and/or disposal are dependent upon many variables such as chemical consistency, local and state regulations, and location of site. Discharge, treatment, and/or disposal of captured and contained purge water must be in accordance with local, state and Federal regulations and shall be specified in the project work documents.

5.5.2 Decontamination Waste Solutions

Decontamination waste solutions that are generated during groundwater sampling include: spent detergent wash solutions; spent tap water rinses; any spent weak acid rinses, any spent methanol rinses; and spent final distilled/deionized water rinses. All spent acid and methanol rinses shall be captured and contained in plastic buckets or drums. Other spent decontamination waste solutions shall be captured and contained in appropriately sized buckets or drums if a reasonable potential exists for the spent solutions to contain hazardous substances. Project work documents shall address, or the Project Manager shall determine, whether spent decontamination solution requires capture and containment.

Captured and contained decontamination waste solutions shall be subject to the same procedures as described for purge water. Some noteworthy differences are as follows: (1) all acid solutions shall be neutralized with lime prior to discharge or disposal; (2) methanol solutions may be able to be evaporated if segregated from other waste solutions, if generated in small enough quantities, and if conditions are favorable; and (3) if quantities are sufficiently small, decontamination waste solutions (detergent washes, rinse waters, neutralized acid solutions) may be added to the captured and contained purge water that corresponds to the same well sampling effort.

5.6 Documentation

Documentation for sampling groundwater includes labeling sample bottles, and completing Groundwater Purging and Sampling Forms, and Chain of Custody Records, and securing individual samples or sample coolers with chain of custody seals.

5.6.1 Sample Labels

Samples shall be immediately labeled. Labels shall be waterproof. Information shall be recorded on each label with indelible ink. All blanks shall be filled in (N/A if not applicable). Groundwater sample designations shall be as specified in the governing workplan or SAP.

5.6.2 Groundwater Purging and Sampling Forms

Groundwater Purging and Sampling Forms (Appendix A) shall be used by the Field Sampling Engineer to record daily activities. Data shall be recorded on the Groundwater Purging and Sampling Form in chronological format. The time of each recorded event shall be included. The original Groundwater Purging and Sampling Form shall be submitted as soon as possible to the Project Manager or Task Leader.

5.6.3 Field Log Book

The field logbook is used by the Field Sampling Engineer to document the official raw field information for each sample that will be chemically analyzed. The original must be submitted as soon as possible to the Project Manager or Task Leader.

5.6.4 Chain of Custody Records

Sampling handling and chain of custody protocols shall be as specified in the governing workplan or SAP.

Chain of custody records will normally be used to record the custody and transfer of samples. Chain of custody forms are normally provided by the selected analytical laboratory. These forms shall be filled in completely (use N/A if not applicable). Tamper-proof seals shall be placed on either sample bottles or shipping coolers. The seal number shall be recorded on the Chain of Custody Form. The original form must accompany the samples to the analytical laboratory to be completed and returned to MWH for filing. A copy of the Chain of Custody Record documenting the transfer of samples from the field shall be submitted to the Project Manager or Task Leader.

6.0 REFERENCES

ASTM, 1995, ASTM Standards On Environmental Sampling - (PCN - 03-418095-38). ASTM, Philadelphia, PA.

U.S. EPA, 1986, RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, U.S. EPA/ Office of Solid Waste, Washington D.C.

U.S. EPA, 1986, Test Methods for Evaluating Solid Waste - (SW-846). U.S. EPA/Office of Solid Waste, Washington, D.C.

Wood, W.W. (1976), Guidelines for Collection and Field Analysis of Ground-Water Samples for Selected Unstable Constituents, Techniques of Water-Resources Investigations of the United states Geological Survey, Book 1, Collection of Water Data by Direct Measurement, Chapter D2.

APPENDIX A

GROUNDWATER PURGING AND SAMPLING FORM

WELL SAMPLING FORM

Page: _____ of _____

Project No.: _____ Client: P4 Date: _____ Well ID: _____

Task: _____ Field Crew: _____

Measuring Point (MP): _____ Water Level (WL): _____ ft below MP

Sampling Method (see applicable calculations below): _____ Device: _____

Low Flow Sampling

Minimum Purge = 2* (feet * vol tubing + vol pump): _____ Starting PSI = (½ WL + 20): _____
Tubing vol = 0.03L/ft, Bladder pump vol = 0.5L]

3-Volume Purge Sampling

Total Depth (TD): _____ ft below MP Water Height (H): _____ ft below MP Casing Radius (r): _____ ft

Three Purge Volumes = 3*[Pi * r^2 * H * 7.48]: _____ gal Required Run Time: _____ min/ hr
H= TD-WL, Pi=3.1415, 1gal = 3.785L, 4in casing=0.653gal/ft, 6in casing = 1.469gal/ft

STABILIZATION READINGS									
Time (24 Hr)	Water Level (ft below MP)	Purge Rate (gal or L/min)	Cumulative Purge Vol (gal or L)	Temp (C)	pH	ORP/Eh (mv)	DO (mg/L)	Specific Conductivity (µS/cm)	Turbidity (NTU)
Stabilization Limit	+/- 0.3 ft	Low flow ≤ 1L 3-vol = no limit	2 tube vol or 3 well vol	+/- 1C	+/- 0.1	+/- 10	+/- 0.2	+/- 3%	≤ 5
Continue stabilization readings on additional pages if necessary									

FINAL READINGS									
Time (24 Hr)	Water Level (ft below MP)	Purge Rate (gal or L/min)	Cumulative Purge Vol (gal or L)	Temp (C)	pH	ORP/Eh (mv)	DO (mg/L)	Specific Conductivity (µS/cm)	Turbidity (NTU)

SAMPLE ID: _____ **TIME:** _____

Final Low Flow Sampling Settings: PSI: _____ Charge: _____ sec Exhaust: _____ sec

WELL SAMPLING FORM
Continued Stabilization Readings

Page: _____ of _____

Project No.: ________________ Client: P4________________ Date: _______________ Well ID: ______________

STABILIZATION READINGS									
Time (24 Hr)	Water Level (ft below MP)	Purge Rate (gal or L/min)	Cumulative Purge Vol (gal or L)	Temp (C)	pH	ORP/Eh (mv)	DO (mg/L)	Specific Conductivity (µS/cm)	Turbidity (NTU)
Stabilization Limit	+/- 0.3 ft	Low flow ≤ 1L 3-vol = no limit	2 tube vol or 3 well vol	+/- 1C	+/- 0.1	+/- 10	+/- 0.2	+/- 3%	≤ 5

SOP-5

Groundwater Level Data Collection

Note: This document is proprietary, revision controlled, and is intended strictly for use by MWH and its teaming partners or subcontractors in support of specific contractual responsibilities. Copying and further dissemination in any manner is not permitted without written authorization by the responsible MWH Project Manager, except as may be agreed upon by MWH and its clients in the terms and conditions of applicable contracts.

1.0 PURPOSE

The purpose of this technical procedure is to establish a uniform and consistent procedure for measuring water levels in wells, piezometers and boreholes.

The groundwater level measurement procedures presented in the following sections were developed using standard industry-accepted practices, as well as international organization and agency guidelines and standard practices. The same care must be exercised in implementing field investigations and sampling events that are exercised in planning the program design and analyzing samples in the laboratory. No analytical result is better than the sample from which it was obtained.

Specific organizations and agencies with guidelines and standard procedures that were used include:

- U.S. Environmental Protection Agency (EPA)
- State of Nevada (U.S.) Division of Environmental Protection (NDEP)
- American Society of Testing and Materials (ASTM)

2.0 DEFINITIONS

2.1 Electric Water Level Sounder (EWS)

An electronic water-level sounder (EWS) is an instrument for measuring water levels in wells, piezometers and boreholes. An EWS is essentially an open circuit involving an ammeter and battery mounted on a reel to which an insulated two-wire electric cord (calibrated by length) is wound. The circuit is closed, and a buzzer sounds when the electrodes on the probe are immersed in water. Depth to water is recorded at the depth where the buzzer sounds.

3.0 DISCUSSION

Measurement of static water levels may constitute a separate task or be performed in conjunction with groundwater sampling. Prior to any purge sampling activity at each monitor well, a water level measurement is required to be taken. Measurement of the static water level is important in determining the hydrogeologic characteristics of the groundwater system.

Prior to taking the EWS to the field, check that the EWS is functioning properly and that the batteries are in working order by turning instrument on and pushing the test button located on the side of the instrument. Also verify that the probe is functioning properly by submerging it in tap water. Both the audio and visual signals should function.

Decontaminate the probe and cord of the EWS using Alconox or equivalent non-phosphate detergent and distilled water. Rinse a minimum of three (3) times with distilled water. At a minimum decontaminate the probe and the length of reel you believe will be in the well plus an additional 3 meters.

The measurement will be referenced from the reference point marked on the top of the well casing; this is typically located on the north side of the casing. The measurement to the static water level in the well will be to the nearest 0.01-foot interval. The measurement will be immediately repeated to verify the accuracy of the initial reading. The depth to water measurement will be compared in the field to previous measurements to verify that the measurement is reasonable. Record the depth to water level on the field logbook and Record of Water Level Readings form (see Appendix A). Other items to record include well identification number, casing diameter, vertical height of measuring point above ground surface, and time and date of measurement.

If depth to water is measured in an open borehole, note that the reference level is ground surface. Also note, especially if the ground is uneven, from which side of the borehole (i.e. north, etc.) the measurement was referenced.

In addition, it is good practice to periodically measure total well depth, since silt can build up and decrease the total depth of the well. Measure the total depth of the well following determination of static water level. If using the EWS to determine the depth of the well, make sure that the additional cable that will be submerged has been decontaminated and that the probe tip length is added to the total depth measured. Total well depth measurement also ensures that the well is in good condition to total depth.

4.0 EQUIPMENT AND MATERIAL

The following is a list of equipment that should be available in the field to perform water level measurements.

- Electric Water-level Sounder or Measuring Tape with a Wetable Surface
- Folding Rule
- Field logbook or field data sheet (see Record of Water Level Readings form, found in Appendix A)
- Data on Well Identification Number and Locations
- Spare Battery for Electric Water-level Meter
- Permanent, waterproof pens

5.0 PROCEDURES

- Record well identification number and measuring device type and serial number.

- Each water level sounder or measuring tape used for recording water levels should have the depth graduations checked with an independent folding rule or measuring tape for calibration prior to field use.

- Clean all downhole instruments and equipment before and after measurements between wells. Cleaning should be with a non-phosphate detergent rinse followed by a rinse with approved tap water, then rinse with organic free distilled or deionized water.
- Measure and record distance from ground level to top of casing or standpipe. Measure the vertical distance from the top of casing or standpipe to the point of the elevation survey mark (if different from top of casing or standpipe).

- If an EWS is used, turn on the EWS, check the battery, lower the wire into the borehole or standpipe and stop at the depth where the EWS meter indicates a repeatable, completed circuit. Record the length of the wire below the casing collar or top of the standpipe to the nearest 0.01-foot.

- If a measuring tape is used, lower the tape (with a weight attached) into the borehole. The tape must be lowered a sufficient depth into the well to ensure the wetable surface section of the tape is partially submerged. The total length of the tape within the well (from the top of casing or standpipe) and the length of the wetted surface to the submerged end of the tape will be recorded.

- Record date, time, well designation, measuring device and all measurements on a Record of Water Level Readings form (Appendix A), and bound logbook. The personnel making the measurement will initial or sign each measurement recorded. All water level measurement records will be maintained in the project records files.

6.0 REFERENCES

American Society of Testing and Materials (ASTM). 1994. *Standards on Ground Water and Vadose Zone Investigations*. Second Edition. ASTM Committee D-18 on Soil and Rock. Philadelphia, Pa.

American Society of Testing and Materials (ASTM), 1995. *ASTM Standards on Environmental Sampling*. PCN-03-418095-38, Philadelphia, Pa.

U.S. Environmental Protection Agency (EPA), 1994. *Standard Operating Procedures for Field Sampling Activities, Version 2*. EPA Region VIII, Denver, Colorado.

U.S. Environmental Protection Agency (EPA), 1995. *Standard Operating Procedures for Water Level and Non-Aqueous Phase Liquid (NAPL) Measurements in Boreholes and Monitoring Wells, Version 1*. EPA Region VIII, Denver, Colorado.

SOP-NW-9.2a

Surface Water Flow Measurements Using Man-Portable Devices or Estimation Techniques

Note: This document is proprietary, revision controlled, and is intended strictly for use by MWH and its teaming partners or subcontractors in support of specific contractual responsibilities. Copying and further dissemination in any manner is not permitted without written authorization by the responsible MWH Project Manager, except as may be agreed upon by MWH and its clients in the terms and conditions of applicable contracts.

1.0 SCOPE

This Standard Operating Procedure (SOP) provides general techniques for obtaining valid, representative flow measurements from natural open-channels using estimation techniques or man- portable devices. Direct methods for flow measurements in open channels using weirs, flumes, or other devices and direct and indirect flow measurements in pipes are provided in SOP-NW-9.2, *Surface Water Flow Measurements*. This SOP applies to any work performed by MWH or subcontractor personnel for any portion of stream flow measurements and is intended to be used in conjunction with governing work plans or sampling and analysis plans (SAPs). Modifications to this SOP may be made with the approved by the Project Manager or Task Leader and the Quality Assurance (QA) Manager.

Stream flow measurement methods in this SOP assume the following conditions:

- No control structures will be used for flow measurements at surface water stations;
- A current meter will be the preferred method for discharge measurements; and
- A few locations may be appropriate for engineering estimates or volumetric measurement.

2.0 DEFINITIONS

Discharge is defined as the volume rate of flow of water, expressed in cubic feet per second (cfs), including any substances suspended or dissolved in the water. Methods for measuring discharge are based on a variety of flow conditions. Many discharge measurement methods are required because flow conditions differ from site to site.

3.0 RESPONSIBILITIES

3.1 Field Sampling Engineer

The Field Sampling Engineer is responsible for field testing, total and accurate completion of data sheets, and delivery of data to the Project Manager and designated project secretary, all as described in this technical

procedure. All staff are responsible for reporting deviations or nonconformance of the procedure to the Field Team Leader, Project Manager, or Quality Assurance (QA) Manager, in compliance with the governing workplan or SAP requirements.

3.2 Field Team Leader

The Field Team Leader is responsible for supervising the Field Sampling Engineers. Supervision includes ensuring that stream flows are measured as specified in project work documents and this technical procedure.

3.3 Project Manager

The Project Manager has overall management responsibilities for the project, is responsible for designing the sampling program, for arranging the logistics of the program, and for providing any required clarifications in the use of this procedure. The Project Manager may assume the responsibilities of the Field Team Leader on smaller projects.

The Project Manager is also responsible for maintaining project files and filing project documents, project correspondence, chain of custody forms, surface water flow measurement forms, generated data, and other associated and pertinent project information.

3.4 QA Manager

The QA Manager is responsible for developing and managing procedures outlined in the SOPs and in site specific SAPs, QA plans, and/or work plans.

4.0 DISCUSSION

The methods described by this procedure may be used to measure flow in open channels. Methods should be selected at the discretion of the Field Team Leader or Project Manager in accordance with any specific provisions of governing SAPs, QA plans, and/or work plans.

Because of the dynamic nature of surface water behavior, flow measurement by the methods described in this document may, on occasion, be impossible at some sites. It is understood that if unmeasurable flow conditions are encountered at any of the surface water data collection sites which are to be measured in this program, the field team will attempt to measure flow at a point upstream or downstream of the site and will

note this point relative to the marked data collection point in the field logbook. Whether or not a measurement is made, the team will note the conditions that inhibited accurate flow measurement. This situation will be brought to the attention of the MWH Project Manager and QA Manager using the field change protocol established in the governing workplan or SAP.

5.0 PROCEDURES

5.1 Velocity-Area Method

Surface flow in stream channels that are greater than one foot wide, or where flow is estimated to exceed 2.0 cfs, will be measured by using the traditional stream gaging technique, the velocity-area method.

When using the velocity-area method to perform discharge measurements of flowing surface water streams, a current meter will be used. The most common current meters are vertical axis meters, such as the Price meter Type AA, and horizontal axis or propeller-type meters. The standard Price meter has a rotor 5 inches in diameter and 2 inches high with six cone-shaped cups mounted on a stainless steel shaft. A pivot bearing supports the rotor shaft. In addition to the type AA meters, a Price pygmy meter is used in shallow depths. The pygmy meter is scaled two-fifths as large as the standard meter and has no tailpiece. Propeller-type current meters employ a propeller turning about a horizontal axis. Variable flow conditions may require the use of the pygmy meter, the Price AA meter, or a propeller- type meter, depending on the amount of runoff contributing to streamflow.

5.1.1 Theoretical Considerations

The volume rate of flow of water, which is commonly called discharge (Q), is the product of multiplying the average velocity (V) times the total cross-sectional area (A):

$$Q = V * A$$

The current meter measures velocity at a point. The velocity-area method of making discharge measurements at a cross-section requires measurement of the mean velocity in multiple portions of the cross-section at each of the selected verticals. These are taken at subsections of the cross-section. A complete discussion of area-velocity methods is found in *Measurement and Computation of Streamflow: Volume 1 - Measurement of Stage and Discharge* (Rantz, 1982)

By dividing the stream width into subsections, total discharge becomes the total of discharges measured in each subsection. Velocity (v) is estimated using the two-point method, or for shallow streams, the six- tenths-depth method, measured at each subsection. The two-point method consists of measuring the velocity at

0.2 and then at 0.8 of the depth from the water surface, and using the average of the two measurements. For streams shallower than 2 feet, the six-tenths-depth method will be used. The six- tenths-method consists of measuring the velocity at 0.6 of the depth from the water surface.

Discharge becomes the sum of the products of each point velocity and cross-sectional area of each subsection:

$$Q = \Sigma (v * a)$$

where:

Q = total discharge (in cubic feet per second),

v = point velocity (feet per second), and,

a = area of the subsection (square feet).

In measuring discharges for developing stream ratings for litigation and flood-plain insurance purposes, federal agencies typically base the number of subsections on the criterion that each subsection contain no more than 5 percent of the total discharge. While this method has reportedly resulted in measurement accuracy as high as 98 percent, factors such as the characteristics of the measurement section reduce this accuracy. Furthermore, measurements limiting flow to 5 percent of the total discharge are time-consuming. Therefore, in the interests of conserving time while maximizing measurement accuracy, current-meter measurements performed in channelized streams will be based on selecting subsections to contain approximately 10 percent or slightly more of the total discharge.

However, the stream should not be partitioned into sections that are significantly greater than 10 percent of the total stream flow because individual measurements that may be in error will then have a significant impact on the overall average velocity determination.

In general, depending on average depth and velocity distribution, a stream less than 2 feet wide will require no more than 8 to 10 subsections. A stream up to 4 feet wide will require about 10 to 12 subsections. Streams wider than 4 feet will require more subsections. Further, subsections need not be of identical width. For example, because velocities near banks are generally lower than velocities near the center of streams, these subsections may be wider than subsections near the center. Subsections will also be more closely spaced if a stream has an unusually deep portion in the cross-section, or if velocities are higher than usual for the cross-section.

Velocity will be observed by current meter at each point for a period that ranges from 40 to 70 seconds.

The stage of a stream is the height of the water surface above an established datum plane. The water-surface elevation referred to is an arbitrary gage datum called the "gage height." Stage or gage height is to be measured and recorded in feet and tenths of a foot.

5.1.2 Required Measurement Conditions

In order to make a velocity-area discharge measurement using a flowmeter, the following conditions are required:

- The stream must be channelized: that is, observable banks must channel the stream flow;
- Depth must be greater than 0.2 foot across most of the cross-section being measured; and
- The stream must have measurable velocity of at least 0.2 feet per second in most of the cross-section, although the pygmy meter is capable of measuring velocity as low as 0.070 feet per second.

The first two conditions can often be met in streams of very low discharge by conservatively modifying the stream channel to produce a narrower and slightly deeper cross-section in order to meet measurement requirements. These modifications will include removal of aquatic growth or ice, moving large stones which impact velocity upstream or downstream of the cross-section, and narrowing or deepening of the cross-section. By rearranging small amounts of native rock or sand, the technician will produce a measurable cross-section. When such modifications are made, great care will be exercised to avoid unnecessary movement of sediments or the splashing of sediments or water onto field team members. After clearing the cross-section, flow will be allowed to stabilize before the current-meter measurement of velocities begins.

If depths of 0.2 feet or greater cannot be found or made, the velocity will be estimated by recording the time required for a floating object to cover a known length of the stream. The object velocity will be calculated by multiplying the fixed distance with the average stream area and a correction factor of .9 and dividing by the recorded time. At a minimum, three separate trials will be completed and a mean object velocity calculated.

Current meter measurements are best made by wading, if conditions permit. The Price AA, Pygmy, or propeller-type meters are used for wading measurements. Vertical axis current meters do not register velocities accurately when placed close to a vertical wall. A Price meter held close to a right-bank vertical wall will under-register because the slower water velocity near the wall strikes the effective (concave) face of the cups. The converse is true at a left-bank vertical wall. (The terms "left-bank" and "right-bank" designate direction from the center of a stream for an observer facing downstream). The Price meter also tends to under-register when positioned close to the water surface or close to the streambed.

5.1.3 Equipment

Current meters, timers, depth and width measuring devices, and a means of counting meter revolutions are needed for measurement of discharge. The equipment includes:

- Depth-measuring device
- Current meter
- Width-measuring devices, either engineer's tape or tagline
- Stopwatch
- Marker

Depth-Measuring Device. The depth-measuring device may consist of a graduated rod or staff in feet and tenths. If a Price-type meter is used, the depth-measuring device will be the topsetting 2-inch diameter hexagonal wading rod.

Current Meter. A current meter is an instrument used to measure the velocity of flowing water. The principle of operation is based on the proportionality between the velocity of the water and the resulting angular velocity of the meter's rotor. By placing the current meter at a point in a stream and counting the number of revolutions of the rotor during a measured interval of time, the velocity of water at that point is determined.

Engineer's Tape or Tagline. Steel tapes, metallic tapes, or premarked taglines are used for width determinations during discharge measurements made by wading. Direct measurement of width using tapes or taglines can be accurate with proper precautions. Orientation normal to the flow pattern of the river and elimination of most of the sag, through support or tension, are recommended for improved accuracy.

Stopwatch. A stopwatch is used to measure time during which velocity is measured at each point in the cross-section. Velocity at each point is measured for a period greater than or equal to 40 seconds and less than or equal to 70 seconds.

Marker. Each location where the velocity-area method will be consistently applied will be marked with a field stake.

If a Price meter is used, additional equipment includes a headset. A headset attaches to an electronic connection at the upper end of the wading rod.

5.1.4 Maintenance and Calibration Procedures

Prior to use of the current meter and following use of the meter, tests will be conducted to ensure that the unit performs acceptably. For Price-type and propeller-type meters, calibration tests will be conducted prior to and after fieldwork. Calibration tests will be conducted in streams fitted with Parshall flume or equivalent measurement device. Stream velocities derived from flume readings will be compared to the meter readings.

In addition, for Price-type meters, spin tests will be conducted. The spin test will be performed in an enclosed area, such as in the cab of a truck or in the enclosed rear of a truck, to prevent wind interference. The test is to be performed prior to attaching the current meter to the wading rod. While holding the meter steady in an area sheltered from breezes, the technician will spin the rotor and then press the start button on the stopwatch. The technician will observe the meter until the rotor ceases to spin. The duration of the spin for the pygmy meter should be more than 40 seconds and for the Price AA meter should be more than 90 seconds. If the meter fails to meet the time-of-spin criteria, the meter will be cleaned and oiled before use. If the meter continues to spin well beyond these time limits, the record will indicate that the meter spun for 40+ seconds, in the case of the pygmy meter, or for 90+ seconds, in the case of the Price AA meter.

To ensure reliable observations of velocity, it is necessary that the selected current meter be kept in good condition. Before and after each discharge measurement, meter cups, vanes, or propellers, pivot and bearing, and shaft should be examined for damage, wear, or faulty alignment. During measurements, the meter will be observed periodically when it is out of the water to be sure that the rotor or propeller spins freely.

Meters will be cleaned and, if applicable, oiled daily when in use. If measurements are made in sediment-laden water, the meter will be cleaned immediately after each measurement. After cleaning and lubrication, the rotor or propeller will be spun to make sure that it operates freely. If the rotor or propeller stops abruptly, the cause of the trouble will be sought and corrected before using the meter.

In addition to meter maintenance, the entire meter unit will be checked before departure to the field each day as follows:

- For Price-type meters, attach the current meter and digital counter/headset to the wading rod. Test the headset by:

1. Spinning the current meter to ensure that audible clicks occur.
2. If audible clicks do not occur, the following steps should correct the problem:

    - Check that electronic connections are tight;
    - Check that the cat's whisker lightly contacts the upper part of the shaft;
    - Spin again and if audible clicks still do not occur, check that the battery in the headset is properly aligned. Replace the battery, if necessary.

- For propeller-type meters, activate the sensor and test by:

    1. Moving the propeller through the air to ensure that electronic readings are displayed.
    2. If no display is visible, the following steps should be completed:

        - Check that the batteries have power. Replace if necessary;
        - Check that sensor and display are connected to the shaft wire.

5.1.5 General Considerations

Based on approximate depths, either the Price-type or propeller-type meter will be used to perform a velocity-area measurement. If depths or velocities under natural conditions are too low for a dependable current meter measurement, the cross-section will be modified, if practical, to provide acceptable conditions. A shovel will be used to remove aquatic vegetation, ice, or rocks that may interfere with meter operation or discharge measurement.

At each measurement point (or station) across the stream cross-section, depth is measured prior to measurement of velocity. Therefore, it is recommended that the measuring rod be set with the current meter suspended out of the water and above the tagline, which is used to measure width and to identify stations across the cross-section. Placement of the rod about 0.5 feet downstream from the tagline prevents contact between the tagline and the current meter when the meter is lowered into measuring position. The measuring rod will be placed in the stream so the base rests on the streambed, and the depth of water will then be read from the graduated main rod. The main rod is graduated into 0.l-foot increments.

The meter operator reads water depth directly from the depth-measuring rod. In high velocity areas, it is recommended that depth be read as the value between the depth on the upstream side of the rod and the

depth on the downstream side of the rod. Depth is measured to the nearest 0.02 foot. This depth is used to set the vertical location on the current meter.

The meter operator will stand in a position that least affects the velocity of the water passing the current meter. The meter operator will face upstream while holding the depth-measuring rod vertically and close to the tagline or measuring tape. The meter operator stands at about a 45-degree angle downstream from the measuring rod and at least 1.5 feet from the depth-measuring rod. This angle is an imaginary angle between the extended arm holding the depth-measuring rod and the tagline or measuring tape. The meter operator should avoid standing in the water if his or her feet and legs occupy a significantly large percentage of a narrow cross-section. For narrow streams, it is often possible to stand astride the stream.

The depth-measuring rod should be held in a vertical position with the meter parallel to the direction of flow while the velocity is being observed. When measuring streams that have shifting beds, the soundings or velocities can be affected by the scoured depressions left by the hydrographer's feet. For such streams, the meter should be placed ahead of and upstream from the operator's feet.

5.1.6 Discharge Calculations

A stream discharge is the summation of the products of the subsection areas of the stream cross-section and their respective average velocities. The formula $Q = \Sigma (v * a)$ represents the computation, where Q is the total discharge, a is an individual subsection's area, and v is the corresponding mean velocity of flow normal to the subsection. The summation of the discharges for all the subsections is the total discharge of the stream. The order for calculating discharge is:

- Use the distances from initial point to compute width for each section. The first width is computed by subtracting the first distance from the second distance, and dividing this quantity by two. The second width will be the quantity of difference between the third distance and the first distance, divided by two. For each subsequent width, subtract the distance on the line above the line you are calculating from the distance on the line below the line you are calculating, and divide this quantity by two. This procedure is carried out for each line until you reach the final width calculation. This is calculated as the quantity of the difference between the final distance and the second-to-the-last distance, divided by two.

- Subsequent calculations will be performed as follows:

- Calculate each discharge for each subsection by multiplying the width of the subsection times the depth times the velocity.

- Sum the discharges for each subsection to arrive at total discharge for the entire cross-section.

- Check your math by summing the subsection widths. Their total should equal the value obtained by taking the difference between the left and right bank station distances from initial point.

- Initial at the line "Comp. by" to identify yourself as the person responsible for performing the discharge calculation.

5.2 VOLUMETRIC METHOD

The volumetric method is a simple and accurate method for measuring flow from small discharges such as gravity flow discharges from pipe outlets.

5.2.1 Theoretical Considerations

This method involves measuring the time required to fill a container of known capacity, or the time required to partly fill a calibrated container to a known volume. Alternatively, in the case of measuring discharge remotely in a sump or standpipe setting, the volumetric method may be performed by capturing flow in a container for a set period of time. This volume of water is then measured and discharge is determined.

5.2.2 Required Measurement Conditions

Conditions must be such that all discharge from an outlet can be captured in the volumetric container during the period of measurement.

5.2.3 Equipment

The bucket and stopwatch technique is particularly useful for the measurement of small flows. Equipment required to make this measurement is a calibrated container and a stopwatch. Calibrated containers of varying sizes will include:

- 5-gallon bucket
- 2-liter graduated cylinder

- 1-liter graduated cylinder
- 1-liter bucket
- 500-milliliter beaker
- 250-milliliter beaker

5.2.4 Maintenance and Calibration Procedures

Graduated cylinders are incremented in terms of milliliters and can be easily converted to gallons. The incremental volume of a 5-gallon bucket can be determined by adding known volumes of water and recording the depth after each addition.

5.2.5 General Considerations

Upon arrival at the site, the sampling personnel will evaluate the flow conditions to select the appropriate method for flow measurement. If flow conditions are appropriate for volumetric measurement, the sampling personnel will observe and use judgment in approximating the flow volume and will select an appropriately sized volumetric container.

Sampling personnel will use a stopwatch to measure the time required to fill a volumetric container. The sampler will time flow into the container for a minimum of 10 seconds. Three consecutive measurements will be made and noted, and the results will be averaged to determine the discharge.

5.2.6 Discharge Calculations

Discharge will be determined initially in gallons per second (gal/s) or in milliliters per second (ml/s). These values will be noted, but the average value will be reported in cubic feet per second. Calculations will be performed as follows:

- Record each of the three measurements in terms of gallons per second or milliliters per second, depending on the volumetric container.

- If one of the three measurements is 50 percent or more different from the other two measurements, then this value will not be used. Instead, three additional measurements will be taken and, provided that none of these three measurements differs by greater than 50 percent from the other two measurements, these values will be used.

- Average the three values.

- Convert the averaged value to cfs as follows:

  - To convert ml/s to cfs, multiply by 3.5. x 10^{-5}

  - To convert gal/s to cfs, multiply by 0.134

- Record discharge in cfs.

6.0 REFERENCES

Linsley R.K, Kohler, M.A., Paulhus, J.L.H. 1982. *Hydrology for Engineers*. McGraw-Hill Book Company. New York, N.Y.

Rantz, S.E., et al. 1982. *Measurement and Computation of Streamflow: Volume 1; Measurement of Stage and Discharge*. Geological Survey Water-Supply Paper 2175. U.S. Government Printing Office. Washington, D.C.

Rantz, S.E., et al. 1982. *Measurement and Computation of Streamflow: Volume 2: Computation of Discharge*. Geological Survey Water-Supply Paper 2175. U.S. Governmental Printing Office. Washington, D.C.

Robertson, J.A., Cassidy, J.J., and Chaudhry, M.H. 1988. *Hydraulic Engineering*. Houghton Mifflin Company. Boston, MA

SURFACE WATER FLOW MEASUREMENT FORM

Project:_____ **Project Number:** _____

Date:____/____/____ **Time:** _____

Field Personnel: _____ **Signatures:** _____

_____ _____

SITE DESCRIPTION

Site Location: _____

Station Number:_____

GPS Coordinates Latitude N____° ____ ′ ____ ′′ **Longitude W**____° ____ ′ ____ ′′

Datum:_____ **Elevation**_____ **ft.**

Description of Stream Cross-Section: _____

Total Width_____**ft R.E.W.**_____**ft L.E.W.**_____**ft**

Distance from Reference	Measure-ment #	Width	Depth	Velocity Depth (60%)	Velocity (30 secs)	Width	Depth	Velocity Depth (60%)	Velocity (30 secs)
	1								
	2								
	3								
	4								
	5								
	6								
	7								
	8								
	9								
	10								
	11								
	12								
	13								
	14								
	15								
	16								

Distance from Reference	Measure-ment #	Width	Depth	Velocity Depth (60%)	Velocity (30 secs)	Width	Depth	Velocity Depth (60%)	Velocity (30 secs)
	20								
	21								
	22								
	23								
	24								
	25								
	26								
	27								
	28								
	29								
	30								
	31								
	32								
	33								
	34								
	35								
	36								
	37								
	38								
	39								
	40								
	41								
	42								

Comp. by: _____ Checked: _____

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN

FOR

LONG-TERM MONITORING OF

SURFACE WATER AND GROUNDWATER

DRAFT

Revision 0

March 2015

Quality Assurance Project Plan Approval Sheet

Approved by:

Program Manager MWH	Date
Quality Manager MWH	Date
Program Manager P4	Date
Project Manager USEPA	Date
QA Manager USEPA	Date

Executive Summary

This Quality Assurance Project Plan (QAPP) presents in specific terms the policies, organization, functions, and quality assurance/quality control (QA/QC) requirements designed to meet the data quality objectives in support of continuing monitoring conducted by P4 Production, LLC (P4) at the inactive properties of Ballard, Henry and Enoch Valley Mines (collectively known as the Sites). This detailed QAPP has been prepared to ensure the data are scientifically valid and defensible, and establishes the analytical protocols and documentation requirements to ensure the data are collected, reviewed, and analyzed in a consistent manner.

The procedures detailed in this QAPP are in accordance with applicable professional technical standards. In addition, this QAPP was prepared in accordance with the following guidance:

- *Sampling and Analysis Plan for Long-Term Monitoring of Surface Water and Groundwater* (MWH, 2015).
- *USEPA Requirements for Quality Assurance Project Plan*, EPA QA/R-5, (USEPA, 2001).
- *Guidance for Quality Assurance Project Plan*, EPA QA/G-5, December, (USEPA, 2002).
- *Administrative Settlement Agreement and Order of Consent/Consent Order for Performance of Remedial Investigation and Feasibility Study at the Enoch, Henry, and Ballard Mine Sites in Southern Idaho*. Effective Date of November 30, 2009. (USEPA, 2009a).
- *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*, EPA-540-R-08-005. USEPA, 2009b).
- *National Functional Guidelines for Inorganic Superfund Data Review*. EPA-540-R-013-001. August (USEPA, 2010) and prior version for methods not updated.
- *USEPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5 (USEPA, 2014.).

TABLE OF CONTENTS

TABLE OF CONTENTS i

1.0 PROGRAM MANAGEMENT 1-1

1.1 INTRODUCTION 1-1
1.2 PURPOSE 1-1
1.3 PROGRAM ORGANIZATION 1-1
1.4 PROGRAM DESCRIPTION 1-2
1.5 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA 1-2
1.5.1 Data Quality Objectives 1-2
1.5.2 Measurement Performance Criteria 1-2
1.6 TRAINING REQUIREMENTS 1-4
1.7 DOCUMENTATION AND RECORDS REQUIREMENTS 1-4
1.7.1 Field Logbooks 1-4
1.7.2 Field Forms 1-5
1.7.3 COC Records 1-6
1.7.4 Analytical Laboratory Records 1-6

2.0 MEASUREMENT/DATA ACQUISITION 2-1

2.1 SAMPLING PROCESS 2-1
2.1.1 Sampling Design 2-1
2.2 SAMPLING ACTIVITIES 2-1
2.2.1 Sampling Activity Summary 2-1
2.2.2 Sample Collection and Preparation Protocols 2-1
2.2.3 In-Process Sampling Data Review and Change Control 2-1
2.3 SAMPLE CUSTODY REQUIREMENTS 2-2
2.3.1 Sample Labeling and Handling 2-2
2.3.2 Chain of Custody 2-3
2.3.3 Holding Time Considerations 2-4
2.3.4 Laboratory Operations 2-4
2.3.5 Sample Storage and Security 2-5
2.3.6 Sample Tracking 2-5
2.3.7 Sample Custody Records 2-6
2.4 ANALYTICAL METHOD REQUIREMENTS 2-6
2.4.1 Sample Preparation 2-6
2.4.2 Analytical Methods and Target Analyses 2-6
2.5 QUALITY CONTROL REQUIREMENTS 2-7
2.5.1 Field Quality Control Samples 2-7
2.5.2 Laboratory Quality Control Samples 2-8
2.6 INSTRUMENT/EQUIPMENT CALIBRATION AND MAINTENANCE REQUIREMENTS 2-9
2.6.1 Field Equipment 2-9

2.6.2 Laboratory Equipment 2-9
2.7 DATA ACQUISITION, VALIDATION, REVIEW AND REPORTING 2-10
2.7.1 Field Data 2-10
2.7.2 Analytical Data Validation 2-10
2.7.3 Data Analysis and Reporting 2-12
2.7.4 Data Storage and Retrieval 2-12
3.0 ASSESSMENT/OVERSIGHT 3-1
3.1 INDEPENDENT TECHNICAL REVIEW 3-1
3.2 CORRECTIVE AND PREVENTIVE ACTION 3-1
4.0 REFERENCES 4-1

LIST OF TABLES

Table 1-1 Program Contacts

Table 1-2 Summary of Quality Control Parameters Evaluated

Table 1-3 Data Quality Characteristics Formulae

Table 2-1 P4 Sites Sampling Activities Summary

Table 2-2 Requirements for Containers, Preservation Techniques, Sample Volumes and Holding Times

Table 2-3 Achievable Laboratory Limits and Applicable Project Screening Values, Surface Water and Groundwater Parameters

Table 2-4 Quality Control Sample Summary

Table 2-5 Calibration and Maintenance Requirements for Field Equipment

Table 2-6 Summary of Calibration and QC Procedures for EPA Method 6020A (ICP-MS)

Table 2-7 Summary of Calibration and QC Procedures for EPA Method 6010C (ICP-AES)

Table 2-8 Summary of Calibration and QC Procedures for EPA Method 300.0 (IC)

LIST OF FIGURES

Figure 1-1 Organization Chart

Figure 1-2 Chain of Custody Form (Microbac)

Figure 2-1 Data Acquisition and Data Management Flow Diagram

ATTACHMENTS

Attachment A Laboratory Standard Operating Procedures

Attachment B Laboratory Quality Assurance Plan (Microbac)

Attachment C Electronic Data Deliverable Format

Attachment D Third Party Validation Sample Report

ACRONYMS AND ABBREVIATIONS

AOC	Administrative Order on Consent
ASTM	American Society for Testing and Materials
COC	chain of custody
DO	dissolved oxygen
DQOs	data quality objectives
EDD	electronic data deliverable
FCR	Field Change Request
FTL	Field Team Leader
FSP	Field Sampling Plan
GPS	global positioning system
HSP	Health and Safety Plan
IC	Ion chromatography
ICP-AES	Inductively coupled plasma-atomic emission spectroscopy
ICP-MS	Inductively coupled plasma-mass spectrometry
LCS	laboratory control sample
LDC	Laboratory Data Consultants, Inc.
LTM	Long Term Monitoring
Microbac	Microbac Laboratories Inc.
MP	Management Procedures
MS	matrix spike
MSD	matrix spike duplicate
NTU	nephelometric turbidity unit
ORP	oxygen reduction potential
P4	P4 Production, LLC
PARCC	precision, accuracy, representativeness, completeness, and comparability
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RL	reporting limit
RPD	relative percent difference
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SI	site investigation
SOP	standard operating procedure
USEPA	U. S. Environmental Protection Agency

Quality Assurance Project Plan Distribution List

Distributed to:

Program Manager MWH	Date
Quality Manager MWH	Date
Program Manager P4	Date
Project Manager USEPA	Date
QA Manager USEPA	Date
Laboratory Manager Microbac	Date
Third Party Validation Manager LDC	Date
Project Chemist MWH	Date

1.0 PROGRAM MANAGEMENT

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents in specific terms the policies, organization, functions, and quality assurance/quality control (QA/QC) requirements designed to meet the data quality objectives in support of continuing monitoring conducted by P4 Production, LLC (P4's) inactive properties of Ballard, Henry and Enoch Valley Mines (collectively known as the Sites). The *Sampling and Analysis Plan for Long-Term Monitoring (LTM) of Surface Water and Groundwater (2015 LTM SAP)* presents the LTM objectives and sampling activities. The complementary documents to the QAPP are the Field Sampling Plan (FSP) and Health and Safety Plan (HSP). The FSP, QAPP, and HSP are included as Appendices A through C of the *2015 LTM SAP*, respectively.

1.2 PURPOSE

This QAPP describes the measures that shall be employed during the Site-specific LTM to assure that data generated are of a known and defensible quality in relation to the overall objectives of the investigation. This document is prepared in accordance with the *United States Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans* (QA/R-5, March 2001) and *USEPA Guidance for Quality Assurance Project Plans* (QA/G-5, December 2002). The various plans and procedures cited in this document govern the program sample acquisition efforts, sample handling and chain-of-custody protocols, laboratory and field analyses, data handling and data management, field documentation, management, quality auditing of field operations, corrective/ preventive action processes, and the management of program quality record.

1.3 PROGRAM ORGANIZATION

The project team for the analysis, data validation/verification and analytical summary reporting for the LTM consists of MWH personnel and a subcontracted analytical laboratory, Microbac Laboratories Inc. (Microbac) of Marietta, OH.

Third (3rd) party data validation will be used for all sampling programs. Laboratory Data Consultants, Inc. (LDC), located in Carlsbad, California has been approved by the Agencies/Tribes (A/Ts) to validate samples collected in support of the comprehensive investigation and monitoring program.

The organization chart for this project is provided in Figure 1-1. Contact information for the program team is provided in Table 1-1.

Changes in program staff may be made as circumstances dictate. The MWH PM will advise the P4 PM in advance of any such changes in the key personnel.

1.4 PROGRAM DESCRIPTION

The overall scope and purpose of the LTM program are discussed in Section 1.0 of the *2015 LTM SAP*. At the discretion of the MWH PM and the P4 PM, any required scope modifications or additions may be accommodated either by revisions to the *2015 LTM SAP* documents or through the use of the field change request (FCR) process described in Section 5.5 of the FSP.

1.5 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

1.5.1 Data Quality Objectives

The data quality objectives (DQOs) for this program are described in Section 3.0 of the *2015 LTM SAP*. These DQOs are consistent with USEPA guidelines (USEPA, 2006), which describe the systematic planning of data collection activities to assure that the proper type, quality, and quantity of data are collected. The DQOs shall be fulfilled by following specific procedures and requirements for sampling; sample handling and chain-of-custody (COC); field and laboratory QC samples; a laboratory-specific preventative maintenance measures; data review and reduction by the destination laboratory; data validation by LDC (a third party); analysis by MWH; and reporting; and quality auditing and corrective/preventive action processes, as described in the *2015 LTM SAP* and this QAPP.

1.5.2 Measurement Performance Criteria

DQOs may be expressed in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters. The PARCC parameters are established as a benchmark for evaluating the attainment of the program objectives. PARCC parameters are summarized in the following subsections and in Table 1-2 and the formulae are presented in Table 1-3.

1.5.2.1 Precision

Precision of field data and laboratory analytical data shall be determined by the analysis of field duplicates, laboratory replicate, matrix spike and matrix spike duplicate results and evaluation of the relative percent difference (RPD). The RPD goals shall be less than or equal to 20 percent. Precision of field measurements and laboratory results of these samples shall be evaluated by means of relative standard deviation (RSD), or the coefficient of variation. The RSD is calculated as shown in Table 1-3. The RSD goal for samples shall be less than or equal to 20 percent.

1.5.2.2 Accuracy

Sample accuracy shall be determined by the collection and analysis of field blanks and shall include at least one trip blank and equipment rinsate blank for every 10-field samples collected or at least one per sample area. Laboratory accuracy is determined by the analysis of calibration and method blanks, calibration verification samples, laboratory control samples, and matrix spike samples. Method blank goals shall be that blanks contain less than the reporting limit (RL) for each target parameter. The accuracy goals shall be the same as the precision goals.

The amount spiked into matrix spike samples may be adjusted to provide more meaningful results. For example, the spike concentration for a given target analyte may be adjusted to a level that is at or near a cleanup or risk-based concentration. These matrix spike results will be more meaningful relative to the task-specific DQOs, and the appropriateness of the spike concentration will be incorporated into the review and data validation process.

1.5.2.3 Representativeness

Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations. Sampling methods are established by the Standard Operating Procedures (SOPs) contained Attachment A of the FSP. Representativeness is also evaluated using RSD between the parent sample and field duplicate and by a review of the results of field blanks, equipment blanks, and trip blanks (water matrices only).

Laboratory representativeness shall be evaluated by reviewing COC forms, sample receipt forms, and sample preparation documentation to ensure that samples are stored, processed, and reported properly.

1.5.2.4 Completeness

The completeness goal of this program shall be 90 percent for each of the samples collected. Care shall be taken to provide duplicate samples from areas determined to be of particular concern during the course of the program, or from areas to which access may be limited.

1.5.2.5 Comparability

Comparability shall be ensured by analyzing water samples obtained in accordance with appropriate SOPs. Data shall be reported in the units specified in the governing analytical methods, generally in mg/L for water matrices.

Laboratory data shall be validated by LDC, a third-party validation firm, in compliance with this QAPP and the current *USEPA National Functional Guidelines for Inorganic Superfund Data Review (NFG)* (USEPA, 2014).

1.5.2.6 Goals

This section identifies numerical goals for precision and accuracy for the various environmental media. The completeness goal for measurements is 90 percent. Failure to meet these goals shall be considered in the data validation process described in Section 2.0. If data are unusable, as defined by the validation procedure, the data shall be qualified as rejected. Further evaluations shall be made and resampling or other appropriate actions initiated

1.6 TRAINING REQUIREMENTS

MWH field personnel shall be trained in the requirements of the *2015 LTM SAP* in a program meeting prior to the initiation of field activity. All personnel shall read the *2015 LTM SAP* documents prior to the start of field work, and shall acknowledge completion of reading training at the time of the program meeting. Meeting notes and attendance sheets shall be kept and forwarded to the program. In addition, prior to conducting each day's sampling activities, the field team leader (FTL), or designee shall conduct a "tailgate" meeting with field staff to review field procedures and sampling requirements, in order to better ensure that samples are collected and handled according to *2015 LTM SAP* and QAPP requirements. Tailgate meeting discussion subjects and attendees shall be documented in field logbooks as noted in Section 1.7.

One hard copy of the current approved version of the entire *2015 LTM SAP* shall be maintained by the FTL for ready-reference purposes, in the field vehicle or field office. All field team members shall have access to .pdf format files of the complete *2015 LTM SAP* through their personal laptop computers.

1.7 DOCUMENTATION AND RECORDS REQUIREMENTS

1.7.1 Field Logbooks

Sample collection activities shall be documented in permanently bound, page-numbered, weather-resistant field logbooks assigned to the FTL, or, if multiple sampling teams are used, to a designee in charge of each team. Each notebook shall be identified to the program, task, and to the individual assigned custody of the logbook. For all sampling to be performed, the appropriate SOP, appended to the FSP shall also be employed. If logbook custody is transferred to another individual, such transfer shall be noted in the logbook and signed and dated by both parties. All entries shall be made in indelible ink; errors shall be corrected by one single line through the text being revised, and all such corrections shall be initialed and dated.

With the exception of the information contained in the appropriate SOP, governing the media to be sampled, bound field logbooks shall be used to record the following information, as appropriate for the type of sampling being performed:

- date, time, subjects, and attendees of daily tailgate training sessions;
- sample date, time, types, numbers, and quantities;
- sample container preservation steps performed;
- sample locations, including global positioning system (GPS) coordinates;
- numbers of associated photographs, with appropriate cross-references to the affected camera;
- sampling equipment used;
- decontamination steps performed;
- calibration information for field equipment (e.g., turbidity meters, conductivity meters), cross-referenced to equipment identification number, model/manufacturer, serial number, or other unique identifier; and,
- acknowledgements that COC forms and express shipment information (see Figure 1-2) were properly completed.

In addition, other ancillary information to be recorded, may include:

- time of arrivals/departures of MWH personnel and/or other visitors to the sampling site(s);
- weather conditions;
- presence of livestock or wild game;
- time and subject of any incoming our outgoing telephone/radio contacts; and,
- any unusual events.

The logbooks shall be kept up to date on a daily basis; backup copies of each day's entries shall be made on a weekly basis and forwarded separately to the program quality, in addition to copies of all field forms, outgoing COCs, and sample shipping documents.

1.7.2 Field Forms

In addition to field logbooks, field forms will be required to be filled out by the sampling team conducting the sampling. All efforts will be made to fill out the information at the sampling location. Field forms for various media include sampling of surface water and groundwater. These forms are used as a supplement to

field logbooks. The appropriate forms for sampling of various media are located in the applicable SOP, appended to the FSP.

1.7.3 COC Records

Documentation of sample custody must be maintained from the time the samples are collected through receipt at the destination laboratory; sample homogenization, preparation, and analysis; data recording and reduction; data validation; and final release of laboratory analytical data. Initial information concerning sample collection shall be recorded in the field logbook as described in Section 1.7.

Information on the custody, transfer, handling, and shipping of samples shall be recorded by field personnel on a Microbac COC form (see Figure 1-2). A COC form shall be completed for each set of samples collected daily and shall contain the following information:

- sampler's signature and affiliation;
- program name and identification number;
- date and time of collection;
- sample identification number and matrix;
- analyses requested;
- number of containers;
- signature of persons relinquishing custody, dates, and times;
- signature of persons accepting custody, dates, and times;
- method of shipment; and,
- shipping papers/waybill identification number (as appropriate).

A copy of each as-transmitted COC form shall be retained in the program records.

1.7.4 Analytical Laboratory Records

The subcontractor analytical laboratory shall be responsible for preparing analytical laboratory reports that are reviewed and approved by the laboratory's QA manager prior to submittal to MWH, as noted in the QA plans provided by the laboratory (see Attachment B).

The hard-copy deliverable ("data package" or "report") will be issued (i.e., published or printed) in one of two formats: a Level 3 or Level 4 data package deliverable. For approximately 90 percent of samples

collected, hard-copy reports will be issued with summary data (or "Level 3" package, as defined below), and the remaining hard-copy reports will be issued with summary data and back-up raw instrument data (or "Level 4" package, as defined below). Level 4 data packages will be provided for all samples (all data packages) in scanned (pdf) format. At the end of the project sample analysis, MWH will request the laboratory to print one out of five data packages as a hard-copy Level 4 deliverable to ensure there are sufficient and representative packages for selection of 10 percent of samples for a USEPA Stage 4 Validation (USEPA, 2009). The 10 percent of samples will be randomly selected from those printed Level 4 data packages. The scanned Level 4 packages containing raw data for 100 percent of samples are required for the project record and may be needed if problems are encountered during review of the Level 3 packages (see Section 1.7.4.2).

The hard-copy report will be paginated and organized with a table of contents. The hard-copy deliverable will contain a cross reference that correlates the client or field identification as provided on the chain-of-custody document with the laboratory's sample identification. Results should be presented on a form equivalent to the USEPA Contract Laboratory Program (CLP) "Form 1". Results from QC samples associated with each distinct analytical method are to be presented all together on QC summary sheets for ease of review. A case narrative will be provided for each analytical method. The case narrative discusses any problem related to sample-receipt, corrective action taken by the laboratory, QC outliers or other problems, method deviations, and/or clarifications or anomalies observed by the laboratory.

1.7.4.1 Sample Results (CLP "Form 1")

This form contains all required data for field samples. The Form 1 (or equivalent) will provide the following information:

- Field sample identification
- Laboratory sample identification
- Sample result(s) for metals and general chemistry parameters will be reported with appropriate units, method detection limit, and reporting limit. Concentrations equal to or greater than the method detection limit (MDL) must be reported. Concentrations between the MDL and reporting limit will be flagged as estimated ("J" flagged). Parameters that are not detected or not present at concentrations equal to or greater than the MDL are flagged as "U" and interpreted to be not detected at a value equal to or greater than the MDL. Any non-detected value ("U" flagged) will be reported with its reporting limit and MDL. Do not report "not detected" (or "ND").
- Sample collection and receipt dates
- Sample preparation date/time

- Analysis date/time
- Dilution factor
- Preparation batch number or identification
- Analysis batch number or identification
- Sample matrix and instrument
- Percent moisture determination
- For solid-matrix samples, identify basis of reporting (i.e., wet-weight or dry-weight basis)

1.7.4.2 Summary or "Level 3" Data Deliverable Package

All other summary forms need to be present, following the Form 1s, with clear association of the QC batch to each sample (on the CLP form specified or equivalent):

- Summary of all field sample results (as described above)
- Sample results and preparation blank (Form IA-IN and IB-IN)
- Initial calibration verification (ICV), and continuing calibration verification (CCV) (Form IIA-IN)
- Initial calibration blanks (ICB), continuing calibration blank (CCB), and preparation blanks (Form III-IN)
- Inductively coupled plasma (ICP) interference check sample (Form IVA-IN) or inductively coupled plasma-mass spectrometer (ICPMS) interference check sample (Form IVB-IN)
- Matrix spike and matrix spike duplicate (MS/MSD) sample recovery and MS/MSD RPD (Form VA-IN)
- Post-digest spike sample recovery (Form VB-IN)
- Laboratory duplicate precision (Form VI-IN)
- Laboratory control sample (LCS) recovery (Form VII-IN)
- ICP and ICPMS serial dilution percent differences (Form VIII-IN)
- MDLs (Form IX-IN)
- ICP interelement correction factors (Forms XA-IN and XB-IN)
- ICP and ICPMS linear ranges (Form XI-IN)

- Preparation log (Form XII-IN)
- Analysis run Log (Form XIII-IN)
- ICPMS tunes (Form XIV-IN)
- ICPMS internal standards relative intensity summary (Form XV-IN)
- Sample log-in sheet (Form DC-1)
- Deliverables inventory sheet (Form DC-2)
- Case narrative
- COC forms

1.7.4.3 Full Raw Data or "Level 4" Data Deliverable Package

The full raw data package includes all items specified for the summary data package (Level 3), plus instrument raw data and/or documentation of the following:

- Calibration standards (including source, preparation date)
- Blanks (ICB, CCB, and preparation)
- ICV, CCV standards
- Interference check samples
- Serial dilution samples
- LCS
- Diluted and undiluted Samples
- Dilution factors
- Sample volumes
- Laboratory duplicates
- Matrix spikes (source, concentration, volume)
- Post-digest spikes (source, concentration, volume)
- Method of standard addition results
- Instrument identification

- Analysis date and time
- All inorganic methods: full raw data print outs from instruments
- Full run log for each analysis
- ICPMS to include: internal standard recoveries, tune data (atomic mass unit [amu] and peak width), and molecular interference check data

2.0 MEASUREMENT/DATA ACQUISITION

2.1 SAMPLING PROCESS

2.1.1 Sampling Design

The QAPP is written for the LTM monitoring sampling events. The number and type of samples that shall be collected and general sampling locations are summarized in Tables 4-1 and 4-4 of the *2015 LTM SAP*.

2.2 SAMPLING ACTIVITIES

2.2.1 Sampling Activity Summary

Sampling activities are summarized in Table 2-1, and consist of surface water bodies and groundwater monitoring wells at the P4 Sites.

Table 2-1
P4 Sites LTM Sampling Activities Summary

Sampling Type	No. of Sample Locations	General Comments/Notes
Surface Water	36	Analyzed in accordance with the *2015 LTM SAP* "LTM Surface Water and Groundwater Analytical List" (Table 4-2).
Groundwater	45	Analyzed in accordance with the *2015 LTM SAP* "LTM Surface Water and Groundwater Analytical List" (Table 4-2).

2.2.2 Sample Collection and Preparation Protocols

Sampling activities and field parameter measurements shall be performed in accordance with the *2015 LTM SAP*; and the related surface water and groundwater SOPs are in Attachment A of the FSP. Sample containers, preservation (if required), and holding times are presented in Table 2-2. All samples will be bagged, placed on wet ice, and stored in coolers prior to storage in a locked refrigerator, and subsequent shipping to the appropriate laboratory for analysis.

2.2.3 In-Process Sampling Data Review and Change Control

Review of field collection data shall be conducted by the FTL. Based on this review, the FTL may request re-sampling or re-analysis of a particular sample or group of samples if omissions or discrepancies are found. Any changes in sampling procedures that may be required in order to address unanticipated field

situations shall be processed as an FCR as discussed in the FSP. Other discrepancies shall be discussed with MWH PM and corrective and preventive action request initiated as appropriate.

2.3 SAMPLE CUSTODY REQUIREMENTS

2.3.1 Sample Labeling and Handling

Samples will be labeled with all necessary information on laboratory supplied labels using waterproof ink. At a minimum, each sample label shall contain the following information:

- site location;
- sample identification;
- date and time of sample collection, with sampler's initials;
- analyses required;
- filtered or unfiltered;
- method of preservation, if used; and,
- sample matrix.

Each sample shall be assigned a unique identification number. This number shall be coded based on the sample location in the following format:

AABBSW or **GWMYYaaa-b-c**

- **AA** indicates the year (two digits) the sampling event started
- **BB** indicates the month (two digits) the sampling event started
- **SW** or **GW** denotes that surface water or groundwater is sampled.
- **M** designates "M" for Monsanto and is used to differentiate from other sample stations identified by MWH for Idaho Mine Association (IMA) mine-specific investigations.
- **YY** denotes the station type; station type which is ST for streams, DS for dump seeps, SG for spring, BW for borehole well, and MW for monitoring well.
- **aaa** denotes the specific station number/location.
- **b** denotes the replicate number (blank shall indicate no replicate samples) if there are QA/QC replicate samples, then 1, 2 and 3 represent the replicate samples.

- **c** denotes whether the sample involved special field handling or is to be handled in a specific manner; handling codes are as follows:

  F: Filtered

- U: Unfiltered

Sample containers shall be sealed in ziplock plastic bags or plastic bags with wire ties supplied by Microbac and immediately placed on ice or "blue ice" in an insulated cooler chilled to at least 4°C. Insulated coolers shall be provided by the contract laboratories or purchased locally. All samples will be stored in the coolers until the end of the day when all of the samples will be placed in a locked refrigerator at the Fox Hills Machine Shed.

Samples will be shipped to the Microbac with blue ice or bagged wet ice in coolers secured with packing tape, via overnight Federal Express. MWH will fill out appropriate COC forms supplied by the laboratory. The chain-of-custody will be included with the sample shipment, and copies of all chains-of-custody along with Federal Express waybills will be kept by MWH field personnel.

All samples will be sent to Microbac or other approved laboratory. Microbac's address and contact person are listed below:

Microbac
158 Starlite Drive
Marietta, OH 45750
(740) 373-4071
Attn: Kathy Albertson

Supplies including sample containers and coolers will be sent to the Monsanto Plant:

Monsanto Company
1853 HWY 34
Soda Springs, ID 83276 (208) 547-1439
Attn: Rachel Roskelley

MWH's FedEx account number is 1000-6314-0.

2.3.2 Chain of Custody

Field personnel responsible for the collection of samples shall sign and retain a copy of the COC form (see Figure 1-2), document the method of shipment, and send the original with the samples. The original signed COC form shall be sealed in a watertight plastic envelope and attached to the inside lid of the cooler. Coolers shall be secured with strapping tape and a container seal applied that is covered with clear strapping tape. After samples are packed and sealed, coolers shall be transferred from the field to a central, secured

area at the Fox Hills machine shop for storage and overnight express carrier pickup. Samples shall be stored in a refrigerator fitted with a keyed lock; the key location shall be made known only to the FTL or designees.

Samples shall be shipped by overnight express carrier for delivery to Microbac in Marietta, OH in time to meet all required holding times. Copies of the COCs and waybills shall be forwarded to the program quality records by the FTL. Immediately after the sample coolers containers are delivered to the carrier, the FTL or MWH project chemist shall notify the analytical laboratory to expect sample deliveries. Samples shall not be shipped on Friday without making prior arrangements with the destination laboratories.

2.3.3 Holding Time Considerations

Immediately after collection, samples shall be placed in field coolers with wet ice and/or blue ice. If there is no likelihood that a holding time will be violated, samples may be stored in the locked refrigerator at the Fox Hills machine shed for one or more days. Constituent-specific holding times are presented in Table 2-2. Sample shipment shall be scheduled to prevent the exceedance of any required holding period. Failure to conduct analyses within the required holding times may potentially require the qualification of associated analytical results and shall prompt appropriate corrective and preventive action measures as discussed in Section 3.0.

2.3.4 Laboratory Operations

2.3.4.1 Laboratory Receipt and Logging

Coolers containing samples to be analyzed shall be shipped via overnight express courier to Microbac, in Marietta, OH. When samples arrive at the laboratory, the designated laboratory custodian receiving the sample cooler shall inspect the cooler custody seal. The custodian shall sign the shipping COC and attach the carrier billing. The shipping COC and waybill shall be archived in the laboratory program file; a copy of the shipping COC shall be forwarded to MWH's Salt Lake City office for filing in the program quality records. The custodian shall then open the cooler to inspect the samples for integrity and compare the number of containers and label information with the COC form attached to the inside of the cooler lid. Cooler temperatures shall be checked and documented on the COC form. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the COC form, and analytical request discrepancies shall be noted on the COC form. The MWH project chemist shall be notified of any such problems; the MWH PM and FTL shall be advised in turn if the issue constitutes a nonconformance. In any case, discrepancies or nonconformance shall be resolved and addressed prior to the samples being released to the laboratory for analysis.

Once any discrepancies are resolved, the laboratory custodian shall enter the samples into an analytical custody log and shall assign each sample a unique identification number that is cross-referenced to the sample number assigned in the field. The identification number shall be used by the laboratory in its internal tracking system and the status of any given sample can be checked at any time by referring to the laboratory numbers on the COC form and in the laboratory log books. Both the laboratory and sample numbers shall be cited when the analytical results are reported. The laboratory custodian shall then sign the COC form. The original COC shall be routed to the laboratory's data management group. Copies of the COC forms documenting custody changes and documentation shall be received and kept in the laboratory program files. The original COC forms shall remain with the samples until final disposition of the samples by the laboratory. Samples, extracts, or digestates shall not be sent to another laboratory without the written authorization of the MWH project chemist. If so authorized, such samples shall be transmitted using a COC form approved by MWH.

2.3.5 Sample Storage and Security

While in the laboratory, the samples and aliquots that require storage at no more than 4°C, but above freezing, shall be kept in a locked refrigerator prior to analysis. At a minimum, the following procedures shall be applied:

- samples and extracts shall be stored in a secure area controlled by the laboratory's designated sample custodian;
- samples shall be removed from the shipping container and stored in their original containers unless damaged; damaged samples shall be disposed in an appropriate manner after notifying the MWH project chemist, and authorization to dispose is received and documented;
- whenever samples are removed from storage, removal shall be documented;
- sample transfers shall be documented on internal COC records;
- samples and extracts shall be stored after completion of analyses in accordance with contractual requirements, or until instructed otherwise by the MWH project chemist; and,
- samples shall not be stored with standards or sample extracts.

2.3.6 Sample Tracking

Laboratory personnel shall use COC records or databases to generate backlist reports of any unanalyzed samples. The reports shall include the collection times along with the program name and laboratory sample number, and shall include the program name, field sample identifications, and sample matrix. Sample analyses shall be scheduled on the basis of holding time considerations. Analytical assignments shall be

reviewed on a daily basis to ensure that holding times are not exceeded. If holding times are exceeded during laboratory custody, the MWH project chemist shall be immediately notified.

2.3.7 Sample Custody Records

Minimum requirements for laboratory sample COC controls are as follows:

- samples shall be stored in a secured area;
- access to the laboratory shall be through a monitored area; other outside access doors to the laboratory shall be kept locked in accordance with local fire requirements;
- a visitor's log shall be maintained, and visitors shall be escorted while in the laboratory;
- refrigerators, freezers, and other sample storage areas shall be securely locked or maintained in a secured area;
- only authorized personnel shall have keys to locked sample storage area(s);
- samples shall remain in secure sample storage until removed for preparation or analysis;
- sample transfers into and out of storage shall be documented; and,
- custody records shall be maintained by the laboratory's sample management group.

Samples, extracts, and digestates shall be retained at the laboratory for at least 60 days after the laboratory's final analytical data report has been submitted to MWH, so that any potential analytical problems can be properly addressed. The samples, extracts, and digestates may then be discarded unless in an approved and environmentally safe manner otherwise directed by the MWH project chemist.

2.4 ANALYTICAL METHOD REQUIREMENTS

2.4.1 Sample Preparation

Sample preparations shall be in accordance with standard laboratory practices as described in the laboratory SOPs and laboratory quality assurance plan (see Attachments A and B). Subsamples shall be obtained for matrix spikes prior to sample preparation and analysis. A subsample shall consist of a proportion of a homogenized liquid sample that is at least equal to the minimum size requirement for the analytical test.

2.4.2 Analytical Methods and Target Analyses

The surface water and groundwater monitoring parameters are listed on Table 2-3. The target parameters for any given surface water and groundwater monitoring event will be identified prior to the event and will be comprised of one or more of the monitoring parameters listed on Table 2-3.

The possible applicable screening values for surface water and groundwater sample results are summarized on Table 2-3. The project-required reporting limits and generally-achievable MDLs are listed on Table 2-3.

With the following exceptions, the reporting limit and MDL for a given target parameter are less than its lowest screening value:

- Aluminum: The reporting limit (0.2 mg/L) and the MDL (0.1 mg/L) are greater than the Surface Water Nation Recommended Water Quality Criteria Screening Level of 0.087 mg/L.
- Beryllium: The reporting limit (0.01 mg/L) and the MDL (0.005 mg/L) are greater than the Surface Water - Chronic freshwater screening level in NOAA SQuiRT of 0.00066 mg/L.
- Boron: The reporting limit (0.1 mg/L) and the MDL (0.05 mg/L) are greater than the Surface Water - Chronic freshwater screening level in NOAA SQuiRT of 0.0016 mg/L.

2.5 QUALITY CONTROL REQUIREMENTS

2.5.1 Field Quality Control Samples

2.5.1.1 Equipment Blanks

Blanks are defined as sample material that is free of reportable concentrations of target analytes; the blanks are introduced at various stages of sample handling to monitor possible contamination introduced by various field activities. Only rinsate (or equipment) blanks shall be used for this program. An equipment rinsate blank is a sample of the distilled water being used by the field team that is collected using decontaminated sampling equipment. The sample is then submitted to the laboratory as a blind sample. Equipment blanks will be collected on a daily basis, and, if more than one team collects samples on a given day, also by each sampling team. For the purpose of data validation (see Section 2.7), primary field samples will be associated to the equipment rinsate blank sample collected on the same day by a given sampling team. Equipment rinsate samples are not required if the sampling equipment is dedicated to the location being sampled (e.g., water samples collected with disposable bailers).

2.5.1.2 Duplicates

A field duplicate is a subsample that has been divided from the primary sample at some step in the sampling process. The primary and duplicate samples are carried through the remaining steps of the process by the analytical laboratory to evaluate analytical performance. Field duplicate samples provide information on the precision of the sampling, transfer, and analytical process. Field duplicates shall be collected for every 10 field samples for all matrices.

2.5.1.3 Matrix Spike and Matrix Spike Duplicate Samples

Site-specific samples need to be used for MS/MSDs. Field sampling personnel will collect extra volume and designate (on the COC forms) the samples that are to be used for the MS/MSD. Every effort will be made

to ensure that these samples are representative of the general sample matrix of samples collected on that sampling date. Equipment rinsate blank samples will not be designated for MS/MSDs.

2.5.2 Laboratory Quality Control Samples

Laboratory quality control samples shall be introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Control samples may be prepared from environmental samples or be generated from standard materials in the laboratory. Laboratory quality control samples used in each analytical method are discussed in the following sections and are summarized in Table 2-4.

2.5.2.1 Method Blanks

Method blanks shall be used for the laboratory processes. A method blank is a volume of deionized water that is carried through the entire sample preparation and analysis procedure. The method blank volume or weight shall be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment. A method blank is prepared and analyzed with each analytical batch of 10 or fewer samples prepared.

2.5.2.2 Matrix Spikes

A spike is a sample to which is added a known amount of analyte(s) before analysis. From the concentrations of the analyte in the spiked and unspiked samples, a percent recovery is calculated. Many samples show matrix effects in which other sample components interfere with the determination of the analyte. The value of the percent recovery indicates the extent of the interference. A matrix spike is prepared by adding an analyte to a subsample of a field sample before sample preparation and analysis. For multi-analyte methods, a representative suite of the analytes is used in the matrix spike. An analytical spike is prepared by adding analyte to an aliquot of a processed sample prior to analysis, and is used to determine whether the analysis system provides results that are representative of the sample when a matrix spike is outside its limits.

2.5.2.3 Laboratory Control Samples (Verification Solutions)

A laboratory control sample (LCS), or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the program samples. An LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix for a stock solution that is different than that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the

sample preparation and analytical steps are in control, apart from sample matrix effects. LCSs contain the target analytes identified in the method.

2.5.2.4 Duplicate Samples

Duplicate samples are samples that have been divided into two portions at some step in the measurement process. Each portion is then carried through the remaining steps of the process. Duplicate samples provide information on the precision of the operations involved. Analytical duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analysis procedure; any difference between the results indicates the precision of the entire method in the given matrix. Under the laboratory protocols, the matrix spike is duplicated to provide a matrix spike duplicate. Matrix spike duplicates shall be prepared for every analytical batch of at least 10 samples.

Analytical duplicates are prepared by taking two aliquots of a process sample and analyzing them in the same manner. Both matrix and analytical spike duplicates are used to monitor the precision of the analytical process.

2.5.2.5 Surrogates

Since surrogates are typically used in chromatographic organic analyses but not in inorganic analyses, surrogates shall not be used.

2.6 INSTRUMENT/EQUIPMENT CALIBRATION AND MAINTENANCE REQUIREMENTS

2.6.1 Field Equipment

Field meters will be used in accordance with the manufacturer's instructions and calibration performed in accordance with Table 2-5. Conductivity (SW only) and specific conductivity, dissolved oxygen, turbidity, and pH meter performance will be calibrated and turbidity meters will be checked each morning prior to field sampling as well as at the end of each day

2.6.2 Laboratory Equipment

Laboratory equipment shall be calibrated according to the manufacturer's instructions, USEPA method criteria, the laboratories' SOPs (see Attachment A), Tables 2-6 through 2-8, and NFG.

2.7 DATA ACQUISITION, VALIDATION, REVIEW AND REPORTING

2.7.1 Field Data

Figure 2-1 provides a flow chart that summarizes all of the data acquisition, data validation, and data management activities of the program. Raw field data shall be entered in field notebooks; and/or sample collection record forms, which shall be reviewed for completeness by the FTL at the end of each day.

The overall quality of the field data from any given sampling round shall be further evaluated during the process of data validation as described below.

2.7.2 Analytical Data Validation

Any anomalies or limitations for the use of the data shall be documented as part of the laboratories analytical data packages. The laboratories shall establish calibration curves and calculation of sample concentrations from instrument responses. Raw laboratory data shall be converted to sample concentrations using formulas defined by applicable laboratory SOPs (Attachment A). The calculations shall be performed by computerized data systems interfaced to the instruments, by personnel calculators, or through programs installed on stand-alone personnel computers. Each laboratory analyst shall be responsible for the reduction of the raw data that they generate. At a minimum, such activities shall include:

- reduction of raw data generated to reportable values;
- an initial review of analytical and quality control data;
- performance of manual calculations and transfer of data onto forms, laboratory reports, and laboratory databases;
- preparation of computer files for instrumental calculations;
- generation of data forms for the analytical reports;
- copying of relevant forms and logs for inclusion in the laboratory reports;
- submittal of the laboratory report to a supervisor for a QA/QC review; and,
- resolution of discrepancies noted during the QA/QC review.

For non-instrument methods and for methods using instruments without computerized data systems that require manual calculations, the responsible analyst shall enter bench-generated data into bound laboratory workbooks with form-specific instrument responses, standard and spike concentrations, sample numbers, and other pertinent information. For instruments that are directly coupled to computerized data systems, raw data consist of instrument responses in the form of printer output or computer-generated data files. Printer output shall be filed by sample batch, and the data files archived on disk or magnetic tape.

Computer data files shall be identified by unique, sequential descriptors cross-referenced in the run logs to the analysis sequence. At a minimum, strip chart recordings shall be labeled with the following information:

- sample identification number;
- date and time of analysis;
- instrument identification;
- name of analyst(s);
- applicable operational parameters;
- date file identification; and,
- positively identified elements or compounds.

Completed analytical packages for each sampling round shall be routed to LDC for verification completeness and validation as stated in this QAPP and the current *NFG* and a copy of the analytical packages and coordinating electronic data deliverable in the format included in Attachment C shall be provided to the MWH Analytical Task Manager. All data shall be screened to ensure that data packages are complete and do not contain any obvious entry errors; any observed problems shall be referred to the affected laboratory for resolution.

At a minimum the MWH project chemist, or designee shall verify:

- COC forms were properly maintained from the field, to the laboratory, and through all analytical procedures;
- laboratory reports are complete and contain no transcription errors or omissions;
- holding time requirements were met;
- method blanks met method requirements; and,
- quantitation limits did not exceed established values.

Following third party data validation the data shall be qualified with regard to its usability as stated in the *NFG* and this QAPP, and copies of the validation report (a sample LDC report is included in Attachment D) will be provided to the MWH project chemist. Any other questions that involve the laboratory shall be resolved and the data package annotated as appropriate. The validated electronic data deliverable (EDD) information shall then be entered into the program database, and the validated data package forwarded to the program quality records pending preparation of final program submittals.

2.7.3 Data Analysis and Reporting

A final program report shall be prepared at the direction of the MWH PM to address the information needs of P4 PM and the A/Ts in accordance with the 2009 CO/AOC.

2.7.4 Data Storage and Retrieval

All field and laboratory records with accompanying data assessment summary forms shall be archived as part of the program quality records. All records shall be stored for a minimum of five years. In addition, MWH shall maintain an electronic database to support the hard- copy record system, using Microsoft Access™ relational database management program or Microsoft Excel, as needed.

3.0 ASSESSMENT/OVERSIGHT

3.1 INDEPENDENT TECHNICAL REVIEW

An independent technical review shall be performed by the MWH PM or their designee for all draft and final program reports. All comments shall be resolved and incorporate prior to any submittals to P4 and the A/Ts.

3.2 CORRECTIVE AND PREVENTIVE ACTION

Any nonconformance with approved *2015 LTM SAP* requirements that may be observed in the technical review discussed in Section 3.1; the data validation activities discussed in Sections 2.7; or that may be reported by MWH personnel or other sources in the course of this program shall be promptly evaluated and resolved and documented on an FCR form in the FSP.

.

4.0 REFERENCES

USEPA, 2001. *EPA Requirements for Quality Assurance Project Plan,* EPA QA/R-5, March.

USEPA, 2002. *Guidance for Quality Assurance Project Plan,* EPA QA/G-5, December.

USEPA, 2009a. *Administrative Settlement Agreement and Order of Consent/Consent Order for Performance of Remedial Investigation and Feasibility Study at the Enoch, Henry, and Ballard Mine Sites in Southern Idaho.* United States Environmental Protection Agency, U.S. EPA Region 10, Idaho Department of Environmental Quality, United States Department of Agriculture, Forest Service Region 4, United States Department of the Interior, Bureau of Land Management, Shoshone-Bannock Tribes, in the Matter of Enoch Valley Mine, Henry Mine, Ballard Mine, P4 Production L.L.C., Respondent. Effective Date of November 30, 2009.

USEPA, 2009b. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use,* EPA-540-R-08-005, January.

USEPA. 2014. *National Functional Guidelines for Inorganic Superfund Data Review.* EPA-540-R-013-001. August and prior version for methods not updated.

TABLES

TABLE 1-1
PROJECT CONTACTS

Company or Agency	Contact	Title	Telephone
P4 Production	Rachel Roskelley	P4 Program Manager	208-547-1248
USEPA	Dave Tomten	Remedial Project Manager	208-378-5763
MWH	Vance Drain	MWH Project Manager	801-617-3250
	Betty VanPelt	Project Chemist	801-617-3243
	Emily Jackson	On-Site Safety Officer/ Field Team Leader	801-617-3232
	Jennifer Barnett	Project Coordinator	801-617-8251
Microbac Laboratories Inc.	Kathy Albertson	Project Manager (primary laboratory)	800-373-4071
Laboratory Data Consultants, Inc	Stella Cuenco	Project Manager (data validation subcontractor)	760-634-0437

TABLE 1-2
SUMMARY OF QUALITY CONTROL PARAMETERS EVALUATED

PARCC	Quality Control Parameter	Evaluation Criteria	Applicable Methods
Precision	Laboratory Replicates	RPD between parent and laboratory replicated sample	All Methods
Precision	Field Duplicate/Replicates	RPD or concentration difference between parent and duplicate/replicate samples	All Methods
Accuracy	Instrument Tune Results	Method Criteria	ICP/MS
Accuracy	Initial Calibration	Relative standard deviation or correlation coefficient	All Methods
Accuracy	Initial Calibration Verification	%D or % Drift	All Methods
Accuracy	Calibration Verification Standard	%D or % Drift	All Methods
Accuracy	Matrix Spike	% Recovery	All Methods
Accuracy	Laboratory Control Sample	% Recovery	All Methods
Accuracy	Internal Standards	% Recovery	ICP/MS
Representativeness	QAPP Compliance	Qualitative, Degree of Confidence	All Methods
Representativeness	Sample Preservation	Cooler Temperature, Sample pH	All Methods
Representativeness	Holding Time	Days form sample collection to sample preparation and/or analysis	All Methods
Representativeness	Laboratory Method Blanks	Comparison of blank results to sample results	All Methods
Representativeness	Reporting Limits (RLs)	Comparison of RLs to cleanup levels	All Methods
Representativeness	Field Duplicates/Replicates	RPD or concentration difference between parent and duplicate/replicate samples	All Methods
Comparability	Standard Field Procedures	Qualitative, Degree of Confidence	All Methods
Comparability	Standard Analytical Methods	Qualitative, Degree of Confidence	All Methods
Comparability	Standard Units of Measure	Qualitative, Degree of Confidence	All Methods
Completeness	Sampling Completeness	% of samples collected versus samples scheduled	All Methods
Completeness	Analytical Completeness	% of samples analyzed versus sample collected	All Methods
Completeness	Valid Data	% Acceptable Data	All Methods

Notes:

%	percent
%D	Percent difference
ICP/MS	Inductively coupled plasma /mass spectrometry
PARCC	precision, accuracy, representativeness, comparability, completeness
RPD	Relative percent difference

TABLE 1-3
DATA QUALITY CHARACTERISTICS FORMULAE

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	RPD = (xi-xj)/((xi+xj)/2) x 100	xi, xj: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	RSD = (s/$\bar{x}$) x 100	s: sample standard deviation $\bar{x}$: sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples)	R = (x/t) x 100	x: sample value t: true or assumed value
Accuracy (as percent recovery, R, for measurements in which a known amount of analyte, a spike, is added to an environmental sample)	R = ((x_s - x)/t) x 100	xs: value of spiked sample x: value of unspiked sample t: true or assumed value
Completeness (as a percentage, C)	C = (n/N) x 100	n: number of valid data points produced N: total number of samples taken

TABLE 2-2
REQUIREMENTS FOR CONTAINERS, PRESERVATION TECHNIQUES, SAMPLE VOLUMES, AND HOLDING TIMES

Method & Parameter	Sample Preparation Method	Sample Container	Preservative	Minimum Sample Volume	Maximum Holding Time (Days)
Water Matrices (Surface Water, Groundwater)					
EPA SW-846 6020A-ICP/MS (Cd, Se)*	Dissolved (Field Filtered)	125 mL HDPE	HNO3	20 mL	180
EPA SW-846 6020A-ICP/MS (Se)*	Total	125 mL HDPE	HNO3	20 mL	180
EPA SW-846 6010C-ICP (Ca, Mg)	Dissolved (Field Filtered)	125 mL HDPE	HNO3	20 mL	180
EPA M300- Ion Chromatography (SO4)	Dissolved (Field Filtered)	250 mL HDPE	None	50 mL	28
EPA 160.1 – Gravimetric (TDS)	NA	500 mL HDPE	None	100 mL	7
SM1240B – Calculation (Hardness)	NA	NA	NA	NA	NA
*Specific parameters are media or sample type specific, see appropriate table in SAP.					

TABLE 2-3
ACHIEVABLE LABORATROY LIMITS AND APPLICABLE PROJECT SCREENING VALUES
SURFACE WATER AND GROUNDWATER PARAMETERS

Method	Analyte	Units	Achievable Laboratory Limits*		Surface Water Screening Value	Groundwater Screening Value
			RL	MDL		
EPA 6020A	Antimony	mg/L	0.001	0.0005	0.0056[c]	0.006[b]
	Arsenic	mg/L	0.001	0.0005	0.01[c]	0.01[d]
	Barium	mg/L	0.003	0.0015	0.004[e]	2[b]
	Cadmium	mg/L	0.0006	0.0003	0.0006[c]	0.005[b]
	Chromium	mg/L	0.002	0.001	0.035[f]	0.1[b]
	Cobalt	mg/L	0.001	0.0005	0.006[f]	0.006[f]
	Copper	mg/L	0.002	0.001	0.011[c]	1.3[b]
	Lead	mg/L	0.001	0.0005	0.0025[c]	0.015[b]
	Manganese	mg/L	0.002	0.001	0.05[a]	0.05[b]
	Nickel	mg/L	0.004	0.002	0.052[c]	0.39[f]
	Selenium	mg/L	0.001	0.0005	0.005[c]	0.05[b]
	Silver	mg/L	0.001	0.0005	0.0034[c]	0.1[b]
	Thallium	mg/L	0.0002	0.0001	0.00024[c]	0.002[b]
	Uranium	mg/L	0.001	0.0005	0.0026[h]	0.03[d]
	Vanadium	mg/L	0.001	0.0005	0.02[h]	0.086[f]
	Zinc	mg/L	0.025	0.0125	0.12[c]	5[b]
EPA 6010C	Aluminum	mg/L	0.2	0.1	0.087[a]	0.2[b]
	Beryllium	mg/L	0.01	0.005	0.00066[e]	0.004[b]
	Boron	mg/L	0.1	0.05	0.0016[e]	4.0[f]
	Iron	mg/L	0.1	0.05	0.16[g]	0.3[b]
	Molybdenum	mg/L	0.1	0.005	0.1[f]	0.1[f]
EPA 7470	Mercury	mg/L	0.0002	0.0001	0.00077[a]	0.002[b]
EPA 300	Chloride	mg/L	0.2	0.1	--	250[b]
	Fluoride	mg/L	0.2	0.1	--	4[b]
	Nitrate	mg/L	0.6	0.1	--	10[b]
	Nitrite	mg/L	0.4	0.1	--	1[b]
	Sulfate	mg/L	1	0.5	--	250[b]
EPA 160.1	TDS	mg/L	20	10	--	500[b]
Field	pH	pH units	--	--	--	6.5-8.5[b]

TABLE 2-3
ACHIEVABLE LABORATROY LIMITS AND APPLICABLE PROJECT SCREENING VALUES
SURFACE WATER AND GROUNDWATER PARAMETERS

Method	Analyte	Units	Achievable Laboratory Limits*		Surface Water Screening Value	Groundwater Screening Value
			RL	MDL		

Notes:

mg/kg milligram per kilogram

mg/L milligram per liter

* Generally achievable laboratory reporting limits; method detection limits will vary annually and by laboratory.

a National Recommended Water Quality Criteria (USEPA, 2009b, 2013b); Freshwater Standards for Chronic Criteria (CCC)

b State of Idaho Ground Water Quality Rule (IDAPA 58.01.11). Note that secondary MCLs are listed for pH, aluminum, chloride, iron, manganese, pH, silver, sulfate, TDS and zinc (IDEQ, 1996)

c State of Idaho Surface Water Quality for Aquatic Life (IDAPA 58.01.02); Chronic Criteria (CCC) or Water & Organisms (IDEQ, 2013a)

g USEPA Region 3 BTAG freshwater sediment screening benchmarks (USEPA, 2012)

d USEPA primary and secondary Maximum Contaminant Level (MCL), National Primary Drinking Water Regulations

e Chronic freshwater screening value in NOAA SQuiRT (Buchman, 2008)

f USEPA Regional Screening Levels (RSLs) for Water (USEPA, 2015)

g Lowest Chronic Value (LCV) observed in freshwater daphnids. Source: ORNL, 1996a.

h Tier II Secondary Chronic Value. Source: ORNL, 1996a.

TABLE 2-4
QUALITY CONTROL SAMPLE SUMMARY

Quality Control Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
Field QC Samples				
Rinsate or Equipment Blank	Identify whether target analytes may have been introduced into samples during sample collection.	One per day of sampling for each media when dedicated or disposable equipment is not used.	Sample collected from decontaminated non-dedicated equipment in the same container type as primary sample.	Assess whether target analyses were introduced into the blanks during sample collection. A measure of accuracy.
Field Duplicate	Assess sampling and analytical precision.	One per 10 samples for each media	Sample is submitted blind to the laboratory. Duplicate of a specific performance sample collected in the field and submitted blind to the laboratory.	Data for the primary sample(s) and the duplicate shall be compared to assess consistency during sampling and analysis. A measure of precision.
Laboratory QC Samples				
Method Blank	Identify target analyses that may have been introduced into the sample during preparation and analysis.	One per 10 samples for each media or per sample delivery group (SDG) (whichever is more frequent).	Reagent-grade water that is carried through the same analytical procedures as the media being analyzed.	Assess whether target analyses were introduced into the blank during the analysis. A measure of accuracy.
Matrix Spike (MS)	Identify media interference during analysis.	One per 10 samples for each media or per SDG (whichever is more frequent).	Two subsamples shall be obtained from primary water or homogenized solid sample. One container shall be labeled for MS analysis; the other for MS duplicate (MSD) analysis. The samples shall be spiked in accordance with lab standard operating procedures (SOPs).	The percent of recovery and the RPD for each spiked analyte shall be reviewed for compliance with the laboratory QC limits. A measure of precision and accuracy.

TABLE 2-4
QUALITY CONTROL SAMPLE SUMMARY

Quality Control Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
Laboratory QC Samples (continued)				
Laboratory Control Sample (LCS)	Assess laboratory precision and accuracy.	One per 10 samples for each media or per SDG (whichever is more frequent).	LCS are prepared by the laboratory and consist of samples spiked with analyses specified in the laboratories' SOPs.	The percent recovery and the RPD for each spiked analyte shall be reviewed for compliance with the laboratory QC limits. A measure of precision
Analytical Duplicate	Assess analytical precision.	One per 10 samples for each media or per SDG (whichever is more frequent).	Duplicate of prepared sample and analyzed and reported by the laboratory.	Data for the sample and its duplicate shall be compared to assess consistency during sample preparation and analysis. A measure of precision.

TABLE 2-5
CALIBRATION AND MAINTENANCE REQUIREMENTS FOR FIELD EQUIPMENT

Field Parameter	Required Procedure	Minimum Frequency	Required Equipment or Calibration Fluids
pH	2-point calibration	Each day prior to sampling and end of day	One pH buffer (7 pH), Reference Standard
Temperature	N/A	N/A	N/A
Dissolved Oxygen	Atmospheric calibration	Each day prior to sampling and end of day	Atmospheric oxygen with elevation
Conductivity and Specific Conductivity	1-point reference	Each day prior to sampling and end of day	Reference Standard
ORP	1-point reference	Each day prior to sampling and end of day	Reference Standard
Turbidity	1-point calibration	Each day prior to sampling and end of day	Reference Standard
Flow	Velocity equivalence and spin test (Price meters) or equivalent	When the rotor is changed	Stopwatch, measuring tape

Notes:

*In the event of a discrepancy, the manufacturer's instruction manual shall take precedence.

TABLE 2-6
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6020A (ICP-MS)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
MS Tuning sample	Prior to initial calibration, solution as specified in Section 7.10 of method (e.g., ^{7}Li, ^{59}Co, ^{115}In, and ^{205}Tl)	Mass calibration ≤ 0.1 µ over the range of 6 – 210 µ, Resolution < 0.9 amu full width at 10% peak height. Stability: RSD ≤ 5% for at least five replicate analysis.	Retune instrument then reanalyze tuning solution	Per Section II of ICP-MS NFG, except substitute with method acceptance limits.	RSD > 5% = J/UJ (professional judgment on criteria related to non-target analytes).
Initial calibration (ICAL) for all target analytes (minimum five standard and a blank, one between RL and MDL	Each time the instrument is set up and after continuing calibration failure.	Correlation coefficient (r) ≥ 0.995 and non-zero standards ±30% of true value of the standard.	Correct problem then repeat initial calibration.	Per Section III of ICP-MS NFG.	r < 0.995 = J/UJ and non-zero standards ±30% of true value of the standard.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within ±10% of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL	Per Section III of ICP-MS NFG.	%R < 90 or >110% = J/UJ Per Table 14 in NFG
Initial Calibration Blank (ICB)	After ICV	No analyte detected > RL	Correct problem and reanalyze.	Per Section IV of ICPMS NFG, except U at detected value if result>MDL < RL.	Per Table 15 in NFG, except U at detected value if result > MDL < RL.
Interference Check Sample A & AB (ICS-A & ICS-AB)	At the beginning of an analytical sequence between the ICV and CCV	< 2X RL or within ± 20% of expected value (whichever is greater).	Correct problem and reanalyze ICS-A and ICS-AB.	Per Section V of ICP-MS NFG.	ICS < 80% or > 120% = J/UJ per Table 16 in NFG.
Continuing Calibration Verification (CCV)	Prior to samples, every 2 hours and the end of the analytical sequence (at a mid_calibration range concentration differenct from ICV concentration)	Every analyte within ±10% of expected value.	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Per Section III of ICP-MS NFG.	CCV < 90 or > 110% = J/UJ; per Table 14 in NFG

TABLE 2-6
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6020A (ICP-MS)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Continuing Calibration Blank (CCB)	Following each CCV and at the end of the analytical sequence.	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and previous 2 hours of samples. Apply "B" flag to all associated positive results for the specific analyte(s) as appropriate.	Per Section IV of ICPMS NFG, except U at detected value if result>MDL < RL.	Per Table 15 in NFG, except U at detected value if result > MDL < RL.
Method blank (or preparation blank)	One per analytical batch, each matrix, or with every SDG whichever is more frequent.	No analyte detected ≥ RL	Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank, Apply B flag to all associated positive results for the specified analyte(s) in the preparation batch.	Per Section IV of ICPMS NFG, except U at detected value if result > MDL<RL	Per Table 15 in NFG, except U at detected value if result > MDL<RL
Laboratory Control Sample (LCS) for all analytes at 2X RL	One LCS per matrix per SDG or analytical batch, whichever is more frequent	Vendor-specified or laboratory-determined control limits (but not wider than 80-120% recovery). If LCS/LSC duplicate (LCSD) used, then use RPD ≤ 20.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Per Section VI of ICP-MS NFG, except substitute 80-120% recovery and ≤ 20 RPD limits.	%R < 80 or > 120% for water = J/UJ; < 50% = J detects, R non-detects
Duplicate Sample	One per matrix per batch or per SDG	Both results > 5X RL within ± 20%	Flag as estimated value ("J" flag)	Per Section VII of ICP-MS NFG	Both results > 5X RL within ± 20%. Table 18 in NFG
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every per matrix per batch or SDG (whichever is more frequent)	Laboratory-determined control limits (but not wider than 70-130% recovery and RPD ≤ 20).	Flag associated sample results and perform post-digestion spike addition.	Per Section VIII of ICP-MS NFG, except substitute 75-125% recovery and ≤ 20 RPD limits.	%R < 75 or > 125% for water = J/UJ; < 30% = J detects, R non-detects. Water RPD <20%, soil < 35%. Low level (< 5 X RL, use + RL water, 2 X RL for soil). For MS, if %R < 30% and post spike< 75% or not run, J detects, R non-detects. If post spike > 75 %, UJ non-detects and J detects
Post-digestion spike addition	If MS/MSD fails	Recovery within 75-125% of expected results	Perform dilution test.	Per Section VIII of ICP-MS NFG	Per Table 19 ICP-MS NFG

TABLE 2-6
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6020A (ICP-MS)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Serial dilution (SD) test	One SD per matrix per batch or SDB (whichever is more frequent).	Five-fold dilution. Must agree with in ±10% of the original determination.	Flag associated sample results and discuss in case narrative.	Per Section IX of ICP-MS NFG.	%D < 90 >110% = J/UJ
Internal Standards (ISs)	Every sample; internal standards selected from list specified in Section 1.4 of method.	IS intensity ≥ 70% < 130% of intensity of the IS in the ICAL	Perform corrective action as described in Section 9.6 of method.	Per Section X of ICP-MS NFG, except substitute 70-130% limits	IS %R < 70% > 130 % = J/UJ
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("J" flag)	Not applicable	Not applicable

a National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2014.).
b Refer to NFG for detailed evaluation protocols.

MDL	method detection limit
RL	reporting limit
RPD	relative percent difference
RSD	relative standard deviation

TABLE 2-7
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6010C (ICP-AES)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Tune	Prior to calibration	Run at least 5X consecutively, peak width within instrument manufacturer's specifications, mass resolution 0.1 µ over range of 6-210 µ, % RSD < %5 for all analytes	Correct problem	Per Section II of the ICP-MS NFG.	R is tune not performed. J hits, UJ non-detects Resolution not 0.1 µ or %RSD>5%
Initial calibration (ICAL) for all target analytes (minimum five standard and a blank, one between RL and MDL	Each time the instrument is set up and after continuing calibration failure.	Correlation coefficient (r) ≥ 0.995 and non-zero standards ±30% of true value of the standard.	Correct problem then repeat initial calibration.	Per Section III of ICP-MS NFG.	r < 0.995 = J/UJ and non-zero standards ±30% of true value of the standard.
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within ±10% of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL	Per Section III of ICP-MS NFG.	%R < 90 or >110% = J/UJ Per Table 14 in NFG
Initial Calibration Blank (ICB)	After ICV	No analyte detected > RL	Correct problem and reanalyze.	Per Section IV of ICPMS NFG, except U at detected value if result>MDL < RL.	Per Table 15 in NFG, except U at detected value if result > MDL < RL.
Interference Check Sample A & AB (ICS-A & ICS-AB)	At the beginning of an analytical sequence between the ICV and CCV.	< 2X RL or within ± 20% of expected value (whichever is greater).	Correct problem and reanalyze ICS-A and ICS-AB.	Per Section V of ICP-MS NFG.	ICS < 80% or > 120% = J/UJ per Table 16 in NFG.

TABLE 2-7
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6010C (ICP-AES)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Continuing Calibration Verification (CCV)	Prior to samples, every 2 hours and the end of the analytical sequence (at a mid_calibration range concentration differenct from ICV concentration)	Every analyte within ±10% of expected value.	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Per Section III of ICP-MS NFG.	CCV < 90 or > 110% = J/UJ; per Table 14 in NFG
Continuing Calibration Blank (CCB)	Following each CCV and at the end of the analytical sequence.	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and previous 2 hours of samples. Apply "B" flag to all associated positive results for the specific analyte(s) as appropriate.	Per Section IV of ICPMS NFG, except U at detected value if result>MDL < RL.	Per Table 15 in NFG, except U at detected value if result > MDL < RL.
Method blank (or preparation blank)	One per analytical batch, each matrix, or with every SDG whichever is more frequent.	No analyte detected ≥ RL	Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank, Apply B flag to all associated positive results for the specified analyte(s) in the preparation batch.	Per Section IV of ICPMS NFG, except U at detected value if result > MDL<RL	Per Table 15 in NFG, except U at detected value if result > MDL<RL

TABLE 2-7
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 6010C (ICP-AES)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Laboratory Control Sample (LCS) for all analytes at 2X RL	One LCS per matrix per SDG or analytical batch, whichever is more frequent	Vendor-specified or laboratory-determined control limits (but not wider than 80-120% recovery). If LCS/LSC duplicate (LCSD) used, then use RPD ≤ 20.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Per Section VI of ICP-MS NFG, except substitute 80-120% recovery and ≤ 20 RPD limits.	%R < 80 or > 120% for water = J/UJ; < 50% = J detects, R non-detects
Duplicate Sample	One per matrix per batch or per SDG	Both results > 5X RL within ± 20%	Flag as estimated value ("J" flag)	Per Section VII of ICP-MS NFG	Both results > 5X RL within ± 20%. Table 18 in NFG
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every per matrix per batch or SDG (whichever is more frequent)	Laboratory-determined control limits (but not wider than 70-130% recovery and RPD ≤ 20).	Flag associated sample results and perform post-digestion spike addition.	Per Section VIII of ICP-MS NFG, except substitute 75-125% recovery and ≤ 20 RPD limits.	%R < 75 or > 125% for water = J/UJ; < 30% = J detects, R non-detects. Water RPD <20%, soil < 35%. Low level (< 5 X RL, use + RL water, 2 X RL for soil). For MS, if %R < 30% and post spike< 75% or not run, J detects, R non-detects. If post spike > 75 %, UJ non-detects and J detects
Post-digestion spike addition	If MS/MSD fails	Recovery within 75-125% of expected results	Perform dilution test.	Per Section VIII of ICP-MS NFG	Per Table 19 ICP-MS NFG
Serial dilution (SD) test	One SD per matrix per batch or SDB (whichever is more frequent)	Five-fold dilution. Must agree with in ±10% of the original determination.	Flag associated sample results and discuss in case narrative.	Per Section IX of ICP-MS NFG.	%D < 90 >110% = J/UJ
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("J" flag)	Not applicable	Not applicable

a National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2014).
b Refer to NFG for detailed evaluation protocols.

MDL	method detection limit	RPD	relative percent difference
RL	reporting limit	RSD	relative standard deviation

TABLE 2-8
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 300.0 (IC)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Initial calibration (ICAL) for all target analytes (minimum five standards)	Daily intial calibration prior to sample analysis	correlation coefficient (r) ≥ 0.995 or relative standard deviation ≤ 10%.	Correct problem then repeat initial calibration.	Evaluate r or RSD against control limits.	r < 0.995 = J/UJ or RSD > 10% = J/UJ
Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run (at a concentration other than used for calibration and from a second source)	All analytes within □10% of expected value. Retention time (RT) window set at ± 3 times the standard deviation for each analyte in runs within a 24-hour period; daily midpoint established from RT of analyte in ICV.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Evaluate percent recovery .	%R < 90 or >110% = J/UJ if %R > 130% or < 70% then J/R.
Initial Calibration Blank (ICB)	After ICV	No analyte detected > 2X MDL	Correct problem and reanalyze.	Evaluate blank result against critierion.	ICB or CCB > MDL U at RL or U at detected value if analyte detected ≥ RL.
Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the sequence (at mid-calibration range concentration)	Every analyte within ±10% of expected value.	Correct problem then repeat CCV and reanalyze all samples since last successful CCV.	Evaluate percent recovery .	%R < 90 or >110% = J/UJ if %R > 130% or < 70% then J/R.
Continuing Calibration Blank (CCB)	Following each CCV and at the end of the analytical sequence.	No analyte detected > 2X MDL	Correct problem and reanalyze.	Evaluate blank result against critierion.	ICB or CCB > MDL U at RL or U at detected value if analyte detected ≥ RL.

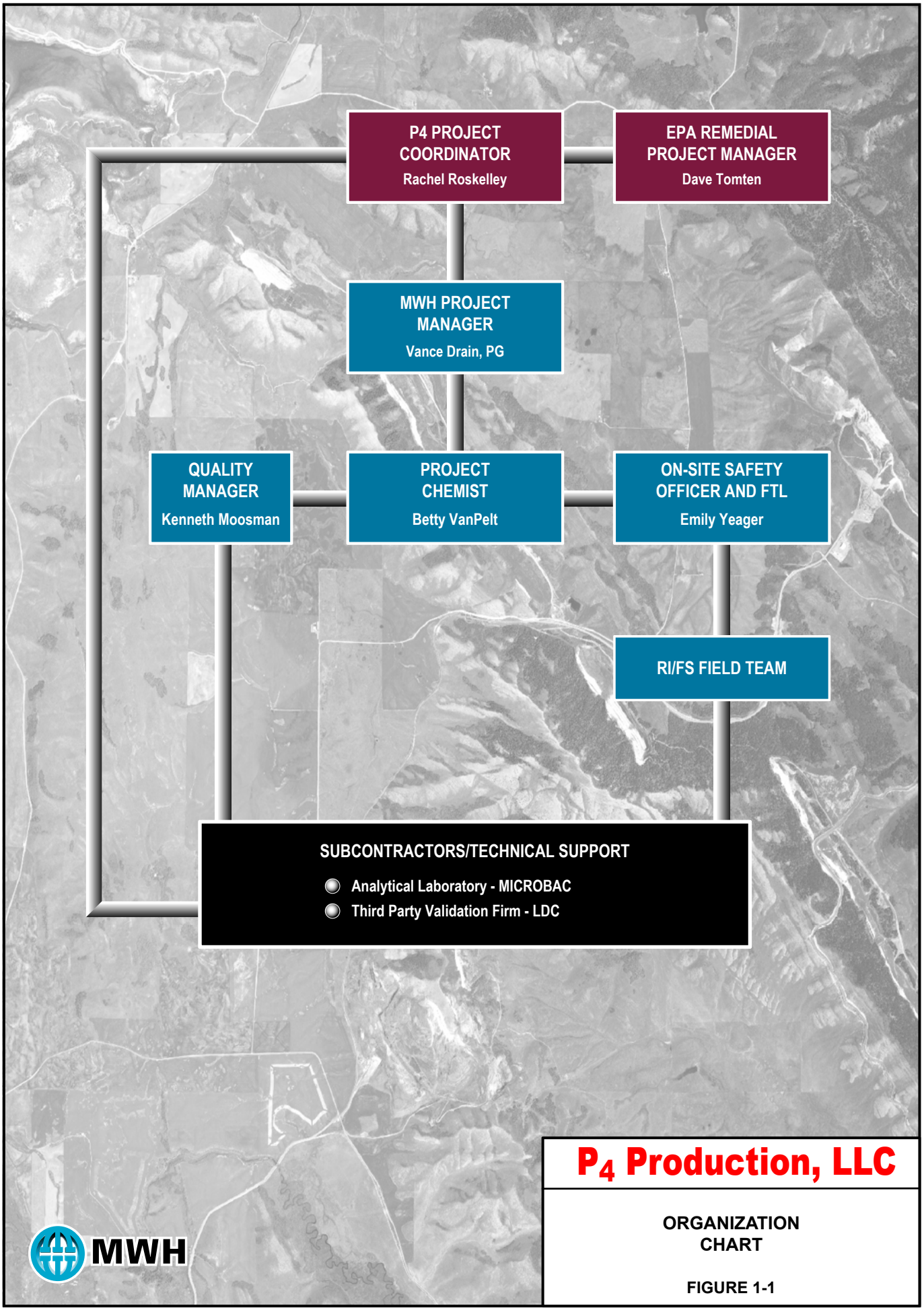
TABLE 2-8
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 300.0 (IC)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section[a]	Data Validation Qualification[b]
Method blank (or preparation blank)	One per analytical batch, each matrix, or with every SDG whichever is more frequent.	No analyte detected ≥ RL	Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank, Apply B flag to all associated positive results for the specified analyte(s) in the preparation batch.	Evaluate blank result against critierion.	MB/PB > MDL = U at RL or U at detected value if analyte detected ≥ RL.
Laboratory Control Sample (LCS) for all analytes at 2X RL	One LCS per matrix per SDG or analytical batch, whichever is more frequent	Vendor-specified or laboratory-determined control limits (but not wider than 80-120% recovery). If LCS/LSC duplicate (LCSD) used, then use RPD ≤ 20.	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Evaluate %R against control limits.	%R < 80 or > 120% for water = J/UJ; < 50% = J detects, R non-detects
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every per matrix per batch or SDG (whichever is more frequent)	Laboratory-determined control limits (but not wider than 80-120% recovery and RPD ≤ 20).	Flag associated sample results and perform post-digestion spike addition.	Per Section VIII of ICP-MS NFG, except substitute 75-125% recovery and ≤ 20 RPD limits.	%R < 80 or > 120% for water = J/UJ; < 30% = J detects, R non-detects
Post-digestion spike addition	If MS/MSD fails	Recovery within 75-125% of expected results	Perform dilution test.	Per Section VIII of ICP-MS NFG	Per Table 19 ICP-MS NFG

a National Functional Guidelines (NFG) for Inorganic Data Review (USEPA, 2014).
b Refer to NFG for detailed evaluation protocols.

IC	ion chromatography		
MDL	method detection limit	RPD	relative percent difference
RL	reporting limit	RSD	relative standard deviation

FIGURES



P_4 Production, LLC

ORGANIZATION CHART

FIGURE 1-1

MWH

Drawn by D. Severson

17 Mar 2015

J:\Industrial Projects\P4 Monsanto\10506883 2015 LTM SAP - FW and DSR\DELIVERABLES\2015 LTM SAP_QAPP\Draft\Figures\Figure 1-1_Organizatin Chart_Mar2015

COC No. A 05083

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project ID:	
Sampler (print):	Signature:

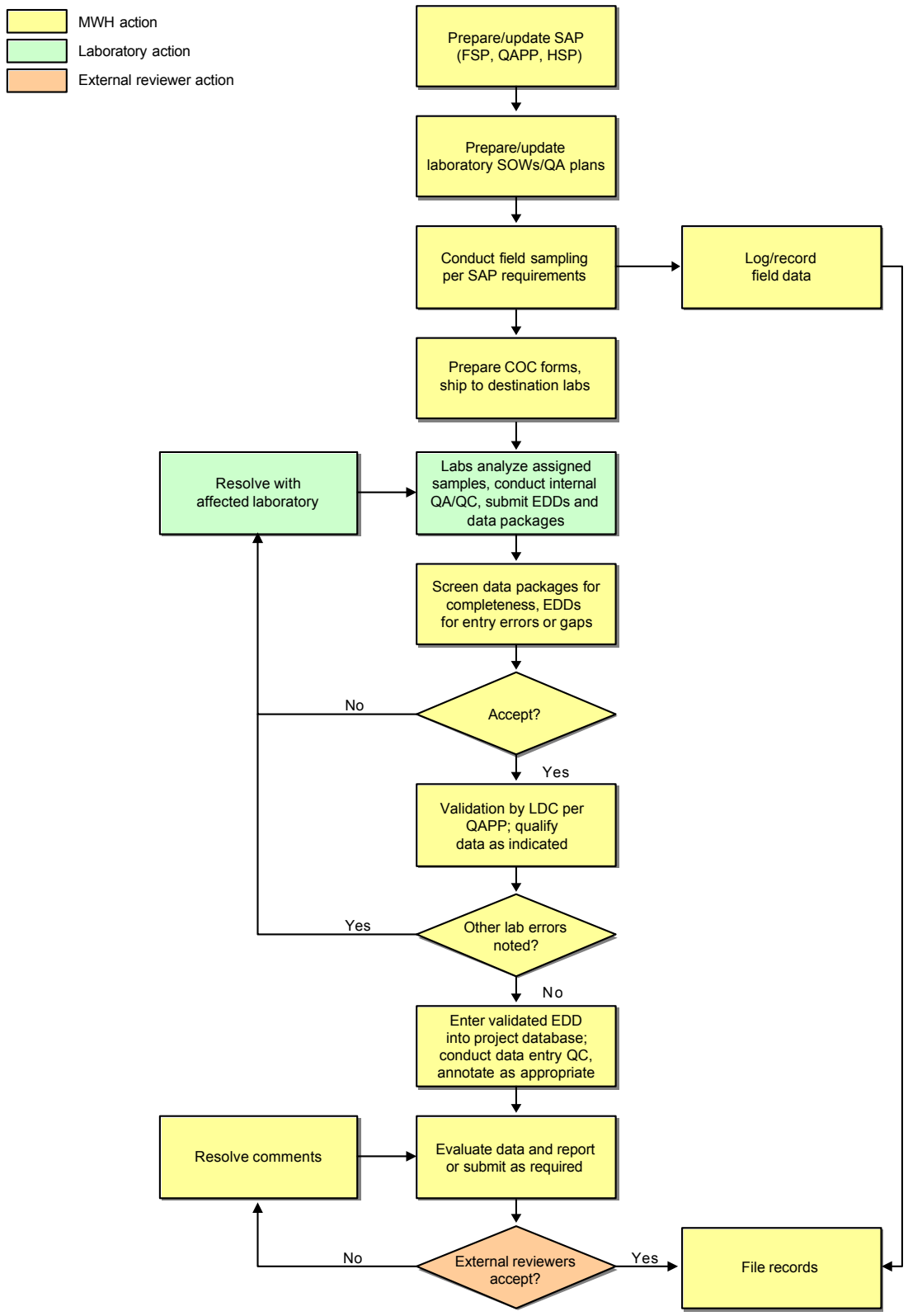
Program
- ☐ CWA
- ☐ RCRA
- ☐ DOD
- ☐ AFCEE
- ☐ Other _____

ADDITIONAL REQUIREMENTS

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold																TOTAL # (LAB USE)

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)	
Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Remarks:		

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)



P_4 Production, LLC

DATA ACQUISITION AND DATA MANAGEMENT FLOW DIAGRAM

FIGURE 2-1

MWH

17 Mar 2015

J:\Industrial Projects\P4 Monsanto\10506883 2015 LTM SAP_ FW and DSR\DELIVERABLES\2015 LTM SAP_QAPP\Draft\Figures\Figure 2-1_Data Acquisition and Data Management Diagram_Mar2015

ATTACHMENTS

ATTACHMENT A
LABORATORY SOPS

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURES
DETERMINATION OF INORGANIC ANIONS
BY ION CHROMATOGRAPHY
METHOD 9056/300.0

Issue/Implementation Date: 15 February 2015

Last Review Date: 15 February 2015

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

John Richards
John W. Richards, Primary Analyst — 02/10/2015 Date

Wade T. DeLong
Wade T. DeLong, Quality Assurance Officer — 2-10-15 Date

Leslie S. Bucina
Leslie S. Bucina, Laboratory Manager — 2/10/15 Date

Uncontrolled Copy

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	5
5.0	Interferences and Corrective Action	5
6.0	Equipment and Supplies	6
7.0	Standards and Reagents	6
8.0	Diagram or Table to Outline Procedures	8
9.0	Sample Preparation	9
10.0	Calibration Procedures	11
11.0	Analytical Procedures	13
12.0	Details of Calculations	15
13.0	Quality Control Requirements	17
14.0	Data Review and Reporting Requirements	19
15.0	Preventive Maintenance	20
16.0	Waste Management and Pollution Control	21
17.0	References	21
	Tables	23
	Figures	25

1.0 SCOPE AND APPLICATION

1.1 This method is used for the analysis of the anions fluoride, chloride, nitrite, bromide, nitrate, and sulfate in water and soil samples and in solutions collected from the bomb combustion (Microbac SOP K5050) of waste samples. This SOP complies with the requirements of SW 846 Method 9056/9056A and EPA Method 300.0.

1.2 A small volume of sample is injected into the ion chromatograph to flush and / or fill a sample loop. The sample is then injected into a carbonate-bicarbonate eluent, pumped through two different ion exchange columns and a conductivity detector. The two columns (guard and analytical) are packed with low capacity, strong basic anion exchanges. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. A suppressor reduces the background conductivity of the eluent and converts the anions into their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. The anions are identified based on their retention times compared to known standards, then compared to a calibration curve.

1.3 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCB	Continuing calibration blank
CCV	Continuing calibration verification
COA	Certificate of analysis
IC	Ion chromatograph
ICAL	Initial calibration
ICV	Initial calibration verification
LCR	Linear Calibration Range
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
LQAP	Laboratory Quality Assurance Program
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report
QC	Quality control

RGT	Reagent
RL	Reporting limit
RSD	Relative standard deviation
SD	Sample duplication
SOP	Standard operating procedure
STD	Standard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 As with any sample of unknown composition, proper safety procedures must be followed. This includes but is not limited to the use of protective glasses, lab coats, gloves, and fume hoods.

2.2 Follow the manufacturer's guidelines for safe and proper use of the ion chromatograph in order to prevent personal injury and instrument damage.

2.3 Gloves must be worn at all times during any handling of samples as well as the materials used in preparing their dilutions.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples may be collected in plastic or glass bottles and are cooled to ≤ 6° C until time of analysis. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis and laboratory fortified matrix analysis, if required, and minimize waste disposal.

3.2 If sample combustion is required at least 10.0 g will be needed for Microbac SOP K5050.

3.3 The holding time is 48 hours for nitrate and nitrite. The other anions have a 28 day hold time.

4.0 METHOD PERFORMANCE

4.1 Table 2 summarizes the performance data for water and soil/solid waste analysis. This table includes the analyte list, ranges for accuracy and precision, nominal laboratory RLs, and the current laboratory MDLs.

4.2 The laboratory performed an initial assessment of the LOD/LOQ using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\Data\Common\MDL. The laboratory verified LOD/LOQ is found in Table 2. The LOD/LOQ is evaluated annually, or when necessary and verified quarterly. The LOQ Verification consists of analyzing a fortified blank (RL check standard) spiked at a concentration equal to Level 1 or 2 (depending on analyte) in Section 7.4.1. The LOQ check standard is used to verify that the laboratory RL is routinely achievable over the course of time. Additional details on LOQ verification is found in Microbac SOP 45.

4.3 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.4 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Large amounts of any anion can overlap with adjacent anions. If this should happen then samples may be diluted.

5.2 Samples are filtered at 0.45 um and eluents are filtered at 0.20 um to prevent damage to the instrument flow system.

5.3 Prior to any injection into the instrument flow system, all samples must be screened for known matrix interferences that can damage components of the instrument (i.e. chloride or sulfate > 200 ppm, samples that generate biological waste matter, samples that cause a precipitation reaction with the working eluent, samples that have a strong organic solvent odor). See Section 9.1 for screening procedures.

Uncontrolled Copy

6.0 EQUIPMENT AND SUPPLIES

6.1 Dionex DX-320, Dionex DX-500, or Dionex ICS1100 chromatography system with a 25 uL sample loop.

6.2 GP50 gradient pump

6.3 CD20 conductivity detector

6.4 LC25 column oven

6.5 AS 40 or AS-DV automated sampler

6.6 Dionex Peaknet software

6.7 Dionex AG14A- 4 mm guard column

6.8 Dionex AS14A- 4 mm analytical column

6.9 Dionex ASRS 500 or AERS 500 regenerating suppressor (4 mm)

7.0 STANDARDS AND REAGENTS

7.1 All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 DI water

7.3 AS14A eluent concentrate: a solution of 800 mM sodium carbonate and 100 mM sodium bicarbonate is prepared by dissolving 84.8 g sodium carbonate (J.T. Baker 3604 or equivalent) and 8.4 g sodium bicarbonate (J.T. Baker 3508 or equivalent) in DI water in a 1 L class A volumetric flask and stirring for at least 30 minutes.

7.3.1 Working eluent is prepared from the eluent concentrate by diluting 20 mL of the eluent concentrate in DI water in a 2 L volumetric flask, bringing to volume and stirring for approximately 20 minutes. The final concentration of this solution will be 8 mM sodium carbonate and 1 mM sodium bicarbonate.

7.4 Stock multiple anion standard (I-Cal Standard from Inorganic Ventures Inc.) at the following concentrations:

ANION	Concentration (ug/mL)
F	100
Cl	100
NO_2	200
Br	100
NO_3	300
SO_4	500

7.4.1 Working calibration standards are prepared from the stock mix using the following scheme:

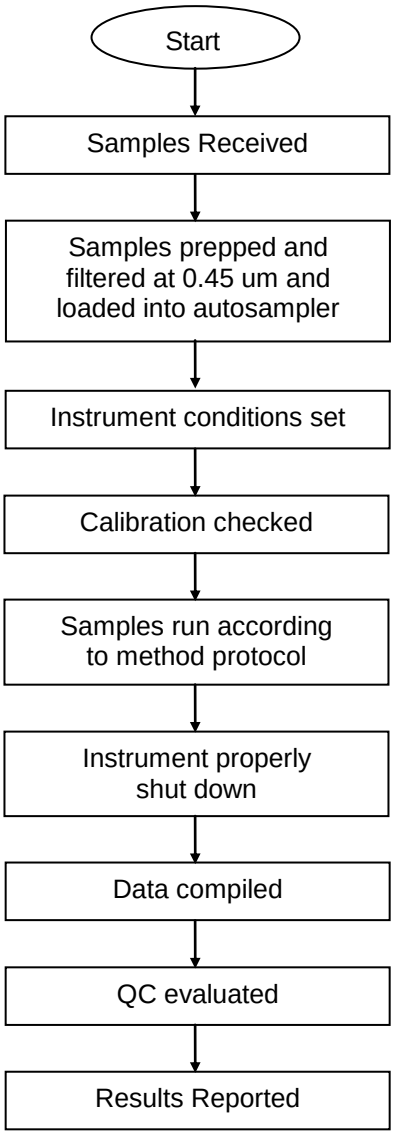
Standard	Standard Amount	Final Volume	Concentration (ug/mL)			
			F, Cl, Br	NO_2-N	NO_3-N	SO_4
Level 6	24 mL stock std.	100 mL (DI water + 1 mL eluent)	24	14.6	16.3	120
Level 5	12 mL stock std.	100 mL (DI water + 1 mL eluent)	12	7.31	8.13	60
Level 4	8 mL stock std.	100 mL (DI water + 1 mL eluent)	8	4.87	5.42	40
Level 3	4 mL stock std.	100 mL (DI water + 1 mL eluent)	4	2.44	2.72	20
Level 2	25 mL std #3	100 mL (working eluent)	1	0.609	0.678	5
Level 1	20 mL std #2	100 mL (working eluent)	0.2	0.122	0.136	1

7.4.2 The lowest standard must be at or below the LOQ.

7.5 Alternate stock multiple anion standard having a different lot number than the calibration standard (7.4) (purchased from Inorganic Ventures, Inc. or elsewhere) to be used for ICV.

7.6 Conditioning reagent (for screening): prepared using the procedure in EPA method 375.4 Section 5.1.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Sample screening: If sample history is not available, samples may be screened in order to determine levels of interferences prior to analysis.

9.1.1 Chloride screening: Syringe filter 14 mL of sample into a 17x100 mm disposable polystyrene culture tube. Using a Chloride Titrator Test Strip (Hach low range: 30-600 ppm), determine the chloride concentration units (ppm). If the sample's chloride concentration exceeds the upper limit of the test strip, dilute the sample and retest with a new strip. If the chloride test results are now within calibration range, use this dilution for analysis.

9.1.2 Sulfate screening: Syringe filter 14 mL of sample into a 17x100 mm disposable polystyrene culture tube. Add 0.5 mL of conditioning reagent to the sample and mix. Add approximately 0.1 g of barium chloride (dihydrate, granular) to the sample and mix. If sulfate is present, the clear sample will turn a turbid cloudy white color as barium sulfate is precipitated. Directly compare the turbidity of the sample in its culture tube to that of various levels of sulfate standards (i.e. 100, 200, 300, 500, 1000 ppm prepared the same way.) If the sample's turbidity is greater than the 300 ppm sulfate standard by comparison, perform a 2x dilution of the sample with DI water and compare its turbidity to the sulfate standards once again. If a further dilution is required, perform a 3x dilution in the same manner and compare to the sulfate standards. If a sample requires more than a 4x dilution, the sample must be diluted prior to the addition of conditioning reagent and barium chloride. If a dilution was necessary, multiply the final sulfate concentration range determined by comparison to the sulfate standards by the total dilution factor used.

9.1.3 Precipitation reaction screening: Syringe filter 14 mL of sample into a 17x100 mm disposable polystyrene culture tube. Make sure that the filtered sample is not turbid at the outset. Add 0.5 mL of eluent concentrate to the filtered/clear sample. Allow this mixture to sit for at least 15 minutes. If the sample gains turbidity at any point during this time, a precipitation reaction has occurred. A turbid white color typically indicates the presence of either dissolved calcium or aluminum; a turbid orange color typically indicates the presence of dissolved iron. Any sample whose matrix undergoes a precipitation reaction with the eluent used for chromatographic analysis will require at least one treatment with the eluent concentrate prior to injection. Samples are treated by adding 1 mL of eluent concentrate per 100 mL of sample directly into their sample bottles. Invert the sample and shake repeatedly. Allow this treated sample to sit for at least 30 minutes or until the precipitate settles to the bottom of the bottle. If the turbidity of this screen is greater than the turbidity of the 200 ppm sulfate standard used for sulfate screening (Section 9.1.2), the treated sample will

need to be screened again. If a second treatment (or more) of the sample is required and the sample is to be analyzed neat (i.e. chloride and sulfate screens < 200 ppm), the sample will need to be treated until the precipitation reaction is no longer observed. If the sample is to be analyzed at a dilution (i.e. chloride or sulfate > 200 ppm), the sample can be diluted with working eluent and filtered prior to injection.

9.1.4 The presence of a hydrocarbon odor or bubbles resulting from biological activity may also indicate that an initial dilution may be warranted in order to avoid instrument damage.

9.2 Water sample preparation: Fill a 10 mL syringe with sample and filter through a 0.45 micron filter disposing the first 5 mL of sample. Place the next 5-6 mL of sample into a disposable 5 mL sample vial and top with a filter cap. If the area of an anion exceeds the area for the level 6 standard for that anion, then the sample must be diluted and analyzed again.

9.2.1 A method blank is prepared for each batch of 20 samples by filtering DI water in the same manner as for the samples.

9.2.2 A LCS is prepared in the same manner as level 4 of the calibration and filtered. In the absence of an MS/MSD, an LCS DUP may be analyzed as well.

9.2.3 MS and MSDs are prepared by adding 0.8 mL of stock standard to 9.2 mL of sample and filtering.

9.3 Soil samples are prepared as indicated in EPA method 300. Weigh 5 g of soil into a 50 mL screw-capped vial and add 50 mL of DI water. Using a shaker, mix for 30 minutes. After shaking, filter through a 0.45 micron filter and dispose of the first 5 mL of extract. The filtered extract is loaded into a 5 mL vial for analysis.

9.3.1 A soil method blank is prepared in the same manner as for samples, using lab sand as a matrix. The sand may be solvent rinsed and dried beforehand to eliminate contamination.

9.3.2 A soil LCS is prepared in the same manner as for samples, using lab sand as a matrix and adding 46 mL of DI water and 4 mL of stock standard. In the absence of an MS/MSD, an LCS DUP may be analyzed as well.

9.3.3 Soil MS/MSD and are prepared in the same manner as for samples, using 46 mL of DI water and 4 mL of stock standard.

Uncontrolled Copy

10.0 CALIBRATION PROCEDURES

10.1 Recommended instrument conditions:

Column:	Dionex AS14A/AG14A analytical/guard
Suppressor:	Dionex ASRS 500 or AERS 500
Conductivity detector:	50 ma
Cell temperature:	30 degrees C
Injection volume:	25 uL
Flow rate:	1 mL/minute
Pressure:	3000 psi maximum

10.2 Initial calibration is performed every 6 months, after major maintenance or when the continuing calibration is out of control.

10.3 The initial calibration is accomplished by injecting each of the calibration standards from Section 7.4.1. After the analysis of the calibration standards is complete, the data is used to plot a linear regression curve using the PEAKNET data system. The correlation coefficient (R) must be ≥ 0.995.

10.4 ICV: Immediately following the initial calibration an ICV is analyzed. The ICV is prepared in the same manner as the Level 4 calibration standard (7.4.1) using the alternate stock solution (7.5). The ICV must have %D ≤ 10 before sample analysis can proceed

10.5 LCR Verification: Immediately following the ICV, the Level 6 and Level 2 calibration standards (7.4.1) and an eluent blank are analyzed to verify the linear calibration range.

10.6 CCV: At the beginning of each daily analysis and after every ten (10) samples, the Level 4 calibration standard is analyzed to evaluate the response of the analytical system. The CCV must have a %D ≤ 10 before sample analysis can proceed. If the CCV fails to meet this criterion, a second CCV may be analyzed. Failure of two consecutive CCVs requires that the problem be discerned and recalibration may be required. Any samples not bracketed by passing CCVs must be reanalyzed.

10.7 After each CCV, a CCB, prepared in the same manner as a method blank, is analyzed and evaluated to one-half of the RL.

New ICAL Insertion into QNT-Editor for 300-9056

10.8 Copy the retention times from the mid-level standard to the Peak Table:

1. View the mid-level chromatogram from the new ICAL.
2. Left click on fluoride's retention time and drag down to sulfate.
3. Right click and copy them.
4. Left click on the QNT-Editor icon on the toolbar.
5. Select the Peak Table tab at the bottom of the page.
6. Left click on fluoride's retention time and drag down to sulfate.
7. Right click and paste them into the table.
8. Hit the save icon on the toolbar.

Copy the Amount Table concentrations from the last ICAL. (These values will need to be pasted into the Amount Table for the newly analyzed ICAL later on):

1. Select the Amount Table tab at the bottom of the page.
2. Left click on the top left (fluoride) concentration within the table and drag across the entire Amount Table to the bottom right (sulfate) concentration within the table.
3. Right click and copy them.

Insert the new ICAL WG#'s into the Peak Table so that new Amount Table Columns can be generated for them:

1. Select the Peak Table tab at the bottom of the page.
2. Double click on the Amount Column Header for STD#7 within the Peak Table. (A message will appear saying, "There are unassigned standards. ..") Hit OK.
3. A table titled "Edit Amount Columns" will be displayed showing the old WG#'s on the left side (these have amount columns already associated with them) and the new WG#'s on the right side (since these don't have amount columns associated with them yet, they are simply called standards). Hit the Autogenerate macro at the bottom of this table and select the option to "Generate a separate amount column for EACH standard …" that appears below.
4. Hit the Apply macro.
5. A statement saying, "Amount data associated with 7 columns will be lost." Then it will ask if you wish to continue. (Remember, the amount data associated with the 7 columns was copied above (at **Copy the Amount Table**). As long as nothing else has been copied since then, there is nothing to worry about. Hit OK.

Paste the Amount Table concentrations from the last ICAL (which were previously copied) into the Amount Table for the newly analyzed ICAL:

1. Select the Amount Table tab at the bottom of the page.

2. Left click on the top left (fluoride) concentration within the table and drag across the entire Amount Table to the bottom right (sulfate) concentration within the table.
3. Right click and paste them.
4. If the WG#'s are in a different order than they were when you copied the last ICAL's amount table values, you will have to make sure that each level (column) has the correct concentration values under it per anion.
5. Hit the save icon on the toolbar.

Verify that the old WG#'s have been removed from the Calibration Table before recalibrating:

1. Select the Calibration Tab at the bottom of the page.
2. Check the WG#'s in the name column within the Calibration Table.
3. If the WG#'s in the Calibration Table are the new ICAL WG#'s skip step 4.
4. If the calibration table shows the old WG#'s from the last ICAL, they will need to be removed from it. Remove these by right clicking on the old WG# at the top of the name column. Select "Delete Standards." Perform this operation on each old WG# until they are all gone. After removing all of them, right click on the empty name column field and select "Insert Standard." Select the new ICAL WG# that corresponds to the highest concentration level. Repeat this operation until all new ICAL WG#'s have been inserted into the Calibration Table in the order in which they were analyzed. Hit the save icon on the toolbar.

Recalibrate (New Calibration Table, with new ICAL WG#'s, new Amount Columns, and new Retention Times):

1. Select the General Tab at the bottom of the page.
2. Hit the Calibrate macro under Global calibration settings.
3. Hit the save icon on the toolbar.
4. Exit.

11.0 ANALYTICAL PROCEDURES

11.1 Samples are extracted as in Section 9.0 or bombed if necessary and filtered to 0.45 microns.

11.2 The instrument is calibrated as in Section 10.0 of this document.

11.3 At the beginning of each analysis the working eluent is injected to ensure that the mobile phase is free from contaminants and DI water is injected to verify the suppressor is in good working condition.

11.4 After calibration, batch QC and up to 10 field samples are analyzed, followed by a CCV and CCB (batch and instrument QC need not be included in the count of 10). If the second set of standards is acceptable, analysis may continue with another set of 10 samples, etc. All samples must be bracketed by passing check standards.

11.5 After sample analysis, the data system generates a quantitation report listing target analytes and concentrations found in the samples. A compound is considered identified if it falls within the calculated retention time window. Retention time windows are calculated by analyzing 3 injections of a standard over a 24 hour period and calculating the standard deviation of each target analyte's retention time and multiplying each standard deviation by 3. The +/- value is the retention time window for that analyte. The retention times of the daily CCV are used for adjusting the position of the window.

11.5.1 Retention time window studies are performed during method setup and after each column change.

11.6 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. It is also recommended that no single anion load exceed 200 ppm and that the total anion load should not exceed 400 ppm. These anion load limits are established in order to protect the analytical column and suppressor membrane from over saturation and dehydration respectively. Prior to IC analysis, samples may be screened for their chloride and sulfate concentrations in order to avoid anion overloading as much as possible. Initial sample dilutions are based on their chloride and sulfate screen results for these reasons. Further dilutions should be prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration.

Dilutions are prepared using 5 mL and 10 mL disposable pipets, 50 mL and 100 mL graduated cylinders, and 7 oz. plastic cups. All measuring and mixing materials shall be rinsed with DI water immediately prior to their use. The graduated cylinders are only used for measuring DI water volumes. Dilutions are prepared by using pipets to transfer aliquots of filtered samples into the appropriate volume of DI water in 7 oz. plastic cups.

Examples are presented below:

Dilution	Filtered Sample (mL)	DI Water (mL)	Total Volume (mL)
1/2	10	10	20
1/3	20	40	60
1/4	10	30	40
1/5	10	40	50
1/10	5	45	50
1/20	5	95	100

Dilutions greater than 1/20 are prepared by performing serial dilutions, e.g. for a 1/100 dilution, a 1/10 dilution would be made on a 1/10 dilution.

11.7 At the end of a run load the "shutdown" method which will turn off the suppressor, currents, and pump.

12.0 DETAILS OF CALCULATIONS

12.1 Linear regression is used for all sample calculations. Results are calculated using the following formula:

12.1.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response(area) = R
x = Concentration = C_i

And m and b are the slope and intercept from the regression equation
For a given response ratio we can solve for C_i:

12.1.2 $$C_i = [R - b]/m$$

Use equations 12.5 or 12.6 to calculate the sample concentration, C_x.

12.2 Recovery for a laboratory control sample:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.3 Calculating recovery for matrix spikes (MS/MSD):

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.4 Relative percent difference (RPD):

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.5 Solving for the concentration in water samples using linear regression.

For a given concentration, compute the unknown, C_x

$$C_x = (C_i)(V_f/V_i)(D)(1000)$$

where:
C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
D = dilution factor
C_x = concentration in the sample (ug/L)

12.6 Solving for the concentration in soil samples using linear regression:

$$C_x = (C_i)(V_f / W_i)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = initial sample weight (g)
D = dilution factor
C_x = concentration of the sample (ug/kg) (as received)

13.0 QUALITY CONTROL REQUIREMENTS

13.1 The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), LCS, MS, MSD and sample duplicates (SD). Additional procedures were defined in Section 10.0 for calibration and are included in the overall review process. The procedures, required frequency, acceptance criteria and corrective action measures are outlined in Table 1.

13.2 A batch is defined as a group of 20 samples or less which are prepared and analyzed together. With each batch of samples, a LCS, method blank, SD, MS and MSD are prepared (an LCSD may be used in lieu of an MS/MSD if there is insufficient sample volume). All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch. The LCS, MS and MSD are spiked so as to yield a concentration that fall near the mid-point of the curve.

(**NOTE**: Some projects require an LCSD. Consult the project QAPP for guidance).

13.3 Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Table 2 are used in the absence of a project QAPP, or program specified control limits. If any of the LCS analytes required for reporting are out, the analyst must stop the analysis, prepare an NCR, and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare an NCR to be reviewed by the department supervisor. Corrective action will consist of re-extraction and re-analysis of the affected

samples for, at a minimum, the COC for which a result was derived outside control limits, unless the client's representative and the Quality Assurance Officer (QAO) approve of another course of action.

13.4 The method blank and CCB cannot contain amounts of any target analytes, which are over the RL. If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL for the presents of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch. Some projects require that blanks are evaluated to a concentration of ½ the RL, consult the specific QAPP for guidance.

13.5 The MS and MSD results are included in the QC summary report and are used to monitor matrix accuracy and precision.

13.6 A sample duplicate is analyzed every twenty (20) samples. The RPD should be less than 20%.

13.7 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.7.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 1 does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 1.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)

- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.7.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.7.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data review uses the attached checklist (Figure 1). All data is reviewed by the analyst. The supervisor (or designated person) performs a second review before entry into the LIMS system.

14.2 Data Reporting

The reporting requirements depend upon the need of the client. Microbac offers four levels of data reporting which are described in some detail below.

14.2.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.2.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.2.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.2.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

14.2.5 Results for water samples are reported in ug/L to three significant figures.

14.2.6 Nitrate and nitrite are calculated and reported as nitrogen due to each respective anion. Phosphate is calculated and reported as phosphorus due to phosphate.

15.0 PREVENTIVE MAINTENANCE

15.1 The manufacturer's guidelines for operation of the ion chromatograph should be followed to prevent damaging the instrument.

15.2 All eluents should be filtered to 0.2 um if needed.

15.3 All samples should be filtered to 0.45 um to prevent damage to the flow system.

15.4 Sufficient eluent should be available for a complete run to prevent pumping air into the system.

15.5 An eluent sample waste container should be available and regularly emptied.

15.6 Guard columns may be changed as necessary to maintain the symmetry and resolution of anion peaks.

15.7 System pressure should be monitored to look for changes which could include leaks, plugs, and inappropriate eluents.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

16.4 The ion chromatograph produces aqueous waste with a relatively neutral pH. This waste is disposed in the municipal sewer system as per agreement with the city.

17.0 REFERENCES

17.1 EPA Test Methods for Evaluating Solid Waste, Laboratory Manual Physical/Chemical Method; February, 2007, Revision 1. Method 9056A

17.2 EPA 40 CFR Part 136, Method 375.4.

Uncontrolled Copy

17.3 Environmental Protection Agency: Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography. EPA Method 300.0. EPA-600/4-84-017. 1993.

17.4 Dionex, IC 16 Operation and Maintenance Manual, PN 30579, Dionex Corp., Sunnyvale, CA 94086.

17.5 Microbac SOP K5050, “Standard Operating Procedures Total Halides”

17.6 Microbac SOP K0005, “Standard Operating Procedures Extraction of Soils and Sludges”

17.7 Microbac SOP LQAP, “Laboratory Quality Assurance Plan”

17.8 Microbac SOP45, “Method Validation Procedures”

17.9 Microbac SOP GP-CAPA, “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.10 Microbac SOP GP-RCA, “Root Cause Analysis”

17.11 Microbac SOP33, “Laboratory Waste Management”

Table 1
Quality Control Criteria
Method 9056/300

Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	When continuing calibration is out of control or after major maintenance	r ≥ 0.995	Correct problem, repeat ICAL.
Initial Calibration Verification (ICV)	After initial calibration	≤ 10% drift	Reanalyze ICV; upon second failure, repeat initial calibration.
Continuing Calibration Verification (CCV)	At beginning of daily analytical sequence and after every 10 field samples.	≤ 10% drift	Reanalyze CCV; upon second failure perform maintenance. Two consecutive CCVs must pass following maintenance.
Continuing Calibration Blank (CCB)	After every CCV	Target analytes ≤1/2 RL.	Reanalyze samples not bracketed by passing CCB unless results > RL.
Method Blank	One per matrix/batch.	Target analytes ≤1/2 RL.	Reprep and reanalyze blank and all associated samples
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS)	One per matrix/batch.	Recovery within 90-110%	Reprep and reanalyze LCS and all associated samples
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch if specified by client.	Recovery within 90-110%. RPD ≤ 20%.	Qualify data and/or address in the report narrative
Sample Duplicate (SD)	Extracted with every batch of 20 samples.	RPD ≤ 20%	Qualify data and/or address in the report narrative
Limits of Quantitation (LOQ)	Quarterly and after major instrument maintenance	50-150%	Reanalyze LOQ; upon second failure, repeat initial calibration.

Uncontrolled Copy

Table 2
Quality Assurance Objectives
Method 9056/300

PARAMETER	ACCURACY (% REC)*	PRECISION (%RPD)*	WATER VERIFIED MDL (mg/L)	WATER REPORTING LIMIT (mg/L)	SOLID VERIFIED MDL (mg/kg)	SOLID REPORTING LIMIT (mg/kg)
Fluoride (F)	90-110	0-20	0.1	0.2	1	2
Chloride (Cl)	90-110	0-20	0.1	0.2	1	2
Nitrite (NO_2) (as N)	90-110	0-20	0.1	0.4	1	4
Bromide (Br)	90-110	0-20	0.1	0.2	1	2
Nitrate (NO_3) (as N)	90-110	0-20	0.1	0.6	1	6
Sulfate (SO_4)	90-110	0-20	0.5	1.0	5	10

* Values are statistically derived from laboratory control samples and are updated annually.
Actual control limits may vary.

Uncontrolled Copy

Figure 1
IC Checklist

Checklist ID: 34006

Microbac Laboratories Inc.

Data Checklist

Date: 20-NOV-2008
Analyst: JWR
Analyst: NA
Method: 300/9056
Instrument: IC1
Curve Workgroup: NA
Runlog ID: 25449
Analytical Workgroups: 11-0537

ANALYTICAL	
System Performance Check	X
DFTPP (MS)	NA
Endrin/DDT breakdown (8081/MS)	NA
Pentachlorophenol/benzidine tailing (MS)	NA
Eluent check (IC)/system pressure (HPLC)	X
Window standard (FID)	NA
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	NA
Continuing calibration blank (CCB) (IC)	X
Special standards	NA
Blanks	X
TCL hits	X
Surrogate recoveries	NA
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	NA
MS/MSD/Sample duplicates	X
Recoveries	X
%RPD	X
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	NA
Surrogate recoveries	NA
Internal standard areas (MS)	NA
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	X
Reruns	X
Manual integrations	X
Project/client specific requirements	X
REPORTING	
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	JWR
SUPERVISORY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer:
26-NOV-2008
John Richards

Secondary Reviewer:
01-DEC-2008
Michael Cohen

CHECKLIST1 - Modified 03/05/2008
Generated: DEC-01-2008 12:17:42

Microbac

Figure 2
IC Run Log

Run Log ID: 25449

Microbac Laboratories Inc.
Instrument Run Log

Instrument: IC1 Dataset: 112008 9056 IC1.SEQ
Analyst1: JWR Analyst2: NA
Method: 9056/300 SOP: IC1 Rev: 8

Maintenance Log ID: 26753

Column 1 ID: AS14A-4MM Column 2 ID: NA
Workgroups: WG288668
Internal STD: NA Surrogate STD: NA Calibration STD NA

Comments: Samples 0537-01-04,06 were analyzed at dilutions only, due to their high screen results for CL, which were greater than 200 ppm.

Seq.	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	I11120081352.01	ELUENT	1	1		11/20/08 13:52
2	I11120081409.02	DI WATER	1	1		11/20/08 14:09
3	I11120081427.03	WG288672-01 ANION CCV	1	1		11/20/08 14:27
4	I11120081444.04	WG288672-02 ANION CCB	1	1		11/20/08 14:44
5	I11120081501.05	WG288668-01 ANION BLANK	1	1		11/20/08 15:01
6	I11120081519.06	WG288668-02 ANION LCS	1	1		11/20/08 15:19
7	I11120081536.07	L08110537-01 RS (F,CL,NO2,NO3,SO4) 1/20	1	20		11/20/08 15:36
8	I11120081554.08	WG288668-04 DUP 0537-01 1/20	1	20		11/20/08 15:54
9	I11120081611.09	L08110537-02 MS (F,CL,NO2,NO3,SO4) 1/20	1	20		11/20/08 16:11
10	I11120081629.10	L08110537-03 MSD (F,CL,NO2,NO3,SO4) 1/20	1	20		11/20/08 16:29
11	I11120081646.11	L08110537-04 DS (F,CL,NO2,NO3,SO4) 1/20	1	20		11/20/08 16:46
12	I11120081703.12	L08110537-06 (F,CL,NO2,NO3,SO4) 1/25	1	25		11/20/08 17:03
13	I11120081721.13	DI WATER	1	1		11/20/08 17:21
14	I11120081738.14	WG288672-03 ANION CCV	1	1		11/20/08 17:38
15	I11120081756.15	WG288672-04 ANION CCB	1	1		11/20/08 17:56
16	I11120081813.16	L08110537-01 RS (RR CL 1/100)	1	100		11/20/08 18:13
17	I11120081830.17	L08110537-04 DS (RR CL 1/100)	1	100		11/20/08 18:30
18	I11120081848.18	L08110537-06 RR CL 1/200	1	200		11/20/08 18:48
19	I11120081905.19	WG288672-05 ANION CCV	1	1		11/20/08 19:05
20	I11120081923.20	WG288672-06 ANION CCB	1	1		11/20/08 19:23
21	I11120081940.21	END	1	1		11/20/08 19:40

Comments

Seq.	Rerun	Dil.	Reason	Analytes

Page: 1 Approved: 01-DEC-08

Microbac®

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
HARDNESS, TOTAL (TITRIMETRIC, EDTA)
EPA 130.2, SM2340B,C-1997
(2011 EDITORIAL REVISION)

Issue/Implementation Date: 20 March 2014

Last Review Date: 20 March 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Deanna I. Hesson, Conventionals Supervisor — Date: 3/17/14

Wade T. DeLong, Quality Assurance Officer — Date: 3-17-14

Leslie S. Bucina, Laboratory Manager — Date: 3/17/14

Uncontrolled Copy

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	4
5.0	Interferences and Corrective Action	5
6.0	Equipment and Supplies	6
7.0	Standards and Reagents	6
8.0	Calibration Procedures	8
9.0	Sample Preparation	9
10.0	Diagram or Table to Outline Procedures	9
11.0	Analytical Procedures	10
12.0	Details of Calculations	10
13.0	Quality Control Requirements	12
14.0	Data Review and Reporting Requirements	14
15.0	Preventive Maintenance	14
16.0	Waste Management and Pollution Control	14
17.0	References	15
	Tables	17
	Figures	18

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface, and saline waters. Hardness is defined as the hardness that can be determined using the EDTA titration method, and expressed as mg/L $CaCO_3$. Calcium and magnesium ions form a chelated soluble complex with the EDTA molecule (Ethylenediamine tetraacetic acid). Calmagite indicator (solid or liquid) is added to a sample (pH 10) containing the metal cations. The color is red when the cations are present, and after the sample is titrated with EDTA and the cations are complexed, the color is blue.

1.2 For waste waters and polluted waters, the sample must be digested first, and then titrated.

1.3 This method references EPA Method 130.2, and SM 2340B,C-1997 (2011 Editorial Revision).

1.4 Hardness may also be calculated by using the concentration of Ca and Mg. See Section 12.0.

1.5 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

COA	Certificate of Analysis
DI water	Deionized Water
DUP	Duplicate
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LOD	Limits of Detection
LOQ	Limits of Quantitation
LQAP	Laboratory Quality Assurance Program
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
QA	Quality Assurance
QC	Quality Control
RGT	Reagent
RL	Reporting Limit

RPD	Relative Percent Difference
SDS	Safety Data Sheet
SOP	Standard Operating Procedure
STD	Standard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Standard Laboratory Safety Procedures must be followed when working with unknown samples.

2.2 Lab coats and safety glasses with sideshields are required. Gloves are recommended since ammonium hydroxide, nitric acid and unknown samples are being used in the procedure.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 The sample must be collected in plastic or glass containers and preserved with nitric acid to pH < 2. The sample must be kept at ≤ 6° C for a period not exceeding 6 months from the time of collection before analysis is performed. After analysis, samples are stored in a designated area of the archive room. (See Section 16.0)

3.2 This procedure requires a minimum of 100 mL. If less than the recommended volume is available, the analysis will be performed on a dilution and the detection limit elevated proportionately.

4.0 METHOD PERFORMANCE

4.1 This method uses an RL or LOQ of 5 mg/L. RLs are nominal laboratory values, but project RL's may vary. The LOQ are nominal limits and were established per Microbac SOP 45.

4.2 The laboratory performed an initial assessment of the method detection limit using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\Data\Common\MDL. The laboratory verified MDL or LOD is 2.5 mg/L. The MDL/LOD is verified annually (using 7 replicates) or when necessary and verified quarterly (using 1 replicate). Verification consists of

analyzing a fortified blank (MDL check standard) spike at a concentration one to three times the concentration of the MDL/LOD. The MDL/LOD check standard is used to verify that the laboratory MDL is routinely achievable over the course of time. Additional details on MDL studies may be found in Microbac SOP 45.

4.3 The current limits are 90 -110% for the LCS and MS/MSD and the RPD is 10%. Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from laboratory control samples to assess the precision and accuracy and to annually evaluate the associated control limits.

4.4 The linear range of this method is 5 - 150 mg/L CaCO3. The range may be extended on the upper end by using less sample or by diluting.

4.5 Each analyst is required to perform an initial demonstration of precision and accuracy. Annually, each analyst is re-certified by performing four LCS analyses. See Microbac SOP 45 which describes the procedure in detail.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Excessive heavy metals can cause interference by causing an indistinct end point. Complexing the metals with certain inhibitors such as sodium cyanide can prevent this interference. Most drinking and surface waters do not contain levels high enough to interfere. See EPA Method 130.2 for levels.

5.2 Magnesium ion must be present to yield a satisfactory endpoint. A small volume of EDTA salt (MgEDTA) is added to the buffer to allow for this fact.

5.3 The sharpness of the endpoint is proportional to the increasing pH. Excessive pH however may cause the precipitation of either magnesium hydroxide, $Mg(OH)_2$, or calcium carbonate, $CaCO_3$. therefore, using a pH of 10.0 ± 0.1 will achieve the desired results.

5.4 A slower endpoint is noticeable as the temperature decreases. Performing the titration at room temperature will give a distinct endpoint.

Uncontrolled Copy

6.0 EQUIPMENT AND SUPPLIES

6.1 Standard titrating equipment. (Magnetic stirrer, burette, stir bar)

6.2 pH meter (Orion 4 Star meter, or equivalent.)

6.3 All glassware is washed with soap and water as described in Microbac SOP K0001.

7.0 STANDARDS AND REAGENTS

NOTE: All standards and reagents are prepared using Class A volumetric flasks and pipets unless otherwise stated differently.

NOTE: All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 $CaCO_3$ standard (purchased as $CaCl_2$) Discard after stated expiration date on the container. This standard must be kept at 4° C.

7.2 Hardness LCS (1000 mg/L): Weigh 1.000 g $CaCO_3$ (pre-dried) into a 500 mL Erlenmeyer flask. Add drop wise 1:1 HCl until $CaCO_3$ is dissolved. Add 200 mL DI Water and boil to expel CO_2. Cool, and add a few drops of methyl red indicator. Add 3 N NH_3OH and/or 1:1 HCl to adjust to the intermediate orange color. Transfer stock to a 1 L flask and dilute to the mark. Discard after 6 months.

7.3 Ammonium Hydroxide: 1 N. Dilute 35 mL of concentrated NH_4OH to 500 mL with DI water. Discard after 6 months.

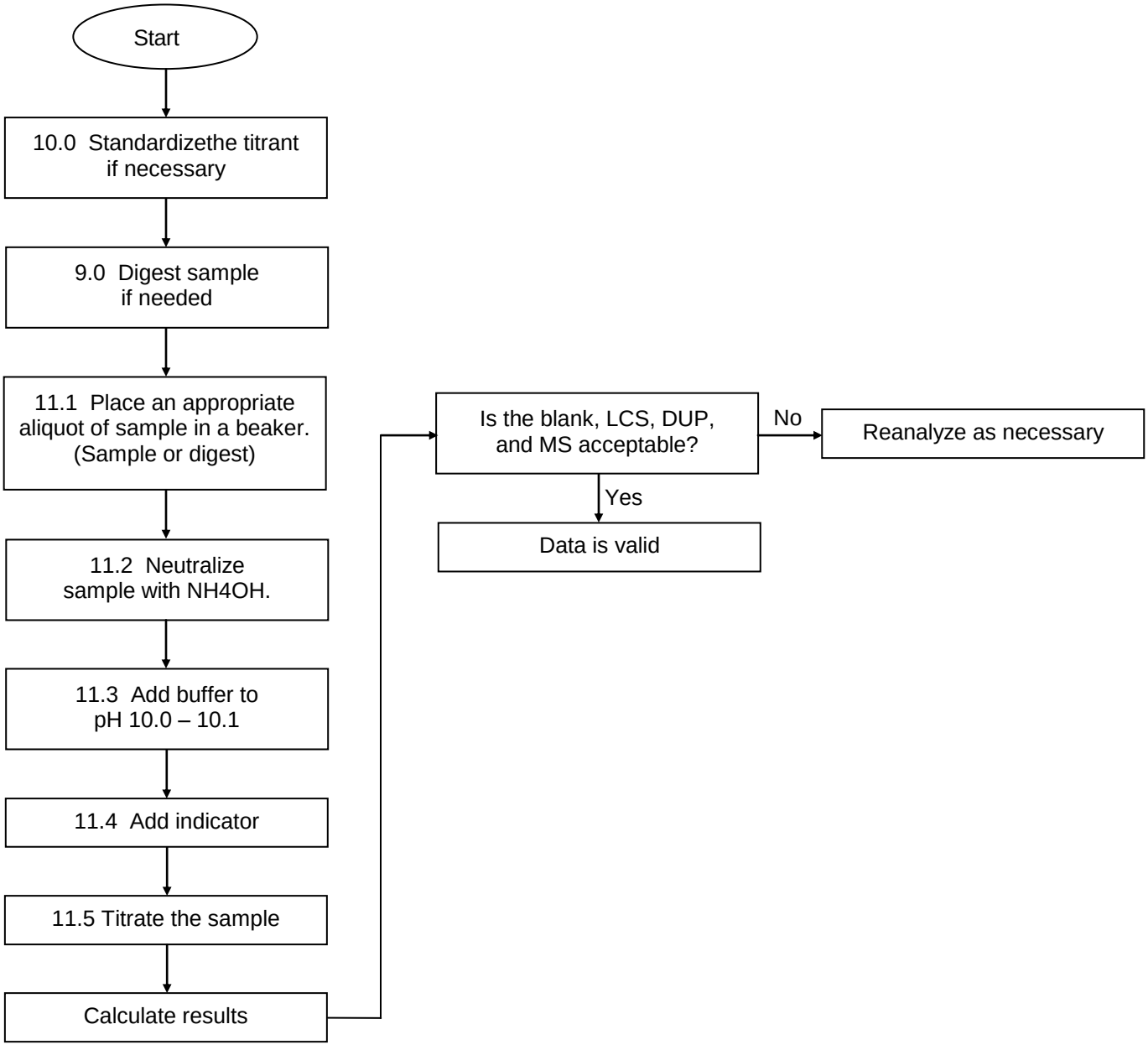
7.4 Hardness Buffer Solution (purchased): This buffer contains ammonium chloride, concentrated ammonium hydroxide, and the magnesium salt of EDTA. Discard at the expiration date on the container. (Discard if it fails to bring the pH between 10.0 -10.1)

7.5 Calmagite indicator: May be purchased as either a solid or liquid solution. Use before expiration date on the container.

7.6 EDTA titrant: 0.02 N (0.01 M) Purchased. Standardize monthly. (North Carolina requires daily stardardization) (see Section 10.0)

7.7 For reagent disposal see Section 16.0.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



Uncontrolled Copy

9.0 SAMPLE PREPARATION

9.1 Pretreatment of waste waters and polluted waters is needed before titrating.

9.2 Digest the sample as in Microbac SOP ME401A Section 11.0.

10.0 CALIBRATION PROCEDURES

10.1 The EDTA titrant is purchased pre-standardized by the manufacturer. The titrant needs re-standardized upon opening and once per month thereafter.

10.2 The following is the standardization procedure.

10.2.1 Using a volumetric pipet, deliver 20 mL of the standard $CaCO_3$ (see Section 7.1) to a beaker.

10.2.2 Add 30 mL DI water, 2 mL of buffer and 0.5 mL of indicator.

10.2.3 Titrate with EDTA (see Section 7.6) to the distinct blue endpoint. This should be done within 5 minutes after adding the buffer solution.

10.2.4 Record the volume of titrant in the Titrant Standardization Logbook.

10.2.5 Repeat steps 10.2.1 - 10.2.4, with another portion of standard.

10.2.6 Run a blank on 30 mL DI water.

10.2.7 Calculate the normality using the following equation:

*(NS*VS)/VT = Normality of the EDTA titrant*

where:

NS = Normality of the standard (see Section 7.1)
VS = Volume of the standard used (see Section 7.1)
VT = Volume of titrant used (see Section 7.6)

Uncontrolled Copy

11.0 ANALYTICAL PROCEDURES

11.1 Choose an aliquot of sample that will require less than 15 mL of titrant. Use no more than 100 mL of sample. Dilute to 50 mL if less than 50 mL of a sample is used. (The original sample or the digest (see Section 9.0) may be used)

11.1.1 Dilutions are made using disposable 1 mL, 5 mL or 10 mL pipets, and graduated cylinders.

11.1.2 If the DI water to be used is greater than 15 mL, the water is measured with a graduated cylinder, otherwise a pipet is used.

11.1.3 If less than 25 mL of the sample is needed, a pipet is used otherwise a graduated cylinder is used.

11.1.4 See Table 1 for examples.

11.2 Neutralize the sample with ammonium hydroxide (see Section 7.3). **Check the pH with a pH meter.**

11.3 Add 1 mL of buffer solution (see section 7.4), for each 25 mL of sample used. If the pH of the sample is not 10.0-10.1, new buffer needs to be used.

11.4 The titration must be completed within 5 minutes of the addition of the buffer.

11.5 Add 2 shots of solid calmagite indicator (see Section 7.5), or 0.5 mL of liquid indicator.

11.6 Titrate the sample with EDTA titrant (see section 7.6) until the last reddish tint disappears. Normally the sample is blue at the endpoint.

11.7 The following should be included on the benchsheet: Parameter, analyst, date, time, reference numbers for standards and LCS, sample numbers, volume, dilution, normality of titrant, volume of titrant and any other information necessary for the calculation of data and traceability of standards.

12.0 DETAILS OF CALCULATIONS

12.1 The following formula is used in the calculation of hardness:

$$\left(\left[ml\ titrated * N * 50{,}000\right] * dilution\right) / volume = Hardness\ as\ m/L\ CaCO_3$$

where:

mL titrated = volume of titrant used
N = normality of EDTA (0.02): (see Section 10.2.7)
50,000 = equivalent weight
volume = total volume of sample used
dilution = dilution of sample (example: a 1:5 dilution = 0.5)

12.2 The following equation is used to calculate the LCS recovery:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
%R = percent recovery

12.3 The following equation is used to calculate the duplicate RPD:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.4 The following equation is used to calculate the spike recovery:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
%R = percent recovery

12.5 To calculate hardness using the concentration of calcium (Ca) and magnesium (Mg), use the following equation from SM2340B:

$$2.497\ (Ca) + 4118\ (Mg) = mg/L\ hardness$$

where:

(Ca) = mg/L calcium
(Mg) = mg/L magnesium

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Blank: Analyze 100 mL of DI water exactly like the samples. Results will be evaluated down to one-half of the RL. The analyst will prepare an NCR to determine the appropriate correction, if the blank exceeds this level.

13.2 LCS/LCSD (100 mg/L): Dilute 10 mL of Hardness standard (see section 7.2) to 100 mL, and analyze exactly like the samples. The recovery must be within the control limits (see Section 4.0), otherwise the analyst will prepare an NCR to determine the appropriate correction.

13.3 Spike: Add 5 mL of LCS standard (see Section 7.2) to 50 mL of sample for a spike of 100 mg/L, or add 5 mL to 100 mL of sample for a spike of 50 mg/L. The hardness concentration in the sample will dictate which spike to use. The recovery must be within the statistically determined limits (see Section 4.0) or a case narrative will be written.

13.4 Duplicate: Rerun any sample at the same volume and/or dilution. The RPD should be less than the statistically determined limits (see Section 4.0) or the run will be examined and reanalyzed as necessary.

13.5 A batch (workgroup) is defined as a blank, LCS, duplicate, spike and up to 20 samples. Titration only and digested samples need to be in separate workgroups.

13.6 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective

Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.6.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Section 13.1-13.4, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Section 13.1-13.4.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.6.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.6.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.

- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review uses the attached checklist. All data is calculated and reviewed by the analyst. The supervisor (or designated person) performs a second review (approval) before final entry into the LIMS system.

14.2 Hardness is reported in mg/L CaCO3 for liquids using a maximum of three significant digits. The analyst, date and time are reported. QA/QC data is entered onto special forms and reported to the client upon request.

15.0 PREVENTIVE MAINTENANCE

15.1 See Microbac SOP K1501 for maintenance and troubleshooting the pH meter.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Laboratory policies and procedures for management of hazardous w aste are found in Microbac SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOP's contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then

combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.3 All reagents are disposed of down the sink after neutralization (near or in a hood) and flushed with a large amount of water.

16.4 Upon completion of the analysis, the samples are stored in a designated area of the archive storage room to await proper disposal by the waste management team.

16.5 For further information on waste management, consult Microbac SOP 33 (Laboratory Waste Management).

17.0 REFERENCES

17.1 "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020, Method 130.2 (Titrimetric, EDTA), 1983.

17.2 Standard Methods. Method 2340 C-1997 (2011 Editorial Revision)

17.3 Standard Methods. Method 2340 B-1997 (2011 Editorial Revision)

17.4 U.S. Environmental Protection Agency, *Test Method for Evaluating Solid Waste,* SW-846, November, 1986, Third Edition, Method 3005A

17.5 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.6 Microbac SOP 45 “Method Validation Procedures”

17.7 Microbac SOP 33 “Laboratory Waste Management”

17.8 40 CFR Part 136

17.9 Microbac SOP GP-CAPA, “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.10 Microbac SOP GP-RCA, “Root Cause Analysis”

17.11 Microbac SOP K0001 “Glassware Washing”

17.12 Microbac SOP ME401 “Digestion Procedure”

Uncontrolled Copy

Table 1
Examples of Common Dilutions

Dilution	Total Volume Needed	Sample (mL)	Deionized water (mL)
1/2	100	50	50
1/4	100	25	75
1/10	100	10	90
1/50	100	2	98
1/100	100	1	99

*If less than 100 mL is needed for analyses, use a smaller portion of the diluted sample.
*Dilutions over 1/100 will need to be made in a series of dilutions.
Ex: 1/5000 would be made as a 1/50 on a 1/100.

Figure 1

Checklist ID: 27469

Microbac Laboratories Inc.

Data Checklist

Date: 14-APR-2008
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Calibration\Linearity	
Second Source Check	
ICV\CCV (std)	
ICB\CCB	
Blank	
LCS\LCS Dup	
MS\MSD	
Duplicate	
Upload Results	
Client Forms	
QC Violation Sheet	
Case Narratives	
Signed Raw Data	
STD\LCS on benchsheet	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Primary Reviewer	
Secondary Reviewer	DIH
Comments	

Primary Reviewer:
14-APR-2008

Secondary Reviewer:
14-APR-2008

CHECKLIST1 - Modified 03/05/2008
Generated: APR-14-2008 12:26:45

Microbac

Uncontrolled Copy

STANDARD OPERATING PROCEDURE
VOLATILE SOLIDS
(TOTAL / DISSOLVED / SUSPENDED)
GRAVIMETRIC, IGNITION 550° C
EPA METHOD 160.4/SM 2540E, G-1997
(2011 EDITORIAL REVISION)

Issue/Implementation Date: 15 May 2011

Last Review Date: 15 April 2014

Microbac Laboratories, Inc
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Deanna I. Hesson, Conventionals Supervisor — Date: 4/29/14

Wade T. DeLong, Quality Assurance Officer — Date: 4-30-14

Leslie S. Bucina, Laboratory Manager — Date: 5/1/14

Document Control # 179

Issued to: Document Master File

Uncontrolled Copy

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	4
5.0	Interferences and Corrective Action	5
6.0	Equipment and Supplies	5
7.0	Diagram or Table to Outline Procedures	6
8.0	Calibration Procedures	7
9.0	Analytical Procedures	7
10.0	Details of Calculations	7
11.0	Quality Control Requirements	8
12.0	Data Review and Reporting Requirements	10
13.0	Preventive Maintenance	10
14.0	Waste Management and Pollution Control	10
15.0	References	11
	Figures	12

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of the amount of solids combustible at 550° C. This test can give a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments. Total Volatile Solids is defined as the total (dissolved and suspended) solids that are lost upon combustion at 550° C. Total Volatile Dissolved Solids is defined as the dissolved solids that are lost upon combustion at 550° C. Total Volatile Suspended Solids is defined as the suspended solids that are lost upon combustion at 550° C.

1.2 Total, dissolved, or suspended solids are determined by the appropriate method. The residue left in the crucible, or on the filter paper is then placed in a 550° C muffle furnace for 15 minutes. The amount of solids lost upon combustion is considered to be volatile solids.

1.3 This method references EPA Method 160.4 and SM 2540E, G-1997 (2011 Editorial Revision).

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

COA	Certificate of analysis
DI water	Deionized water
DUP	Duplicate
LIMS	Laboratory Information Management System
LOQ	Limits of Quantitation
LQAP	Laboratory Quality Assurance Program
MB	Method blank
MSDS	Material Safety Data Sheet
NCR	Nonconformance report
QA	Quality assurance
QC	Quality control
RGT	Reagent
RL	Reporting limit
RPD	relative Percent Difference
SOP	Standard Operating Procedure
STD	Standard
TS	Total Solids
TDS	Total Dissolved Solids
TSS	Total Suspended Solids

Uncontrolled Copy

TVDS	Total Volatile Dissolved Solids
TVS	Total Volatile Solids
TVSS	Total Volatile Suspended Solids

For a more comprehensive list of common terms and definitions, consult Appendix A in SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Standard Laboratory safety procedures should be followed when working with unknown samples.

2.2 Lab coats and safety glasses with side shields are required. Gloves are recommended since unknown samples are being used in this procedure.

2.3 The crucibles, ovens, and furnace used in this procedure are hot and extreme care should be taken.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 The sample may be collected in glass or plastic containers. The sample must be kept at ≤ 6° C for a period not exceeding 7 days from the time of collection before analysis is performed. After analysis, samples are stored in a designated area of the archive room. See Section 16.0 for disposal protocol.

3.2 This procedure requires 200 mL for TVSS, 50 mL each for TVDS and TVS and a minimum of 25.0 g for a solid (TVS-S).

3.3 If less than the recommended volume is available the analyses will be performed on a lesser amount and the detection limit elevated proportionately.

4.0 METHOD PERFORMANCE

4.1 This method uses RLs or LOQ as listed below. RLs are nominal laboratory values, but project RL's may vary. The LOQ are nominal limits and were established per Microbac SOP 45.

4.1.1 TVS: 10 mg/L or 1% (for solids)

4.1.2 TVDS: 10 mg/L

Uncontrolled Copy

4.1.3 TVSS: 5 mg/L

4.2 The linear range of this method depends on the parameter being analyzed. The range may be extended on the upper end by using less sample.

4.3 The duplicate results must be within 5% of the average results. This is close to an RPD of 10%.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion, can result in low recovery.

5.2 The main cause of high or low recovery is failure to obtain a representative sample.

6.0 EQUIPMENT AND SUPPLIES

6.1 Apparatus from the appropriate solids method (TS, TDS, TSS): drying crucibles, filter paper, vacuum, ovens (103° C, 180° C). See Microbac Methods K1601, K1602, and K1603.

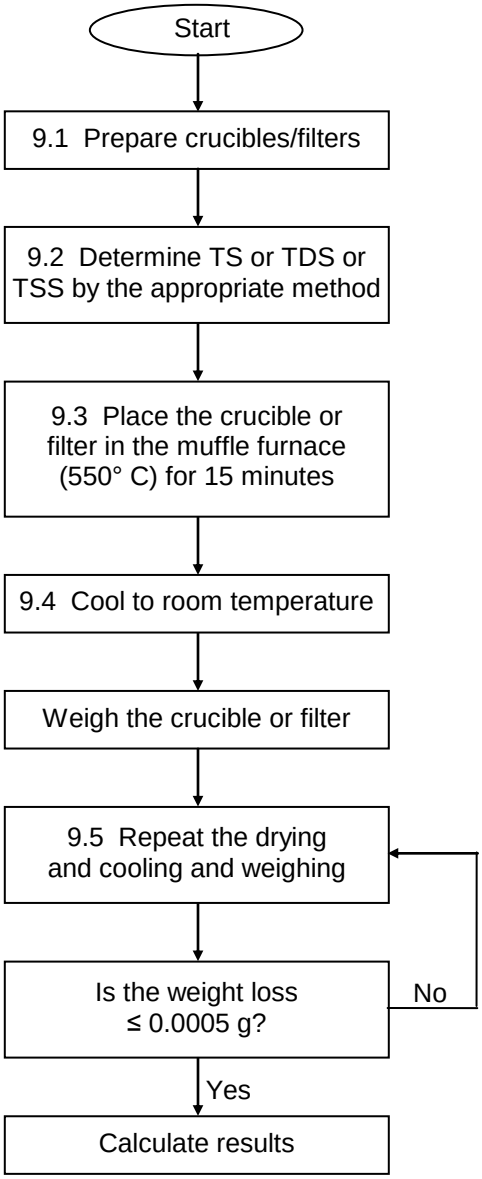
6.2 Muffle furnace: 550 ± 50° C (Thermolyne 62700 or equivalent).

6.3 Analytical Balance, accurate to 0.0001 g

6.4 All glassware is washed with soap and water as described in Microbac SOP K0001.

6.5 Desicctor with active desiccant

7.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



Uncontrolled Copy

8.0 CALIBRATION PROCEDURES

8.1 Ovens must have thermometers checked quarterly for digital and yearly for others.

8.2 Follow Microbac Method K0002 for this procedure.

9.0 ANALYTICAL PROCEDURES

9.1 Drying crucibles and filters must be washed and muffled at 550° C for 1 hour before using.

9.2 Determine TS, TDS, or TSS on the sample, retaining the residue for volatile solids analysis. See Microbac Methods K1601, K1602, and K1603 or K0003.

9.3 Place the crucibles or filter paper containing the residue in a 550° C muffle furnace for 15 minutes, and then remove from the furnace.

9.4 Allow the crucible or filter to cool partially in air, then transfer to a desiccator to cool completely, then weigh quickly.

9.5 Repeat the drying (Step 9.3) and cooling (Step 9.4) procedures until weight loss is less than or equal to 0.0005 g.

9.6 The following should be included on the benchsheet: Parameter, analyst, date and time (on and off), time, sample numbers, volume, weights, and any other information necessary for the calculation of data and traceability of standards.

10.0 DETAILS OF CALCULATIONS

10.1 The following formula is used in the determination of volatile solids:

$$\frac{[(WT2 - WT3)1{,}000{,}000]}{volume} = mg/L\ TVS, TVDS\ or\ TVSS$$

$$\frac{[(WT2 - WT3)100]}{WT2 - WT1} = \%\ TVS, TVDS\ or\ TVSS$$

where:

WT1 = weight (g) of empty crucible or filter

Uncontrolled Copy

WT2 = weight (g) of crucible or filter and dried residue, from TS, TDS, or TSS analysis
WT3 = weight (g) of crucible or filter and ignited residue volume is in mL
1,000,000 = factor to get units as mg/L
100 = factor to get units as % (Percent weight)

10.2 Duplicate Criteria:

10.2.1 Average method

$$(C_1 + C_2)/2 = AVE$$

Sample and duplicate should be within +/- $(5\% \, of \, AVE)$

10.2.2 The Relative percent difference (RPD) is calculated as follows:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)} \right] 200$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

11.0 QUALITY CONTROL REQUIREMENTS

11.1 Method Blank: DI water is analyzed exactly like the samples: The result must be < RL or analyst will prepare an NCR to determine the appropriate correction.

11.2 Duplicate samples will be analyzed at the frequency of one per batch of 20 samples or less. Results are evaluated as described in Section 4.0.

11.3 A batch (workgroup) is defined as a duplicate and up to 20 samples.

11.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective

Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

11.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Steps 11.1-11.2, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Step 11.1.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

11.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RAC, corrections, corrective action(s) and evidence of effectiveness.

11.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.

- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

12.0 DATA REVIEW AND REPORTING REQUIREMENTS

12.1 Data review uses the attached checklist. All data is calculated and reviewed by the analyst. The supervisor (or designated person) performs a second review before final entry into the LIMS system.

12.2 TVS, TVDS, and TVSS are reported in mg/L using a maximum of three significant digits TVS (for solids) is reported as percent weight using a maximum of three significant digits. The analyst, date and time are reported.

13.0 PREVENTIVE MAINTENANCE

13.1 Ovens must remain free of dirt and liquids to prevent contamination of TDS, TS, or TSS.

13.2 Crucibles must be cleaned immediately to help with removal of solids.

14.0 WASTE MANAGEMENT AND POLLUTION CONTROL

14.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA, and Ohio VAP rules allow the suspension of our certification for failure to comply with these laws.

14.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are

responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

14.3 All reagents (unless noted below) are disposed of down the sink (near or in a hood) and flushed with a large amount of water.

14.4 For further information on waste management, consult Microbac SOP 33 (Laboratory Waste Management).

15.0 REFERENCES

15.1 "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020, Method 160.4 (Gravimetric, Ignition at 550° C), 1983.

15.2 Standard Methods 2540E, G-1997 (2011 Editorial Revision)

15.3 40 CFR Part 136

15.4 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

15.5 Microbac SOP 45 “Method Validation Procedures”

15.6 Microbac SOP 33 “Laboratory Waste Management”

15.7 Microbac SOP GP-CAPA “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

15.8 Microbac SOP GP-RCA “Root Cause Analysis”

15.9 Microbac SOP K0001 “Glassware Washing”

Uncontrolled Copy

Figure 1

Checklist ID: 27469

Microbac Laboratories Inc.

Data Checklist

Date: 14-APR-2008
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Calibration/Linearity	
Second Source Check	
ICV/CCV (std)	
ICB/CCB	
Blank	
LCS/LCS Dup	
MS/MSD	
Duplicate	
Upload Results	
Client Forms	
QC Violation Sheet	
Case Narratives	
Signed Raw Data	
STD/LCS on benchsheet	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Primary Reviewer	
Secondary Reviewer	DIH
Comments	

Primary Reviewer:
14-APR-2008

Secondary Reviewer:
14-APR-2008

Dianna Hixson

CHECKLIST1 - Modified 03/05/2008
Generated: APR-14-2008 12:26:45

Microbac

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
PREPARATION OF DISSOLVED METALS IN WATERS FOR DIRECT ANALYSIS BY INDUCTIVELY COUPLED PLASMA SPECTROSCOPY (EPA 200.7)

Issue / Implementation Date: 22 August 2013

Last Review Date: 15 August 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Robert E. Kyer, Digest Group Leader — Date 8/5/14

Maren M. Beery, Metal Supervisor — Date 08/12/2014

Wade T. DeLong, Quality Assurance Officer — Date 8-13-14

Leslie S. Bucina, Laboratory Manager — Date 8/14/14

Document Control # 1330

Issued to: Document Master File

Uncontrolled Copy

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	5
5.0	Interferences and Corrective Action	5
6.0	Equipment and Supplies	5
7.0	Standards and Reagents	5
8.0	Diagram or Table to Outline Procedures	7
9.0	Sample Preparation	8
10.0	Calibration Procedures	8
11.0	Analytical Procedures	8
12.0	Details of Calculations	9
13.0	Quality Control Requirements	9
14.0	Data Review and Reporting Requirements	11
15.0	Preventive Maintenance	11
16.0	Waste Management and Pollution Control	11
17.0	References	12
	Tables	13
	Figures	14

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This method is a procedure used to prepare filtered wastewater samples that contain no suspended solids for analysis by Inductively Coupled Plasma Spectroscopy (ICP). Samples prepared by this method may be analyzed by ICP for the metals listed in Table 1.1. Samples prepared by this method may be analyzed by ICP for additional metals if the QC criteria of the method can be achieved. This method complies with EPA Method 200.7.

1.2 Dissolved metals: The sample is filtered through a 0.45 micrometer filter at the time of collection. The liquid phase is then acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested as long as acid concentrations have been adjusted to be the same as the instrument standards.

1.3 The acid concentrations and volumes in Section 11.2 are consistent with the requirements of EPA Method 200.7.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

DI water	Deionized water
HCl	Hydrochloric acid
HNO_3	nitric acid
ICP	Inductively Coupled Plasma
LCS	Laboratory control sample
LCSD	Laboratory control sample Duplicate
LIMS	Laboratory Information Management System
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
QA	Quality Assurance
QC	Quality Control
RGT	Reagent
SDS	Safety Data Sheets
SOP	Standard Operating Procedure
STD	Standard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

Uncontrolled Copy

2.0 SAFETY PRECAUTIONS

2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by the means available (i.e.: hood, gloves, faceshield, etc.). The laboratory maintains SDS sheets for handling and exposure information.

2.2 Chipped and broken glassware must be disposed of in the broken glass waste.

2.3 Care must be taken to avoid burns and electrical shock from hotblock operation.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 All sample containers must be pre-cleaned. Glass or plastic are both acceptable.

3.3 Aqueous samples need to be preserved to pH less than 2 with HNO_3. The pH is checked by the sample receiving department prior to login. For determinations of dissolved metals, the sample must be filtered before preservation on site. Samples that are received unpreserved are preserved by the digestion laboratory personnel on site and must sit for 24 hours prior to digestion. The pH determined upon receipt, after adjustment and after 24 hours is recorded in the Laboratory Preservation Log book. If the pH after resting 24 hours is found to be noncompliant, additional acid is added and the sample must sit for another 24 hours prior to a further pH check

3.4

Measurement	Digestion Volume/ Weight Requirement	Collection Volume/ Weight Requirement	Preservative/ Holding Time*
Dissolved	100 mL	600 mL	Filter on site HNO_3 to pH <2; 6 months

Holding time is the storage time allowed between sample collection and analysis when properly preserved and stored.

Uncontrolled Copy

4.0 METHOD PERFORMANCE

4.1 For estimated quantitation limit and working linear range, refer to Section 4.0 of the ME600 series SOPs. Method performance data are acquired as per Microbac SOP45.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 The analyst should be cautioned that this procedure may not be vigorous enough to destroy some metal complexes.

6.0 EQUIPMENT AND SUPPLIES

6.1 Filter paper and filter funnels. (Whatman No. 41) or equivalent 0.45 micrometers

6.2 Digestion hood

6.3 Repeat pipettors

6.4 Polypropylene digestion cups (50 mL, tested to be ± 0.2 mL)

6.5 Class A pipettes.

7.0 STANDARDS AND REAGENTS

NOTE: Acids used in the preparation of standards and for sample processing must be reagent grade or better.

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Spiking Solutions:

Custom Multielement Solution MIC-SPK-1A from Inorganic Ventures or equivalent containing:

Uncontrolled Copy

250 mg/L:	K, Na
50 mg/L:	Al, Ca, Mg
25 mg/L:	Si
20 mg/L:	Fe
6 mg/L:	Sb
5 mg/L:	Ba, Li, Mo, Ti, V, Zn, Sr
2.5 mg/L:	Cr, Cu, Mn, Ni, Pb, Tl,
2 mg/L:	Ag, As, Se
1 mg/L:	Co
0.25 mg/L:	Be, Cd
10 mg/L:	B

7.2 Single element Tin: 1000 ug/mL from Inorganic Ventures or equivalent

7.3 Single element Phosphorus: 1000 ug/mL from Inorganic Ventures or equivalent

7.4 Single element Zirconium: 1000 ug/mL from Inorganic Ventures or equivalent

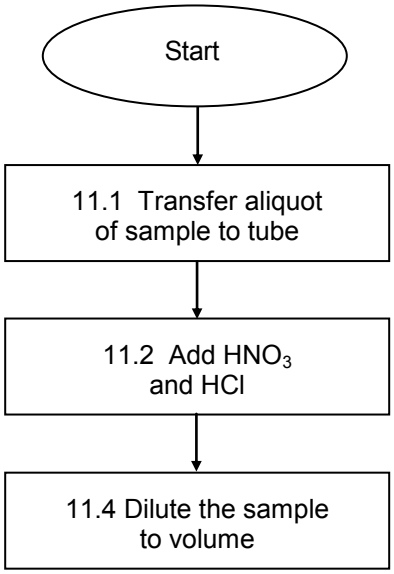
7.5 Nitric acid (concentrated), HNO_3: Baker Instra Analyzed grade or equivalent.

7.6 Hydrochloric acid (concentrated), HCl: Baker Instra analyzed grade or equivalent.

7.7 DI water

Uncontrolled Copy

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



Uncontrolled Copy

9.0 SAMPLE PREPARATION

9.1 Upon receipt, the pH of the sample is taken and recorded. If the pH is greater than 2, additional nitric acid must be added and the final pH brought to less than 2 and recorded. A note must be made to the client in the final report. The preserved sample must sit 24 hours prior to digestion.

9.2 Samples are organized into similar matrix batches of no more than 20.

10.0 CALIBRATION PROCEDURES

10.1 For instrument calibration, refer to appropriate analytical method.

10.2 Mechanical pipettes are certified with a 10 point calibration upon receipt. The calibration is verified daily thereafter.

11.0 ANALYTICAL PROCEDURES

11.1 Choose no more than 20 samples of like matrix. Transfer the sample to a polypropylene tube up to the 40 mL mark. Each batch of up to 20 samples must contain a method blank, LCS, LCSD (optional), duplicate, 2 MS samples or an MS/MSD (See Section 13.1). The reagents are entered electronically when the preparation workgroup is made. The spike amounts, are recorded in the sample data. If insufficient volume is available for the MS or MSD analysis, an LCSD is required.

11.1.1 As per method 200.7, there must be a sample duplicate for every twenty (20) samples or less and a sample MS for every ten (10) samples.

11.2 If 40 mL is the initial volume, then add 1.5 mL concentrated HNO_3 and 2.5 mL of concentrated HCl to the sample with a repeat pipettor.

11.3 Use DI water to volume to 50 mL in the polypropylene tube.

11.4 Enter initial and final volumes into general prep logbook.

11.5 See Figure 11.1 for an example of the Metals Digest Log.

Uncontrolled Copy

12.0 DETAILS OF CALCULATIONS

12.1 Refer to individual methodology.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Each batch of up to 20 samples requires the following:

13.1.1 Method blank - a 50 mL aliquot of DI water that is acidified and diluted with the sample batch.

13.1.2 LCS - a 50 mL aliquot of DI water that is spiked with 5 mL spiking solution acidified and diluted with the sample batch. 25 uL of tin is added to each LCS. 25 uL of phosphorus and zirconium are added when needed.

13.1.3 LCSD - a LCS prepared in duplicate. This is only required if the batch has no MS/MSD due to lack of volume.

13.1.4 MS/MS or MS/MSD - two additional aliquots of a sample that are spiked with 5 mL spiking solution acidified and diluted with the sample batch. 25 uL of Sn spike is added to each MS/MSD. 25 uL of phosphorus and zirconium are added when needed. Prepare 1 MS for every 10 samples.

13.2 Results of the analysis of the QA/QC samples are kept for easy reference.

13.3 All batch quality control samples are subjected to exactly the same preparation procedures as those used on actual samples in the digestion batch.

13.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13-1 and 13-2 in Microbac Sops ME600E and ME600G, does not meet acceptance criteria. When nonconforming data occurs the

employee initiates an NCR and proceeds with indicated corrections as Tables 13-1 and 13-2 in Microbac SOPs ME600E and ME600G.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 14.1 of Microbac SOPs ME600E and ME600G). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

Uncontrolled Copy

13.4.4 Any item not addressed here can be found in Section 13.0 Microbac Laboratories Inc.'s Laboratory Quality Assurance Plan.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 The appropriate log books will be checked and signed by the department supervisor for completeness.

15.0 PREVENTIVE MAINTENANCE

15.1 The hood area periodically needs to be cleaned.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Each laboratory in the sample preparation department generates specific waste streams which are segregated and collected in labeled satellite containers. Each analyst in the sample preparation department is responsible for proper disposal of spent samples and chemical waste in the specified satellite container daily or as needed.

16.2 The following are waste streams in the sample preparation area.

16.2.1 Non-Halogentated solvents: acetone

16.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

16.2.3 Acid: Dilute acid waste from soak tanks.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

Uncontrolled Copy

17.0 REFERENCES

17.1 U.S. Environmental Protection Agency, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma – Atomic Emission Spectrometry", Method 200.7 (Rev. 4.4 / 1994)

17.2 Microbac SOP ME600E "Perkin Elmer Optima 4300 Inductively coupled Plasma Atomic Emission Spectroscopy"

17.3 Microbac SOP ME600G "Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy"

17.4 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.5 Microbac SOP 45 "Method Validation Procedures"

17.6 Microbac SOP 33 "Laboratory Waste Management"

17.7 Microbac SOP GP-CAPA "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.8 Microbac SOP GP-RCA "Root Cause Analysis"

17.9 Microbac SOP K0002 "Calibration Techniques"

17.10 40CFR Part136.3 Table II – Required Containers, Preservation Techniques, and Holding Times, footnote 19

An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (See footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

Footnote 2 defines immediately as within 15 minutes of collection.

Table 1.1
Method Analytes

Name	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Calculated		
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	72608-12-9
Silica (as SiO_2)	SiO_2	99439-28-8

Figure 11

Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG442226
Analyst: REK
Spike Analyst: REK
Run Date: 08/22/2013 06:09
Method: 200.7
Balance: BAL002
Instrument: MW-2
Instrument Start: 08/22/2013 06:09

SOP: ME401 Revison 15
Spike Solution: STD59554
Spike Witness: ERP
HNO3 Lot #: COA16631
Digestion Tubes Lot #: COA16971
HCL Lot #: COA17017
ICP Filters- fisher-Lot#: RGT26836

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG442226-03	BLANK	2	40 mL	50 mL				
2	WG442226-04	LCS	2	40 mL	50 mL			5 mL	
3	WG442226-01	REF	2	40 mL	50 mL				
4	L13080863-03	SAMP	2	40 mL	50 mL				08/23/13
5	L13080863-04	SAMP	2	40 mL	50 mL				08/23/13
6	L13080863-05	SAMP	2	40 mL	50 mL				08/23/13
7	L13080863-06	SAMP	2	40 mL	50 mL				08/23/13
8	L13080863-07	SAMP	2	40 mL	50 mL				08/23/13
9	L13080863-08	SAMP	2	40 mL	50 mL				08/23/13
10	L13080863-09	SAMP	2	40 mL	50 mL				08/23/13
11	L13080863-10	SAMP	2	40 mL	50 mL				08/23/13
12	L13080863-11	SAMP	2	40 mL	50 mL				08/23/13
13	L13080863-12	SAMP	2	40 mL	50 mL				08/23/13
14	WG442226-02	REF	2	40 mL	50 mL				
15	L13080863-18	SAMP	2	40 mL	50 mL				08/23/13
16	L13080863-19	SAMP	2	40 mL	50 mL				08/23/13
17	L13080874-01	SAMP	2	40 mL	50 mL				08/23/13
18	L13080874-02	SAMP	2	40 mL	50 mL				08/23/13
19	L13080874-03	SAMP	2	40 mL	50 mL				08/23/13
20	L13080874-04	SAMP	2	40 mL	50 mL				08/23/13
21	L13080874-06	SAMP	2	40 mL	50 mL				08/23/13
22	L13080874-07	SAMP	2	40 mL	50 mL				08/23/13
23	L13081030-01	SAMP	2	40 mL	50 mL				08/30/13
24	WG442226-05	DUP	2	40 mL	50 mL				
25	WG442226-06	MS	2	40 mL	50 mL			5 mL	
26	WG442226-07	MS	2	40 mL	50 mL			5 mL	

Analyst: [signature]

Reviewer: [signature]

MW_DIG - Modified 09/30/2009
PDF ID: 3046395
Report generated: 08/22/2013 06:09

Microbac

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
MICROWAVE DIGESTION OF
SEDIMENTS, SLUDGES, SOILS AND OILS (3051A)

Issue / Implementation Date: 10 December 2014

Last Review Date: 10 December 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Robert E. Kyer, Digestion Group Leader — Date 12/11/2014

Maren M. Beery, Metal Supervisor — Date 12/11/2014

Wade T. DeLong, Quality Assurance Officer — Date 12-11-14

Leslie S. Bucina, Laboratory Manager — Date 12/14/14

Document Control # 253

Issued to: Document Master File

Uncontrolled Copy

Table of Contents

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	4
5.0	Interferences and Corrective Action	4
6.0	Equipment and Supplies	5
7.0	Standards and Reagents	5
8.0	Diagram or Table to Outline Procedures	8
9.0	Sample Preparation	9
10.0	Calibration Procedures	9
11.0	Analytical Procedures	9
12.0	Details of Calculations	11
13.0	Quality Control Requirements	11
14.0	Data Review and Reporting Requirements	13
15.0	Preventive Maintenance	13
16.0	Waste Management and Pollution Control	13
17.0	References	14
	Appendices	16
	Tables	17
	Figures	18

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This procedure references SW-846 Method 3051A and is applicable to the microwave assisted acid digestion of sediments, sludges, soils, and oils. Samples prepared by this method are analyzed by Inductively Coupled Argon Plasma Spectroscopy (ICP), or by Inductively Coupled Argon Plasma Spectroscopy-Mass Spectrometry (ICP-MS) for the metals listed in Table 1.1. Samples prepared by this method may be analyzed by ICP for additional metals if the QC criteria of the method can be achieved.

1.2 Homogenize and transfer a representative sample aliquot to a digestion vessel. The appropriate acids are added and the vessels sealed. The samples are microwave digested, cooled and transferred to centrifuge tubes. Following centrifugation the supernatant is transferred to a clean digestion tube and brought to volume. In the event of centrifuge mechanical failure, the samples may be optionally filtered using the filter paper described in Section 6.3.1.

1.3 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

DI water	Deionized water
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LQAP	Laboratory Quality Assurance Plan
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
QC	Quality Control
RGT	Reagent
RL	Reporting Limit
SOP	Standard Operating Procedure
STD	Stadard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

1.4 Updates that effect concentration, vendor choices, reagents, MDLs, RLs and QC limits are subject to change without notice.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated HNO_3 and HCl to the vessels and when manually venting the vessels to avoid exposure to toxic fumes.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in SW-846.

3.2 All sample containers are certifiable pre-cleaned from the manufacturer.

3.3 Samples must be refrigerated upon receipt and kept at a temperature ≤ 6° C.

3.4 The 180 day holding time for digestion and analysis begins at the time of sampling and ends after 180 days. Samples processed or analyzed after the expiration of 180 days are out of hold.

4.0 METHOD PERFORMANCE

4.1 For estimated RLs and working linear ranges refer to Section 4.0 of the Microbac ME600 and ME700 series SOPs. Method performance data are acquired as per Microbac SOP 45.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds

may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

6.1.1 MARS Express microwave unit or equivalent. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power.

6.1.2 75 mL vessel for the Mars Express.

6.2 Apparatus or Equipment

6.2.1 Beckman GS-6 centrifuge or equivalent.

6.2.2 Analytical balance (600 g capacity) or equivalent.

6.3 Other Supplies

6.3.1 0.45 micron syringe filters or 0.45 micron filter paper

6.3.2 Volumetric pipettes

6.3.3 50 mL and 100 mL disposable centrifuge tubes or equivalent.

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 ICP-MS Spiking Solutions

QC-MS-1, CPI International, or equivalent containing:
10 ug/mL As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb, Se, Ag, Tl, V, Zn, U

7.2.1 LCS – for ICP-MS analysis, 0.25 g or approximately 3 Teflon chips, that is spiked with 0.25 mL spiking solution and digested with the sample batch.

7.2.2 LCSD – a LCS prepared in duplicate. This is only required if the batch has no MS/MSD due to lack of sample volume.

7.2.3 MS and MSD – two additional aliquots of a sample that are spiked with 0.25 mL spiking solution and digested with the sample batch.

7.3 ICP Spiking Solutions

7.3.1 MIC-SPK-1A Inorganic Ventures, or equivalent containing:

250 mg/L:	K, Na
50 mg/L:	Al, Ca, Mg
25 mg/L:	Si
20 mg/L:	Fe
6 mg/L:	Sb
5 mg/L:	Ba, Li, Mo, Ti, V, Zn, Sr
2.5 mg/L:	B, Cr, Cu, Mn, Ni, Pb, Tl
2 mg/L:	Ag, As, Se
1 mg/L:	Co
0.25 mg/L:	Be, Cd

7.3.2 Single element Tin: 1000 ug/mL from Inorganic Venutres, or equivalent

7.3.3 Single element Phosphorus: 1000 ug/mL from Inorganic Ventures, or equivalent

7.3.4 Single element Zirconium: 1000 ug/mL from Inorganic Ventures, or equivalent

7.3.5 LCS – For ICP analysis, use 1.0 g of Teflon chips, approximately 12 Teflon chips, that is spiked with 5 mL spiking solution MIC-SPK-1A, and 25 uL of tin. 25 uL of phosphorus and zirconium are added when needed. The LCS is digested with the sample batch.

7.3.6 LCSD – a LCS prepared in duplicate. This is only required if the batch has no MS/MSD due to lack of sample volume.

7.3.7 MS and MSD – two additional aliquots of a sample that are spiked as in 7.3.5 and digested with the sample batch.

Uncontrolled Copy

7.4 Nitric acid (concentrated), HNO_3. Baker Instra analyzed grade or equivalent.

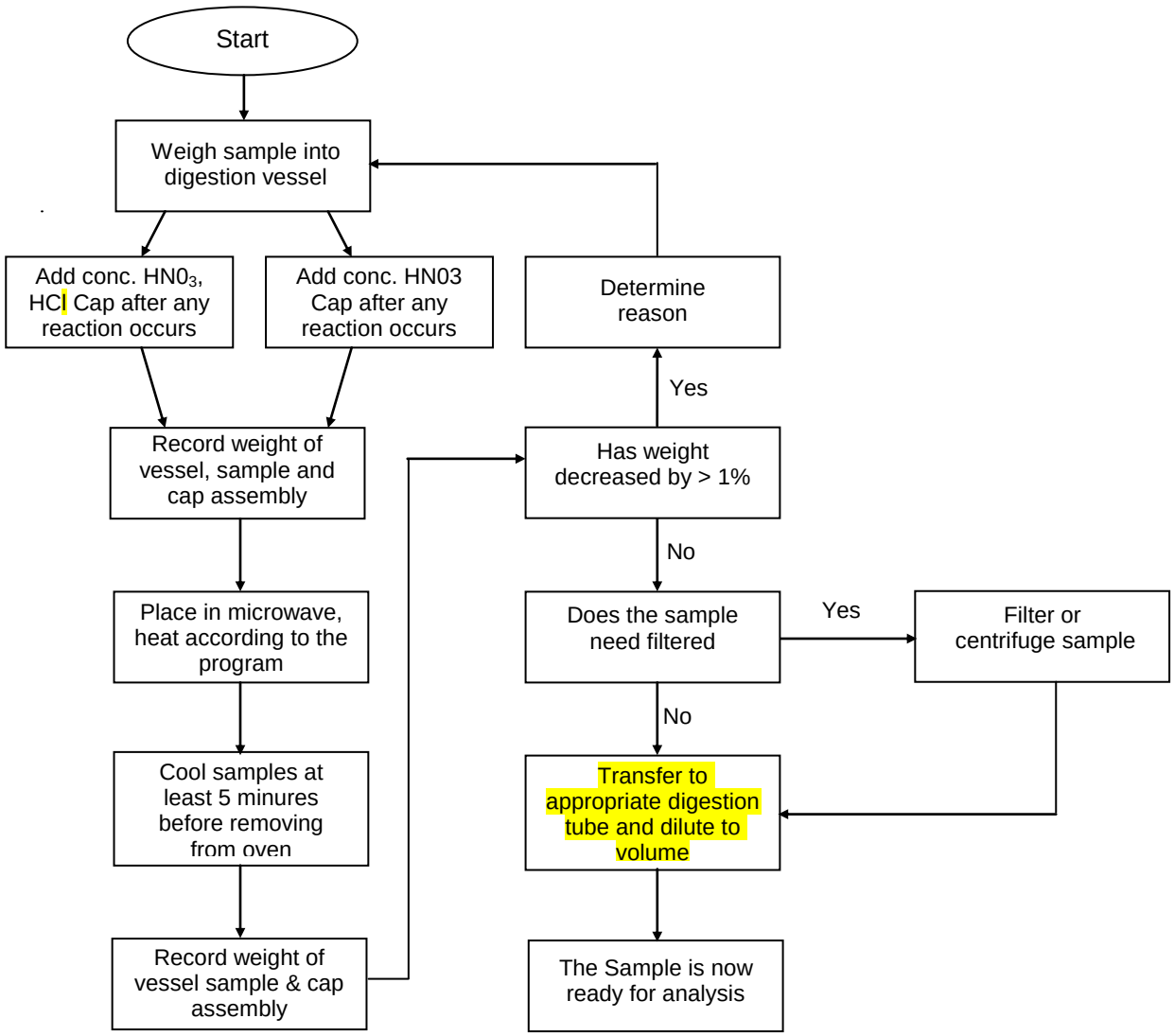
7.5 Hydrochloric acid (concentrated), HCl. Baker Instra analyzed grade or equivalent.

7.6 ASTM type II Water (ASTM D1192): Water must be monitored for impurities.

7.6.1 MB – for ICP-MS analysis except antimony and silver, weigh 0.25 g or approximately 3 Teflon chips. 5 mL of concentrated nitric acid is digested with the sample batch. For ICP-MS analysis of antimony and silver the acids added are 4.5 mL HNO_3 and 1.5 mL HCl. For ICP analysis, weigh 1.0 g or approximately 12 Teflon chips, 9 mL of concentrated HNO_3 and 3 mL of concentrated HCl is used.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES

8.1 Microwave Digestion of Sediments, Sludges, Soils and Oils



Uncontrolled Copy

9.0 SAMPLE PREPARATION

9.1 Samples are digested using SW-846 Method 3051A utilizing the appropriate acids.

9.2 Soil subsampling

a) Remove sample bottle contents and place in a tray lined with wax paper mix sample with an inert rod or scoop and break up lumps. Remove all large stones, sticks, leaves, etc. Do not over mix the sample
b) Obtain representative sample either by random removal of 3-10 portions of the sample from the pan or by using a "standard" scoop designed to retrieve a linear cross-section of the pan contents
c) The analyst will not attempt to target an exact weight once a method specified minimum amount is weighed.
d) The remaining sample will be returned to the sample container.

10.0 CALIBRATION PROCEDURES

10.1 The Mars Express is calibrated annually by the service engineer.

11.0 ANALYTICAL PROCEDURES

11.1 MARS Express – follow these guidelines.

11.1.1 Choose no more than 20 soil samples in a preparation batch.

11.1.2 Following making of workgroup, go to "Sample Data", "Digestions", then "Microwave". Here is where initial weights are recorded. This opens the electronic digestion log template where the initial weights are recorded.

11.1.3 For ICP-MS analysis except antimony and silver, weigh a 0.25 g portion of a well mixed sample into a digestion vessel. Add 5 mL of HNO_3 to each vessel. For Blanks and LCS/LCS Dup weigh 0.25 g or approximately 3 Teflon chips. Add 5 mL of HNO_3 to the blank and LCS. The LCS, MS, and MSD are spiked with 0.25 mL of ICP-MS spiking solution (7.2). In addition, when digestion for antimony and/or silver, perform digestion as per the paragraph above with the exception of adding 4.5 mL of HNO_3 and 1.5 mL of HCl to each vessel.

For ICP analysis, weigh out 1.3 - 1.5 g of a well mixed sample into a digestion vessel. The LCS, MS, and MSD are spiked with 5 mL of MIC-SPK-1A-REV1

(7.3.1). 25 uL of zirconium is added when needed. Teflon chips are used for blanks and LCS, approximately 12 Teflon chips. Add 9 mL of HNO_3 and 3 mL of HCl to each vessel.

For oils weigh out 0.25 g of sample. Add 5 mL of HNO_3 to the vessels and let samples sit under a hood for approximately 10 minutes to pre-digest samples. For Blank, LCS and LCS Dup weigh 0.25 g or approximately 3 Teflon chips. Add 5 mL of HNO_3. Spike LCS/LCS Dup with 5 mL of MIC-SPK-1A (7.3.1).

Seal all samples, blanks and LCS with rubber stoppers and also the vessel cap. Tighten vessel caps with either a torque wrench or a capping station.

Weigh vessels and record weight in Microwave electronic digestion log template.

Place vessels in carousel making sure they are pushed down completely. Then place the carousel in the microwave oven.

Click the “Run Date/Time:” box in the electronic digestion log template to insert the current date and time.

Select “Load Method” from main menu of microwave.

Select “User Directory”.

Select appropriate 3051 method to be used (EPA 3051-8V-Xpress, - 16 Xpress, -24 Xpress).

Push start button.

11.1.4 After the program has run, allow samples to cool down.

11.1.5 Remove Carousel from unit and reweigh vessels and record weight under “Sample Data”. If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

11.1.6 Transfer the solution to a graduated centrifuge tube. Rinse the microwave vessel two times with DI water and transfer the rinsates to the tube.

11.1.7 Centrifugation: Place sample in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean 50 mL or 100 mL digest tube and volume up to 50 mL for ICP analysis or 100 mL for ICP-MS analysis with the DI water. Digestion tubes are calibrated volumetrically by lot number when

received as per Microbac SOP K0002 “Calibration Procedures”. The sample is now ready for analysis unless filtration is required.

11.1.8 If filtration is needed after centrifugation, filtering is done either by 0.45 micron filter paper and vacuum pump or by the 0.45 micron syringe filter. The sample is now ready for analysis.

11.2 See Figure 11 for an example of the Metals Digest Log.

12.0 DETAILS OF CALCULATIONS

12.1 Refer to Microbac SOPs ME600E, ME600G, and ME700A.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Each batch of up to 20 samples requires the following:

13.1.1 Method blank

13.1.2 LCS

13.1.3 LCSD – an LCS prepared in duplicate. This is only required if the batch has no MS/MSD due to lack of sample volume.

13.1.4 MS and MSD

13.2 Results of the analysis of the QA/QC samples are kept for easy reference.

13.3 All batch QC samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

13.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Section 13.0 of Microbac SOPs ME600E or G and Microbac SOP ME700A, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per in Section 13.0 of Microbac SOPs ME600E or G and Microbac SOP ME700A.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 14.1 of Microbac SOPs ME600E and G and ME700A). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP or program requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.

- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 The appropriate log books are checked and signed by the department supervisor for completeness.

15.0 PREVENTIVE MAINTENANCE

15.1 Monthly examine the door, door seals and door interlocks to verify they are clean and working properly. Ensure that the door closes securely.

15.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

15.3 Clean the exhaust outlet by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

15.4 Troubleshooting procedures for the MARS Express Microwave can be found at pages 55-92 of the MARS Operation Manuals, Document Control ID #s 55 and 57.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management

technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 The following are waste streams in the sample preparation area.

16.2.1 Non – Halogenated solvents: Acetone

16.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

16.2.3 Acid: Dilute acid waste from soak tanks.

NOTE: 16.2.1 and 2 are all kept in satellite containers in each lab and are combined into the proper 55 gallon waste drums in our explosion proof waste building located outside of the Microbac laboratory facility by a waste disposal technician. 16.2.3 is neutralized and disposed in the municipal sewer system as per agreement with the city.

17.0 REFERENCES

17.1 Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils, US EPA SW-846 Method 3051A, Revision 1, February 2007, EPA Publication SW-846.

17.2 Microbac SOP LQAP “Laboratory Quality Assurance Plan“

17.3 Microbac SOP 45 “Method Validation Procedures“

17.4 Microbac SOP 600E “Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy”

17.5 Microbac SOP 600G “Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy”

17.6 Microbac SOP ME700A “Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer (SW-846 6020 / EPA METHOD 200.8)

17.7 Microbac SOP K0002 “Calibration Techniques”

17.8 Microbac SOP 33 “Laboratory Waste Management”

17.9 Microbac SOP GP-CAPA “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.10 Microbac SOP GP-RCA “Root Cause Analysis”

17.11 Black, C. A. Editor-in-Chief and Evans, D. D.; Ensminger, L. E.; White, J. L. and Clark, F. E. Associate Editors: Methods of Soil Analysis Part 2 Chemical and Microbiological Properties, 1965. American Society of Agronomy Inc., Publisher, Madison, Wisconsin, USA.

Appendix I

California ELAP samples digested by method 3051A (SOP ME406) will not have valid results reported below the nominal reporting limit. Should such "J" flagging of results occur, they must be considered insignificant.

Appendix II
Hot Water Selemium Method (80 - 3.2.2 MOD)

1.0 Weigh out 5.0 g of sample into microwave vessel.

2.0 Spike LCS, MS, MSD with 0.25 mL of MS Spike.

3.0 **NO ACID WILL BE ADDED**

4.0 Add 50 mL of DI water to blank, LCS, and all samples.

5.0 Stopper and cap vessels

6.0 Weigh vessels into LIMS system.

7.0 Digest in microwave at 100° C.

7.1 Ramp time is 15 mins, and control time is 30 mins.

8.0 Allow samples to cool to room temperature.

9.0 Transfer digestate to a centrifuge tube and centrifuge samples for 10 mins at 3500 RPM.

10.0 Transfer digestate to clean 100 mL digestion tube and volume up to 100 mL.

TABLE 1.1
METHOD ANALYTES

Name	Symbol	Wavelength	Cas Number
Aluminum	Al	308.2	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	455.4	7440-39-3
Beryllium	Be	313.1	7440-41-7
Boron	B	249.6	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	422.6	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	224.7	7440-50-8
Iron	Fe	261.1	7439-89-6
Lead	Pb	220.3	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	279.0	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Phosphorus	P	214.9	7723-14-0
Potassium	K	766.4	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	212.4	7440-21-3
Silver	Ag	328.0	7440-22-4
Sodium	Na	589.5	7440-23-5
Strontium	Sr	407.7	7440-24-6
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	337.2	7440-32-6
Vanadium	V	292.4	7440-62-2
Zinc	Zn	206.2	7440-66-6
Zirconium	ZR	339.1	7704-67-7
Calculated			
Hardness, Calculated (as $CaCO_3$)	CaCO3	NA	72608-12-9
Silica (as SiO_2)	SiO_2	NA	99439-28-8

Figure 11

Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG345781
Analyst: REK
Spike Analyst: ERP
Run Date: 10/15/2010 10:27
Method: 3051A

SOP: ME406 Revison 12
Spike Solution: STD41851
Spike Witness: VC
HNO3 Lot #: COA14980
HCL Lot #: COA14983
Digestion Tubes Lot #: COA14948

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG345781-02	BLANK	7	1 g	50 mL	177.387 g	177.377 g		
2	WG345781-03	LCS	7	1 g	50 mL	177.812 g	177.807 g	.5 mL	
3	L10100261-01	SAMP	7	1.462 g	50 mL	176.633 g	176.6 g		10/18/10
4	L10100261-02	SAMP	7	1.306 g	50 mL	180.067 g	179.99 g		10/18/10
5	L10100261-03	SAMP	7	1.315 g	50 mL	179.901 g	179.867 g		10/18/10
6	L10100261-04	SAMP	7	1.345 g	50 mL	179.905 g	179.887 g		10/18/10
7	L10100261-05	SAMP	7	1.022 g	50 mL	179.612 g	179.439 g		10/18/10
8	L10100261-06	SAMP	7	1.361 g	50 mL	178.824 g	178.787 g		10/18/10
9	L10100261-07	SAMP	7	1.405 g	50 mL	179.439 g	179.401 g		10/18/10
10	L10100261-08	SAMP	7	1.329 g	50 mL	176.486 g	176.483 g		10/18/10
11	L10100322-24	SAMP	7	1.317 g	50 mL	176.677 g	176.617 g		10/20/10
12	L10100322-25	SAMP	7	1.406 g	50 mL	179.139 g	178.781 g		10/20/10
13	L10100322-26	SAMP	7	1.404 g	50 mL	175.564 g	175.561 g		10/20/10
14	L10100322-27	SAMP	7	1.308 g	50 mL	175.457 g	175.44 g		10/20/10
15	L10100322-28	SAMP	7	1.381 g	50 mL	178.337 g	178.284 g		10/20/10
16	WG345781-01	REF	7	1.348 g	50 mL	177.654 g	177.655 g		
17	L10100322-29	RS02	7	1.348 g	50 mL	177.654 g	177.655 g		10/20/10
18	WG345781-04	MS	7	1.348 g	50 mL	180.688 g	180.683 g	.5 mL	
19	L10100322-30	MS02	7	1.348 g	50 mL	180.688 g	180.683 g	.5 mL	10/20/10
20	WG345781-05	MSD	7	1.348 g	50 mL	177.045 g	177.024 g	.5 mL	
21	L10100322-31	SD02	7	1.348 g	50 mL	177.045 g	177.024 g	.5 mL	10/20/10
22	L10100322-32	SAMP	7	1.323 g	50 mL	176.44 g	176.435 g		10/20/10
23	L10100322-33	SAMP	7	1.382 g	50 mL	176.367 g	176.3 g		10/20/10
24	L10100322-34	SAMP	7	1.419 g	50 mL	177.258 g	176.863 g		10/20/10
25	L10100322-35	SAMP	7	1.333 g	50 mL	178.678 g	178.633 g		10/20/10
26	L10100322-36	SAMP	7	1.324 g	50 mL	176.391 g	176.375 g		10/20/10
27	L10100322-37	SAMP	7	1.301 g	50 mL	178.277 g	178.093 g		10/20/10

Analyst: [signature]

Reviewer: Brenda Gregory

MW_DIG - Modified 09/30/2009
PDF ID: 1808924
Report generated: 10/26/2010 16:33

Microbac

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
MICROWAVE DIGESTION - AQUEOUS
SW846-3015A

Issue/Implementation Date: 10 December 2014

Last Review Date: 10 December 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158Starlite Drive
Marietta, Ohio 45750

Approved By:

Robert E. Kyer, Digest Group Leader — Date 12/11/2014

Maren M. Beery, Metal Supervisor — Date 12/11/2014

Wade T. DeLong, Quality Assurance Officer — Date 12-11-14

Leslie S. Bucina, Laboratory Manager — Date 12/14/14

Document Control # 258

Issued to: Document Master File

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	4
4.0	Method Performance	5
5.0	Interferences and Corrective Action	5
6.0	Equipment and Supplies	5
7.0	Standards and Reagents	6
8.0	Diagram or Table to Outline Procedures	7
9.0	Sample Preparation	8
10.0	Calibration Procedures	8
11.0	Analytical Procedures	8
12.0	Details of Calculations	9
13.0	Quality Control Requirements	10
14.0	Data Review and Reporting Requirements	12
15.0	Preventive Maintenance	12
16.0	Waste Management and Pollution Control	12
17.0	References	13
	Tables	14
	Figures	15

1.0 SCOPE AND APPLICATION

1.1 This procedure utilizes SW-846 Method 3015A and is an acid digestion procedure used to prepare surface water, groundwater, TCLP and mobility procedure extracts, and waste samples that contain suspended solids for analysis by Inductively Coupled Argon Plasma Spectroscopy (ICP) or by ICP-MS. Samples prepared by this method may be analyzed by ICP or ICP-MS for the metals in Table 1.

1.2 For the analysis of dissolved metals, the sample is filtered at the time of collection, prior to acidification with nitric acid.

1.3 Forty milliliters or less of a well shaken sample is transferred to a digestion vessel. Nitric acid is added and in the case of TCLP (SW846 1311) and SW846 6010/EPA 200.7 batches hydrochloric acid is also added. The closed vessels are microwave digested. The cooled digestates are transferred to graduated digestion tubes and brought to final volume. Samples are filtered and/or centrifuged prior to transfer and voluming if necessary.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

DI water	Deionized water
DIG-ICP	Digest Inductively Coupled Plasma
HCl	hydrochloric acid
HNO_3	nitric acid
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectroscopy
QA/QC	Quality Assurance/Quality Control
RGT	Reagent
SOP	Standard Operating Procedure
STD	Standard
TCLP	Toxicity Characteristic Leachate Procedure

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated Nitric acid (HNO_3) or hydrochloric acid (HCl) to the vessels.

2.3 Venting of the vessels must only be done when contents are at room temperature inside a hood with shield lowered to avoid the potential for chemical burns.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Volume Requirement (mL)	Collection Volume (mL)	Preservative/ Holding Time*
Total	40	600	HNO_3 to pH <2 / 6 months
Dissolved	40	600	Filter on site; HNO_3 to pH <2 / 6 months
Suspended	40	600	Filter on site / 6 months

* Holding time is the storage time allowed between sample collection and analysis when properly preserved and stored.

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 All sample containers must be pre-cleaned. Glass or plastic are both acceptable.

3.3 Total recoverable metals: All samples must be acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.4 Dissolved metals: All samples must be filtered through a 0.45 micrometer filter and then acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.5 Aqueous samples must be preserved to pH less than 2 with HNO_3. The pH is checked by the sample receiving department prior to login. For determinations of

dissolved and suspended metals, the sample must be filtered before preservation on site. Samples that are received unpreserved are preserved by the digestion laboratory personnel on site and must sit for 24 hours prior to digestion. The pH determined upon receipt, after adjustment and after 24 hours is recorded in the Laboratory Preservation Log book. If the pH after resting 24 hours is found to be noncompliant, additional acid is added and the sample must sit for another 24 hours prior to a further pH check

4.0 METHOD PERFORMANCE

4.1 For estimated quantitation limit and working linear range, refer to Section 4.0 of Microbac SOPs ME600 and ME700. Method performance data are acquired as per Microbac SOP 45.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

- Mars Xpress unit. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power.
- 75 mL Vessels for the Mars Express

6.2 Apparatus or Equipment

- Beckman GS-6 centrifuge or equivalent
- Analytical balance (600g capacity) or equivalent

6.3 Other Supplies

- Graduated digest tubes (50 mL or 100 mL Capacity)

- Quantitative filter paper, Whatman 41 or equivalent
- Volumetric pipettes
- VWR 50 mL disposable centrifuge tubes or equivalent

7.0 STANDARDS AND REAGENTS

Acids used in the preparation of samples must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 QC-MS-1 Spike – 10 mg/L, As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb,Se, Ag, Tl, U, V and Zn in 2% HNO3 and tr HF – CPI International or equivalent.

7.2 Custom multi-element solution MIC-SPK-1A-REV2 from Inorganic Ventures or equivalent containing:

250 mg/L:	K, Na
50 mg/L:	Al, Ca, Mg, P
25 mg/L:	Si
20 mg/L:	Fe
6 mg/L:	Sb
5 mg/L:	Ba, Li, Mo, Ti, V, Zn, Sr, SN
2.5 mg/L:	Cr, Cu, Mn, Ni, Pb, Tl,
2 mg/L:	Ag, As, Se
1 mg/L:	Co
0.25 mg/L:	Be, Cd
10 mg/L:	B

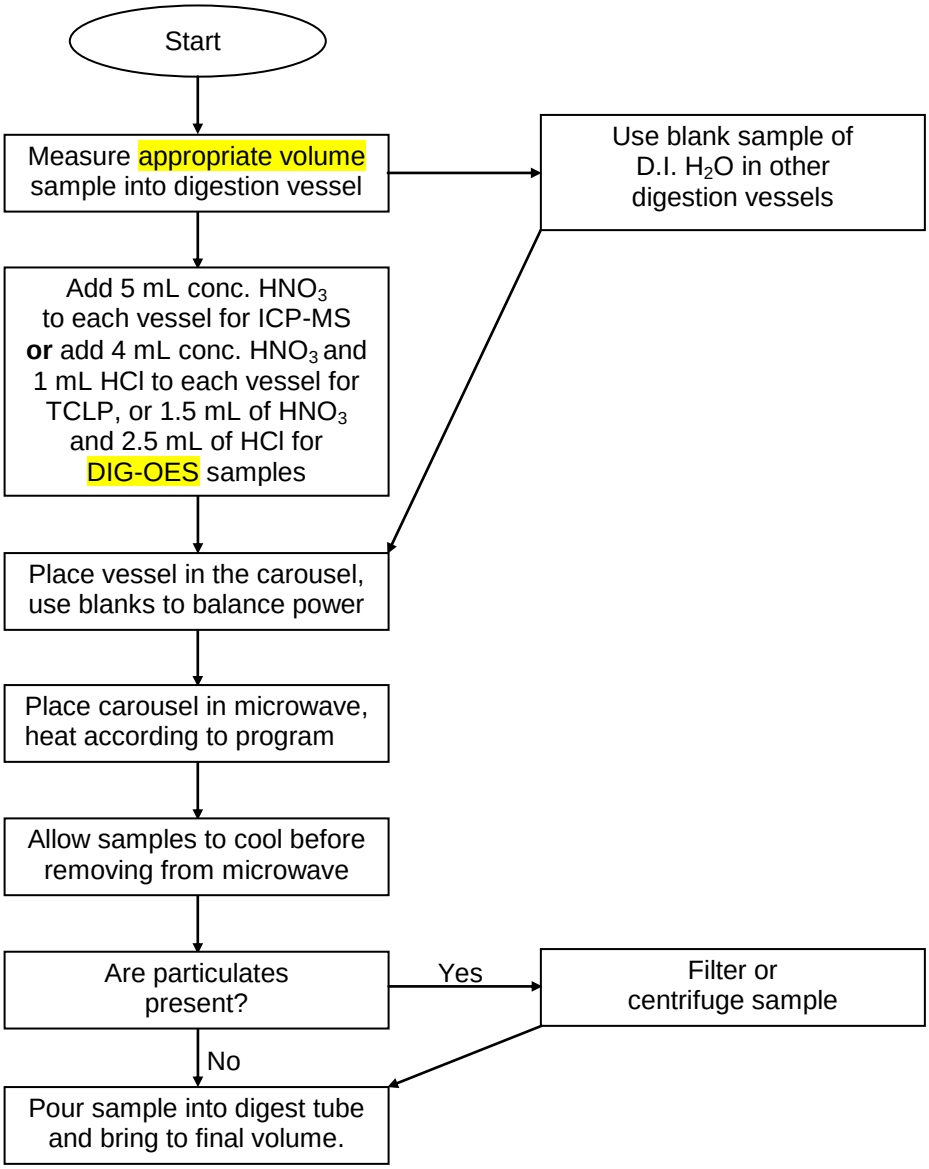
7.3 1000 ug/mL: Zr

7.4 Concentrated HNO_3 (Baker Instra analyzed or equivalent).

7.5 ASTM Type II Water (ASTM D1192): Water must be monitored for impurities.

7.6 Concentrated HCl (Baker Instra analyzed or equivalent)

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Samples are shaken to homogenize prior to digestion.

10.0 CALIBRATION PROCEDURES

10.1 The Mars Express is calibrated annually by the Manufacturer.

11.0 ANALYTICAL PROCEDURES

11.1 Choose 20 samples of similar matrix for the preparation batch.

11.2 For ICP-MS samples, measure 20 mL of a well shaken sample into a 100 mL digest tube and transfer the aliquot into a digestion vessel. 20 mL of DI water are used for the blank and LCS. Add 2.5 mL of HNO_3 to each vessel including blank and LCS.

For ICP samples, measure 40 mL of a well shaken sample into a 50 mL digest tube and transfer the aliquot into a digestion vessel. 40 mL of DI water are used for the blank and LCS.

NOTE: If a high organic content is suspected, such as TCLP extracts, 5 mL or less of sample may be used (the difference is made up with DI water).

For TCLP and DIG-ICP samples, add 3.5 mL of HNO_3 and 1.0 mL of HCl to all sample including blank and LCS.

11.2.1 Water batches for ICP-MS analysis are spiked with 0.25 mL of QC-MS-1 spike (7.1) CPI International or equivalent.

11.2.2 200.7 and 6010 Batches are spiked with 5 mL of custom multielement solution MIC-SPK-1A-REV2.

11.2.3 As per methods 200.7 and 200.8, there must be a sample duplicate per every batch of twenty (20) samples or less and a sample MS per every ten (10) samples or less.

11.3 Mars Xpress – Follow these guidlines

11.4 Seal all samples with rubber stoppers and also the vessel cap. Hand tighten vessel cap only.

Weigh vessels and record weight in microwave electronic digestion log template.

Place vessels in carousel making sure they are pushed down completely. Then place the carousel in the microwave oven.

Click the "Run Date/Time:" box in the electronic digestion log template to insert the current date and time.

Select "Load Method" from main menu of microwave.

Select "User Directory".

Select appropriate 3015 method to be used (EPA 3015-8V-Xpress, - 16 press, -24 Xpress).

Push start button.

11.4.1 After the program has finished, allow sample to cool down.

11.4.2 Remove carousel from unit and reweigh vessels and record weight in electronic digestion log template. If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

11.4.3 Transfer the solution to a graduated digest tube and bring up to a 50 mL volume with DI H_2O.

Digestion tubes are calibrated volumetrically by lot number when received as per Microbac SOP K0002 "Calibration Procedures".

11.4.4 Centrifugation: Transfer sample in 50 graduated centrifuge tube and place in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean digest tube and bring up to a 50 mL volume with DI Water. The sample is now ready for analysis. If filtration is needed after centrifugation, this is done by filtering sample through a .45 uL filter into a clean digest tube and bringing up to a 50 mL volume with DI water. The sample is now ready for analysis.

11.5 See Figure 11.1 and 11.2 for examples of the Metals Digest Logs.

12.0 DETAILS OF CALCULATIONS

12.1 Refer to individual methodology.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Each batch of up to 20 samples requires the following:

13.1.1 Method blank (MB) – an aliquot of DI water that is digested with the sample batch.

13.1.2 LCS – an aliquot of DI water that is spiked with spiking solution and digested with the sample batch.

13.1.3 Sample duplicate – Batches that include samples for Methods 200.7 and 200.8 will include a sample prepared in duplicate, both carried through the batch digestion.

13.1.4 MS and MSD – two additional aliquots of a sample that are spiked with spiking solution and digested with the sample batch. Batches that include samples for method 200.8 will include a spiked sample for every ten (10) 200.8 samples.

13.2 Results of the analysis of the QA/QC samples are kept for easy reference.

13.3 All batch QC samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

13.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1 of Microbac SOPs ME600E, ME600G and ME700. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 The appropriate log books must be checked and signed by the department supervisor for completeness.

15.0 PREVENTIVE MAINTENANCE

15.1 Monthly examine the door, door seals and door interlocks of the microwaves to verify they are clean and working properly. Ensure that the door closes securely.

15.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

15.3 Clean the exhaust outlet of the microwave by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 The following are waste streams in the sample preparation area.

16.2.1 Non – Halogenated solvents: Acetone

16.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

16.2.3 Acid: Dilute acid waste from soak tanks.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee received training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846, Method 3015A, Revision 1, February 2007, EPA Publication SW-846.

17.2 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.3 Microbac SOP 33 "Laboratory Waste Management"

17.4 Microbac SOP 45 "Method Validation Procedures"

17.5 Microbac SOP GP-CAPA "Corrective Action and Preventive Action; Initiating, Tracking, and Monitoring"

17.6 Microbac SOP GP-RCA "Root Cause Analysis"

17.7 Microbac SOP ME600E "Perkin Elmer OPTIMA 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy"

17.8 Microbac SOP ME600G "Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy"

17.9 Microbac SOP ME700A "Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer (SW-846 6020 / EPA METHOD 200.8)

17.10 Microbac SOP K0002 "Calibration Procedures"

17.11 40CFR Part136.3 Table II – Required Containers, Preservation Techniques, and Holding Times, footnote 19

An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (See footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

Footnote 2 defines immediately as within 15 minutes of collection.

17.12 Microbac SOP TCLP01 "Toxicity Characteristic Leachate Procedure"

Table 1
Method Analytes

Name	Symbol	Cas Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Phosphorus	P	7723-14-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Zirconium	Zr	7704-67-7
Calculated		
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	72608-12-9
Silica (as SiO_2)	SiO_2	99439-28-8

Figure 11.1

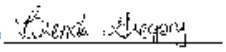
Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG419896
Analyst: REK
Spike Analyst: REK
Run Date: 02/05/2013 05:40
Method: 3015
Balance: BAL016
Instrument: MW-2
Instrument Start: 02/05/2013 05:48

SOP: ME407 Revison 13
Spike Solution: STD56032
Spike Witness: VC
Digestion Tubes Lot #: COA16400
HCL Lot #: COA16547
HNO3 Lot #: COA16631
6010 h2o mdl verfs LOQ L STD55795

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG419896-01	BLANK	1	40 mL	50 mL	202.949 g	202.943 g		
2	WG419896-02	LCS	1	40 mL	50 mL	207.665 g	207.643 g	5 mL	
3	L13010746-01	ML01	1	40 mL	50 mL	203.512 g	203.485 g	40 mL	02/08/13
4	L13010746-02	ML02	1	40 mL	50 mL	204.145 g	204.125 g	40 mL	02/08/13
5	L13010746-03	ML03	1	40 mL	50 mL	203.801 g	203.789 g	40 mL	02/08/13
6	L13010746-04	ML04	1	40 mL	50 mL	204.308 g	204.293 g	40 mL	02/08/13
7	L13010746-05	ML05	1	40 mL	50 mL	204.184 g	204.168 g	40 mL	02/08/13
8	L13010746-06	ML06	1	40 mL	50 mL	203.302 g	203.289 g	40 mL	02/08/13
9	L13010746-07	ML07	1	40 mL	50 mL	205.097 g	205.087 g	40 mL	02/08/13

Analyst:

Reviewer: 

MW_DIG - Modified 09/30/2009
PDF ID: 2744411
Report generated: 03/04/2013 11:01

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
PERKIN ELMER OPTIMA 4300
INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY SW-846 6010/EPA 200.7

Issue/Implementation Date: 25 August 2014

Last Review Date: 25 August 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Maren M Beery — 08/27/2014
Maren M. Beery, Metal Supervisor — Date

Wade T. DeLong — 8-29-14
Wade T. DeLong, Quality Assurance Officer — Date

Leslie S. Bucina — 8/29/14
Leslie S. Bucina, Laboratory Manager — Date

Document Control # 263

Issued to: Document Master File

Uncontrolled Copy

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	5
4.0	Method Performance	6
5.0	Interferences and Corrective Action	7
6.0	Equipment and Supplies	7
7.0	Standards and Reagents	8
8.0	Diagram or Table to Outline Procedures	11
9.0	Sample Preparation	12
10.0	Calibration Procedures	12
11.0	Analytical Procedures	13
12.0	Details of Calculations	17
13.0	Quality Control Requirements	19
14.0	Data Review and Reporting Requirements	23
15.0	Preventive Maintenance	24
16.0	Waste Management and Pollution Control	25
17.0	References	26
	Appendices	28
	Tables	36
	Figures	51

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure covers the operation of a Perkin Elmer Optima 4300 Inductively Coupled Argon Plasma spectrometer according to methods 6010B, 6010C, and 200.7 for the analysis of metals in digested soils, sludges, wastes, extracts and waters. Filtered waters preserved in acid may also be analyzed by this method. Samples originating from the states of North and South Carolina will be analyzed by Method 6010C in lieu of Method 6010B.

1.2 For elements for which this method is applicable see Table 1.1.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Section 4.0 provides concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods (see Microbac SOPs ME401A, ME406 and ME407). The instrument is prepared for operation and stabilized through a warmup period. Once stabilized, the instrument is calibrated and initial quality control elements are analyzed. When initial quality control requirements have been met, sample analysis begins. Samples are analyzed for metals content by optical emission spectroscopy. The Perkin Elmer Optima 4300 optical design combines an eschelle polychromator with a solid state detector in an integrated system optimized for ICP-OES. The polychromator allows for simultaneous multi-element analysis. The Perkin Elmer Optima 4300 contains a custom, two dimensional SCD array detector and RF generator. Perkin Elmer software is completely Windows based and extremely easy to use. A basic understanding of windows operation enables the analyst to move throughout the software.

1.4 Background correction points are selected according to compared background scans of standards, blanks, and samples.

1.5 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
COC	Coefficient of Correlation
DI water	Deionized Water
HCl	Hydrochloric Acid

HNO_3	Nitric Acid
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	interference Check Sample
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IECs	Interelement Corrections
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LLCCV	Low Level Continuing Calibration Verification
LLICV	Low Level Initial Calibration Verification
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectroscopy
QC	Quality Control
RGT	Reagent
RL	Reporting Limit
SCD	Segmented-array Charge-coupled Device
SOP	Standard Operating Procedure
STD	Standard

Calibration blank – a calibration standard prepared with DI water (7.8) used in establishing the calibration curve.

1.6 For a comprehensive list of common terms and definitions, consult Appendix A in SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 The Optima 4300 is equipped with safety interlocks to protect the analyst and instrument from harm if something out of the ordinary happens. The system checks water flow, argon pressures, sample compartment door interlocks, and plasma stability. These interlocks are constantly monitored and displayed on the screen. If any interlock is interrupted, the plasma is automatically shut down. Never attempt to defeat any interlocks.

2.1.1 The following interlocks must be satisfied in order to ignite the plasma:

- Front and side doors on the sample compartment must be closed
- Argon pressures for the torch must be correct
- Cooling water must be flowing to the RF coil

2.1.2 The following interlocks must not be interrupted while the system is operating:

- Purge gas for the detectors and the spectrometer optics must be functioning properly.
- The temperature of the optics housing must be 36-40° C
- The temperature of the detector must be less than -30° C

2.2 **WARNING:** Due to the high voltages and temperature, caution must be used in maintenance and troubleshooting.

2.3 A red Emergency Plasma Off switch located on the left front of the instrument allows the analyst to shut off the plasma in an emergency.

2.4 **WARNING:** Use gloves, safety glasses, lab coats and/or other appropriate safety precautions when handling samples and reagents.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1

Measurement	Digestion Vol./Wt.Req*	Collection Vol./Wt.	Preservative	Holding Time**
Total recoverable	50 mL	250 – 1000 mL p	HNO3 to pH <2	6 months
Dissolved	50 mL	250 – 1000 mL p	HNO3 to pH <2 Filter on-site	6 months
Suspended	50 mL	250 – 1000 mL p	Filter on-site	6 months
Total	50 mL	250 – 1000 mL p	HNO3 to pH <2	6 months
Soil	1 g	200 g glass	≤6° C	6 months

p = Plastic

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

3.2 Water samples received unpreserved will be acid preserved in the laboratory and must rest for at least 24 hours prior to digestion.

Uncontrolled Copy

4.0 METHOD PERFORMANCE

Instrument Detection Limits (IDLs)

4.1 IDLs are calculated by multiplying the standard deviation obtained from analysis of a reagent blank solution with seven consecutive measurements by the one-sided 98% confidence level t-statistic (3.14 is the t-statistic for seven samples). Each measurement is performed as though it were a separate sample (i.e, with rinsing in between).

IDLs must be determined quarterly. The IDL is required to be numerically less than the associated MDL.

Method Detection Limits (MDLs)

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 4-1. These limits were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project reporting limits may be higher.

4.5 Precision and accuracy data in Tables 4.2a and 4.2b were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 MDLs must be re-determined whenever there is any change to the sample preparation procedure, or any significant change to the instrument

Linear Calibration Ranges

4.7 Calibrate the instrument, as described in Section 10.0.

4.8 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

4.9 The linear range is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard. The values reported in Table 4-1 are 90% of the verified upper linear range.

Uncontrolled Copy

4.10 Linear Dynamic Range verifications are analyzed quarterly and edited in KOBRA and in the instrument software.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 IEC's (InterElement Corrections) are minimized by the use of the eschelle grating. However this grating does not eliminate all interferences due to spectral overlap. Table 5-1 lists the approximate IEC's necessary of analyte per unit (mg/L) of interferant for the Optima 4300. These IEC's are subject to change with modifications made to operating conditions, such as changes in coolant flow, power, nebulizer or even new torches.

5.2 The use of a peristaltic pump reduces physical interferences. However, samples with high dissolved solids, high acid concentration or high viscosity may need to be diluted.

6.0 EQUIPMENT AND SUPPLIES

6.1 Perkin Elmer Optima 4300 equipped with a CETAC ASXpress-520 Autosampler.

6.2 Argon gas supply (liquid).

6.3 Dell Pentium 4 computer with Microsoft Windows 2000 Professional and Perkin Elmer WinLab32 ICP Continuous Software Version 4.0.0.0303.

6.4 ESI Microflow PFA-ST3-84 Nebulizer

6.5 Peristaltic pump tubing

6.5.1 Black/Black – 0.32 mL/min (Sample and flush station).

6.5.2 Yellow/Orange – 0.51 mm I.D (Internal Standard)

6.5.3 Red/Red – 1.14 mm I.D. (drain)

6.6 Mixing block

6.7 Calibrated mechanical pipettes:

6.7.1 10 -100 uL

6.7.2 100-1000 uL

6.7.3 1000-5000 uL

6.8 Metal-free plastic pipette tips (for the pipettes) specified in 6.7

6.9 Metal-free 15 mL plastic test tubes

6.10 Metal-free 50 mL plastic test tubes

6.11 Class A glass pipettes – for preparation of standard solutions

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS System and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Concentrated hydrochloric acid (HCl). Baker Instra Analyzed Grade or better.

7.3 Concentrated nitric acid (HNO_3). Baker Instra Analyzed grade or better.

7.4 DI water – ASTM Type II or equivalent (ASTMD 1193)

Calibration Solutions

7.5 Mixed calibration stock standards are purchased from Inorganic Ventures as KEM-CONC-1,2,3 standards set. From this the High Standard and CCV are made for two different matrices.

7.6 Working Calibration Solutions – For the High Standard water matrix, dilute 5 mL from each bottle of stock into 500 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 5 mL from each bottle of stock into 500 mL 5% HCl and 5% HNO_3 in DI water. From the High Standard the following dilutions are made with the appropriate matrix matched acid water:

For Optima 4300:

S1: 250X dilution of high standard
S2: 125X dilution of high standard
S3: 2X dilution of high standard
S4: High Standard

Standard S0 is a matrix matched blank (7.8). Concentrations of each element in the calibration standards are given in Table 7.4.1 for Optima 4300.

7.7 Working CCV Solutions – The CCV is prepared for 2 different matrices. For the water matrix dilute 5 mL from each bottle of calibration stock into 1000 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 5mL from each bottle of calibration stock into 1000 mL 5% HCl and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

7.8 Working Calibration Blank Solution - A calibration blank of 5% HNO_3 , 5% HCl for soil and 2% HNO_3, 5% HCl for water is used to establish the analytical curve and is analyzed following each initial and continuing calibration standard analysis.

Initial Calibration Verification Solutions

7.9 The ICV stock solution is purchased from SPEX as XKES-5 and SCP Science as Plasmacal1 and 3. The ICV is at the same level as the midpoint standard used in the calibration and is a separately prepared, quality control analyzed and certified source from that of the calibration standards.

7.10 1000 ug/mL silicon – Inorganic Ventures

7.11 The ICV is prepared for 2 different matrices. For the water matrix dilute 1 mL from each bottle of ICV stock into 200 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 1 mL from each bottle of ICV stock into 200 mL 5% HCl and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

Interference Check Sample Solutions

7.12 ICS Stock – An ICS A (ICSA) stock solution is purchased from Inorganic Ventures as CLPP-ICS-A.

ICS AB (ICSAB) standards are purchased from Inorganic Ventures as CLPP-ICS-A, CLPP-ICS-B, and KEM-ICS-B-1A.

7.13 Working ICS Solutions – The ICSA is prepared for 2 different matrices. For the water matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 2%

HNO_3 in DI water. For the soil matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSA solution will contain only aluminum, calcium, iron and magnesium in high concentrations (see Table 7.7.1).

The ICSAB is made for 2 different matrices. For the water matrix dilute 10 mL of CLPP-ICS-A, 1 mL of CLPP-ICS-B and 1 mL of KEM-ICS-B-a into 200 mL 5% HCl and 2% HNO_3 in DI water. For the soil matrix dilute same volumes of the ICSAB into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSAB will have the same concentrations for aluminum, calcium, iron and magnesium as the ICSA with additional metals spiked at detectable levels (see Table 7.7.1).

Internal Standard Solutions

7.14 10,000 ug/mL Yttrium – Inorganic Ventures

7.15 10,000 ug/mL Gallium – Inorganic Ventures

7.16 Working Internal Standard Solution – a 5 mg/L Y and 25 mg/L Ga internal standard solution for Optima 4300, prepared by diluting 5 mL gallium and 1 mL yttrium into 2000 mL of the appropriate matrix matched acid water.

Low Level Calibration Verification Solutions

7.17 The low level ICV and low level CCV (LLICV and LLCCV) are analyzed for 6010C only. The LLICV and LLCCV are analyzed using the same solution with concentrations at or below the RL.

7.18 The LLICV/LLCCV solution is custom prepared from single element standards (Inorganic Ventures or equivalent) or by appropriate dilution of the CCV solution (7.7) into 5% HCl and 2% HNO_3 for the water matrix and 5% HCl and 5% HNO_3 for the soil matrix.

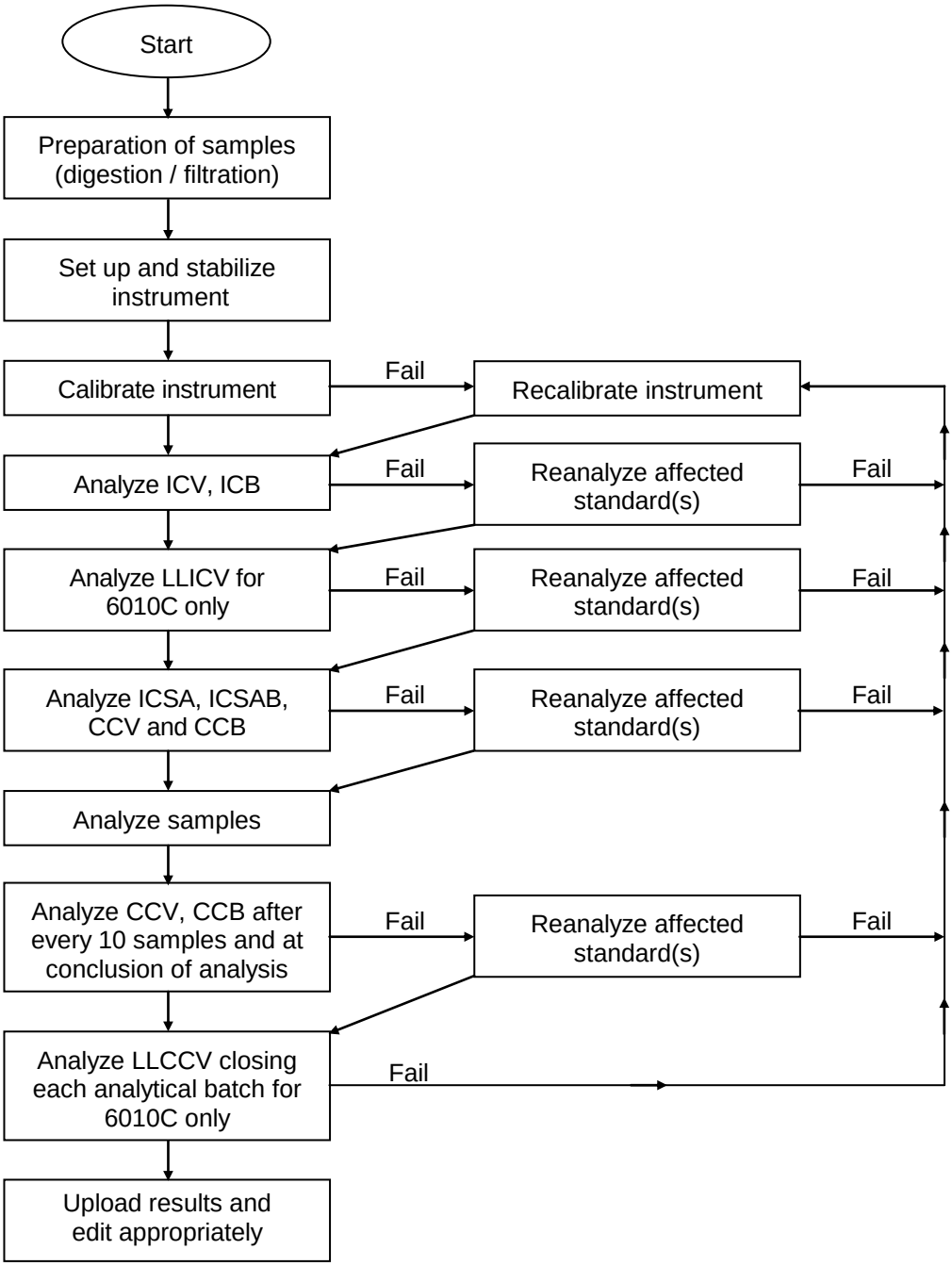
7.19 Concentrations of each target metal must be at or below the respective RL for the metal.

7.20 4 % HCl Solution – To approximately 500 mL of DI water add 40 mL conc. HCl. Dilute to 1000 mL final volume with DI water.

7.21 Custom Multielement Solution MIC-SPK-1A from Inorganic Ventures or equivalent.

Uncontrolled Copy

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



Uncontrolled Copy

9.0 SAMPLE PREPARATION

9.1 Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

ME401A – Microwave Digestion – Dissolved Aqueous, SW-846 Method 3015/EPA 200.7

ME406 – Microwave Digestion of Sediments/Sludges/Soils/Oils, SW-846 Method 3051

ME407 – Microwave Digestion – Aqueous, SW-846 Method 3015

10.0 CALIBRATION PROCEDURES

10.1 Initial Calibration – The instrument is calibrated before analysis of any samples with a blank and four calibration standards or alternatively with a blank and the high standard, S4 from 7.6 (6010C only). Calibration Standards are prepared from Inorganic Ventures Stock. The dilutions of the calibration standards are listed in Section 7.6. The concentrations are listed in Table 7.4.1.

For the multipoint calibration, the instrument performs a linear or a weighted linear regression for all elements except potassium and sodium which have a non-linear curve fit. For the single point calibration the instrument performs a linear regression with a calculated intercept for all analyzed elements. (See Appendix A for calibration algorithms.) The correlation coefficients are printed out when calibration is complete. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

When performing the multipoint calibration the low calibration standard must contain the elements of interest at concentrations at or below the reporting limit or a low level calibration check standard at or below the reporting limit must be analyzed after calibration and before sample analysis. The LLICV must always be analyzed for 6010C. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action for the curve and low level calibration verification standard.

10.2 ICV Analysis – ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

Uncontrolled Copy

10.3 CCV Analysis – The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.4 ICB and CCB Analysis – The solution used is the calibration blank (7.8). See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.5 ICSA and ICSAB Analysis – Required at the beginning of analytical run. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.6 Low Level Calibration Verification (LLICV and LLCCV) Analysis – For 6010C only is required at the beginning of the run (LLICV) and at a minimum after every analytical batch (LLCCV). See Table 13.3 for acceptance criteria and corrective action.

10.7 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices.

11.2 Startup procedures

11.2.1 Pump tubing on instrument must be pliable and have only slight discoloration. If tubing is worn, replace.

11.2.2 Torch must be clean and dry.

11.2.3 The instrument's main power source, RF generator, water recirculator and vent must be on at all times unless there is an emergency situation or for maintenance purposes.

11.2.4 Turn on the computer and login to the system. Click on WinLab 32. While the instrument goes through its systems check, attach all tubing to the pump and engage tension arms. Double click on Xpress Config 3.0 located on the desk top to open up the autosampler software.

Click on connect to ASXpress and minimize the window.

11.2.5 Click on plasma icon at the top of the screen. This opens up the plasma control window. Click on box that says pump. This starts the sample, rinse and internal standard flows.

11.2.6 To ignite plasma, click on the "on" button in the plasma control window, close window.

11.3 Set up the instrument with the proper operating conditions. These conditions are found under Plasma control.

- Power 1300 – 1450 W
- Plasma 15 -17 L/min
- Nebulizer 0.45 – 0.6 L/min
- Auxiliary 0.2 – 0.8 L/min
- Pump rate 2.0 - 2.5 mL/min for samples 4.0 mL/min rinse
- Water circulation on

11.4 The ICP must be allowed to become thermally stable before beginning analysis. (This usually requires approximately 30 minutes of operation prior to calibration.)

11.5 Pumps must be on before beginning calibration.

11.6 Set up of analytical sequence

11.6.1 After igniting plasma click on file (top left of screen) open, sample info File, click on Default .SIF, open. To call up method click on Method (top right of screen) click on method desired for current analysis, ok. Click on Auto, Open Results Data Set Name, change date, click on ok.

11.6.2 To enter the analytical sequence click on SamInfo. Enter all sample labels.

11.6.3 Enter 2,3,4,5,6,7 into the "Analyze QCs Before" Column and 6,7 in the same column after every 10 samples. This tells the instrument to read check standards at the beginning and check standards after every 10 samples.

11.6.4 To have the instrument calculate percent difference and percent recoveries, click on "Matrix Check Samples" column. Click on Duplicate for a duplicate sample, Recovery Set Number for LCS, MS/MSD and post digestion spike. Click on Remarks and enter 5 for the dilution factor for a serial dilution. Reference the

correct sample for calculation of recoveries and RPD. Enter dilution factor in the remarks column.

11.6.5 To save all run information, click on file (top left of screen), Save As, Sample info File, type in run date and click on Save.

11.7 Calibration

11.7.1 Standards are prepared from Inorganic Ventures stock. The concentrations are listed in Tables 7.4.1.

11.7.2 For the Optima 4300 model, the prepared blank and standards are placed in positions one through five in the standard rack (larger tubes). The ICV solution is located in position 11. The ICSA and ICSAB are in positions 12 and 13 respectively. The CCV is placed in position six following calibration.

11.7.3 Prior to calibration you must align the Hg lamp. To do this click on Tools, Spectrometer Control, Hg Realign. This is done axially and radially. Record the results for entry into the maintenance log. You must also perform the Background Equivalent Concentration test (BEC).

To run the BEC Test:

1) Warm-up the instrument for at least 5 minutes
2) Open method **MnBEC**
3) Open the workspace by clicking on: File – Open – Workspace – MnBEC
   This will bring up the Manual Analysis Control Window, Results Window and Spectrometer Control Window.
4) In the Manual Analysis Control Window, enter a **Results Data Set Name** (e.g. date + BEC)
5) Click on ASXpress Configuration to open aurosampler window.
6) Switch the valve to load position.
7) Connect the probe to the peripump channel leading to the rinse port on the valve.
8) Put probe into a cup of deionized water.
9) Disable ASXpress operation through xpress configuration by unclicking the top check box.
10) Click save configuration to ASXpress.
11) Aspirate the blank, click on **Analyze Blank** button
12) Aspirate the 1 mg/L Mn standard, click on **Analyze Standard** button RSD <1%
13) Aspirate rinse solution
14) In the Spectrometer Control Window select **Shutter: Closed**

15) In the Manual Analysis Control Window type in **BEC** next to ID, click on the **Analyze Sample** button
16) The absolute value of the result is the **BEC**
17) Compare **BEC** value to the acceptable value of < 0.04 mg/L
18) In the Spectrometer Control Window select **Shutter: Auto**
19) Record BEC value for entry-in the maintenance log and close the windows.
20) Enable ASXpress operation through xpress configuration by unclicking the top check box.
21) Click save configuration to ASXpress.

11.7.4 To initiate calibration, click on Auto, Analyze and Analyze All.

11.7.5 The prepared calibration standards ICV, CCV, ICB/CCB. ICSA/ICSAB and LLICV/LLCCV (if running 6010C) are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

11.7.6 For a multipoint calibration, the instrument software performs a linear or a weighted linear regression with a calculated intercept for all analyzed metals except potassium and sodium which exhibit a nonlinear response. The software performs a nonlinear regression with a calculated intercept for potassium and sodium. The instrument software performs a linear regression with a calculated intercept for single point calibration. (See Appendix A for the calibration algorithms). Upon completion of the calibration, check printout to verify that all correlation coefficients are 0.995 (0.998 for 6010C) or better before continuing.

11.8 Upon completion of the calibration begin following the analytical sequence as described below. For acceptance criteria and corrective actions, see Tables 13.1, 13.2 and 13.3:

Calibration
ICV
ICB
LLICV (6010C)
ICSA
ICSAB
CCV
CCB
10 or less samples
CCV
CCB
Continue with the last three (3) steps until the end of run,
always ending with a CCV/CCB and LLCCV (6010C).

11.8.1 The sample results are an arithmetic mean (average) of three replicate readings per analyte. For any analyte with a result greater than the reporting detection limit, the % RSD between the replicate readings must be less than ten.

11.9 Flush the system with the rinse solution for at least one (1) minute before the analysis of each sample. Analyze the CCV and CCB after each 10 samples at a minimum, and at the end of each analytical run.

11.10 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values.

11.11 Dilute and reanalyze samples that are more concentrated than the linear dynamic range.

11.12 After analysis, let the instrument rinse for fifteen minutes then extinguish plasma and release pump tension. For the Optima 4300 model, press F11 to raise probe.

11.13 Exit software and turn off computer.

12.0 DETAILS OF CALCULATIONS

12.1 All calculations for samples and standards are computed from the mean of three exposures. Each metal has a specified linear range. Refer to Table 4-1 for upper limits.

12.2 After the multipoint calibration is complete, the software performs a linear or a weighted linear regression with a calculated intercept for all analyzed metals except potassium and sodium which exhibit nonlinear responses.

The software performs a nonlinear regression with a calculated intercept for potassium and sodium. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance.

Following a single point calibration, the software performs a linear regression with a calculated intercept. Calculated correlation coefficients in this case are all equal to 1.0. (See Appendix A for the calibration algorithm). All sample results are calculated from the calibration curve.

12.3 Dilution factors and preparation factors are calculated into the final result which is computed from the mean of three exposures.

12.3.1 For Liquid Samples:

$$mg/L\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume(mL)}{Inital\ Volume(mL)} * \frac{Total\ Diluted\ Volume}{Sample\ aliquot}$$

12.3.2 For Solid Samples:

$$mg/Kg\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume(mL)}{Inital\ Volume(g)} * \frac{Total\ Diluted\ Volume}{Sample\ aliquot}$$

12.3.3 LCS/LCS Duplicate % Recovery

$$\%\ recovery = \frac{C_s}{C_t} * 100\%$$

where:

C_s = the LCS sample result
C_t = the LCS true value

12.3.4 MS/MSD % Recovery

$$\%\ recovery = \frac{(C_s - C_a)}{C_t} * 100\%$$

where:

C_s = the MS/MSD sample result
C_a = the reference sample result
C_t = the MS/MSD true value

12.3.5 Post Digestion Spike Recovery

$$\%\ recovery = \frac{(C_s - BC_a)}{C_t} * 100\%$$

where:

C_s = the spike sample result
C_a = the reference sample result
B = a factor to account for the dilution of the spiked sample relative to the reference sample (usually B = 0.9)
C_t = the spike true value

12.3.6 Duplicate RPD

$$RPD = \frac{|C_a - C_b|}{(C_a + C_b)/2} * 100\%$$

where:

C_a = the reference sample result
C_b = the duplicate sample result

12.3.7 Serial Dilution % Difference

$$\%D = \frac{|C_a - 5C_b|}{C_a} * 100\%$$

where:

C_a = the reference sample result
C_b = the diluted sample result

12.4 Calcium and magnesium results can be used to calculate water hardness results. The LIMS will use the uploaded calcium and magnesium results to calculate the hardness results. The automated hardness calculation is manually verified annually.

$$Hardness(mg/L) = 2.497[Ca(mg/L)] + 4.118[Mg(mg/L)]$$

12.5 Silicon results can be used to calculate silica results.

The calculation assumes all silicon recovered from 6010 is in the form Si02. KOBRA converts the results as follows:

$$silica(mg/L) = silicon(mg/L) * 2.13923$$

12.6 See Figure 12.1 for a sample calculation summary

13.0 QUALITY CONTROL REQUIREMENTS

Overview

13.1 Refer to Microbac SOP 45 for Details of Demonstration of Capability. (Method Validation Procedures).

Uncontrolled Copy

13.2 Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC samples (see Section 13.3 for list of required QC). The QC samples are prepared and digested identically to the analytical samples.

13.3 The following QC are digested with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Tables 13.1, 13.2 and 13.3.

Batch Quality Control

13.3.1 Method blank (MB) an aliquot of DI water that is digested with the sample batch and contains all reagents identical with the samples.

13.3.2 LCS - for water matrix, a spiked DI water that is digested with the sample batch. The LCS is prepared by diluting 5 mL of KEM-SPK-1F (see Table 13.3.2a for initial concentrations) and 0.025 mL each of single element tin, phosphorus and zirconium into a 50 mL volume of DI water.

For soil matrix; 1.0 g of PTFE Boiling Stones (Teflon) Chemware is spiked with 5 mL of KEM-SPK-1F and 0.025 mL each of single element tin, phosphorus and zirconium prior to digestion.

A LCSD may also be analyzed with the batch and is prepared identically to the LCS.

(**NOTE:** Both the LCS and LCSD must pass acceptance criteria or the samples will have to be re-digested/re-analyzed for the analyte in question.)

The final concentration of the aqueous and soil LCS are given in Table 13.3.2b:

13.3.3 Sample duplicate - a sample prepared in duplicate, both carried through the digestion procedure. (By client request only)

13.3.4 MS and MSD - a sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spiked with 5 mL of KEM-SPK-1F and 0.025 mL each of single element tin, phosphorus and zirconium for each 50 mL of sample. The final concentration spiked into the two spiked samples will be the same as the aqueous and soil LCS level in Table 13.3.2.

Interference Tests – Post Digestion

13.4 With every analytical batch a post digestion spike and a serial dilution must be analyzed. If the matrix spike fails the 75 - 125% acceptance criteria, one of the following interference tests must pass for the analyte outlier:

13.4.1 Serial dilution: If the analyte concentration is sufficiently high (at least a factor of 50 above the method detection limit before dilution), analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.4.2 Post Digestion Spike: an analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% (80-120% for 6010C) of the known value. The spike addition should produce a minimum level of 10 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.4.3 Method of standard additions: This method must be employed for certain TCLP extracts and can also be used to compensate for sample constituents which enhance or depress the analyte signal producing a slope different from that of the calibration standards. It will not correct for additive interferences which cause a base line shift.

The simplest version of these techniques is the single-addition method, in which two identical aliquots of the sample, each of volume Vx are taken. To the first (A) a small volume (V_s) of a standard analyte concentration (C_s) is added. To the second (B) a volume (V_s) of the solvent is added. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration (C_x) is calculated:

$$C_x = (S_b)(V_s)(C_s)/(S_a - S_b)(V_x)$$

where:

S_a and S_b are the analytical signals of A and B respectively. V_s and C_s should be chosen so that S_a is roughly twice S_b on the average. It is best if V_s is made much less than V_x, and thus C_s is much greater than C_x, to avoid excess dilution of the sample matrix. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected concentration from the endogenous analyte in the sample.

Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample concentration. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero concentration, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

13.5 Linear Range Analysis/High-Level Standard

A linear range analysis is performed quarterly to determine the highest concentrations for each analyte at which the instrument yields a result within 10% of the true concentration. When use of the Department of Defense Version 3 QAP is indicated, a high-level check standard with analyte concentrations at the linear range of the instrument may be analyzed subsequent to sample analysis for any analyte which exceeds the calibration range. The recovery must be within 10% of the true concentration.

13.6 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.6.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1, 13.2 and 13.3, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Tables 13.1, 13.2 and 13.3.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 14.1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval

- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.6.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RAC, corrections, corrective action(s) and evidence of effectiveness.

13.6.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that may be invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An ICV, CCV or LCS recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.7 All data is scrutinized by the analysts for method and project specific compliance. Check lists are utilized and accompany each data batch (Figure 14.1).

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client

samples into KOBRA. This is done via MICROBAC's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and RLs. Any elements that are not to be reported must be checked as excluded. This will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1, 13.2 and 13.3. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating**.** The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and KOBRA workgroup reports. The Data Review Checklist acts as a cover page and will be archived with the hardcopy data. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. This review consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness. The supervisor (or designate) also signs and dates the Data Review Checklist.

When all levels of review have been completed. The elements being reported on each sample are taken to a done status in KOBRA.

15.0 PREVENTIVE MAINTENANCE

15.1 Check the torch and nebulizer every day and clean when needed depending on sample load.

15.2 Tubing needs to be changed when it loses pliability and is worn.

15.3 Drain compressor daily.

15.4 The instruments are under service contracts so that every year a service representative will perform a systems check.

15.5 The water in the recirculator/cooler must be changed yearly.

15.6 Troubleshooting procedures for the Perkin Elmer Optima 4300 can be found in Chapter 6 of the Optima 4000 Series Hardware Guide, Document Control ID #65, and in Chapter 10 of the WinLab32 Instrument Control Software Guide, Document Control ID #67.

15.7 Performance problems (such as loss of signal or poor precision) are often related to the sample introduction system.

15.7.1 Perform the sodium bullet test to monitor sample flow.

15.7.2 Check pump tubing for flat spots, leaks or discoloration.

15.7.3 Check pump rollers for grooves or binding.

15.7.4 Check pump tubing for crimps, pinching and clogging.

15.7.5 Check nebulizer and spray chamber for leakage and proper drainage.

15.7.6 Check nebulizer spray pattern. The spray must not be uneven or sputtering.

15.7.7 Check to torch and injection for deposits, deformation and security of fit. O-rings must not be cracked or worn.

15.7.8 Check that the purge window is not cloudy or dirty.

15.8 The instrument configuration for the OPTIMA 4300 can be found in the electronic maintenance log. The template maintenance log is found at number 13195 in KOBRA. Replacement of instrument components will be recorded in the relevant maintenance log and updated in the log header as needed.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are

responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams are as follows:

The metals laboratory waste is neutralized with sodium bicarbonate and flushed down the drain with tap water as per agreement with the Marietta Department of Water and Waste Water.

17.0 REFERENCES

17.1 Inductively Coupled Plasma – Atomic Emission Spectrometry, US EPA SW-846 Method 6010B, Revision 2, December 1996, EPA Publication SW-846

17.2 Us Environmental Protection Agency, EPA Method 200.7, Revision 4.4, 1994, 40CFR pt.136.

17.3 Inductively Coupled Plasma-Atomic Emission Spectrometry, US EPA SW-846 Method 6010C, Revision 3, February 2007, EPA publication SW-846.

17.4 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.5 Microbac SOP 45 “Method Validation Procedures”

17.6 Microbac SOP ME406 “Microwave Digestion of Sediments, Sludges, Soils and Oils (3051)”.

17.7 Microbac SOP ME407 “Microwave Digestion – Aqueous SW846-3015”.

17.8 Microbac SOP 33 “Laboratory Waste Management”

17.9 Microbac SOP GP-CAPA “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.10 Microbac SOP GP-RCA “Root Cause Analysis”

17.11 Microbac SOP ME401A “Preparation of Dissolved Metals in Waters for Direct Analysis by Inductively Coupled Plasma Spectroscopy (EPA 200.7)”

Appendix A

Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$$Y=MX+B$$

where:

X = concentration
Y = intensity
M = slope of the calibration curve
B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

N = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$M = \frac{(n)\sum_{n}^{i=1}(X_i Y_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}(X_i^{\,2}) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

[2] $$B = \frac{\sum_{n}^{i=1}(X_i^{\,2})\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_i Y_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}(X_i^{\,2}) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

Uncontrolled Copy

Linear Calibration Algorithm (continued)

$$COC = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sqrt{\left(\sum x^2 - \frac{(\sum x)^2}{n}\right)\left(\sum y^2 - \frac{(\sum y)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

Uncontrolled Copy

Weighted Linear Calibration Algorithm

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_i x$$

Where y is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

where:

x = concentration value of the standard
y = measured intensity of the standard
n = number of standards
i = index for the standards
b_0 = intercept
b_1 = slope
COC = correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:

$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:

$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Uncontrolled Copy

Weighted Linear Calibration Algorithm (continued)

Correlation Coefficient: $$COC = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^{\,2}\right]\left[(w_1 w_5) - w_4^{\,2}\right]}}$$

Non-Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is a curvilinear one that can be described by the following polynomial equation:

$$Y = AX^2 + BX + C$$

where:

X = concentration
Y = intensity
A = curvature
B = slope
C = y-axis intercept

Given 3 or more data points, the values for A, B and C are calculated using the following equations [1, 2, 3].

n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$A = \frac{\left[\frac{a_2}{a_0} - \frac{a_5}{a_3}\right]}{\left[\frac{a_1}{a_0} - \frac{a_4}{a_3}\right]}$$

[2] $$B = \frac{a_5}{a_3} - \frac{(A \times a_4)}{a_3}$$

[3] $$C = \frac{c_2}{n} - \frac{(B \times c_0)}{n} - \frac{(A \times c_1)}{n}$$

where:

Uncontrolled Copy

Non-Linear Calibration Algorithm (continued)

$$a_0 = \frac{c_0}{n} - \frac{c_1}{c_0} \qquad c_0 = \sum_{n}^{i=1}(X_i)$$

$$a_1 = \frac{c_1}{n} - \frac{c_3}{c_0} \qquad c_1 = \sum_{n}^{i=1}(X_i^{\,2})$$

$$a_2 = \frac{c_2}{n} - \frac{c_4}{c_0} \qquad c_2 = \sum_{n}^{i=1}(Y_i)$$

$$a_3 = \frac{c_1}{c_0} - \frac{c_3}{c_1} \qquad c_3 = \sum_{n}^{i=1}(X_i^{\,3})$$

$$a_4 = \frac{c_3}{c_0} - \frac{c_5}{c_1} \qquad c_5 = \sum_{n}^{i=1}(X_i^{\,4})$$

$$a_5 = \frac{c_4}{c_0} - \frac{c_6}{c_1} \qquad c_6 = \sum_{n}^{i=1}(X_i^{\,2}Y_{ii})$$

Uncontrolled Copy

Appendix B

Simultaneously Extracted Metals (SEM)

SEM are operationally defined as metals (arsenic, cadmium, copper, lead, mercury, nickel, silver and zinc for example) that form sulfides less soluble than those of iron or manganese and which are at least partially released from the sediment under the conditions for generating Acid Volatile Sulfides (AVS) (see Microbac SOP KAVS). Since sulfide is a precipitant of heavy metals under anoxic conditions, the SEM to AVS ratio obtained by the method is an indicator of heavy metal bioavailability in the sediment sample.

AVS Generation and Filtration

AVS is generated in a reaction cell per the procedure in Microbac SOP KAVS. Once sulfide generation is complete the sediment and water from the reaction cell (Microbac SOP KAVS Section 11.10) is vacuum filtered through Whatman TCLP Glass microfiber Filters Acid Treated Low Metal (or equivalent) into a filtration apparatus that has been washed, rinsed in 0.1 M HNO_3 and then DI water rinsed prior to use.

The filtrate is transferred to B-D 60 mL sterile luer-lok syringes equipped with Corning 0.2 um membrane syringe filters (or equivalent) and filtered into an acid washed, DI water rinsed graduated cylinder.

The filter flask is rinsed with DI water and the rinsates are similarly 0.2 um filtered and added to the cylinder.

DI water is then used to bring the filtrate to a final volume of 250 mL. The sample is transferred to a labeled, certified precleaned 250 mL Nalgene bottle C and G Containers (or equivalent) and analyzed within two weeks.

Metal concentrations are determined by ICP-OES.

Batch QA/QC Samples

Method Blank – An aliquot of 4% HCl in DI water is filtered in the same manner as the samples. This filtrate will be used as the method blank.

LCS – In a digestion tube containing about 20 mL 4% HCl spike 5 mL of spike solution (7.25) and bring to a 50 mL final volume with 4% HCl. Filter the LCS in the same manner as the samples.

MS/MSD – If there are no client designated spike samples, choose one sample from the batch. Into two digestion tubes pour approximately 20 mL of the filtered sample. Add 5 mL of spike solution (7.25) and bring to final 50 mL volume with the additional filtered sample.

Interference Tests – A post digestion spike will be prepared as in Section 13.8. Additionally, if appropriate, a serial dilution will be analyzed as in 13.7.

Calibration Solutions

Calibration Solutions, ICV and Interference Check Solutions are prepared as in Sections 7.2 through 7.13 and 7.17 through 7.18 except that the diluents used in 4% HCl (7.24).

The working calibration blank solution used is a 4% HCl (7.24).

Analysis and Reporting

Metal concentrations are determined by ICP-OES following the procedures in Section 11.0.

The instrument is calibrated as in 10.1 followed by ICV/ICB, ICSA/ICSAB and CCV/CCB analysis. Acceptance criteria are found in Table 13.1. Samples are analyzed in groups of ten or less followed by CCV/CCB analyses. Concentrations of the metals are reported as umole per gram dry sediment (umole/g).

SEM is then calculated as the sum of the metal concentrations:

$$SEM = \sum[metals]$$

Uncontrolled Copy

Table 1.1
Method Analytes

Name	Symbol	Wavelength (4300 Model)	Cas Number
Aluminum	Al	396.1	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	233.5	7440-39-3
Beryllium	Be	234.9	7440-41-7
Boron	B	249.7	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	227.5	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	327.4	7440-50-8
Iron	Fe	239.6	7439-89-6
Lead	Pb	220.4	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	279.1	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Phosphorus	P	214.9	7723-14-0
Potassium	K	766.5	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	251.6	7440-21-3
Silver	Ag	328.1	7440-22-4
Sodium	Na	589.6	7440-23-5
Strontium	Sr	407.8	7440-23-5
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	334.9	7440-32-6
Vanadium	V	290.9	7440-62-2
Zinc	Zn	206.2	7440-66-6
Calculated			
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	NA	72608-12-9
Silica (as SiO_2)	SiO_2	NA	99439-28-8

Table 4-1
Optima 4300 MDLs, RLs and Linear Ranges for 6010/200.7

Metal	Upper Linear Range (mg/L)	Verified MDL Water (mg/L)	Reporting Limit Water (mg/L)	Verified MDL Soil (mg/Kg)	Reporting Limit Soil (mg/Kg)
Aluminum	450	0.1	0.2	10	20
Antimony	45	0.05	0.1	0.5	1.0
Arsenic	9	0.005	0.01	0.5	1.0
Barium	9	0.005	0.01	0.25	0.5
Beryllium	4.5	0.001	0.002	0.05	0.1
Boron	45	0.05	0.1	2.5	5
Cadmium	9	0.0005	0.001	0.05	0.1
Calcium	450	0.25	0.5	25	50
Chromium	45	0.0025	0.005	0.125	0.25
Cobalt	45	0.0025	0.005	0.125	0.25
Copper	45	0.0025	0.005	0.5	1.0
Iron	450	0.025	0.1	1.5	3.0
Lead	90	0.005	0.010	0.5	1.0
Lithium	0.9	0.05	0.1	2.5	5.0
Magnesium	450	.0.25	0.5	12.5	25
Manganese	27	0.005	0.01	0.25	0.5
Molybdenum	45	0.005	0.01	1.5	3.0
Nickel	45	0.01	0.04	1.	2.0
Phosphorus	180	0.5	1.0	25	50
Potassium	72	0.5	1.0	25	50
Selenium	45	0.01	0.02	0.5	1.0
Silica (as SiO_2)	77.0	1.07	2.14	NA	NA
Silicon	36	0.5	1.0	NA	NA
Silver	9	0.005	0.01	0.25	0.5
Sodium	180	0.25	0.5	12.5	25
Strontium	4.5	0.025	0.05	0.25	0.5
Thallium	45	0.1	0.2	1.0	2.0
Tin	45	0.25	0.5	12.5	25
Titanium	45	0.015	0.03	1.0	2.0
Vanadium	45	0.005	0.01	0.25	0.5
Zinc	45	0.01	0.02	0.5	1.0

Uncontrolled Copy

Table 4.2a
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010B/200.7

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	85 - 115	0 - 20	80 - 120	0 - 20
Antimony	85 - 115	0 - 20	80 - 120	0 - 20
Arsenic	85 - 115	0 - 20	80 - 120	0 - 20
Barium	85 - 115	0 - 20	80 - 120	0 - 20
Beryllium	85 - 115	0 - 20	80 - 120	0 - 20
Boron	85 - 115	0 - 20	80 - 120	0 - 20
Cadmium	85 - 115	0 - 20	80 - 120	0 - 20
Calcium	85 - 115	0 - 20	80 - 120	0 - 20
Chromium	85 - 115	0 - 20	80 - 120	0 - 20
Cobalt	85 - 115	0 - 20	80 - 120	0 - 20
Copper	85 - 115	0 - 20	80 - 120	0 - 20
Iron	85 - 115	0 - 20	80 - 120	0 - 20
Lead	85 - 115	0 - 20	80 - 120	0 - 20
Lithium	85 - 115	0 - 20	80 - 120	0 - 20
Magnesium	85 - 115	0 - 20	80 - 120	0 - 20
Manganese	85 - 115	0 - 20	80 - 120	0 - 20
Molybdenum	85 - 115	0 - 20	80 - 120	0 - 20
Nickel	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85-115	0-20	80-120	0-20
Potassium	85 - 115	0 - 20	80 - 120	0 - 20
Selenium	85 - 115	0 - 20	80 - 120	0 - 20
Silicon	85 - 115	0 - 20	80 - 120	0 - 20
Silver	85 - 115	0 - 20	80 - 120	0 - 20
Sodium	85 - 115	0 - 20	80 - 120	0 - 20
Strontium	85 - 115	0 - 20	80 - 120	0 - 20
Thallium	85 - 115	0 - 20	80 - 120	0 - 20
Tin	85 - 115	0 - 20	80 - 120	0 - 20
Titanium	85 - 115	0 - 20	80 - 120	0 - 20
Vanadium	85 - 115	0 - 20	80 - 120	0 - 20
Zinc	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85 - 115	0 - 20	80 - 120	0 - 20
Zirconium	85 - 115	0 - 20	80 - 120	0 - 20

Table 4.2b
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010C

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	80 - 120	0 - 20	80 - 120	0 - 20
Antimony	80 - 120	0 - 20	80 - 120	0 - 20
Arsenic	80 - 120	0 - 20	80 - 120	0 - 20
Barium	80 - 120	0 - 20	80 - 120	0 - 20
Beryllium	80 - 120	0 - 20	80 - 120	0 - 20
Boron	80 - 120	0 - 20	80 - 120	0 - 20
Cadmium	80 - 120	0 - 20	80 - 120	0 - 20
Calcium	80 - 120	0 - 20	80 - 120	0 - 20
Chromium	80 - 120	0 - 20	80 - 120	0 - 20
Cobalt	80 - 120	0 - 20	80 - 120	0 - 20
Copper	80 - 120	0 - 20	80 - 120	0 - 20
Iron	80 - 120	0 - 20	80 - 120	0 - 20
Lead	80 - 120	0 - 20	80 - 120	0 - 20
Lithium	80 - 120	0 - 20	80 - 120	0 - 20
Magnesium	80 - 120	0 - 20	80 - 120	0 - 20
Manganese	80 - 120	0 - 20	80 - 120	0 - 20
Molybdenum	80 - 120	0 - 20	80 - 120	0 - 20
Nickel	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 - 120	0 - 20	80 - 120	0 - 20
Potassium	80 - 120	0 - 20	80 - 120	0 - 20
Selenium	80 - 120	0 - 20	80 - 120	0 - 20
Silicon	80 - 120	0 - 20	80 - 120	0 - 20
Silver	80 - 120	0 - 20	80 - 120	0 - 20
Sodium	80 - 120	0 - 20	80 - 120	0 - 20
Strontium	80 - 120	0 - 20	80 - 120	0 - 20
Thallium	80 - 120	0 - 20	80 - 120	0 - 20
Tin	80 - 120	0 - 20	80 - 120	0 - 20
Titanium	80 - 120	0 - 20	80 - 120	0 - 20
Vanadium	80 - 120	0 - 20	80 - 120	0 - 20
Zinc	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 – 120	0 – 20	80 – 120	0 - 20
Zirconium	80 – 120	0 – 20	80 – 120	0 - 20

Uncontrolled Copy

Table 5-1
Interelement Correction Factors for Optima 4300

Factors per 100 mg/L Interferant							
Analyte	**Al**	**As**	**Ba**	**Be**	**Ca**	**Co**	**Cr**
Aluminum	-	-	-	-	-	-	-
Antimony	0.447967	-	-	-	-	-	4.19
Arsenic	-	-	.189139	-	.0981	-	-4.87
Barium	-	-	-	-	-	-	-
Beryllium	-	-	-	-	-	-	-.0120651
Boron	-	-	-	-	-	4.99	-
Cadmium	-	6.15489	-0.0191992	-	-	-6.26327	-
Calcium	-	-	-	-	-	-	-
Chromium	-	-	-	-	-	-	-
Cobalt	-	-	-1.17228	-	-	-	.159
Copper	-	-	-	-	-.0252062	.218641	-
Iron	-	-	-	-	-	4.06579	-
Lead	-.126299	-	-	-	-.0202022	.106579	-
Lithium	-	-	-	-	-	-	-
Magnesium	-	-	-	-	-	-	-
Manganese	-	-	-	-	-	-	-
Molybdenum	-	-	-	-	-	-	-
Nickel	-	-	-	-	-	.976335	-
Phosphorus		-	-	-	-	-	-
Potassium	.620975	-	-	-	.574622	-	-
Selenium	-	-	-	-	-	-.794408	-
Silicon	-	-	-	-	-	-	-
Silver	-	-	-	-	-.0118481	-	-
Sodium	.718161	-	-	-	.698715	-	-
Strontium	-	-	-	-	-	-	-
Thallium	-0.0169987	-	-	-	-.033338	-.183759	0.4
Tin	-	-	-	-	-.0529445	-	-
Titanium	-	-	-	-	-	-	.27456
Vanadium	-	-	-	-	-	-	-1.433
Zinc	0.001	-	-	-	-	-	-3.34

Uncontrolled Copy

Table 5-1
Interelement Correction Factors for Optima 4300 (continued)

Factors per 100 mg/L Interferant						
Analyte	**Cu**	**Fe**	**Mg**	**Mn**	**Mo**	**Ni**
Aluminum	-	-	-	-	44	-
Antimony	-	-	-	-	-24.4	-.681302
Arsenic	-	-.146	-	-	.742	-
Barium	-	-	-	-	-	-
Beryllium	-	-	-	-	-	-
Boron	-	-4.35839	-	-	-	-
Cadmium	-	0.0014	-	-	.0190091	-.0568649
Calcium	-	-	-	-	-	-
Chromium	-	-.0183539	-	.328	-.26	-
Cobalt	-	.00668691	-	-	-.93	.146461
Copper	-	-.0200902	-	-	.4	-
Iron	-	-	-	-	-	-
Lead	-	.0570054	-	-	-2.56406	-
Lithium	0.0647055	-	-	-	-	-
Magnesium	-	-	-	-4.05	-1.64	-
Manganese	-	-.0170384	.0157809	-	-	-
Molybdenum	-	-0.0444556	-	-	-	-
Nickel	-	.0255009	-	-	-	-
Phosphorus	-	-	-	-	-	-
Potassium	-	.696417	.526097	-	-	-
Selenium	-	-.6	-	.405	.0945449	-
Silicon	-	-.135546	-	-	10.7478	-
Silver	-	-.208152	-	-	-.24288	-
Sodium	-	.837684	.750261	-	-	-
Strontium	-	-	-	-	-	-
Thallium	-	-0.0726809	-	-1.036	.119755	-
Tin	-	-.0519835	-.0314678	-	-	-
Titanium	-	-	.0228899	-	-.250622	-
Vanadium	-	0.1	.1	-	-40	-
Zinc	-	.001	.0001	-	-	-

Uncontrolled Copy

Table 5-1
Interelement Correction Factors for Optima 4300 (continued)

Factors per 100 mg/L Interferant							
Analyte	**Sb**	**Se**	**Sn**	**Ti**	**Tl**	**V**	**Zn**
Aluminum	-	-	-	-	-	-	-
Antimony	-	.568779	-14.4736	2.11867	-	-2.31443	-
Arsenic	-	-	-	-	-	.214681	-
Barium	-	-	-	-	-	-2.27389	-
Beryllium	-	-	-	-.01	-	-0.0329788	-
Boron	-	-	-	-	-	-	-
Cadmium	-.0173125	-	-	-.0249829	-	.0642357	-.0109044
Calcium	-	-	-	-	-	-	-
Chromium	-	-	-	.118925	-	-.333864	-
Cobalt	-	-	-.0434213	2.34234	-	-	-
Copper	-	-	-	-.117406	-	-.21956	-
Iron	-	-	-	-	-	-	-
Lead	-	-	-	-.138344	-	-.0970818	-
Lithium	-	-	-	-	-	-	-
Magnesium	-	-	-	-2.32782	-	-	-
Manganese	-	-	-	-	-	-	-
Molybdenum	-	-	-	-	-	-.202941	-
Nickel	-.302278	-	-	-	.378754	-	-
Phosphorus	-	-	-	-	-	-	-
Potassium	-	-	-	-	-	-	-
Selenium	-	-	.185983	-	-	.250848	.0887204
Silicon	-	-	-	-	-	-	-
Silver	-	-	-	-	-	-2.88494	-
Sodium	-	-	-	-	-	-	-
Strontium	-	-	-	-	-	-	-
Thallium	-	-	-	-5.25518	-	-.406757	-
Tin	-	-	-	-3.02151	.549717	.389475	.269888
Titanium	-	-	-	-	-	-	-
Vanadium	-	-	-	-	-	-	-
Zinc	-	-	-	-	-	-	-

Uncontrolled Copy

Table 7.4.1
Calibration Standards Concentrations in mg/L for OPTIMA 4300

Element	S0	S1	S2	S3	S4	Stock Concentration
Aluminum	0.0	0.08	0.16	10.0	20.0	2000
Antimony	0.0	0.0096	0.0192	1.2	2.4	240
Arsenic	0.0	NA	0.0064	0.4	0.8	80
Barium	0.0	0.008	0.016	1.0	2.0	200
Beryllium	0.0	0.0004	0.0008	0.05	0.1	10
Boron	0.0	NA	0.008	0.5	1.0	100
Cadmium	0.0	0.0004	0.0008	0.5	0.1	10
Calcium	0.0	NA	0.16	10.0	20.0	2000
Chromium	0.0	0.004	0.008	0.5	1.0	100
Cobalt	0.0	0.0016	0.0032	0.2	0.4	40
Copper	0.0	0.004	0.008	0.5	1.0	100
Iron	0.0	0.032	0.064	4.0	8.0	800
Lead	0.0	0.004	0.008	0.5	1.0	100
Lithium	0.0	0.008	0.016	1.0	2.0	200
Magnesium	0.0	0.08	0.16	10.0	20.0	2000
Manganese	0.0	0.004	0.008	0.5	1.0	100
Molybdenum	0.0	0.008	0.016	1.0	2.0	200
Nickel	0.0	0.004	0.008	0.5	1.0	100
Phosphorus	0.0	0.08	0.16	10	20	2000
Potassium	0.0	0.4	0.8	50.0	100.0	10000
Selenium	0.0	NA	0.0062	0.4	0.8	80
Silicon	0.0	0.04	0.08	5.0	10.0	1000
Silver	0.0	0.0032	0.0064	0.4	0.8	80
Sodium	0.0	0.4	0.8	50.0	100.0	10000
Strontium	0.0	0.008	0.016	1.0	2.0	200
Thallium	0.0	NA	0.008	0.5	1.0	100
Tin	0.0	0.008	0.016	1.0	2.0	200
Titanium	0.0	0.008	0.016	1.0	2.0	200
Vanadium	0.0	0.008	0.016	1.0	2.0	200
Zinc	0.0	0.008	0.016	1.0	2.0	200

NA = S1 is not used in construction of the calibration curve for this element.
The concentration of S2 is at or below the reporting limit for this element.

Table 7.5.1
Initial and Continuing Calibration Standards Concentration in mg/L

Element	ICV/CCV	Stock Concentration
Aluminum	10.0	2000
Antimony	1.2	240
Arsenic	0.4	80
Barium	1.0	200
Beryllium	0.05	10
Boron	0.5	100
Cadmium	0.05	10
Calcium	10.0	2000
Chromium	0.5	100
Cobalt	0.2	40
Copper	0.5	100
Iron	4.0	800
Lead	0.5	100
Lithium	1.0	200
Magnesium	10.0	2000
Manganese	0.5	100
Molybdenum	1.0	200
Nickel	0.5	100
Phosphorus	10.0	2000
Potassium	50.0	10000
Selenium	0.4	80
Silicon	5.0	1000
Silver	0.4	80
Sodium	50.0	10000
Strontium	1.0	200
Thallium	0.5	100
Tin	1.0	200
Titanium	1.0	200
Vanadium	1.0	200
Zinc	1.0	200

Table 7.7.1
ICSA and ICSAB Concentration in mg/L

Element	ICSA	ICSAB	Stock Concentration
Aluminum	250	250	5000
Antimony	-	0.5	100
Arsenic	-	0.25	50
Barium	-	0.25	50
Beryllium	-	0.25	50
Cadmium	-	0.5	100
Calcium	250	250	5000
Chromium	-	0.25	50
Cobalt	-	0.25	50
Copper	-	0.25	50
Iron	100	100	2000
Lead	-	0.5	100
Magnesium	250	250	5000
Manganese	-	0.25	50
Nickel	-	0.5	100
Potassium	-	5	1000
Selenium	-	0.25	50
Silver	-	0.5	100
Sodium	-	5.0	1000
Thallium	-	0.5	100
Vanadium	-	0.25	50
Zinc	-	0.5	100

Table 13.1
Quality Control Criteria Total Metals - ICP
Method 6010B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial calibration	Daily at beginning Of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL or < MDL x 2 (1)	Stop analysis, investigate, reanalyze. If still > limit, qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120% Soil 85 – 115 % Water	Stop analysis, investigate, reanalyze. If still outside limits, qualify data and address in narrative
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	80 - 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check	Run at beginning of each run	80 - 120% of true value for EPA check sample element. < RL or project specific criteria for non-spiked elements	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are analyte specific and instrument specific

Table 13.2
Quality Control Criteria Total Metals - ICP
Method 200.7

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning Of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	95 – 105% RSD < 3%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL	Stop analysis, investigate, reanalyze. If still > limit, qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Water 85 – 115 % (STD) 80 – 120 % (DoD) Soil 80-120 %	Stop analysis, investigate, reanalyze. If still outside limits, qualify data and address in narrative
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per ten samples (20 samples maximum per batch)	80- 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check ICS – A ICS -AB	Run at beginning of each run	Spiked Elements 80 – 120 % Nonspiked Elements <RL (STD) < 2 x MDL (DoD)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike	5%, or minimum of 1 per batch	75 – 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are project specific. Consult QAPP

Table 13.3
Quality Control Criteria Total Metals – ICP
Method 6010C

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Daily, at beginning of run	COC ≥ 0.998	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After ICSA/ICSAB and minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCV.
Initial Calibration Blank (ICB)	After ICV	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)	After every CCV, minimum every 10 samples	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCB.
Low Level Initial Calibration Verification (LLICV)	After ICB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Low Level Continuing Calibration Verification (LLCCV)	Minimum following closing batch CCB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant LLCCV.
Method Blank	Once per batch (20 samples maximum per batch)	< RL or < MDL < ½ RL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	Once per batch (20 samples maximum per batch)	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Once per batch (20 samples maximum per batch)	75 - 125%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (DP)	Once per batch (20 samples maximum per batch) upon client request	RPD ≤ 20%	Qualify data and address in narrative if client specified.
ICP Interference Check Samples (ICSA/ICSAB)	After ICB, prior to sample analysis	80 - 120% for spiked analytes; < RL or other project specific criteria for nonspiked analytes	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post Digestion Spike (PS)	One per batch	80 - 120%	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	One per batch, if sample concentration > 50X MDL	± 10% of original determination	Dilute and repeat post digestion spike.

(1) Acceptance criteria are analyte specific and instrument specific.

Table 13.3.2a
Purchased Standard KEM-SPK-1F from Inorganic Ventures
with the following concentrations:

Element	Concentration (mg/L)
Potassium	250
Sodium	250
Aluminum	50
Calcium	50
Magnesium	50
Silicon	25
Iron	20
Antimony	6
Barium	5
Lithium	5
Molybdenum	5
Strontium	5
Titanium	5
Vanadium	5
Zinc	5
Boron	2.5
Chromium	2.5
Copper	2.5
Manganese	2.5
Nickel	2.5
Lead	2.5
Thallium	2.5
Arsenic	2
Selenium	2
Silver	25
Cobalt	1
Beryllium	0.25
Cadmium	0.25

Uncontrolled Copy

Table 13.3.2b
Final LCS/MS Concentrations

ELEMENT	WATER (mg/L)	SOIL (mg/Kg)
Potassium	25	1250
Sodium	25	1250
Aluminum	5	250
Calcium	5	250
Magnesium	5	250
Silicon	2.5	NA
Iron	2	100
Antimony	0.6	30
Barium	0.5	25
Lithium	0.5	25
Molybdenum	0.5	25
Strontium	0.5	25
Titanium	0.5	25
Vanadium	0.5	25
Zinc	0.5	25
Boron	0.25	12.5
Chromium	0.25	12.5
Copper	0.25	12.5
Manganese	0.25	12.5
Nickel	0.25	12.5
Lead	0.25	12.5
Thallium	0.25	12.25
Arsenic	0.2	10
Selenium	0.2	10
Silver	0.2	10
Cobalt	0.1	5
Beryllium	0.025	1.25
Cadmium	0.025	1.25
Tin*	0.5	25
Phosphorus*	5	250
Zirconium*	0.5	25

*Added as needed from single element solutions.

Figure 12.1

Example 6010 Calculations
Perkin Elmer Optima 4300 DV

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and three standards.

2.0 Calculating the concentration © of an element in water using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system in ug/mL (ppm)	0.08
Vf = Final volume (mL)	50
Vi = Initial volume (mL)	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/mL (mg/L)	0.1

3.0 Calculating the concentration © of an element in soil using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where**:**	**Example:**
Cs = Concentration computed by the data system (mg/L) (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial weight (g)	1
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/g (mg/kg)	5

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where**:**	**Example:**
Cx = Concentration calculated as received (wet basis)	5
Px = Percent solids of sample (% wt)	80
Cdry = Concentration calculated as fry weight (mg/kg)	6.25

Figure 14.1

Checklist ID: 62336

Microbac Laboratories Inc.

Data Checklist

Date: 12-DEC-2011
Analyst: KHR
Analyst: NA
Method: 6010
Instrument: PE-ICP2
Curve Workgroup: 384458
Runlog ID: 44204
Analytical Workgroups: 384170, 383839, 384366, 383507, 384440

Calibration/Linearity	X
ICV/CCV	X
ICV RSD <= 3% (EPA 200.7 only)	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	X
Case Narrative	X
Client Forms	X
Level X	
Level 3	208
Level 4	760, 028, 230
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	KHR
Secondary Reviewer	SLP
Comments	

Primary Reviewer: 13-DEC-2011

Secondary Reviewer: 14-DEC-2011

CHECKLIST1 - Modified 03/05/2008
Generated: MAR-02-2012 16:34:18

Microbac

Uncontrolled Copy

MICROBAC®

STANDARD OPERATING PROCEDURE
THERMO iCAP 6000/7000 SERIES
INDUCTIVELY COUPLED PLASMA
ATOMIC EMISSION SPECTROSCOPY
SW-846 METHOD 6010 / EPA 200.7

Issue / Implementation Date: 15 September 2014

Last Review Date: 15 September 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Maren M Beery — Maren M. Beery, Metal Supervisor — 09/03/2014 — Date

Wade T. DeLong — Wade T. DeLong, Quality Assurance Officer — 9-5-14 — Date

Leslie S. Bucina — Leslie S. Bucina, Laboratory Manager — 9/5/14 — Date

Document Control # 265

Issued to: Document Master File

TABLE OF CONTENTS

SECTION		PAGE
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	5
4.0	Method Performance	5
5.0	Interferences and Corrective Action	7
6.0	Equipment and Supplies	7
7.0	Standards and Reagents	8
8.0	Diagram or Table to Outline Procedures	11
9.0	Sample Preparation	12
10.0	Calibration Procedures	12
11.0	Analytical Procedures	13
12.0	Details of Calculations	17
13.0	Quality Control Requirements	19
14.0	Data Review and Reporting Requirements	23
15.0	Preventive Maintenance	24
16.0	Waste Management and Pollution Control	25
17.0	References	26
	Appendices	28
	Tables	33
	Figures	49

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure covers the operation of a Thermo iCAP 6000/7000 Series Inductively Coupled Plasma spectrometer according to methods 6010B, 6010C, and 200.7 for the analysis of metals in digested soils, sludges, wastes, extracts and waters. Filtered waters preserved in acid may also be analyzed by this method.

1.2 For elements for which this method is applicable see Table 1.1.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Section 4.0 provides concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods (See Microbac SOPs ME401A, ME406 and ME407). The instrument is prepared for operation and stabilized through a warmup period. Once stabilized the instrument is calibrated and initial quality control elements are analyzed. When initial quality control requirements have been met sample analysis begins. Samples are analyzed for metals content by optical emission spectroscopy. The Thermo iCAP 6000/7000 optical design combines a high resolution echelle polychromometer with an upgraded charge injection device CID 86 chip detector. It is also equipped with a solid state dual-view RF generator. The instrument software provides real-time results for the analysis and allows for automated upload into the Laboratory LIMS system for editing and reporting purposes. iTEVA software is completely Windows based and easy to use. A basic understanding of windows operation enables the analyst to move throughout the software.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

AVS	Acid Volatile Sulfides
CCB	Continuing calibration blank
CCV	Continuing calibration verification
CID	Charge Injection Devise
COC	Coefficient of Correlation
DI water	Deionized water
HCl	Hydrochloric acid
HNO3	Nitric Acid

ICB	Initial calibration blank
ICV	Initial calibration verification
ICP	Inductively Coupled Plasma
IDL	Instrument Detection Limit
LCS	Laboratory control sample
LCSD	Laboratory control sample Duplicate
LIMS	Laboratory Information Management System
LLCCV	Low level continuing calibration verification
LLICV	Low level initial calibration verification
LOD	Limit of detection
LOQ	Limit of Quantitation
MDL	Method detection limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectrometry
QC	Quality Control
RGT	Reagent
RL	Reporting limit
SEM	Simultaneously Extracted Metals
SOP	Standard Operating Procedure
STD	Standard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP

2.0 SAFETY PRECAUTIONS

2.1 The iCAP 6000/7000 is equipped with safety interlocks to protect the analyst and instrument from harm if something out of the ordinary happens. The system checks water flow, argon pressures, sample compartment door interlocks, and plasma stability. These interlocks are constantly monitored and displayed on the screen. If any interlock is interrupted, the plasma is automatically shut down. Never attempt to defeat any interlocks.

2.1.1 The following interlocks must be satisfied in order to ignite the plasma:

- The front door on the sample compartment must be closed
- Argon pressures for the torch must be correct
- Cooling water must be flowing to the RF coil

2.1.2 The following interlocks must not be interrupted while the system is operating:

- Argon or nitrogen purge for the detectors and the spectrometer optics must be functioning properly.
- The temperature of the optics housing must be 36-40° C
- The temperature of the generator must be less than 24° C
- The camera temperature must be -45°C to -50°C.

2.2 **WARNING:** Due to the high voltages and temperature, caution must be used in maintenance and troubleshooting.

2.3 **WARNING:** Use gloves, safety glasses, lab coats and/or other appropriate safety precautions when handling samples and reagents. Acids used in preparation and analysis of samples for metals are corrosive and may cause severe burns to body tissue. All samples are unknowns and must be treated as potentially hazardous.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Vol./Wt. Req.*	Collection Vol./Wt.	Preservative	Holding Time**
Total recoverable	50 mL	250 - 1000 mL p	HNO3 to pH <2	6 months
Dissolved	50 mL	250 - 1000 mL p	HNO3 to pH <2 Filter on-site	6 months
Suspended	50 mL	250 - 1000 mL p	Filter on-site	6 months
Total	50 mL	250 - 1000 mL p	HNO3 to pH <2	6 months
Soil	1 g	200 g glass	≤6° C	6 months

p = plastic

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

4.0 METHOD PERFORMANCE

Instrument Detection Limits

4.1 6010B/200.7 IDLs

6010B/200.7 IDLs are calculated by multiplying the standard deviation obtained from analysis of a reagent blank solution with seven consecutive measurements by the one-sided 98% confidence level t-statistic (3.14 is the t-statistic for seven

samples). Each measurement is performed as though it were a separate sample (i.e, with rinsing in between).

IDLs must be determined quarterly. The IDL is required to be numerically less than the associated MDL.

6010C IDLs

6010C IDLs are estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement is performed as though it were a separate sample (i.e., with rinsing in between). IDLs must be determined quarterly.

Method Detection Limits

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 4.1a for 6010B/200.7 and Table 4.1b for 6010C. These limits were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project RLs may be higher.

4.5 Precision and accuracy data in Tables 4.2a and 4.2b were derived from an initial demonstration of capability using spiked control samples. Going forward, the laboratory will use results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 MDLs must be redetermined whenever there is any change to the sample preparation procedure, or any significant change to the instrument

Linear calibration ranges

4.6 Calibrate the instrument, as described in Section 10.0.

4.7 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

Uncontrolled Copy

4.8 The linear range is defined as the highest concentration where the measured value is within 10 % of the actual prepared value of the standard. The values reported in Tables 4.1a and 4.1b are 90% of the verified upper linear range.

4.9 Linear Dynamic Range verifications are analyzed quarterly and edited in KOBRA and in the instrument software.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 IEC's (InterElement Corrections) are minimized by the use of the eschelle grating. However this grating does not eliminate all interferences due to spectral overlap. Table 5-1 lists the approximate IEC's necessary of analyte per unit (mg/L) of interferant for the Thermo iCAP 6000/7000. These IEC's are subject to change with modifications made to operating conditions, such as changes in coolant flow, power, nebulizer or even new torches.

5.2 The use of a peristaltic pump reduces physical interferences. However, samples with high dissolved solids, high acid concentration or high viscosity may need to be diluted.

6.0 EQUIPMENT AND SUPPLIES

6.1a Thermo iCAP 6000 with ESI SC-8 DXFAST autosampler.

6.1b Thermo iCAP 7000 with CETAC ASXpress 520 autosampler.

6.2 Argon gas supply (liquid).

6.3 Dell intel Core 2 VPRO or DUO computer with Thermo Fisher Scientific iTEVA Analyst software.

6.4 IBM compatible printer

6.5 Peristaltic pump tubing

6.5.1 Black/Black – 0.32 mL/min (Sample and flush station).

6.5.2 Yellow/Orange – 0.51 mm I.D > (Internal Standard)

6.5.3 Red/Red – 1.14 mm I.D. (drain)

6.6 Mixing tee

6.7 Calibrated mechanical pipettes:

6.7.1 10 - 100 uL

6.7.2 100 - 1000 uL

6.7.3 1000 - 5000 uL

6.8 Metal-free plastic pipette tips (for the pipettes specified in 6.7)

6.9 Metal-free 15 mL plastic test tubes

6.10 Metal-free 50 mL plastic test tubes

6.11 Class A glass pipettes – for preparation of standard solutions

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used.

All purchased stock solutions and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

Calibration Solutions

7.2 Mixed calibration stock standards are purchased from SPEX as XKES-5-250 and from SCP Science as custom mixes Plasmacal 1 and Plasmacal 3A or equivalent.

7.3 10,000 ug/mL silicon - Inorganic Ventures or equivalent.

7.4 10,000 ug/mL phosphorus – Inorganic Ventures or equivalent.

7.5 1000 ug/mL zirconium – Inorganic Ventures or equivalent.

7.6 Working Calibration Solutions - For the high standard water matrix, dilute 5 mL each of 7.2 and 7.3, 1 mL of 7.4 and 1 mL of 7.5 into 500 mL of 5% HCl 2% $HN0_3$ in DI water. For the soil matrix dilute into 500 mL of 5% HCl and 5% HNO_3

in DI water. From the high standard dilutions are made with the appropriate matrix matched water. Standard S0 is a matrix matched blank. Concentrations of each element in the calibration standard are given in Table 7.4.1 for Thermo iCAP 6000/7000.

7.7 Working CCV Solutions – The CCV is prepared for two different matrices. For the water matrix, dilute 5 mL each of 7.2 and 7.3, 1 mL of 7.4 and 1 mL of 7.5 into 1000 mL of 5% HCl and 2% $HN0_3$ in DI water. For soil matrix, dilute into 1000 mL 5% HNO_3 and 5% HCl in DI water. Concentrations for each element are listed in Table 7.5.1.

7.8 Working Calibration Blank Solution - A calibration blank of 5% HNO_3, 5% HCl for soil and 2% HNO_3, 5% HCl for water is used to establish the analytical curve and is analyzed following each initial and continuing calibration standard analysis.

Initial Calibration Verification Solutions

7.9 The ICV concentration is at the same level as the midpoint standard used in the calibration and is a separately prepared, quality control analyzed and certified source from that of the calibration standards.

7.10 Mixed ICV stock solution is purchased from Inorganic Ventures as a KEM-CONC – 1A,2,3A standard set or equivalent.

7.11 10,000 ug/mL phosphorus – CPI or equivalent.

7.12 1000 ug/mL zirconium – CPI or equivalent.

7.13 Working ICV Solution – The ICV is prepared for two different matrices. For the water matrix, dilute 0.2 mL of 7.10, 0.2 mL of 7.11 and 0.2 mL of 7.12 into 200 mL of 5% HCl and 2% HNO_3 in DI water. For soil matrix, dilute into 200 mL 5% HNO_3 and 5% HCL in DI water. Concentrations for each element are listed in Table 7.5.1.

Low Level Calibration Verification Solutions

7.14 The LLICV and LLCCV are analyzed for 6010C only. The LLICV and LLCCV are analyzed using the same solution with concentrations at or below the RL.

7.15 Mixed LLCV stock solution is purchased from Inorganic Ventures or equivalent.

7.16 For the LLICV/LLCCV water matrix, dilute 1 mL of 7.15 into 500 mL of 5% HCL and 2% HNO_3 in DI water. For the soil matrix, dilute 1 mL of 7.15 into 500 mL

of 5% HCL and 5% HNO_3 in DI water. Concentrations of each metal are listed in Table 7.6.1.

Interference Check Sample Solutions

7.17 Interference Check Sample Stock - An ICSA stock solution is purchased from Inorganic Ventures as CLPP-ICS-A or equivalent.

ICSAB standards are purchased from Inorganic Ventures as CLPP-ICS-A, CLPP-ICS-B and KEM-ICS-B-1A or equivalent.

7.18 Working Interference Check Sample Solutions -The ICSA is prepared for 2 different matrices. For the water matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 2% HNO_3 in DI water. For the soil matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSA solution will contain only aluminum, calcium, iron and magnesium in high concentrations. See Table 7.7.1.

The ICSAB is made for 2 different matrices. For the water mixture dilute 10 mL of CLPP-ICS-A, 1 mL of CLPP-ICS-B and 1 mL of KEM-ICS-B-1 into 200 mL 5% HCl and 2% HNO_3 in DI water. For the soil matrix dilute same volumes of the ICSAB into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSAB will have the same concentrations for aluminum, calcium, iron and magnesium as the ICSA with additional metals spiked at detectable levels. See Table 7.7.1.

Internal Standard Solutions

7.19 Internal Standard Stock - single element yttrium 10,000 ug/mL from Inorganic Ventures or equivalent.

7.20 Working Internal Standard Solution – 25 mg/L yttrium prepared by diluting 1 mL of Y stock into 2000 mL of the appropriate matrix matched acid water.

7.21 Concentrated hydrochloric acid (HCl). Baker Instra Analyzed Grade or better.

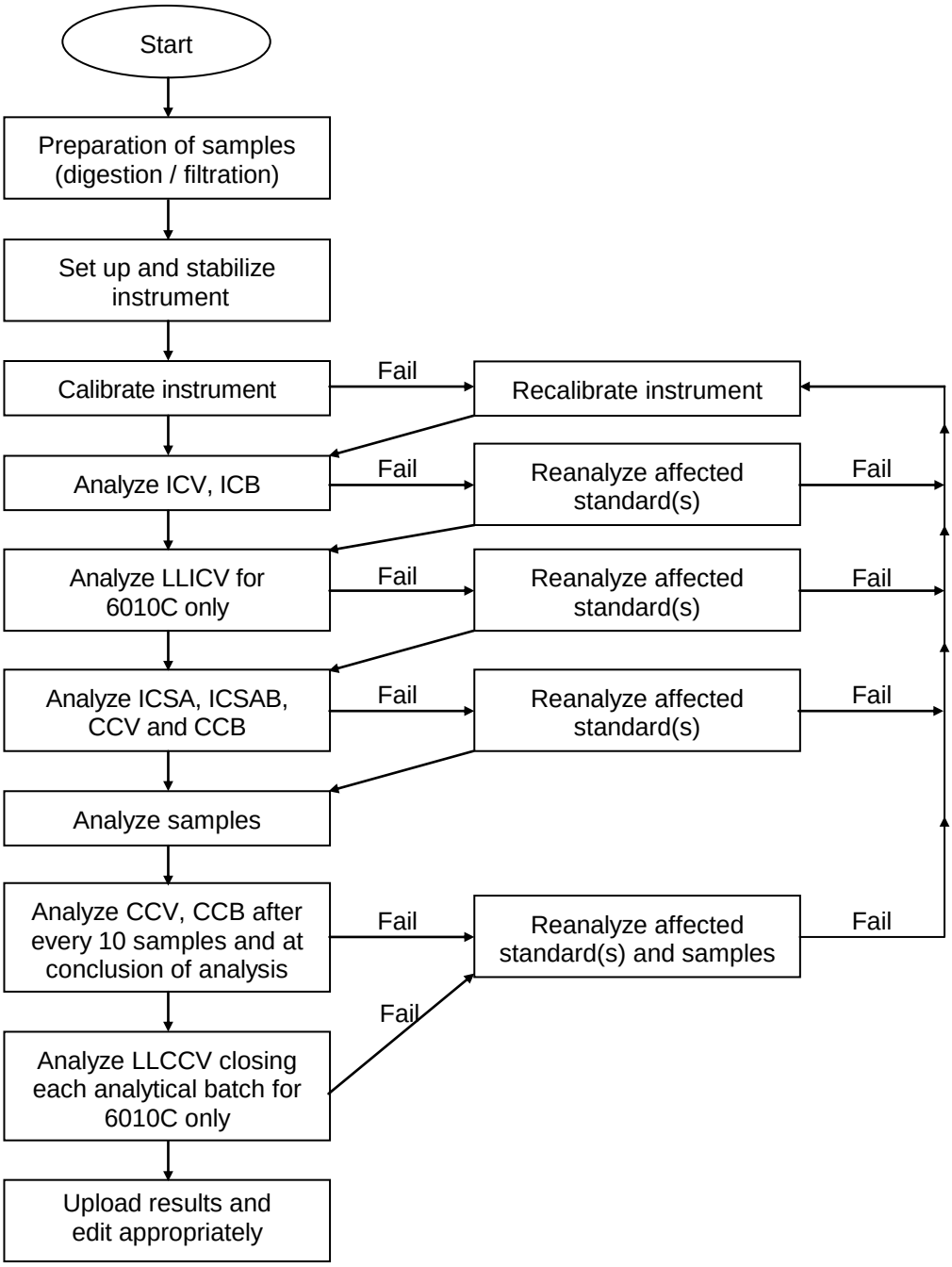
7.22 Concentrated nitric acid ($HNO_{3)}$. Baker Instra Analyzed Grade or better.

7.23 DI water – ASTM Type II or equivalent (ASTMD 1193)

7.24 4 % HCl Solution – To approximately 500 mL of DI water add 40 mL conc. HCl. Dilute to 1000 mL final volume with DI water.

7.25 Custom Multielement Solution MIC-SPK-1A from Inorganic Ventures or equivalent.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

- ME401A - Preparation of Dissolved Metals in Waters for Direct Analysis by Inductively Coupled Plasma Spectroscopy (EPA 200.7)
- ME406 - Microwave Digestion of Sediments/Sludges/Soils/Oils, SW-846 Method 3051
- ME407 - Microwave Digestion – Aqueous, SW-846 Method 3015

10.0 CALIBRATION PROCEDURES

10.1 Initial Calibration – The instrument is calibrated before analysis of any samples with a blank and four calibration standards or alternatively with a blank and the high standard S4 from 7.6 (6010C only). Calibration Standards are prepared from SCP Science and SPEX stock. The dilutions of the calibration standards are listed in Section 7.9. The concentrations are listed in Table 7.4.1. For the multipoint calibration the instrument performs a weighted linear regression with a calculated intercept for all analyzed elements. For the single point calibration the instrument performs a linear regression for all analyzed elements (See Appendix A for calibration algorithms.). The correlation coefficients can be printed out when calibration is complete. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

When performing the multipoint calibration the low calibration standard must contain the elements of interest at concentrations at or below the RL or a low level calibration check standard at or below the RL must be analyzed after calibration and before sample analysis. The LLICV must always be analyzed for 6010C. See Tables 13.1, 13.2 and 13.4 for acceptance criteria and corrective action for the curve and low level calibration check standard.

10.2 ICV Analysis - ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13.1, 13.2, and 13.4 for acceptance criteria and corrective actions.

10.3 CCV Analysis - The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13.1, 1 3.2, and 13.4 for acceptance criteria and corrective action.

10.4 ICB and CCB Analysis - The solution used is the calibration blank (7.11). See Tables 13.1, 13.2, and 13.4 for acceptance criteria and corrective action.

10.5 ICSA and ICSAB Analysis – Required at the beginning of analytical run. See Tables 13.1, 13.2 and 13.4 for acceptance criteria and corrective action.

10.6 LLICV and LLCCV Analysis – For 6010C only, is required at the beginning of the run (LLICV) and at a minimum after every analytical batch (LLCCV). See Table 13.4 for acceptance criteria and corrective action.

10.7 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices.

11.2 Startup Procedures

11.2.1 Pump tubing on instrument must be pliable and have only slight discoloration. If tubing is worn, replace. If tubing appears to be in good condition or once new tubing is installed, then connect the pump winding.

11.2.2 Turn on autosampler and chiller.

11.2.3 Turn on computer. Instrument and argon remain on at all times except when the instrument is to be shut down for an extended period of time.

11.2.4 Open ESI Software by double clicking on the icon. Click "Initialize Autosampler". Open iTEVA Control software by double clicking on the icon, enter analyst user name, click "OK". Wait for the "Initializing Instrument" procedure to complete.

11.2.5 Set up the instrument with the proper operating conditions. These conditions are found under Source Settings:

Plasma Settings:

- Flash Pump Rate 10 rpm

- Analysis Pump Rate 10 rpm
- Pump Stabilization time 0 sec

Source Settings:

- RF Power 1300W
- Auxiliary Gas Flow 0.70 L/min
- Nebulizer Gas Flow 0.65 L/min
- Coolant Gas Flow 12 L/min
- Water circulation on

11.2.6 After instrument is initialized, click on [Ignite] tab. This will open the "Plasma Status" box. Click on the "Plasma On", releasing the argon by lifting up the button from the Argon Humidifier to start the ignition sequence. After plasma has ignited, the setting will read approximately:

R.F. Power	1300 watts
Auxiliary Flow	0.7 L/min
Nebulizer Flow	0.65 L/min
Pump Rate	10 rpm
Coolant Gas Flow	12 L/min
Purge Gas Flow	Normal

11.2.7 The ICP must be allowed to become thermally stable before beginning analysis. (This usually requires approximately 30 minutes of operation prior to calibration.)

11.3 Setup of Analytical Sequence

11.3.1 After igniting plasma click the "Analyst" icon on iTEVA Control Center. This will open the "Select a Method" screen. Highlight the appropriate method and click [OK].

11.3.2 To set up the sample table Click on the "Sequence" tab. Right click on "Untitled(Manual)". Click on "Modify", in "Modify Automation Session" Box, choose "Autosampler", depending on which autosampler is installed choose "CETAC by ESI SC8" (Emulation by ESI) or "CETAC ASX-520 5x12 by Col Racks". Click on [New], this will open "New Sequence" box. Highlight "Import from Delimited Text File" and choose file "FAST". Click [OK] "New Sequence" box, click [OK] on "Modify Automation Session" box. This will open the screen where sample information is entered. Make sure that work group numbers are filled in the "Comment" column and dilution factors are filled in "Custom ID1" column. From "auto-session" tool bar, save the sample file.

Uncontrolled Copy

11.3.3 To edit the Autosampler Table, double click "CETAC by ESI SC8" or "CETAC ASX-520 5x12 by Col Racks" depending on which autosampler is installed, and double click "S_ICP-THERMO". Positions for standards and samples will appear. Connect the autosampler to the PC by clicking on the "Initialize" icon in the sequence tab.

11.3.4 Click "Auto-Session" on the screen to open the appropriate sample file and make sure positions for samples are highlighted. Double click the second "Untitled CETAC by ESI SC8, then double click "S_ICP-THERMO". The number of standards and samples will appear. Right click on "Standards, then click "Auto-Locate All" to make sure positions for standards are highlighted.

11.3.5 Torch Alignment: The torch alignment should only be performed after the torch has been replaced and before the auto peak. Manually place the carrier probe and the internal standard probe into a 2 mg/L zinc solution. Click the "Analysis" tab, select "Torch Alignment" from the instrument menu. Aspirate the zinc solution and click run. This procedure takes approximately ten minutes. After the torch alignment is complete, rinse both the carrier and internal standard probes with DI water before returning them back to their solutions.

11.3.6 Auto Peak: The peak location for the method lines associated with each high standard need to be justified. Manually place the carrier probe in the S4 cup. Click the "Analysis" tab, select "Perform Auto Peak Adjust" from the instrument menu, which presents the "Auto Peak Adjust" dialog box. Select "All Elements", aspirate the high standard, and click run. Allow a delay after failures before re-initializing the auto peak routine. After the auto peak is complete, rinse the probe with DI water, then return the probe back to the carrier solution.

11.3.7 On the method tab, click "Automated Output", change appropriate file name, click "Apply to All Sample Types" to set the new file name. Save the method.

11.4 6010/200.7

The prepared calibration standards, ICV, ICB/CCB, ICSA, ICSAB, and CCV are placed into 50 mL cups and are assigned positions by the analyst. This can be printed out by clicking on the "List View" icon in the "Sequence" screen to show sequence page and clicking on "Print Samples List".

6010C only

The prepared calibration standards, ICV, ICB/CCB, ICSA, ICSAB, CCV, LLICV, and LLCCV are placed in 50 mL cups and are assigned positions by the analyst. This can be printed out by clicking on the "List View" icon in the

"Sequence" screen to show sequence page and clicking on "Print Samples List."

11.4.1 To start calibration, click on "Run-Auto-Session" in the "Sequence" screen.

11.4.2 To print correlation coefficients, click on "Element Calibration Report" from the "Method" screen. Right click on mouse in the [Calibration Report] screen, clicking [Print]. The correlation coefficients must be 0.995 or better for all elements needed for analysis for 6010B/200.7 and 0.998 or better for 6010C. Calibration is automatically stored until a new calibration is performed.

11.5 The prepared calibration standards, ICV, ICB/CCB, ICSA, ICSAB, CCV, LLICV, and LLCCV are analyzed in three replicates with the reported results being the average of the three replicate readings.

11.6 Upon completion of the calibration, begin following the analytical sequence as described below for:

6010B/200.7
ICV
ICB
ICSA
ICSAB
CCV
CCB
10 or less samples
CCV
CCB
Continue with the last three (3) steps until the end of run, always ending with a CCV/CCB.

6010C
ICV
ICB
LLICV
ICSA
ICSAB
CCV
CCB
10 samples or less
CCV
CCB
LLCCV
Continue with the last three (3) steps until the end of the run, always ending with a CCV, CCB, and LLCCV.

11.6.1 The samples are analyzed in three replicates with the reported results being the average of the three replicate readings. For any analyte with a result greater than the reporting detection limit, the %RSD between the replicate readings must be less than ten.

11.7 Flush the system with the rinse solution for thirty (30) seconds before the analysis of each sample. Analyze the CCV and CCB after each 10 samples at a minimum, and at the end of each analytical batch for 6010B/200.7. Analyze the

CCV and CCB after every 10 samples at a minimum, and the CCV, CCB and LLCCV at the end of each analytical batch for 6010C.

11.8 Dilute and reanalyze samples that are more concentrated than the linear dynamic range.

11.9 After all sample analyses are complete, rinse the system by analyzing acid water and DI water, followed by air. Extinguish the plasma and release the pump tension. Turn off the autosampler.

11.10 Exit software and turn off computer.

12.0 DETAILS OF CALCULATIONS

12.1 All calculations for samples and standards are computed from the mean of three exposures. Each metal has a specified linear range. Refer to Section 4.0 for upper limits.

12.2 After the multipoint calibration is complete, the software performs a weighted linear regression with a calculated intercept for all metals analyzed. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance.

Following a single point calibration, the software performs a linear regression with a calculated intercept. Calculated correlation coefficients in this case are all equal to 1.0 (See Appendix A for the calibration algorithms.). All sample results are calculated from the calibration curve.

12.3 Dilution factors and preparation factors are calculated into the final result which is computed from the mean of three exposures.

12.3.1 For Liquid Samples:

$$mg/L\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume\ (mL)}{Initial\ Volume\ (mL)} * \frac{Total\ Diluted\ Volume}{Sample\ Aliquot}$$

12.3.2 For Solid Samples:

$$mg/Kg\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume\ (mL)}{Initial\ Weight\ (g)} * \frac{Total\ Diluted\ Volume}{Sample\ Aliquot}$$

12.3.3 LCS/LCS Duplicate % Recovery

$$\%\,Recovery = \frac{C_s}{C_t} * 100\%$$

where:

C_s = the LCS sample result
C_t = the LCS true value

12.3.4 MS/MSD % Recovery

$$\%\,Recovery = \frac{(C_s - C_a)}{C_t} * 100\%$$

where:

C_s = the MS/MSD sample result
C_a = the reference sample result
C_t = the MS/MSD true value

12.3.5 Post Digestion Spike Recovery

$$\%\,Recovery = \frac{(C_s - BC_a)}{C_t} * 100\%$$

where:

C_s = the spike sample result
C_a = the reference sample result
B = a factor to account for the dilution of the spiked sample relative to the reference sample (usually B = 0.9)
C_t = the spike true value

12.3.6 Duplicate RPD

$$RPD = \frac{|C_a - C_b|}{(C_a + C_b)/2} * 100\%$$

where:

C_a = the reference sample result

C_b = the duplicate sample result

12.3.7 Serial Dilution % Difference

$$\%D = \frac{|C_a - 5C_b|}{C_a} * 100\%$$

where:

C_a = the reference sample result
C_b = the diluted sample result

12.4 Calcium and magnesium results can be used to calculate water hardness results. The LIMS will use the uploaded calcium and magnesium results to calculate the hardness results. The automated hardness calculation is manually verified annually.

$$Hardness(mg/L) = 2.497[Ca(mg/L)] + 4.118[Mg(mg/L)]$$

12.5 Silicon results can be used to calculate silica results. The calculation assumes all silicon recovered from 6010 is in the form Si02. KOBRA converts the results as follows:

$$Silica(mg/L) = silicon(mg/L) * 2.13923$$

12.6 See figure 12.1 for a sample calculation summary.

13.0 QUALITY CONTROL REQUIREMENTS

Overview

13.1 Refer to Section 10.0 for instrument calibration and instrument quality control samples. Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC samples. The QC samples are prepared and digested identically to the analytical samples. The following QC are digested and or analyzed with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Tables 13.1, 13.2 and 13.4.

Batch Quality Control

13.2 Method blank (Prep Blank (PB)) an aliquot of DI water that is digested with the sample batch and contains all reagents identical with the samples.

13.3 LCS - for water matrix, a spiked DI water that is digested with the sample batch. For soil matrix; 1.0 gram of PTFE Boiling Stones (Teflon) Chemware is spiked prior to digestion and carried through the entire digestion procedure.

A LCSD may also be analyzed with the batch and is prepared identically to the LCS.

(**NOTE**: The LCS and LCSD must pass acceptance criteria or the samples will have to be re-digested/re-analyzed for the analyte in question.)

13.4 Sample duplicate - a sample prepared in duplicate, both carried through the digestion procedure. (EPA 200.7 or by client request only)

13.5 MS and MSD - a sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spiked with 5 mL of KEM-SPK-1F for each 50 mL of sample. The final concentration spiked into the two spiked samples will be the same as the aqueous and soil LCS level in Table 13.3.2.

Interference Tests-Post Digestion

13.6 With every analytical batch a post digestion spike and a serial dilution must be analyzed. If the matrix spike fails the 75 to 125% acceptance criteria, one of the following interference tests must pass for the analyte outlier:

13.7 Serial dilution: If the analyte concentration is sufficiently high (at least a factor of 50 above the MDL before dilution), analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.8 Post Digestion Spike: an analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% of the known value for 6010B/200.7 and within 80 to 120% of the known value for 6010C. The spike addition should produce a minimum level of 10 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.9 Method of standard additions: This method must be employed for certain TCLP extracts and can also be used to compensate for sample constituents which enhance or depress the analyte signal producing a slope different from that of the calibration standards. It will not correct for additive interferences which cause a base line shift.

The simplest version of this techniques is the single-addition method, in which two identical aliquots of the sample, each of volume Vx are taken. To the first

(A) a small volume (Vs) of a standard analyte concentration (Cs) is added. To the second (B) a volume (Vs) of the solvent is added. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration (Cx) is calculated:

$$Cx = (Sb)(Vs)(Cs)/(Sa - Sb)(Vx)$$

where:

Sa and *Sb* are the analytical signals of A and B respectively. *Vs* and *Cs* should be chosen so that *Sa* is roughly twice *Sb* on the average. It is best if *Vs* is made much less than *Vx*, and thus *Cs* is much greater than *Cx*, to avoid excess dilution of the sample matrix. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected concentration from the endogenous analyte in the sample.

Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample concentration. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero concentration, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

13.10 Linear Range Analysis/High-Level Standard

A linear range analysis is performed quarterly to determine the highest concentrations for each analyte at which the instrument yields a result within 10% of the true concentration. When use of the Department of Defense version 3 QAP is indicated, a high-level check standard with analyte concentrations at the linear range of the instrument may be analyzed subsequent to sample analysis for any analyte which exceeds the calibration range. The recovery must be within 10% of the true concentration.

13.11 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP

(Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.12 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1, 13.2 or 13.4, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per 13.1, 13.2 or 13.4.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch, (Figure 14-1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.13 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.14 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.15 All data is scrutinized by the analysts for method and project specific compliance. Check lists are utilized and accompany each data batch (figure 14-1).

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client samples into KOBRA. This is done via Microbac's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and RLs. Any elements that are not to be reported must be checked as excluded. This will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1, 13.2, and 13.4. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating**.** The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the hardcopy QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and KOBRA workgroup reports. The Data Review Checklist acts as a cover page. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. This review consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness. The supervisor (or designate) also signs and dates the Data Review Checklist.

When all levels of review have been completed, the elements being reported on each sample are taken to a done status in KOBRA.

15.0 PREVENTIVE MAINTENANCE

15.1 Check the torch and nebulizer every day and clean when needed depending on sample load.

15.2 Tubing needs to be changed when it loses pliability and is worn.

15.3 Clean the valve and all tubing when needed depending on sample load. Replace the rotor every six months or every 15,000 samples, whichever comes first.

15.4 The instruments are under service contracts so that every year a service representative will perform a systems check.

15.5 The water in the re-circulator/cooler must be changed yearly.

15.6 Troubleshooting and maintenance procedures can be found in Chapter 13 of the iCAP 6000 Series ICP-OES Spectrometer Hardware Manual and in the Help Menu of the iTEVA Analyst Software Control Center.

15.7 Performance problems (such as loss of signal or poor precision) are often related to the sample introduction system.

15.7.1 Perform the sodium bullet test to monitor sample flow.

15.7.2 Check pump tubing for flat spots, leaks or discoloration.

15.7.3 Check pump rollers for grooves or binding.

15.7.4 Check pump tubing for crimps, pinching and clogging.

15.7.5 Check nebulizer and spray chamber for leakage and proper drainage.

15.7.6 Check nebulizer spray pattern. The spray must not be uneven or sputtering.

15.7.7 Check the torch and injector for deposits, deformation and security of fit. O-rings must not be cracked or worn.

15.7.8 Check that the purge window is not cloudy or dirty.

15.8 The instrument configurations for the Thermo iCAP 6000/7000 series instruments can be found in the corresponding maintenance run logs. The template maintenance logs are found at numbers 13196 (ICP-Thermo1), 13199 (ICP-Thermo2), and 51236 (ICP-Thermo3) in KOBRA. Replacement of instrument components will be recorded in the relevant maintenance log and updated in the log header as needed.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams for the Metals Laboratory are as follows:

The metals laboratory waste is neutralized with sodium bicarbonate and flushed down the drain with tap water as per agreement with the Marietta Department of Water and Waste Water.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous waste generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Inductively Coupled Plasma – Atomic Emission Spectrometry, US EPA SW-846 Method 6010B, Revision 2, December 1996, EPA Publication SW-846

17.2 Us Environmental Protection Agency, EPA Method 200.7, Revision 4.4, 1994, 40CFR pt.136.

17.3 Inductively Coupled Plasma-Atomic Emission Spectrometry, US EPA SW-846 Method 6010C, Revision 3, February 2007, EPA publication SW-846.

17.4 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.5 Microbac SOP 45 “Method Validation Procedures”

17.6 Microbac SOP ME401A “Preparation of Dissolved Metals in Waters for Direct Analysis by Inductively Coupled Plasma Spectroscopy (EPA 200.7)”

17.7 Microbac SOP ME406 “Microwave Digestion of Sediments, Sludges, Soils and Oils (3051)”.

17.8 Microbac SOP ME407 “Microwave Digestion – Aqueous SW846-3015”.

17.9 Microbac SOP 33 “Laboratory Waste Management”

17.10 Microbac SOP GP-CAPA “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.11 Microbac SOP GP-RCA “Root Cause Analysis”

17.12 Microbac SOP KAVS “Analytical method for Determination of Acid Volatile Sulfide (AVS) in Sediment”

17.13 USEPA, 1991. Draft Analytical method for determination of Acid Volatile Sulfide in Sediment, EPA-821-R-91-100. Office of Water, U.S. Environmental Protection Agency, Washington, D.C.

Appendix A

Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$$Y=MX+B$$

where:

X = concentration
Y = intensity
M = slope of the calibration curve
B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

N = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$M = \frac{(n)\sum_{n}^{i=1}(X_i Y_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}(X_i^{\,2}) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

[2] $$B = \frac{\sum_{n}^{i=1}(X_i^{\,2})\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_i Y_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}(X_i^{\,2}) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

Linear Calibration Algorithm (continued)

$$COC = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sqrt{\left(\sum x^2 - \frac{\left(\sum x\right)^2}{n}\right)\left(\sum y^2 - \frac{\left(\sum y\right)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

Weighted Linear Calibration Algorithm

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_i x$$

Where y is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

where:

x = concentration value of the standard
y = measured intensity of the standard
n = number of standards
i = index for the standards
b_0 = intercept
b_1 = slope
COC = correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:

$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:

$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Uncontrolled Copy

Weighted Linear Calibration Algorithm (continued)

Correlation Coefficient: $$COC = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^{\,2}\right]\left[(w_1 w_5) - w_4^{\,2}\right]}}$$

Appendix B

Simultaneously Extracted Metals (SEM)

SEM are operationally defined as metals (arsenic, cadmium, copper, lead, mercury, nickel, silver and zinc for example) that form sulfides less soluble than those of iron or manganese and which are at least partially released from the sediment under the conditions for generating Acid Volatile Sulfides (AVS) (see Microbac SOP KAVS). Since sulfide is a precipitant of heavy metals under anoxic conditions, the SEM to AVS ratio obtained by the method is an indicator of heavy metal bioavailability in the sediment sample.

AVS Generation and Filtration

AVS is generated in a reaction cell per the procedure in Microbac SOP KAVS. Once sulfide generation is complete the sediment and water from the reaction cell (Microbac SOP KAVS Section 11.10) is vacuum filtered through Whatman TCLP Glass microfiber Filters Acid Treated Low Metal (or equivalent) into a filtration apparatus that has been washed, rinsed in 0.1 M HNO_3 and then DI water rinsed prior to use.

The filtrate is transferred to B-D 60 mL sterile luer-lok syringes equipped with Corning 0.2 um membrane syringe filters (or equivalent) and filtered into an acid washed, DI water rinsed graduated cylinder.

The filter flask is rinsed with DI water and the rinsates are similarly 0.2 um filtered and added to the cylinder.

DI water is then used to bring the filtrate to a final volume of 250 mL. The sample is transferred to a labeled, certified precleaned 250 mL Nalgene bottle C and G Containers (or equivalent) and analyzed within two weeks.

Metal concentrations are determined by ICP-OES.

Batch QA/QC Samples

Method Blank – An aliquot of 4% HCl in DI water is filtered in the same manner as the samples. This filtrate will be used as the method blank.

LCS – In a digestion tube containing about 20 mL 4% HCl spike 5 mL of spike solution (7.25) and bring to a 50 mL final volume with 4% HCl. Filter the LCS in the same manner as the samples.

MS/MSD – If there are no client designated spike samples, choose one sample from the batch. Into two digestion tubes pour approximately 20 mL of the filtered sample. Add 5 mL of spike solution (7.25) and bring to final 50 mL volume with additional filtered sample.

Interference Tests – A post digestion spike will be prepared as in Section 13.8. Additionally, if appropriate, a serial dilution will be analyzed as in 13.7.

Calibration Solutions

Calibration Solutions, ICV and Interference Check Solutions are prepared as in Sections 7.2 through 7.13 and 7.17 through 7.18 except that the diluent used is 4% HCl (7.24).

The working calibration blank solution used is 4% HCl (7.24).

Analysis and Reporting

Metal concentrations are determined by ICP-OES following the procedures in Section 11.0.

The instrument is calibrated as in 10.1 followed by ICV/ICB, ICSA/ICSAB and CCV/CCB analysis. Acceptance criteria are found in Table 13.1. Samples are analyzed in groups of ten or less followed by CCV/CCB analyses. Concentrations of the metals are reported as umole per gram dry sediment (umole/g).

SEM is then calculated as the sum of the metal concentrations:

$$SEM = \sum [metals]$$

Table 1.1

Name	Symbol	Wavelength	Cas Number
Aluminum	Al	308.2	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	455.4	7440-39-3
Beryllium	Be	313.1	7440-41-7
Boron	B	249.6	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	422.6	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	224.7	7440-50-8
Iron	Fe	261.1	7439-89-6
Lead	Pb	220.3	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	279.0	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Potassium	K	766.4	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	212.4	7440-21-3
Silver	Ag	328.0	7440-22-4
Sodium	Na	589.5	7440-23-5
Strontium	Sr	407.7	7440-24-6
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	337.2	7440-32-6
Vanadium	V	292.4	7440-62-2
Zinc	Zn	206.2	7440-66-6
Phosphorus	P	214.9	7723-14-0
Zirconium	ZR	339.1	7704-67-7
Calculated			
Hardness, Calculated (as $CaCO_3$)	CaCO3	NA	72608-12-9
Silica (as SiO_2)	SiO_2	NA	99439-28-8

Uncontrolled Copy

Table 4.1a
iCAP 6000/7000 MDLS and Linear Ranges for 6010B/200.7

Metal	Linear Dynamic Range (mg/L)	Verified MDL Water (mg/L)	Reporting Limit Water (mg/L)	Verified MDL Soil (mg/Kg)	Reporting Limit Soil (mg/Kg)
Aluminum	810	0.1	0.2	10	20
Antimony	90	0.05	0.1	0.5	1.0
Arsenic	90	0.005	0.01	0.5	1.0
Barium	45	0.005	0.01	0.25	0.5
Beryllium	4.5	0.001	0.002	0.05	0.1
Boron	90	0.05	0.1	2.5	5
Cadmium	9.0	0.0005	0.001	0.05	0.1
Calcium	900	0.25	0.5	25	50
Chromium	45	0.0025	0.005	0.125	0.25
Cobalt	9.0	0.0025	0.005	0.125	0.25
Copper	180	0.0025	0.005	0.5	1.0
Iron	900	0.025	0.1	1.5	3.0
Lead	225	0.005	0.01	0.5	1.0
Lithium	90	0.05	0.1	2.5	5.0
Magnesium	900	0.25	0.5	12.5	25.0
Manganese	27	0.005	0.01	0.25	0.5
Molybdenum	9.0	0.005	0.01	1.5	3.0
Nickel	90	0.01	0.04	1.0	2.0
Potassium	360	0.5	1.0	25	50
Selenium	90	0.01	0.02	0.5	1.0
Silicon	81	0.5	1.0	NA	NA
Silver	4.5	0.005	0.01	0.25	0.5
Sodium	360	0.25	0.5	12.5	25
Strontium	4.5	0.025	0.05	0.25	0.5
Thallium	9.0	0.1	0.2	1.0	2.0
Tin	90	0.25	0.5	12.5	25
Titanium	90	0.015	0.03	1.0	2.0
Vanadium	90	0.005	0.01	0.25	0.5
Zinc	45	0.01	0.02	0.5	1.0
Phosphorus	450	0.5	1.0	25.0	50.0
Zirconium	18	0.025	0.05	1.25	2.5
Silica (as SiO2)	193	1.07	2.14	NA	NA

Table 4.1b
iCAP 6000/7000 MDLs & Linear Ranges for 6010C

Metal	Linear Dynamic Range (mg/L)	Verified MDL, Water (mg/L)	Reporting Limit, Water (mg/L)	Verified MDL, Soil (mg/Kg)	Reporting Limit, Soil (mg/Kg)
Aluminum	900	0.1	0.2	10.0	20.0
Antimony	90	0.05	0.1	2.5	5.0
Arsenic	90	0.005	0.01	0.5	1.0
Barium	90	0.0025	0.01	0.125	0.5
Beryllium	9	0.0005	0.002	0.025	0.1
Boron	45	0.05	0.1	2.5	5.0
Cadmium	18	0.0005	0.001	0.05	0.1
Calcium	900	0.25	0.5	25	50
Chromium	90	0.0025	0.01	0.125	0.5
Cobalt	90	0.0025	0.005	0.125	0.25
Copper	180	0.0025	0.005	0.5	1.0
Iron	900	0.025	0.1	2.0	5.0
Lead	180	0.005	0.01	0.5	1.0
Lithium	27	0.05	0.1	2.5	5.0
Magnesium	900	0.25	0.5	12.0	25.0
Manganese	180	0.005	0.01	0.1	0.5
Molybdenum	45	0.005	0.1	2.0	5.0
Nickel	90	0.005	0.04	0.5	2.0
Potassium	315	0.25	1.0	25.0	50.0
Selenium	90	0.01	0.02	0.5	1.0
Silicon	90	0.25	1.0	NA	NA
Silver	9	0.002	0.01	0.25	0.5
Sodium	315	0.25	0.5	5.0	25.0
Strontium	4.5	0.025	0.05	0.25	0.5
Thallium	9	0.1	0.2	5.0	10.0
Tin	81	0.05	0.5	5.0	25.0
Titanium	90	0.005	0.03	0.5	2.0
Vanadium	90	0.005	0.01	0.25	0.5
Zinc	45	0.005	0.02	0.5	1.0
Phosphorus	900	0.5	1.0	25.0	50.0
Zirconium	45.0	0.025	0.05	12.5	25.0

Table 4.2a
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010B/200.7

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	85 - 115	0 - 20	80 - 120	0 - 20
Antimony	85 - 115	0 - 20	80 - 120	0 - 20
Arsenic	85 - 115	0 - 20	80 - 120	0 - 20
Barium	85 - 115	0 - 20	80 - 120	0 - 20
Beryllium	85 - 115	0 - 20	80 - 120	0 - 20
Boron	85 - 115	0 - 20	80 - 120	0 - 20
Cadmium	85 - 115	0 - 20	80 - 120	0 - 20
Calcium	85 - 115	0 - 20	80 - 120	0 - 20
Chromium	85 - 115	0 - 20	80 - 120	0 - 20
Cobalt	85 - 115	0 - 20	80 - 120	0 - 20
Copper	85 - 115	0 - 20	80 - 120	0 - 20
Iron	85 - 115	0 - 20	80 - 120	0 - 20
Lead	85 - 115	0 - 20	80 - 120	0 - 20
Lithium	85 - 115	0 - 20	80 - 120	0 - 20
Magnesium	85 - 115	0 - 20	80 - 120	0 - 20
Manganese	85 - 115	0 - 20	80 - 120	0 - 20
Molybdenum	85 - 115	0 - 20	80 - 120	0 - 20
Nickel	85 - 115	0 - 20	80 - 120	0 - 20
Potassium	85 - 115	0 - 20	80 - 120	0 - 20
Selenium	85 - 115	0 - 20	80 - 120	0 - 20
Silicon	85 - 115	0 - 20	80 - 120	0 - 20
Silver	85 - 115	0 - 20	80 - 120	0 - 20
Sodium	85 - 115	0 - 20	80 - 120	0 - 20
Strontium	85 - 115	0 - 20	80 - 120	0 - 20
Thallium	85 - 115	0 - 20	80 - 120	0 - 20
Tin	85 - 115	0 - 20	80 - 120	0 - 20
Titanium	85 - 115	0 - 20	80 - 120	0 - 20
Vanadium	85 - 115	0 - 20	80 - 120	0 - 20
Zinc	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85 - 115	0 - 20	80 - 120	0 - 20
Zirconium	85 - 115	0 - 20	80 - 120	0 - 20

Table 4.2b
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010C

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	80 - 120	0 - 20	80 - 120	0 - 20
Antimony	80 - 120	0 - 20	80 - 120	0 - 20
Arsenic	80 - 120	0 - 20	80 - 120	0 - 20
Barium	80 - 120	0 - 20	80 - 120	0 - 20
Beryllium	80 - 120	0 - 20	80 - 120	0 - 20
Boron	80 - 120	0 - 20	80 - 120	0 - 20
Cadmium	80 - 120	0 - 20	80 - 120	0 - 20
Calcium	80 - 120	0 - 20	80 - 120	0 - 20
Chromium	80 - 120	0 - 20	80 - 120	0 - 20
Cobalt	80 - 120	0 - 20	80 - 120	0 - 20
Copper	80 - 120	0 - 20	80 - 120	0 - 20
Iron	80 - 120	0 - 20	80 - 120	0 - 20
Lead	80 - 120	0 - 20	80 - 120	0 - 20
Lithium	80 - 120	0 - 20	80 - 120	0 - 20
Magnesium	80 - 120	0 - 20	80 - 120	0 - 20
Manganese	80 - 120	0 - 20	80 - 120	0 - 20
Molybdenum	80 - 120	0 - 20	80 - 120	0 - 20
Nickel	80 - 120	0 - 20	80 - 120	0 - 20
Potassium	80 - 120	0 - 20	80 - 120	0 - 20
Selenium	80 - 120	0 - 20	80 - 120	0 - 20
Silicon	80 - 120	0 - 20	80 - 120	0 - 20
Silver	80 - 120	0 - 20	80 - 120	0 - 20
Sodium	80 - 120	0 - 20	80 - 120	0 - 20
Strontium	80 - 120	0 - 20	80 - 120	0 - 20
Thallium	80 - 120	0 - 20	80 - 120	0 - 20
Tin	80 - 120	0 - 20	80 - 120	0 - 20
Titanium	80 - 120	0 - 20	80 - 120	0 - 20
Vanadium	80 - 120	0 - 20	80 - 120	0 - 20
Zinc	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 – 120	0 – 20	80 – 120	0 - 20
Zirconium	80 – 120	0 – 20	80 – 120	0 - 20

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000

Factors per 100 mg/L Interferant							
Analyte	**Al**	**As**	**Ba**	**Be**	**Ca**	**Co**	**Cr**
Aluminum	--	--	--	--	--	-0.00082	--
Antimony	0.00021	--	--	--	--	--	0.0095
Arsenic	--	--	--	--		--	0.00049
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	--	--	--	--
Boron	--	--	--	--	--	0.0343	--
Cadmium	--	0.0171	--	--	--	--	--
Calcium	--	--	--	--	--	--	--
Chromium	--	--	--	--	--	--	0.00108
Cobalt	--	--	--	--	--	0.000077	--
Copper	--	--	--	--	--	-0.000093	-0.000172
Iron	--	--	--	--	--	--	--
Lead	-0.000081	--	--	--	--	--	--
Lithium	--	--	--	--	--	--	--
Magnesium	--	--	--	--	--	-0.000092	--
Manganese	--	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	0.00016	--
Nickel	--	--	--	--	--	--	--
Potassium	--	--	--	--	--	--	--
Selenium	-0.00024	--	--	--	--	--	--
Silicon	--	--	--	--	--	--	--
Silver	--	--	--	--	--	--	--
Sodium	--	--	--	--	--	--	--
Strontium	--	--	--	--	--	--	--
Thallium	-0.000012	--	--	--	--	0.00397	0.000276
Tin	--	--	--	--	--	--	--
Titanium	--	--	--	--	--	--	--
Vanadium	--	--	--	--	--	--	--
Zinc	--	--	--	--	--	-0.00074	--
Phosphorus	0.00075	--	--	--	--	--	--
Zirconium	--	--	--	--	--	--	--

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000 (continued)

Factors per 100 mg/L Interferant						
Analyte	**Cu**	**Fe**	**Mg**	**Mn**	**Mo**	**Ni**
Aluminum	--	--	--	--	0.0153	--
Antimony	--	--0.000056	--	--	0.00067	--
Arsenic	--	-0.00025	--	--	0.00109	--
Barium	--	--	--	--	--	--
Beryllium	--	--	--	--	--	--
Boron	--	-0.000398	--	--	-0.00169	--
Cadmium	--	0.000002	--	--	0.000022	-0.000128
Calcium	--	--	--	--	--	--
Chromium	--	-0.000006	--	0.00016	--	--
Cobalt	--	--	--	--	-0.000983	0.000175
Copper	--	0.00021	--	--	0.00274	-0.00455
Iron	--	--	---	--	--	--
Lead	0.000809	0.000021	--	--	-0.00183	0.00011
Lithium	--	--	--	--	--	--
Magnesium	--	--	--	--	--	--
Manganese	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	--
Nickel	--	--	--	--	--	--
Potassium	--	--	--	--	--	--
Selenium	--	--	--	--	0.000156	--
Silicon	--	--	--	--	0.0187	--
Silver	--	--	--	--	-0.000044	--
Sodium	--	--	--	--	--	--
Strontium	--	--	--	--	--	--
Thallium	--	--	--	--	--	--
Tin	--	--	--	--	--	--
Titanium	--	--	--	--	-0.000153	--
Vanadium	--	0.00002	--	--	-0.00778	--
Zinc	--	--	--	--	--	--
Phosphorus	0.002	0.00165	--	--	0.008	--
Zirconium	--	-0.000031	--	--	--	--

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000 (continued)

Factors per 100 mg/L Interferant							
Analyte	**Sb**	**Se**	**Sn**	**Ti**	**Tl**	**V**	**Zn**
Aluminum	--	--	--	--		0.003	--
Antimony	--	--	-0.0084	-0.00099	--	-0.00438	--
Arsenic	--	--	--	--	--	0.000107	--
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	-0.00033	--	0.00025	--
Boron	--	--	--	--	--	--	--
Cadmium	--	--	--	-.0249829	--	0.000102	--
Calcium	--	--	--	--	--	--	--
Chromium	--	--	--	0.000055	--	0.00002	--
Cobalt	--	--	--	0.00188	--	--	--
Copper	--	--	--	0.000469	--	--	--
Iron	--	--	--	--	--	--	--
Lead	--	--	--	--	--	--	--
Lithium	--	--	--	--	--	--	--
Magnesium	--	--	--	--	--	--	--
Manganese	--	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	-0.00011	--
Nickel	--	--	--	-	--	--	--
Potassium	--	--	--	--	--	--	--
Selenium	--	--	--	-	--	--	--
Silicon	--	--	--	--	--	--	--
Silver	--	--	--	-0.0062	--	--	--
Sodium	--	--	--	--	--	--	--
Strontium	--	--	--	--	--	--	--
Thallium	--	--	--	-0.0017	--	0.0282	--
Tin	--	--	--	-0.0022	--	--	--
Titanium	--	--	--	--	--	--	--
Vanadium	--	--	--	0.000824	--	--	--
Zinc	--	--	--	--	--	--	--
Phosphorus	--	--	--	--	--	-0.005	--
Zirconium	--	--	--	--	--	--	--

Uncontrolled Copy

Table 7.4.1
Water Matrix
Calibration Standards Concentrations in mg/L for Thermo iCAP 6000/7000

Element	S0	S1	S2	S3	S4	Stock Conc.
Aluminum	0.0	0.08	0.16	10.0	20.0	2000
Antimony	0.0	0.0096	0.0192	1.2	2.4	240
Arsenic	0.0	NA	0.0064	0.4	0.8	80
Barium	0.0	0.008	0.016	1.0	2.0	200
Beryllium	0.0	0.0004	0.0008	0.05	0.1	10
Boron	0.0	NA	0.008	0.5	1.0	100
Cadmium	0.0	0.0004	0.0008	0.05	0.1	10
Calcium	0.0	0.08	0.16	10.0	20.0	2000
Chromium	0.0	0.004	0.008	0.5	1.0	100
Cobalt	0.0	0.0016	0.0032	0.2	0.4	40
Copper	0.0	0.004	0.008	0.5	1.0	100
Iron	0.0	0.032	0.064	4.0	8.0	800
Lead	0.0	0.004	0.008	0.5	1.0	100
Lithium	0.0	0.008	0.016	1.0	2.0	200
Magnesium	0.0	0.08	0.16	10.0	20.0	2000
Manganese	0.0	0.004	0.008	0.5	1.0	100
Molybdenum	0.0	0.008	0.016	1.0	2.0	200
Nickel	0.0	0.004	0.008	0.5	1.0	100
Potassium	0.0	0.4	0.8	50.0	100.0	10000
Selenium	0.0	NA	0.0062	0.4	0.8	80
Silicon	0.0	0.04	0.08	5.0	10.0	1000
Silver	0.0	0.0032	0.0064	0.4	0.8	80
Sodium	0.0	0.4	0.8	50.0	100.0	10000
Strontium	0.0	0.008	0.016	1.0	2.0	200
Thallium	0.0	NA	0.008	0.5	1.0	100
Tin	0.0	0.008	0.016	1.0	2.0	200
Titanium	0.0	0.008	0.016	1.0	2.0	200
Vanadium	0.0	0.008	0.016	1.0	2.0	200
Zinc	0.0	0.008	0.016	1.0	2.0	200
Phosphorus	0.0	0.08	0.16	10.0	20.0	1000
Zirconium	0.0	0.008	0.016	1.0	2.0	1000

*S0 through S4 are used for 6010B and 200.7.
6010C may use S0 and S4 only.

NA = S1 is not used in construction of the calibration curve for this element. The concentration of S2 is at or below the reporting limit for this element.

Table 7.5.1
Initial and Continuing Calibration Standards Concentration in mg/L

Element	ICV/CCV	Stock Concentration
Aluminum	10.0	2000
Antimony	1.2	240
Arsenic	0.4	80
Barium	1.0	200
Beryllium	0.05	10
Boron	0.5	100
Cadmium	0.05	10
Calcium	10.0	2000
Chromium	0.5	100
Cobalt	0.2	40
Copper	0.5	100
Iron	4.0	800
Lead	0.5	100
Lithium	1.0	200
Magnesium	10.0	2000
Manganese	0.5	100
Molybdenum	1.0	200
Nickel	0.5	100
Potassium	50.0	10000
Selenium	0.4	80
Silicon	5.0	1000
Silver	0.4	80
Sodium	50.0	10000
Strontium	1.0	200
Thallium	0.5	100
Tin	1.0	200
Titanium	1.0	200
Vanadium	1.0	200
Zinc	1.0	200
Phosphorus	10.0	1000
Zirconium	1.0	1000

Table 7.6.1
Low Level Calibration Verification Solution Concentrations

Metal	LLICV/LLCCV (mg/L)	Stock Concentration (mg/L)
Aluminum	0.1	50
Antimony	0.012	6
Arsenic	0.01	5
Barium	0.01	5
Beryllium	0.0005	0.025
Boron	0.1	2.5
Cadmium	0.001	0.5
Calcium	0.2	100
Chromium	0.01	5
Cobalt	0.002	1
Copper	0.005	2.5
Iron	0.1	50
Lead	0.01	5
Lithium	0.01	5
Magnesium	0.5	250
Manganese	0.01	5
Molybdenum	0.01	5
Nickel	0.02	10
Potassium	1	500
Selenium	0.02	10
Silicon	0.05	25
Silver	0.01	5
Sodium	0.5	250
Strontium	0.01	5
Thallium	0.2	100
Tin	0.01	5
Titanium	0.01	5
Vanadium	0.01	5
Zinc	0.01	5

Table 7.7.1
ICSA and ICSAB Concentration in mg/L

Element	ICSA	ICSAB	Stock Concentration
Aluminum	250	250	5000
Antimony	--	0.5	100
Arsenic	--	0.25	50
Barium	--	0.25	50
Beryllium	--	0.25	50
Cadmium	--	0.5	100
Calcium	250	250	5000
Chromium	--	0.25	50
Cobalt	--	0.25	50
Copper	--	0.25	50
Iron	100	100	2000
Lead	--	0.5	100
Magnesium	250	250	5000
Manganese	--	0.25	50
Nickel	--	0.5	100
Potassium	--	5	1000
Selenium	--	0.25	50
Silver	--	0.5	100
Sodium	--	5.0	1000
Thallium	--	0.5	100
Vanadium	--	0.25	50
Zinc	--	0.5	100

The final concentration of the aqueous and soil LCS is as follows:

Table 13.1
Quality Control Criteria
Total Metals - ICP
Method 6010B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL x 2 (1)	Stop analysis, investigate, reanalyze. If still > limit re-digest batch or qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120% Soil 85 – 115 % Water	Stop analysis, investigate, reanalyze. If still outside limits re-digest batch or qualify data and address in narrative
Matrix spike/ Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	80 - 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (DP)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check Samples (ICSA/ICSAB)	Run at beginning of each run	80 - 120% of true value for EPA check sample element. < RL or project specific criteria for nonspiked elements	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike (PS)	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are analyte specific and instrument specific

Table 13.2
Quality Control Criteria
Total Metals - ICP
Method 200.7

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	95 – 105% RSD < 3%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or <MDL	Stop analysis, investigate, reanalyze. If still > limit re-digest batch or qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Water 85 – 115 % (STD) 80 – 120 % (DoD) Soil 80-120 %	Stop analysis, investigate, reanalyze. If still outside limits re-digest batch or qualify data and address in narrative
Matrix spike/Matrix Spike Duplicate (MS/MSD)	One per ten samples (20 samples maximum per batch)	80- 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (DP)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP Interference Check Samples (ICSA/ICSAB)	Run at beginning of each run	Spiked Elements 80 – 120 % Nonspiked Elements <RL (STD) < 2 x MDL (DoD)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike (PS)	5%, or minimum of 1 per batch	75 – 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are project specific. Consult QAPP

Uncontrolled Copy

Table 13.3.2
Final Concentration of the Aqueous and Soil LCS

ELEMENT	WATER mg/L	SOIL mg/Kg
Potassium Sodium	25	1250
Aluminum Calcium Magnesium	5	250
Silicon	2.5	NA
Iron	2	100
Antimony	0.6	30
Barium Lithium Molybdenum Strontium Titanium Vanadium Zinc Phosphorus Zirconium Tin	0.5	25
Boron Chromium Copper Manganese Nickel Lead Thallium	0.25	12.5
Arsenic Selenium Silver	0.2	10
Cobalt	0.1	5
Beryllium Cadmium	0.025	1.25

Uncontrolled Copy

Table 13.4
Quality Control Criteria
Total Metals – ICP
Method 6010C

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Daily, at beginning of run	COC ≥ 0.998	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After ICSA/ICSAB and minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCV.
Initial Calibration Blank (ICB)	After ICV	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)	After every CCV, minimum every 10 samples	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCB.
Low Level Initial Calibration Verification (LLICV)	After ICB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Low Level Continuing Calibration Verification (LLCCV)	Minimum following closing batch CCB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant LLCCV.
Method Blank	Once per batch (20 samples maximum per batch)	< RL < ½RL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Laboratory Control Sample /Laboratory Control Sample Duplicate (LCS/LCSD)	Once per batch (20 samples maximum per batch)	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Once per batch (20 samples maximum per batch)	75 - 125%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (DP)	Once per batch (20 samples maximum per batch) upon client request	RPD ≤ 20%	Qualify data and address in narrative if client specified.
ICP Interference Check Samples (ICSA/ICSAB)	After ICB, prior to sample analysis	80 - 120% for spiked analytes; < RL or other project specific criteria for nonspiked analytes	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post Digestion Spike (PS)	One per batch	80 - 120%	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	One per batch, if sample concentration > 50X MDL	± 10% of original determination	Dilute and repeat post digestion spike.

(1) Acceptance criteria are analyte specific and instrument specific.

Uncontrolled Copy

Figure 12.1

Example 6010 Calculations
Thermo Scientific iCAP 6500

1.0 Initial Calibration (ICAL) Parameters
For a multi-point calibration, the system performs linear regression from data consisting of a blank and four standards.

2.0 Calculating the concentration (C) of an element in water using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system in ug/mL (ppm)	0.08
Vf = Final volume (mL)	50
Vi = Initial volume (mL)	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/mL (mg/L)	0.1

3.0 Calculating the concentration (C) of an element in soil using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where**:**	**Example:**
Cs = Concentration computed by the data system (mg/L) (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial weight (g)	1
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/g (mg/kg)	5

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where**:**	**Example:**
Cx = Concentration calculated as received (wet basis)	5
Px = Percent solids of sample (% wt)	80
Cdry = Concentration calculated as dry weight (mg/kg)	6.25

Figure 14.1

Checklist ID: 79093

Microbac Laboratories Inc.

Data Checklist

Date: 16-AUG-2013
Analyst: QX
Analyst: NA
Method: 6010B/6010C/200.7
Instrument: ICP-THERMO2
Curve Workgroup: 441689
Runlog ID: 55099
Analytical Workgroups: 441377, 441535, 441656, 441660, 441661, 441677

Calibration/Linearity	X
ICV/CCV	X
ICV RSD <= 3% (EPA 200.7 only)	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	X
Case Narrative	
Client Forms	
Level X	
Level 3	
Level 4	
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	KHR
Secondary Reviewer	SLP
Comments	

Primary Reviewer:
19-AUG-2013

Secondary Reviewer:
19-AUG-2013

CHECKLIST1 - Modified 03/05/2008
Generated: AUG-19-2013 10:49:37

Microbac

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURE
PERKIN ELMER NexION 300X
INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETER
(SW-846 6020 / EPA METHOD 200.8)

Issue/Implementation Date: 15 April 2014

Last Review Date: 15 April 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Maren M. Beery, Metal Supervisor — Date: 04/04/2014

Wade T. DeLong, Quality Assurance Officer — Date: 4-15-14

Leslie S. Bucina, Laboratory Manager — Date: 4/15/14

Uncontrolled Copy

Table of Contents

Section		Page
1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	5
4.0	Method Performance	5
5.0	Interferences and Corrective Action	7
6.0	Equipment and Supplies	7
7.0	Standards and Reagents	8
8.0	Diagram or Table to Outline Procedures	13
9.0	Sample Preparation	14
10.0	Calibration Procedures	14
11.0	Analytical Procedures	15
12.0	Details of Calculations	19
13.0	Quality Control Requirements	21
14.0	Data Review and Reporting Requirements	24
15.0	Preventive Maintenance	25
16.0	Waste Management and Pollution Control	26
17.0	References	27
	Appendices	28
	Tables	30
	Figures	45

Uncontrolled Copy

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples according to U.S. EPA Methods 6020, 6020A and 200.8 for the elements listed as analytes in Table I using the NexION 300X ICP-MS.

1.2 This method is applicable to the following sample matrices: ground waters, surface waters, industrial wastes, sludges, and soil samples.

1.3 Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and for other solid wastes for which total (acid leachable) elements are required. Sample preparation methods may be found in EPA Publication SW-846 as Methods 3015 and 3051 and are detailed in SOP ME407 and ME406.

1.4 Routine operation and maintenance procedures for the NexION 300X ICP-MS may be found in the NexION Software (Version 1.0) Help Menu.

1.5 Detailed instructions on operating of the NexION 300X ICP-MS operating software may be found in the NexION Software Reference Guide Version 1.0 (Build 1.0.1916.0) for ICP-MS Instrument Control.

1.6 Following the appropriate digestion procedure, aqueous sample digestates, leachates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a R.F. plasma. There the sample is ionized. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector.

1.7 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCB	Continuing calibration blank
CCV	Continuing calibration verification
COA	Certificate of analysis
COC	Coefficient of Correlation
DI water	DI water
HCl	Hydrochloric acid

HNO_3	Nitric Acid
ICB	Initial calibration blank
ICP/MS	Inductively Coupled Plasma Mass Spectrometry
ICS	Interference check sample
ICV	Initial calibration verification
IDL	Instrument Detection Limit
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information Management System
LLCCV	Low level continuing calibration verification
LLICV	Low level initial calibration verification
LOD	limit(s) of detection
LOQ	limits(s) of quantitation
MB	Method blank
MDL	Method detection limit
MS	Matrix spike
MSA	Method of standard additions
MSD	Matrix spike duplicate
NCR	Nonconformance Report
QC	Quality control
RF	Radio frequency
RGT	Reagent
RL	Reporting limit
SOP	Standard Operating Procedure
STD	Standard

Calibration blank – a calibration standard prepared with DI water and carried through the digestion procedure used in establishing the calibration curve.

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

1.8 Updates that effect concentration, vendor choices, reagents, MDLs, RLs and QC limits are subject to change without notice.

2.0 SAFETY PRECAUTIONS

2.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices must be followed at all times.

2.2 Safety glasses and gloves must be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

2.3 Liquid argon represents a potential cryogenic hazard and safe handling procedures must be used when handling liquid argon tanks at all times.

2.4 The NexION 300X is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the NexION if any safety interlock is known to be disabled or malfunctioning.

2.5 Spilled samples, reagents, and water must be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills the acid must be neutralized with sodium bicarbonate solution before cleanup.

2.6 All additional company safety practices must be followed at all times.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1

Measurement	Digestion Vol./Wt. Req.*	Collection Vol./Wt.	Preservative	Holding Time **
Total recoverable	40 mL	250 - 1000 mL	HNO3 to pH <2	6 months
Dissolved	40 mL	250 - 1000 mL	Filter on-site HNO3 to pH <2	6 months
Suspended	40 mL	250 - 1000 mL	Filter on-site	6 months
Total	40 mL	250 - 1000 mL	HNO3 to pH <2	6 months
Soil	0.5 g	200 g		6 months

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

*** Storage at ≤ 6° C if mercury is to be analyzed.

3.2 Water samples received unpreserved will be acid preserved in the laboratory and must then rest for at least 24 hours prior to digestion.

4.0 METHOD PERFORMANCE

Instrument Detection Limits (IDLs)

4.1 IDLs are determined according to the procedure outlined in Section 8.2 of Method 6020 (Section 9.3, Method 6020A). This is accomplished by

calculating the average of the standard deviations of the three runs on three nonconsecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate sample (ie, with rinsing in between).

IDLs must be determined quarterly and kept with the instrument.

Method Detection Limits (MDLs)

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 1 and were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project reporting limits may be higher.

4.5 Precision and accuracy data in Table 1 were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 MDL check samples are analyzed quarterly to verify the MDLs listed in Table 1.

4.7 MDLs must be re-determined whenever there is any change to the sample preparation procedure, or any significant change to the instrument (new detector or different sample introduction system used).

Linear calibration ranges

4.8 Calibrate the instrument, as described in Section 10.0.

4.9 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

4.10 The definition of the Linear Range is not specifically addressed in either SW-846 Method 6020 or in Chapter 1 (Quality Control) of the SW-846 Methods manual. In order to use an established definition of linear range, the definition from EPA Method 200.8 will be used. In this reference, the linear range is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard. The values reported in Table 1 are 90% of the verified upper linear range.

Uncontrolled Copy

4.11 The upper Linear Dynamic Range must be re-determined whenever one of the following occurs. A new detector is installed. The detector (analog and/or pulse count) voltages are changed. A new PA tube is installed in the RF generator. A different sample introduction system (change in nebulizer or spray chamber type) is installed.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Isobaric Interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98). Corrections for isobaric interferences must be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interferences are already present as default interference equations in the NexION software. Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

5.2 Molecular interferences are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.) Predictions about the type of molecular interferences must be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of Ar40Cl35 on As at mass 75 must be made by measuring the intensity of ArCl present at mass 77 (Ar40Cl37) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl37 to Cl35. A list of the corrections used is given in the listing of the isotopes monitored in the NexION 6020A method in Table 2.

6.0 EQUIPMENT AND SUPPLIES

6.1 Perkin Elmer NexION 300X ICP-MS system.

6.1.1 Perkin Elmer NexION ICP-MS

6.1.2 Intel Core 2 Vpro computer with Windows XP Professional, Ienovo enhanced.

6.1.3 NexION Software Version 1.0 (Build 1.0.1916.0)

6.1.4 ESI FAST DX-4 Autosampler

6.2 Peristaltic pump tubing:

6.2.1 Red/Red - 1.14 mm I.D. (for drain)

6.2.2 Green/Orange - 0.38 mm I.D. (for internal standard introduction)

6.2.3 Black/Black – 0.76 mm I.D. (for sample introduction)

6.3 Calibrated mechanical pipettes:

6.3.1 10-100 uL

6.3.2 100 - 1000 uL

6.3.3 1000 - 5000 uL

6.4 Metal-free plastic pipette tips (for the pipettes specified in 6.4).

6.5 Metal-free15 mL plastic test tubes

6.6 Metal-free 50 mL plastic test tubes

6.7 Class A glass pipettes – for preparation of standard solutions.

7.0 STANDARDS AND REAGENTS

7.1 All reagents may contain impurities that may affect the integrity of the analytical results. Due to the high sensitivity of ICP-MS, high-purity reagents, water, and acids must be used whenever possible. All acids used for this method must be of ultra high-purity grade. Nitric acid is preferred for ICP-MS in order to minimize polyatomic interferences. It should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for poly-atomic ion interferences must be used. All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Nitric acid and hydrochloric acid, Instra-analyzed reagent, J.T. Baker or equivalent.

7.3 Reagent water equivalent to ASTM Type II water (ASTM D 1193).

7.4 5% HNO_3 (in reagent water) – 50 mL HNO_3 in 1000 mL reagent grade water.

Internal Standard Stocks

7.5 ICP-MS Internal Standard - 100 mg/L Li, Sc, Y, In, Tb and Bi in 1% HNO_3, CPI International or equivalent.

7.6 Germanium – 1000 mg/L in 2% HNO_3, CPI International or equivalent.

7.7 Internal Standard Working Solutions - Prepare the working solution by pipetting 100 mL of nitric acid into approximately 1000 mL of reagent water in a 2000 mL volumetric flask. Pipette 1 mL of ICP-MS internal standard and 1 mL of germanium single-element standard into the flask. Dilute to 2000 mL with reagent water. The concentrations of the working internal standard solution can be found in Table 7-1.

Tuning Solution and Daily Performance Check Solution

7.8 ICP-MS Internal Standard – 100 mg/L Li, Sc, Tb, Y, In and Bi in 1% HNO_3 CPI or equivalent.

7.9 QC-MS1 – 10 mg/L Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, U, V and Zn in 2% HNO_3, CPI or equivalent.

7.10 QC-MS2 – 1000 mg/L Ca, Fe, Mg, K and Na in 5% HNO_3, CPI or equivalent.

7.11 Cerium (Ce) – 1000 mg/L in 5% HNO_3 – Inorganic Ventures, Inc. or equivalent

7.12 Stock Tuning Solution – Prepare the stock tuning solution by pipetting 5 mL of nitric acid into 80 mL of reagent grade water in a 100 mL volumetric flask. Add 0.02 mL of 1000 mg/L Ce and 0.02 mL of QC-MS2, add 0.2 mL of ICP-MS Internal Standard and 2 mL of QC-MS1, dilute to 100 mL with reagent grade water.

7.13 Working Tuning Solution – Prepare the working tuning solution by pipetting 50 mL of nitric acid into 500 mL of reagent grade water in a 1000 mL volumetric flask. Add 5 mL of stock tuning solution into the flask, dilute to 1000 mL with reagent grade water. The concentrations of the working tuning solution also used as the daily performance check solution can be found in Table 7-2.

Calibration Solutions

7.14 Purchased Standard Mixes:

CAL-MS1 – 10 mg/L Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V and Zn in 3.5% HNO_3 – Inorganic Ventures, Inc. or equivalent

IV-STOCK-35 - 1000 mg/L Ca, Fe, Mg, K and Na in 2% HNO_3 – Inorganic Ventures, Inc. or equivalent

7.15 1000 ug/mL Mo – Inorganic Ventures, Inc. or equivalent

7.16 1000 ug/mL Ti – Inorganic Ventures, Inc. or equivalent

7.17 1000 ug/mL Sn – Inorganic Ventures, Inc. or equivalent

7.18 1000 ug/mL U – Inorganic Ventures, Inc. or equivalent

7.19 Working Calibration Solution - Prepare the working solution (high standard) by pipetting 25 mL of nitric acid into approximately 250 mL DI water in a 500 mL volumetric flask. Pipette 5 mL each of CAL-MS1 and IV-STOCK-35 into the flask. Add 0.1 mL each of the single element solutions Mo and Ti; and 0.05 mL of Sn and U. Dilute to 500 mL with DI water. The concentration of the working solution can be found in Table 7-3.

7.20 Working Calibration Solutions - Prepare calibration standards using a 5% HNO_3 solution (7.4) and the working solution (high standard) (7.19).

7.21 Blank (for establishing internal standard intensities) and S1 (STD 1) – 5 % HNO_3 Solution. (7.4).

7.22 S2 (Std 2) – 1/2000 high standard: 5% HNO_3

7.23 S3 (Std 3) – 1/2 high standard: 5% HNO_3

7.24 S4 (Std 4) – high standard solution (7.19)

7.25 Working Continuing Calibration Verification Solution (CCV) - Prepare the working solution (CCV-QC STD 6) by pipetting 50 mL of nitric acid into approximately 500 mL DI water in a 1000 mL volumetric flask. Pipette 5 mL each of CAL-MS1 and IV-STOCK-35 into the flask. Add 0.1 mL each of the single element solutions Mo and Ti; and 0.05 mL of Sn and U. Dilute to 1000

mL with DI water. The concentration of the working solution can be found in Table 7-3.

Initial Calibration Verification Solutions

7.26 ICV – QCStd 1: This must originate from a different source than that of the calibration standard (i.e. second vendor).

7.27 Purchased Standard Mixes: Microbac QC-MS1 – 10 mg/L As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb, Se, Ag, Tl, U, V and Zn in 2% HNO_3 and tr HF – SCP Science or equivalent

7.28 Microbac QC-MS2-1000 mg/L Ca, Fe, Mg, K and Na in 5% HNO_3 – SCP Science or equivalent

7.29 1000 ug/mL Mo – SCP Science or equivalent

7.30 1000 ug/mL Ti – SCP Science or equivalent

7.31 1000 ug/mL Sn – SCP Science or equivalent

7.32 Working ICV Solution - Prepare working solution by pipetting 25 mL of HNO_3 into approximately 250 mL of DI water into a 500 mL volumetric flask. Pipette 2.5 mL each of QC-MS1 and QC-MS2 into the flask. Add 0.05 mL of the single element solutions Mo and Ti and 0.025 mL of Sn. Dilute to 500 mL with reagent water. The concentrations can be found in Table 7-4.

7.33 ICB – QCStd 2 and Continuing Calibration Blank (CCB – QCStd 7). – 5% HNO_3 solution (7.4).

Interference Check Solutions (ICSA and ICSAB)

7.34 6020ICS-9A – 21215 mg/L Cl^-, 2000 mg/L C, 3000 ppm Ca, 2500 mg/L Fe and Na, 1000 mg/L Al, Mg, P, K and S and 20 ppm Mo and Ti in 1% HNO_3 – Inorganic Ventures, Inc. or equivalent

7.35 Microbac QC-MSI -10 mg/L As, Al, Ba, Be, Cd, Co, Cr, Cu, Pb, Mn, Ni, Sb, Se, Ag, Tl, U, V and Zn in 2% HNO_3 and trHF–CPI International or equivalent

7.36 Working ICSA Solution - Prepare working solution by pipetting 5 mL of HNO3 into approximately 50 mL of DI water in a 100 mL volumetric flask. Pipette 0.5 mL 6020ICS-9A and dilute to 100 mL with DI water. The concentrations can be found in Table 7-5.

Uncontrolled Copy

7.37 Working ICSAB Solution - Prepare the working solution by pipetting 5 mL of HNO3 into approximately 50 mL of DI water in a 100 mL volumetric flask. Pipette 0.5 mL of 6020ICS-9A and 1 mL of Microbac QC-MSI into the flask. Dilute to 100 mL with DI water. The concentrations can be found in Table 7-5.

Low Level Calibration Verification Solutions

7.38 The LLICV Solution is used to establish and/or verify the lower limit of quantitation.

7.39 The solution from 7.38 is also used as the LLCCV solution and is analyzed for 6020A only.

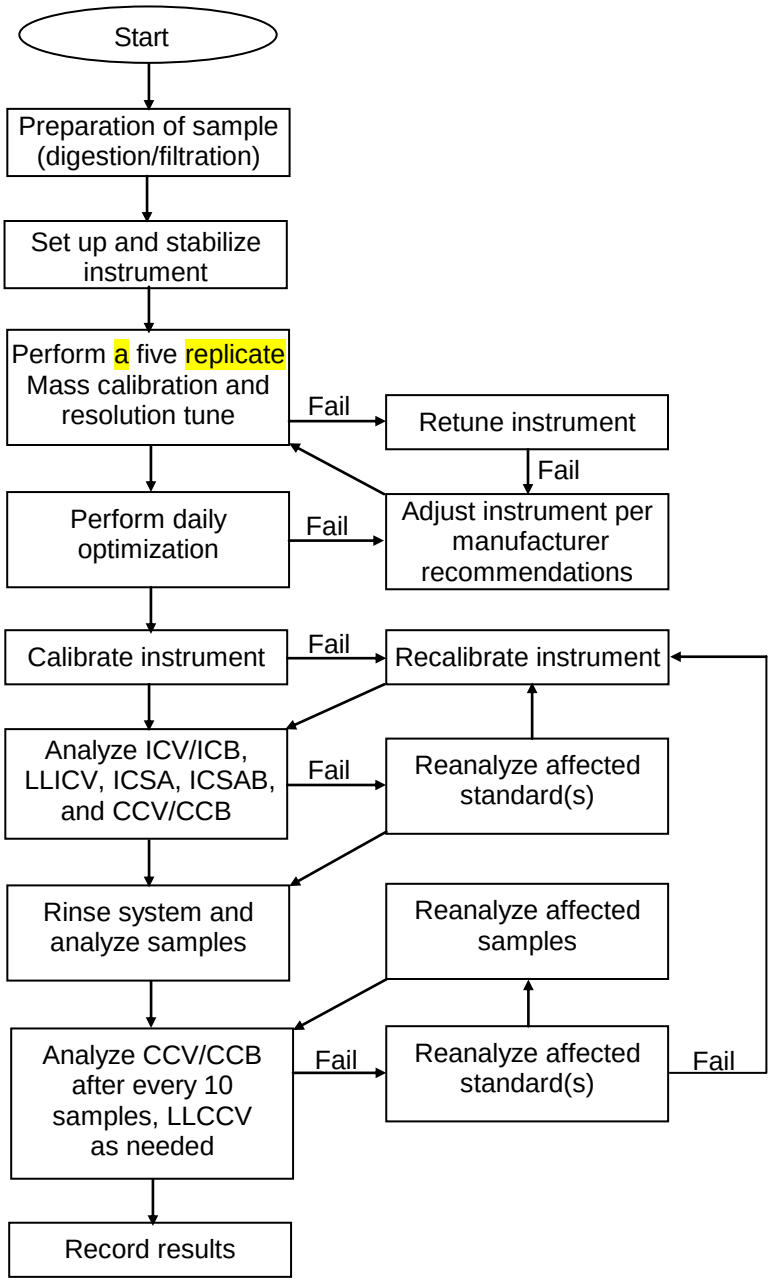
7.40 Single Element Standards 1000 ug/mL As, Ba, Cd, Cr, Co, Cu, Mn, Ni, Se, Tl, Sb, Ag, V, Pb, Zn and U - InorganicVentures .

7.41 Intermediate Low Level Calibration Verification Solutions – Prepare the intermediate solution by pipetting 2.5 mL of HNO_3 into approximately 20 mL of DI water in a 50 mL volumetric flask. Add the individual single element standards in the amounts indicated in Table 7-6 to give the corresponding intermediate concentrations listed in the table after dilution to 50 mL volume with DI water.

7.42 Working Low Level Calibration Verification Solutions (LLICV/LLCCV) – Prepare the working solution by pipetting 25 mL of HNO_3 into approximately 250 mL of DI water in a 500 mL volumetric flask.

Pipette 0.025 mL of the intermediate solution from 7.41 into the flask and bring to 500 mL final volume with DI water. The concentrations can be found in Table 7-6.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



Uncontrolled Copy

9.0 SAMPLE PREPARATION

9.1 Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

Microbac SOP ME406 - Microwave Digestion of Sediments/Sludges/Soils/Oils

Microbac SOP ME407 - Microwave Digestion - Aqueous

10.0 CALIBRATION PROCEDURES

10.1 Initial Calibration - The instrument is calibrated before analysis of any samples with a blank and three calibration standards. The concentrations of the standards used must be entered into the calibration page of the analytical method in the NexION software according to the values of the standards prepared in Section 7.21, 7.22, 7.23 and 7.24. A Weighted Linear (with calculated intercept) curve type must be selected for all analytes. A blank (7.21) will be run as a blank, before the analysis of any actual calibration standards. This blank is used to monitor the internal standard intensities throughout the analytical sequence. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings. See Appendix A for the weighted linear calibration algorithm.

The low calibration standard must contain the elements of interest at concentrations at or below the reporting limit or a low level calibration check standard at or below the reporting limit must be analyzed after calibration and before sample analysis. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective action for the curve and low level calibration check standard.

10.2 ICV Analysis – The ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective actions.

10.3 CCV Analysis - The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective action.

10.4 Initial and CCB Analysis - The solution used is the calibration blank (7.21). See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective action.

10.5 LLICV Analysis – The LLICV analysis is performed after the calibration but prior to sample analysis. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective action.

10.6 LLCCV Analysis – The LLCCV analysis is performed for Method 6020A and as required by specific QAPPS. The LLCCV is analyzed minimally at the end of each relevant batch or more frequently (eg: following every 10 samples) as desired. See Table 13-6 for acceptance criteria and corrective action.

10.7 ICSA and ICSAB Analysis – Required at the beginning of analytical run or every 12 hours, whichever is more frequent. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective action.

10.8 Internal Standards – An internal standard is a non-analyte isotope that is added to standards and samples before a determination. Internal standards allow you to correct for changes in instrument hardware response or for sample-to-sample variations in sensitivity. Intensities must be monitored in all solutions. Intensities of internal standards in all subsequent analyses of CCV and CCB solutions must be greater than 80% and less than 120% of the levels in the original calibration blank. Failure action is to terminate the analysis, correct the problem, re-calibrate, and reanalyze all affected samples. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective actions for analytical and quality control samples. See 12.8 for an example calculation of how the internal standard is used to adjust measured intensities of analyte elements.

10.9 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Initiate the plasma and allow a warm-up of at least 30 minutes. The tuning procedures must be carried out after warm-up

11.2 Perform the mass calibration and resolution at least once a day prior to analysis.

11.2.1 Place internal standard probe and rinse probe in a tube containing working tuning solution.

11.2.2 Open Mass Cal window by clicking on Mass Cal on the NexION Tool Bar. Depress start Mass Cal button. The instrument will now perform the Mass calibration and resolution procedure.

11.2.3 The measured mass of each isotope must be ± 0.05 amu of the exact mass and ± 0.03 amu for resolution optimization to pass.

11.2.4 Peak height (%) for resolution optimization is 10%. Target resolution (amu) is 0.70. The number of replicates is 5 with 5% RSD.

11.2.5 After passing the mass calibration and resolution, click Smart Tune on the NexION Tool Bar for the daily performance check.

11.2.6 Click Optimize for daily performance check.

11.3 The following criteria must be met for best results:

Be intensity > 2000 cps
Mg intensity > 15000 cps
In intensity > 40000 cps
U intensity > 30000 cps
Ce^{+2}/Ce < 0.03 cps
CeO/Ce < 0.025 cps
Bkgd ≤ 1 cps @ mass 220

11.3.1 If these criteria are not met, further optimizations may be needed. See the NexION Software Reference Guide Version 1.0 in the Help Menu..

11.4 Calibrate the instrument at least once a day followed by the analysis of the ICV, ICB, LLICV, ICSA, ICSAB, CCV and CCB prior to the analysis of samples. See Tables 13-1, 13-4, and 13-6 for acceptance criteria and corrective actions.

11.4.1 Prepare the four calibration solutions using the high standard and/or 5% HNO_3 (in polished water). The calibration standards are prepared as follows:

Blank: 5% HNO_3 water
S1: 5% HNO_3 water (analyzed from Blank cup)
S2: 1/2000 high standard in 5% HNO_3 water
S3: 1/2 high standard in 5% HNO_3 water
S4: high standard solution

Load Blank, S1, S2 and S3, S4 into autosampler locations 1,2,3, and 4 respectively.

11.4.2 Pour the ICV solution into a tube and place it in autosampler location 201. Pour the LLICV/IICCV solution into a tube and place in location 202. Pour the CCV solution into a tube and place it in location 101.

11.4.3 Put the ICSA in location 203 and ICSAB in location 204.

11.4.4 Put ICB/CCB in location 102.

11.4.5 Select the sample window from the NexION tool bar. Click on batch tab.

- Enter sample numbers (including digest workgroup numbers) in the Sample ID column.

- Enter the autosampler location number for each sample in the A/S Loc. Column.

- Make sure the appropriate command appears in the measurement action column for each sample. It must read “Run Sample” for samples that will not be auto-diluted by the instrument and “Run Diluted Sample” for samples that will be auto-diluted by the instrument. If the instrument is to be calibrated before the first sample is analyzed, then the measurement action cell for that sample only must read “Run Blank, Stds, and Sample” if the sample is not to be auto-diluted, and “Run Blank, Stds, and Dil. sample” if the sample is to be auto-diluted. This is edited by clicking the right mouse button on the cell that is to be edited and selecting the desired command from the list that appears using the left mouse button.

- Enter the factor which indicates the “fold” of dilution in the description column for each sample. For instance, if the sample is to be analyzed at a 1 to 5 dilution, then enter 5 in the description column for that sample. If the sample is not to analyzed at a dilution, then enter 1 in the description column. This must be entered regardless of whether the operator or the instrument will be performing the dilutions.

- It is not necessary to enter the sample preparation factors. These will be applied during the data upload to LIMS procedure.

- Save the sample file.

- Print the run list by selecting the print command from the file menu and clicking on the print button in the NexION file print window that appears.

11.4.6 Select the method button from the NexION tool bar. Select File/Open. When the open window appears, choose Service 6020 Methods/6020A.mth.

- Select the QC tab. Enter an "X" in the initial column next to the appropriate QC Std # for all the QC standards that must be analyzed after the calibration is finished (if the instrument is to be calibrated). A list of the names and the standards to which they correspond follows:

  QC Std 1 - ICV
  QC Std 2 - ICB
  QC Std 3 - LLICV
  QC Std 4 - ICSA
  QC Std 5 - ICSAB
  QC Std 6 - CCV
  QC Std 7 - CCB
  QC Std 8 - LLCCV

- In the final column, enter an "X" beside the QC Std # that corresponds to the QC standards that must be analyzed after all samples have been analyzed. In the before A/S Loc. Column, enter the number that corresponds to the autosampler position of the sample that will run after the standards. For instance, 12 samples are loaded in autosampler positions 11-22, and a CCV and CCB must be analyzed after the sample in A/S position 20 and at the end. An "X" must be entered next to QC Std 6 and QC Std 7 in the final column and "21" must be entered in the first before A/S Loc. Next to QC Std 6 and QC Std 7.

- Click on the report tab located on the right edge of the window. Edit the cell report filename in the following manner. The file path must read "c:\NexIONdata\ReportOutput\mmddyy.rep" where mmddyy is the current date. Edit only the current date portion of the file path. Do not edit anything else in this section.

- Save the Method.

11.4.7 Prepare samples and load into autosampler positions indicated in sample file.

11.4.8 Click on the sample window from the NexION tool bar. Choose the batch tab.

- Select the samples to be analyzed by highlighting the row of the first number and, while holding down the left mouse button, moving down the row numbers until all the samples to be analyzed have been highlighted.

- Aspirate the rinse for at least 10 minutes after daily optimization before beginning analysis to avoid carry-over and contamination.

- Click on analyze batch button in the upper left corner of the window. The instrument should now begin analysis.

- The sample results are an arithmetic mean (average) of three replicate readings per analyte. For any analyte with a result greater than the reporting detection limit, the % RSD between the replicate readings must be less than 20.

12.0 DETAILS OF CALCULATIONS

12.1 Each metal is analyzed within the calibration range. Refer to Table 1 for upper limits. Dilutions must be performed if the upper limit is exceeded.

12.2 After the calibration is complete, the software performs a weighted linear regression with a calculated intercept. The weighting factor equals the inverse of the square of the concentration of the standard. Weighting emphasizes measurements in the low concentration region of the calibration curve. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance. The results are calculated from the calibration curve. See Appendix A for the weighted linear calibration algorithm.

12.3 Dilution factors and preparation factors are calculated into the final result, which is computed from the mean of three exposures, during the data upload procedure.

12.3.1 For Liquid Samples:

$$mg/L\ metal\ in\ sample = mg/L\ in\ digestate \times \frac{Final\ Prepared\ Volume\ (mL)}{Initial Volume\ (mL)} \times \frac{Total\ Diluted Volume}{Sample\ aliquot}$$

12.3.2 For Solid Samples:

$$mg/kg\ metal\ in\ sample = mg/L\ in\ digestate \times \frac{Final\ Prepared\ Volume\ (mL)}{Initial Weight\ (g)} \times \frac{Total\ Diluted Volume}{Sample\ aliquot}$$

12.4 LCS

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.5 Spike Percent Recovery is calculated as follows:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.6 Relative Percent Difference (RPD) is calculated as follows:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.7 Percent Difference is calculated as follows:

$$\%D = \left[\frac{|C_1 - C_2|}{C_1}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.8 Adjusted Analyte Intensity is calculated from the internal standard as follows:

$$\frac{A_{meas}}{I_{meas}} \times I_{std} = A_{adjusted}$$

where:

A_{meas} = Measured intensity of analyte
$A_{adjusted}$ = Adjusted intensity of analyte
I_{meas} = Measured intensity of the internal standard
I_{std} = Intensity of the internal standard in the blank solution analyzed prior to calibration

12.9 See Figure 12.1 for a sample calculation summary.

13.0 QUALITY CONTROL REQUIREMENTS

Overview

13.1 Refer to Section 10.0 for instrument calibration and instrument QC samples. Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC Samples. The QC samples are prepared and digested identically to the analytical samples. The following QC are digested and or analyzed with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Table 13-1, 13-4, and 13-6.

Batch Quality Control

13.2 Method blank (Prep Blank (PB)) - An aliquot of DI water that is digested with the sample batch and contains all reagents identical with the sample.

13.3 LCS, LCSD - A LCS or LCS/LSCD must be analyzed using the same sample preparations, analytical methods, and QA/QC procedures used for test samples. One LCS must be prepared and analyzed for each sample batch of 20 samples. Acceptance ranges are 80–120% for method 6020 and 85–115% for 200.8. The final concentration is outlined in Table 13-2. QC Acceptance ranges are outlined in Tables 13-3 and 13-5.

13.4 Duplicate sample analysis (200.8 only) - Analyze one duplicate sample in a batch of twenty samples or less. The formulas for calculation of the RPD

between the duplicate determinations is located in Section 12.6. A control limit of 20% RPD must not be exceeded for analyte values greater than 100 times the IDL. If the control limit is exceeded, the reason for the out of control situation must be corrected and any samples analyzed during the out of control condition reanalyzed.

13.5 MS and MSD – A sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spike with 0.5 mL of CAL-MS-1 for each 50 mL of sample. The final concentration spiked into the two spiked samples is outlined in Table 13-2 Batches that include samples for method 200.8 must include a spiked sample for every ten 200.8 samples.

Interference Tests – Post Digestion

13.6 Dilution Test - If the analyte concentration is within the linear range of the instrument and is a factor of at least 100 times the MDL, the analysis of a five-fold dilution of the sample must agree within 10% of the original determination. If not, an interference must be suspected. The dilution is prepared by adding 1 mL of sample to 4 mL of 5% HNO_3.

One dilution test must be performed for each twenty samples or less of each matrix in a sample batch.

13.7 Post digestion spike - An analyte spike added to a portion of a prepared sample or its dilution must be recovered to within 75-125% of the known value of the spike (80-120% for 6020A) or within laboratory derived acceptance criteria. The final concentration spiked into the two spiked samples is outlined in Table 13-2. The spike value should be based upon the indigenous level of the analyte in the sample. If the spike is not recovered within the acceptance limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. The results of the dilution must agree within 10% of the original determination (see Section 13.6). The use of the method of standard additions may be used to compensate for matrix effects.

Control of Nonconforming Data

13.8 The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis.

13.8.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC is an analysis, as outlined in Sections 11.0 and 13.0 and Tables 13-1, 13-4, and 13-6 does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Sections 11.0 and 13.0 and Tables 13-1, 13-4, and 13-6.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.8.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.8.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An ICV, CCV or LCS recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are non-detect.

- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client samples into Kobra. This is done via Microbac's customized upload program. When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and RLs. Any elements that are not to be reported must be checked in the "Excl" box next to the element. This will be determined by the primary analyst through real time review of all QC elements as summarized in Tables 13-1, 13-4, and 13-6. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating**.** The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the hardcopy raw data with QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and Kobra workgroup reports. The Data Review Checklist acts as a cover page and must be archived with the hardcopy data. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. The secondary reviewer also signs and dates the Data Review Checklist.

This review also consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness.

When all levels of review have been completed. The elements being reported on each sample are taken to a done status in Kobra.

14.2 See figure 14.1 for an example date review checklist.

Uncontrolled Copy

15.0 PREVENTIVE MAINTENANCE

The following sections describe some commonly occurring problems and proposed solutions.

15.1 Poor recovery on selected QC Analytes: If poor recoveries are obtained on only particular analytes in a Quality Control Standard and it had been verified that the sample has been properly prepared, it may be possible that the problem could be related to a problem with the internal standard used. Look at the entire list of elements grouped with an internal standard. If the results for all elements are not satisfactory but the results for other elements not grouped with that internal standard are acceptable, there could be a problem with the internal standard used for that grouping.

15.1.1 Try using a different internal standard and reprocessing the data.

15.1.2 Look at the monitored intensities of the internal standards. If the internal standard used for the elements with unacceptable results is not within the allowable range or the percent recovery for this internal standard significantly different that the others, use a different internal standard and reprocess the data.

15.2 Poor relative standard deviation (precision (RSD)) on standards and samples.

15.2.1 Poor RSDs can be caused by many things. First check that the interface cones are in good condition and the orifices of both cones are round and of the proper size.

15.2.2 Check that the nebulizer is operating properly by checking the aerosol with the plasma off and the spray chamber removed. Turn on the nebulizer gas and the peristaltic pump - must be a visible aerosol leaving the spray chamber. If there is not, clean or replace the nebulizer.

15.2.3 Check that the peristaltic pump tubing is in good condition and not worn. When the autosampler probe is removed and reinserted in the wash solution an air bubble will be visible in the tubing. Watch the progress of this bubble and adjust the tension on the pump tubing so the flow is smooth without any pulsations.

15.3 Sequence occurs improperly on startup of a batch analysis. The sample file has either not been saved or re-opened properly. Save the sample file and then reopen this same sample file. The full path name of the method must appear in the Method field of the sample list.

15.4 Further troubleshooting procedures for the NexION 300X can be found in Appendix A of the NexION Software Reference Guide Version 1.0 located in the Help menu in the instrument software.

15.5 The instrument configuration for the NexION 300S can be found in the electronic Maintenance Log header. The template maintenance log is found at number 13197 in KOBRA. Replacement of instrument components will be recorded in the relevant daily maintenance log and updated in the log header template as needed.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams are as follows:

Metals Laboratory - acid waste is neutralized and disposed of in the sewer per agreement with the city of Marietta.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 - Laboratory Waste management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job descriptions. As a hazardous generator, we are subject to inspection from the Ohio EPA.

Uncontrolled Copy

17.0 REFERENCES

17.1 **"**Inductively Coupled Plasma-Mass Spectrometry, US EPA SW-846 Method 6020, Revision 0", September 1994, EPA Publication SW-846.

17.2 **"**Inductively Coupled Plasma-Mass Spectrometry, US EPA SW-846 Method 6020A, Revision 1", February 2007, EPA Publication SW-846.

17.3 NexION 300X ICP-MS Software Reference Guide, 2009 Version 1.0, Perkin-Elmer Corporation.

17.4 "Methods for the Determination of Metals in Environmental Samples - Supplement 1", EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942, Method 200.8 Rev. 5.4.

17.5 Microbac SOP ME407 "Microwave Digestion for Aqueous Matrices – SW3015".

17.6 Microbac SOP ME406 "Microwave Digestion of Sediments, Sludges, Soils and Oils 3051A".

17.7 Microbac SOP LQAP "Laboratory Quality Assurance Plan".

17.8 Microbac SOP 45 "Method Validation Procedures".

17.9 Microbac SOP GP-CAPA "Corrective Action/Preventative Action: Initiating, Tracking and Monitoring".

17.10 Microbac SOP GP-RCA "Root Cause Analysis".

17.11 Microbac SOP 33 "Laboratory Waste Management".

Uncontrolled Copy

Appendix A

Weighted Linear Calibration Algorithm

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_i x$$

Where y is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

where:

x = concentration value of the standard
y = measured intensity of the standard
n = number of standards
i = index for the standards
b_0 = intercept
b_1 = slope
COC = correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:

$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:

$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Uncontrolled Copy

Weighted Linear Calibration Algorithm (continued)

Correlation Coefficient: $$COC = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^{\,2}\right]\left[(w_1 w_5) - w_4^{\,2}\right]}}$$

Uncontrolled Copy

Table 1
NexION 300X IDLs, MDLs, and Linear Ranges

Analyte	Symbol	Mass	IDL* (ug/L)	Linear Range ** (ug/L)	Water MDL (ug/L)	Water RL (ug/L)	Soil MDL (mg/Kg)	Soil RL mg/Kg
Vanadium	V	51	0.036	100	0.5	1.0	0.25	0.5
Chromium	Cr	52	0.124	100	1.0	2.0	0.2	0.4
Manganese	Mn	55	0.017	100	1.0	2.0	0.1	0.2
Cobalt	Co	59	0.008	100	0.5	1.0	0.25	0.5
Nickel	Ni	60	0.020	100	2.0	4.0	0.4	0.8
Copper	Cu	65	0.206	100	1.0	2.0	0.3	0.6
Zinc	Zn	66	0.557	100	12.5	25.0	1.25	2.5
Arsenic	As	75	0.119	100	0.5	1.0	0.15	0.3
Selenium	Se	82	0.258	100	0.5	1.0	0.1	0.2
Silver	Ag	107	0.034	100	0.5	1.0	0.1	0.2
Cadmium	Cd	111	0.039	100	0.3	0.6	0.05	0.1
Antimony	Sb	123	0.026	100	0.5	1.0	0.05	0.1
Barium	Ba	135	0.021	100	1.5	3.0	0.15	0.3
Thallium	Tl	203	0.024	100	0.1	0.2	0.02	0.04
Lead	Pb	208	0.014	100	0.5	1.0	0.1	0.2
Uranium	U	238	0.006	100	0.5	1.0	0.2	0.4

* Updated quarterly
** Values reported are 90% of the verified upper linear range

Uncontrolled Copy

Table 2
Table of Isotopes Monitored and Equations Used

Analyte	Symbol	Isotopes Monitored	Correction Equations
Aluminum	Al	27	
Antimony	Sb	123	Sb 123 = Sb 123 – 0.127189 * Te 125
Arsenic	As	75	As 75 = As 75 – 3.127 * (ArCl 77 – (0.87496 * Se 82))-2.73*Kr 83
Barium	Ba	135	
Beryllium	Be	9	
Cadmium	Cd	111,114	Cd 111 = Cd 111 – 0.0014 * Mo 98
Chromium	Cr	52,53	
Cobalt	Co	59	
Copper	Cu	65	
Lead	Pb	206,207,208	Pb 208 = Pb 208 + 1 * Pb 206 + 1 * Pb 207
Manganese	Mn	55	
Nickel	Ni	60	
Selenium	Se	77,82	Se 82 = Se 82 – 0.6 * Kr 83
Silver	Ag	107	
Thallium	Tl	203,205	
Vanadium	V	51	V 51 = V 51 – 3.127 * [ClO 53 – (0.113 * Cr 52)]
Zinc	Zn	66	
Internal Standards			
Lithium	Li	6	
Scandium	Sc	45	
Yttrium	Y	89	
Rhodium	Rh	103	
Indium	In	115	
Terbium	Tb	159	
Bismuth	Bi	209	
Germanium	Ge	72	
(Information Only)			
Molybdenum	Mo	98	Mo 98 = Mo 98 – 0.110588 * Ru 101
Uranium	U	238	
Calcium	Ca	44	
Magnesium	Mg	24	
Sodium	Na	23	
Potassium	K	39	
Iron	Fe	54	Fe 54 = Fe 54 – 0.028226 * Cr 52

Uncontrolled Copy

Table 7-1
Internal Standard Solution

Analyte	Symbol	Stock Concentration (mg/L)	Secondary Concentration (mg/L)
Lithium	Li	100	0.025
Scandium	Sc	100	0.025
Germanium	Ge	1000	0.25
Yttrium	Y	100	0.025
Indium	In	100	0.025
Terbium	Tb	100	0.025
Bismuth	Bi	100	0.025

Table 7-2
Tuning Solution and Daily Performance Check Solution

Analyte	Symbol	Stock Concentration (mg/L)	Secondary Concentration (mg/L)
Aluminum	Al	10	0.001
Antimony	Sb	10	0.001
Arsenic	As	10	0.001
Barium	Ba	10	0.001
Beryllium	Be	10	0.001
Bismuth	Bi	100	0.001
Cadium	Cd	10	0.001
Calcium	Ca	1000	0.001
Cerium	Ce	1000	0.001
Chromium	Cr	10	0.001
Cobalt	Co	10	0.001
Copper	Cu	10	0.001
Indium	In	100	0.001
Iron	Fe	1000	0.001
Lead	Pb	10	0.001
Lithium	Li	100	0.001
Magnesium	Mg	1000	0.001
Manganese	Mn	10	0.001
Nickel	Ni	10	0.001
Potassium	K	1000	0.001
Scandium	Sc	100	0.001
Selenium	Se	10	0.001
Silver	Ag	10	0.001
Sodium	Na	1000	0.001
Terbium	Tb	100	0.001
Thallium	Tl	10	0.001
Uranium	U	10	0.001
Vanadium	V	10	0.001
Yttrium	Y	100	0.001
Zinc	Zn	10	0.001

Uncontrolled Copy

Table 7-3
Calibration and CCV Solutions

Analyte	Symbol	Stock Conc. (mg/L)	S1 (mg/L)	S2 (mg/L)	CCV/S3 (mg/L)	S4 (mg/L)
Aluminum	Al	10	0	0.00005	0.05	0.1
Antimony	Sb	10	0	0.00005	0.05	0.1
Arsenic	As	10	0	0.00005	0.05	0.1
Barium	Ba	10	0	0.00005	0.05	0.1
Beryllium	Be	10	0	0.00005	0.05	0.1
Calcium	Ca	1000	0	0.005	5	10
Cadmium	Cd	10	0	0.00005	0.05	0.1
Chromium	Cr	10	0	0.00005	0.05	0.1
Cobalt	Co	10	0	0.00005	0.05	0.1
Copper	Cu	10	0	0.00005	0.05	0.1
Iron	Fe	1000	0	0.005	5	10
Lead	Pb	10	0	0.00005	0.05	0.1
Magnesium	Mg	1000	0	0.005	5	10
Manganese	Mn	10	0	0.00005	0.05	0.1
Molybdenum	Mo	1000	0	0.0001	0.1	0.2
Nickel	Ni	10	0	0.00005	0.05	0.1
Potassium	K	1000	0	0.005	5	10
Selenium	Se	10	0	0.00005	0.05	0.1
Silver	Ag	10	0	0.00005	0.05	0.1
Sodium	Na	1000	0	0.005	5	10
Titanium	Ti	1000	0	0.0001	0.1	0.2
Tin	Sn	1000	0	0.00005	0.05	0.1
Thallium	Tl	10	0	0.00005	0.05	0.1
Uranium	U	1000	0	0.00005	0.05	0.1
Vanadium	V	10	0	0.00005	0.05	0.1
Zinc	Zn	10	0	0.00005	0.05	0.1

Uncontrolled Copy

Table 7-4
ICV Solutions

Analyte	Symbol	Stock Concentration (mg/L)	ICV Concentration (mg/L)
Aluminum	Al	10	0.05
Antimony	Sb	10	0.05
Arsenic	As	10	0.05
Barium	Ba	10	0.05
Beryllium	Be	10	0.05
Calcium	Ca	1000	5
Cadmium	Cd	10	0.05
Chromium	Cr	10	0.05
Cobalt	Co	10	0.05
Copper	Cu	10	0.05
Iron	Fe	1000	5
Lead	Pb	10	0.05
Magnesium	Mg	1000	5
Manganese	Mn	10	0.05
Molybdenum	Mo	1000	0.1
Nickel	Ni	10	0.05
Potassium	K	1000	5
Selenium	Se	10	0.05
Silver	Ag	10	0.05
Sodium	Na	1000	5
Titanium	Ti	1000	0.1
Tin	Sn	1000	0.05
Thallium	Tl	10	0.05
Uranium	U	10	0.05
Vanadium	V	10	0.05
Zinc	Zn	10	0.05

Table 7-5
Interference Check Solutions

Analyte	Symbol	Stock Concentration (mg/L)	ICSA Concentration (mg/L)	ICSAB Concentration (mg/L)
Aluminum	Al	1000	5	5
Antimony	Sb	10	0	0.1
Arsenic	As	10	0	0.1
Barium	Ba	10	0	0.1
Beryllium	Be	10	0	0.1
Calcium	Ca	3000	15	15
Cadmium	Cd	10	0	0.1
Chromium	Cr	10	0	0.1
Cobalt	Co	10	0	0.1
Copper	Cu	10	0	0.1
Iron	Fe	2500	12.5	12.5
Lead	Pb	10	0	0.1
Magnesium	Mg	1000	5	5
Manganese	Mn	10	0	0.1
Molybedenum	Mo	20	0.1	0.1
Nickel	Ni	10	0	0.1
Potassium	K	1000	5	5
Selenium	Se	10	0	0.1
Silver	Ag	10	0	0.1
Sodium	Na	2500	12.5	12.5
Thallium	Tl	10	0	0.1
Titanium	Ti	20	0.1	0.1
Uranium	U	10	0	0.1
Vanadium	V	10	0	0.1
Zinc	Zn	10	0	0.1
Chlorine	Cl^-*	21215	106.1	106.1
Carbon	C*	2000	10	10
Phosphorus	P*	1000	5	5
Sulfur	S*	1000	5	5

* Although these are non-metals, they are essential for verifying the absence of mass interferences.

Uncontrolled Copy

Table 7-6
LLICV / LLCCV Solutions

Analyte	Symbol	Volume of Single Element Standard (mL)	Intermediate Concentration (mg/L)	LLICV / LLCCV Concentration (ug/L)
Antimony	Sb	0.4	8	0.4
Arsenic	As	0.4	8	0.4
Barium	Ba	0.75	15	0.75
Cadmium	Cd	0.24	4.8	0.24
Chromium	Cr	0.8	16	0.8
Cobalt	Co	0.4	8	0.4
Copper	Cu	0.8	16	0.8
Lead	Pb	0.2	4	0.2
Manganese	Mn	0.5	10	0.5
Nickel	Ni	1.6	32	1.6
Selenium	Se	0.4	8	0.4
Silver	Ag	0.4	8	0.4
Thallium	Tl	0.08	1.6	0.08
Uranium	U	0.4	8	0.4
Vanadium	V	0.4	8	0.4
Zinc	Zn	6.25	125	6.25

Uncontrolled Copy

Table 13-1
Quality Control Criteria
Total Metals – ICP/MS Method 6020

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	Correlation coefficient must be ≥ 0.998	Investigate, reanalyze the aberrant standard or recalibrate
Initial Calibration Verification (ICV)	After calibration	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Low Level Initial Calibration Verification (LLICV) (at or below RL)	Minimum of once per calibration prior to sample analysis unless multipoint calibration with low std at or below RL is performed	All analyte(s) within ± 50% of expected value for SW-846 and AFCEE QAP 3.1. All analytes within ± 20 % of expected value for DOD Version 4 and AFCEE QAP 4.0	Correct problem then reanalyze
Mass Calibration and Resolution	Daily prior to calibration 5 replicates	Measured mass within ± 0.05 amu of exact mass; Resolution ± 0.03 amu RSD < 5% with 10% peak height	Retune Instrument
Initial Calibration Blank (ICB)	After ICV	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, Redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
ICP Interference Check	Run at beginning of each run (12 hour maximum)	All non-spiked analytes <2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) Spiked analytes ± 20% of expected value.	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
Matrix spike/ Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	75 - 125% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (optional)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike
Internal Standards	Every sample	Intensity >30% < 120% of that of initial calibration blank. See Appendix I for OVAP requirements.	Stop analysis, investigate, dilute sample if interference is apparent or recalibrate and reanalyze affected samples.
Daily Performance Report (Manufacturer's recommendations)	Daily after Mass calibration and Resolution	Background < 1 cps @ Mass 220 Mg sensitivity > 15000 cps CeO/Ce = ≤ 0.025 Ce^{++}/Ce = < 0.03 Be sensitivity > 2000 In sensitivity > 40000 U sensitivity > 30000	Stop analysis, investigate, reanalyze. If still outside limit, examine and replace cones.

(1) Acceptance criteria are project specific. Consult QAPP.

Uncontrolled Copy

Table 13-2
Spike Concentrations
Total Metals – ICP/MS Method 6020

Analyte	Symbol	LCS		MS/MSD		Post Spike	
		Water mg/L	Soil mg/Kg	Water mg/L	Soil mg/Kg	Water mg/L	Soil mg/Kg
Silver	Ag	0.125	10.0	0.125	10.0	0.125	10.0
Antimony	Sb	0.125	10.0	0.125	10.0	0.125	10.0
Arsenic	As	0.125	10.0	0.125	10.0	0.125	10.0
Lead	Pb	0.125	10.0	0.125	10.0	0.125	10.0
Selenium	Se	0.125	10.0	0.125	10.0	0.125	10.0
Thallium	Tl	0.125	10.0	0.125	10.0	0.125	10.0
Barium	Ba	0.125	10.0	0.125	10.0	0.125	10.0
Cadmium	Cd	0.125	10.0	0.125	10.0	0.125	10.0
Chromium	Cr	0.125	10.0	0.125	10.0	0.125	10.0
Cobalt	Co	0.125	10.0	0.125	10.0	0.125	10.0
Copper	Cu	0.125	10.0	0.125	10.0	0.125	10.0
Manganese	Mn	0.125	10.0	0.125	10.0	0.125	10.0
Nickel	Ni	0.125	10.0	0.125	10.0	0.125	10.0
Uranium	U	0.125	10.0	0.125	10.0	0.125	10.0
Vanadium	V	0.125	10.0	0.125	10.0	0.125	10.0
Zinc	Zn	0.125	10.0	0.125	10.0	0.125	10.0

Table 13-3
Microbac's QA Objectives and Analytical Methods for Inorganic Metals Analyses of Groundwater

Analyte	Symbol	CAS #	EPA SW-846 Method	Accuracy (% Recovery)	Precision (% RPD)	MDL Water (ug/L)	Reporting Limits Water (ug/L)
Antimony	Sb	7440-36-0	6020	80-120	20	0.5	1.0
Arsenic	As	7440-38-2	6020	80-120	20	0.5	1.0
Lead	Pb	7439-92-1	6020	80-120	20	0.5	1.0
Selenium	Se	7782-49-2	6020	80-120	20	0.5	1.0
Thallium	Tl	7440-28-0	6020	80-120	20	0.1	0.2
Silver	Ag	7440-22-4	6020	80-120	20	0.5	1.0
Barium	Ba	7440-39-3	6020	80-120	20	1.5	3.0
Cadmium	Cd	7440-43-9	6020	80-120	20	0.3	0.6
Chromium	Cr	7440-47-3	6020	80-120	20	1.0	2.0
Cobalt	Co	7440-48-4	6020	80-120	20	0.5	1.0
Copper	Cu	7440.50-8	6020	80-120	20	1.0	2.0
Manganese	Mn	7439-96-5	6020	80-120	20	1.0	2.0
Nickel	Ni	7440-02-0	6020	80-120	20	2.0	4.0
Uranium	U	7440-61-1	6020	80-120	20	0.5	1.0
Vanadium	V	7440-62-2	6020	80-120	20	0.5	1.0
Zinc	Zn	7440-66-6	6020	80-120	20	12.5	25.0

Table 13-3 (continued)
Microbac's QA Objectives and Analytical Methods for
Inorganic Metals Analyses of Solid Waste

Analyte	Symbol	CAS #	EPA SW-846 Method	Accuracy (% Recovery)	Precision (% RPD)	MDL Soil (ug/Kg)	Reporting Limits Soil (ug/Kg)
Antimony	Sb	7440-36-0	6020	80-120	20	50	100
Arsenic	As	7440-38-2	6020	80-120	20	150	300
Lead	Pb	7439-92-1	6020	80-120	20	100	200
Selenium	Se	7782-49-2	6020	80-120	20	100	200
Thallium	Tl	7440-28-0	6020	80-120	20	20	40
Silver	Ag	7440-22-4	6020	80-120	20	100	200
Barium	Ba	7440-39-3	6020	80-120	20	150	300
Cadmium	Cd	7440-43-9	6020	80-120	20	50	100
Chromium	Cr	7440-47-3	6020	80-120	20	200	400
Cobalt	Co	7440-48-4	6020	80-120	20	250	500
Copper	Cu	7440-50-8	6020	80-120	20	300	600
Manganese	Mn	7439-96-5	6020	80-120	20	100	200
Nickel	Ni	7440-02-0	6020	80-120	20	400	800
Uranium	U	7440-61-1	6020	80-120	20	200	400
Vandium	V	7440-62-2	6020	80-120	20	250	500
Zinc	Zn	7440-66-6	6020	80-120	20	1250	2500

Table 13-4
Quality Control Criteria
Total Metals – ICP/MS Method 200.8

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial Calibration	Daily at beginning of analytical run	Correlation coefficient must be ≥ 0.998	Investigate, reanalyze the aberrant standard or recalibrate
Initial Calibration Verification (ICV)	After calibration	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	85 - 115%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Low Level Initial Calibration Verification (LLICV) (at or below RL)	Minimum of once per calibration prior to sample analysis unless multipoint calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value for SW-846 / EPA Method 200.8 and AFCEE QAP 3.1. All analytes within ± 20 % of expected value for DOD Version 4 and AFCEE QAP 4.0	Correct problem then reanalyze
Mass Calibration and Resolution	Daily, minimum of 5 replicates	Measured mass within 0.05 amu of exact mass; RSD < 5% Peak width < 0.75 amu at 5% peak height Resolution ± 0.03	Retune Instrument
Initial Calibration Blank (ICB)	After ICV	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, redigest batch (required for Ohio VAP projects) or qualify data and address in narrative
ICP Interference check	Run at beginning of each run (12 hour maximum)	All non-spiked analytes < RL (unless they are a verified trace impurity from one of the spiked analytes). Spiked analytes ± 20% of expected value.	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 85 - 115%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per ten 200.8 samples	70 - 130% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike
Internal Standards	Every sample	60 - 125%	Stop analysis, investigate, dilute sample if interference is apparent or recalibrate and reanalyze affected samples.
Daily Performance Report (Manufacturer's recommendations)	Daily after Mass Calibration and Resolution	Background < 1 cps @ Mass 220 Mg sensitivity > 15000 cps CeO/Ce = ≤ 0.025 Ce^{++}/Ce= < 0.03 Be sensitivity > 2000 In sensitivity > 40000 U sensitivity > 30000	Stop analysis, investigate, reanalyze. If still outside limit, examine and replace cones.
Quality Control Sample (QCS) (120 ppb)	Run Quarterly	90-110%	Stop analysis, investigate, reanalyze. If still outside limit, recalibrate.

(1) Acceptance criteria are project specific. Consult QAPP.

Table 13-5
Microbac's QA Objectives and Analytical Methods for Inorganic Metals Analyses

Analyte	Symbol	CAS #	EPA SW-846 Method	Accuracy (% Recovery)	Precision (% RPD)	MDL Water (ug/L)	Reporting Limits Water (ug/L)
Antimony	Sb	7440-36-0	200.8	85-115	20	0.5	1.0
Arsenic	As	7440-38-2	200.8	85-115	20	0.5	1.0
Lead	Pb	7439-92-1	200.8	85-115	20	0.5	1.0
Selenium	Se	7782-49-2	200.8	85-115	20	0.5	1.0
Thallium	Tl	7440-28-0	200.8	85-115	20	0.1	0.2
Silver	Ag	7440-22-4	200.8	85-115	20	0.5	1.0
Barium	Ba	7440-39-3	200.8	85-115	20	1.5	3.0
Cadmium	Cd	7440-43-9	200.8	85-115	20	0.3	0.6
Chromium	Cr	7440-47-3	200.8	85-115	20	1.0	2.0
Cobalt	Co	7440-48-4	200.8	85-115	20	0.5	1.0
Copper	Cu	7440.50-8	200.8	85-115	20	1.0	2.0
Manganese	Mn	7439-96-5	200.8	85-115	20	1.0	2.0
Nickel	Ni	7440-02-0	200.8	85-115	20	2.0	4.0
Uranium	U	7440-61-1	200.8	85-115	20	0.5	1.0
Vanadium	V	7440-62-2	200.8	85-115	20	0.5	1.0
Zinc	Zn	7440-66-6	200.8	85-115	20	12.5	25.0

Uncontrolled Copy

Table 13-6
Quality Control Criteria
Total Metals – ICP/MS Method 6020A

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial Calibration	Daily at beginning of analytical run	Correlation coefficient must be ≥ 0.998	Investigate, reanalyze the aberrant standard or recalibrate
Initial Calibration Verification (ICV)	After calibration	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Low Level Initial and Continuing Calibration Verification (LLICV / LLCCV) (at or below RL)	Following Calibration prior to sample analysis and minimally at the close of each analytical batch	All analyte(s) within ± 30% of expected value or QAPP specific limits	Correct problem then reanalyze
Mass Calibration and Resolution	Daily prior to calibration minimum of 4 replicates	Measured mass within 0.05 amu Of exact mass; Peak within < 0.7 amu at 10% peak height RSD < 5% resolution ± 0.03	Retune Instrument
Initial Calibration Blank (ICB)	After ICV	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RL or < MDL x 2 < 10% of RL, regulatory limit or lowest sample concentration	Stop analysis, investigate, reanalyze. If still > limit, redigest batch or qualify data and address in narrative
ICP interference check	Run at beginning of each run (12 hour maximum)	All non-spiked analytes <2 x MDL (unless they are a verified trace impurity from one of the spiked analytes). Spiked analytes ± 20% of expected value.	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	75 - 125% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (optional)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
Post digestion spike	5%, or minimum of 1 per batch	80 – 120% recovery	Serial dilution
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike
Internal Standards	Every sample	≥ 70% recovery of that of initial calibration blank. No upper limit.	Stop analysis, investigate, dilute sample if interference is apparent or recalibrate and reanalyze affected samples.
Daily Performance Report (Manufacturer's recommendations)	Daily after Mass Calibration and Resolution	Background < 1 cps @ Mass 220 Mg sensitivity > 15000 cps CeO/Ce = ≤ 0.025 Ce^{++}/Ce= < 0.03 Be sensitivity > 2000 In sensitivity > 40000 U sensitivity > 30000	Stop analysis, investigate, reanalyze. If still outside limit, examine and replace cones.

(1) Acceptance criteria are project specific. Consult QAPP.

Uncontrolled Copy

Figure 12.1

Example 6020 Calculations
Perkin Elmer NexION 300X

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and three standards.

2.0 Calculating the concentration (C) of an element in water using data from prep log, run log, and quantitation report (note:the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Final volume	100
Vi = Initial volume	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in (ug/L)	0.25

3.0 Calculating the concentration (C) of an element in soil using data from prep log, run log, and quantitation report (note: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Final volume	200
Vi = Initial volume	0.5
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in (ug/kg)	40

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where:	**Example:**
Cx = Concentration calculated as received (wet basis)	40
Px = Percent solids of sample (%wt)	80
$Cdry$ = Concentration calculated as dry weight (ug/kg)	50

50 ug/kg = 0.050 mg/kg

Uncontrolled Copy

Figure 14.1

Checklist ID: 64277

Microbac Laboratories Inc.

Data Checklist

Date: 06-MAR-2012
Analyst: JYH
Analyst: NA
Method: 6020
Instrument: ICP-MS2
Curve Workgroup: 391549
Runlog ID: 45519
Analytical Workgroups: 389877,391204

Calibration/Linearity	X
ICV/CCV	X
ICV RSD <= 3% (EPA 200.7 only)	
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	X
Case Narrative	
Client Forms	X
Level X	
Level 3	
Level 4	003
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Primary Reviewer	JYH
Secondary Reviewer	
Comments	

Primary Reviewer: Secondary Reviewer:

CHECKLIST1 - Modified 03/05/2008
Generated: MAR-09-2012 19:49:05

Microbac

ATTACHMENT B
LABORATORY QUALITY ASSURANCE PLAN

Uncontrolled Copy

MICROBAC

STANDARD OPERATING PROCEDURES
MICROBAC LABORATORIES, INC.
OHIO VALLEY DIVISION
LABORATORY QUALITY ASSURANCE PLAN

Issue / Implementation Date: 28 March 2014

Last Review Date: 28 March 2014

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750
(740) 373-4071

Wade T. DeLong, Quality Assurance Officer — Date: 3-26-14

Leslie S. Bucina, Laboratory Manager — Date: 3/27/14

David E. Vandenberg, Managing Director — Date: 3/27/14

Uncontrolled Copy

Laboratory Quality Assurance Plan

Table of Contents

Section 1.0 SIGNATURE PAGE 1

Section 2.0 TABLE OF CONTENTS 2

Section 3.0 STATEMENT OF POLICY 6

Section 4.0 ORGANIZATION AND RESPONSIBILITY 7

4.1 Responsibilities and Authorities 8
4.2 Key Personnel 8
4.3 Coverage for Temporary Absence of Key Personnel 8

Section 5.0 GENERAL POLICIES AND PROCEDURES 12

5.1 NELAC Policy Statement 12
5.2 Employee Protection from Undue Pressure 12
5.3 Ethics Training 12
5.4 Departures from Documented Policies and Procedures 12
5.5 Reviewing New Work and Capacity Evaluation 13
5.6 Complaints 13
5.7 Document Control and Maintenance 14
5.8 Laboratory Records 15
5.9 Analyst Training and Demonstration of Capability 15
5.10 Protecting Confidentiality and Proprietary Rights 16
5.11 Identification of Approved Signatories 17
5.12 Monitoring and Controlling System Time 18
5.13 Changes of Ownership/Key Staff 18
5.14 Method References 18
5.15 A2LA Advertising Policy 18
5.16 Purchasing Supplies and Services 19
5.17 Uncertainty – Estimating Uncertainty of Measurements 19
5.18 Review of Requests, Proposals, and Contracts 19
5.19 Measurement of Traceability 20
5.20 Trip Blank hold times 20

Section 6.0 SAMPLING PROCEDURES 21

6.1 General Sampling Guidelines 21
6.2 Microbac Sampling Containers 22
6.3 Sample Preservation 22
6.4 Sample Documentation 23
6.5 Sampling Equipment Decontamination 24
6.6 Field Waste Disposal Practices 25
6.7 Laboratory Sub-Sampling Procedures 25
6.8 Sample Dilutions 26

Uncontrolled Copy

Section 7.0 SAMPLE CUSTODY 31

7.1 Sampling Kits 31
7.2 Field Custody 31
7.3 Sample Transport 32
7.4 Laboratory Custody Procedures 33
7.5 Sample Receipt and Inspection 34
7.6 Sample Logging Procedures 35
7.7 Sample Storage 36
7.8 Sample Distribution and Tracking 37
7.9 Sample Security 37
7.10 Laboratory Building Security 37
7.11 Sample Subcontracting/Shipping 38
7.12 Electronic Data Security 38
7.13 Automated Field Procedures 39

Section 8.0 ANALYTICAL PROCEDURES, STANDARDS, AND REAGENTS 44

8.1 Analytical Methods 44
8.2 Glassware 45
8.3 Reagents and Solvents 46
8.4 Analytical Standards 49
8.5 Standardization of Titrating Solutions 54

Section 9.0 CALIBRATION PROCEDURES AND FREQUENCY 66

9.1 Instrumentation 66
9.2 General Calibration Requirements 70
9.3 Gas Chromatography 70
9.4 Gas Chromatography/Mass Spectrometry 71
9.5 HPLC-VWD/FD (PNA and Explosives) 74
9.6 IC – Conductivity Detector (Anions) 74
9.7 Atomic Absorption – Cold Vapor 75
9.8 ICP-AES, MS 75
9.9 Support Equipment 76
9.10 Logbooks 78

Section 10.0 PREVENTIVE MAINTENANCE 79

10.1 Routine Maintenance Activities 79
10.2 Contingency Plan 79

Section 11.0 QUALITY CONTROL CHECKS, ROUTINES TO ASSESS PRECISION, ACCURACY AND CALCULATION OF METHOD DETECTION LIMIT 82

11.1 Analytical Methods 82
11.2 The Batch Concept 82
11.3 Batch Quality Control Samples 83
11.4 Quality Assurance Summary Reports 85
11.5 Measuring Precision, Accuracy and Method Detection Limits 86
11.6 Statistical Evaluation of Data 88
11.7 General Control Charting Procedure 89

Uncontrolled Copy

11.8 Method Detection Limits 89
11.9 Quantitation Limits and Reporting Limits 90
11.10 Proficiency Testing Studies (PT) 90

Section 12.0 DATA REDUCTION, REVIEW, VERIFICATION AND REPORTING 93

12.1 Data Reduction 93
12.2 Review of Data and Deliverables 94
12.3 Data Entry and Verification 94
12.4 Report Content and Levels 95
12.5 Data Integrity, Storage and Archive 95

Section 13.0 CONTROL OF NONCONFORMING WORK 98

13.1 Nonconforming Work 98
13.2 Corrective Action 98
13.3 Preventive Action 99
13.4 General Analytical Requirements 99
13.5 Calibration Requirements 99
13.6 Method Blanks 100
13.7 Laboratory Control Samples 100
13.8 Matrix Spikes and Duplicates 101
13.9 Nonconformance Reports 101
13.10 Corrective Action/Preventive Action Reports 102

Section 14.0 PERFORMANCE AND SYSTEMS AUDITS 104

14.1 External Audits 104
14.2 Performance Audits and Proficiency Testing 104
14.3 Internal Audits 104
14.4 Management Review Procedures (Managing Director) 105

Section 15.0 QUALITY ASSURANCE REPORTS 105

15.1 Laboratory Quality Assurance Reports 106
15.2 Quality System Review Report 106
15.3 Special Reports 106

Section 16.0 SPECIAL PROGRAM REQUIREMENTS 106

16.1 Department of Defense 106
16.2 Air Force Center for Environmental Excellence 109

Section 17.0 REFERENCES 110

Uncontrolled Copy

List of Appendices

Appendix A - Definitions 112
Appendix B - Standard Operating Procedures (SOP) Table of Contents 121
Appendix C - NELAP Accredited Analytes 130
Appendix D - Key Personnel Job Descriptions 164

List of Tables

6-1 Glassware Cleaning Protocol 22
6-2 Sample Containers, Preservation and Hold Times 27
8-1 Lab Glassware Cleaning Procedures 55
8-2 Reagent Storage 56
8-3 Standard Preparation and Sources 57
8-4 Standardization of Titrating Solutions 60
9-1 Laboratory Equipment List 66
9-2 Key Ions and Abundance Criteria for BFB 73
9-3 Key Ions and Abundance Criteria for DFTPP 73
9-4 Balance Calibration Acceptance Criteria 78
10-1 Laboratory Instrumentation Preventive Maintenance 80

List of Figures

4-1 Microbac Leadership Circle Chart 9
4-2 Microbac Organizational Chart (Ohio Valley Division) 10
4-3 Microbac Laboratory Floor Plan 11
7-1 Microbac Chain-of-Custody Record Form 40
7-2 Microbac Sample Receipt Form 41
7-3 Microbac Internal Sample Custody Form 42
7-4 Microbac Sample Bottle Label 43
8-1 Example – KOBRA LIMS Reagent Record 61
8-2 Example – KOBRA LIMS Certificate of Analysis Record 62
8-3 Example – KOBRA LIMS Standard Record 63
8-4 Example – KOBRA LIMS Standard Template 64
8-5 Example – KOBRA LIMS Standard Group 65
11-1 Control Chart, Laboratory Control Sample 92
12-1 Data Qualifiers 97
13-1 Microbac Nonconformance Report 102
13-2 Microbac Corrective Action/Preventive Action Report 103

3.0 STATEMENT OF POLICY

This Quality Manual summarizes the policies and operational procedures of Microbac Laboratories, Inc. Ohio Valley Division (OVD), located at 158 Starlite Drive in Marietta, Ohio. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, correction action, and reporting are described. All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) standards adopted in 2003 and 2009 (current as of the date of this publication) and applicable Environmental Protection Agency (EPA) requirements, regulations, guidance, and technical standards. This manual has been prepared in accordance with the guidance documents listed in Section 17.0. Further details on these policies and procedures are contained in Standard Operating Procedures (SOP) and related documents. This Quality Manual, SOPs, and related documentation describe the quality system for Microbac. Top management makes the following quality policy statements and commitments:

- Laboratory management is committed to good professional practice, to the quality of our testing, and to providing excellent service to our customers.

- Microbac's management commitment to providing the highest standard of service, with respect to data quality and information to our clients, is embodied in Microbac's corporate policy on quality assurance.

- The purpose of our management system is to ensure that all of our work shall be performed in an absolutely professional manner, and all data shall be scientifically valid, legally defensible, of known precision and accuracy.

- Management will assure through documented, annual refresher training that all laboratory staff are familiar with relevant and applicable aspects of the quality management, and will implement these policies and procedures in their work.

- Laboratory management has established policies and procedures to comply with the quality system requirements of ISO/IEC 17025:2005, and is committed to a mission of continuous quality improvement.

Every level of management, the laboratory staff, and project scientists are committed to the quality assurance (QA) program described in this Quality Manual and as such ensure that the appropriate facilities and resources are available before producing any analytical results.

All of our work will be performed in accordance with standards developed by the NELAC and any applicable state or EPA regulations or requirements.

This program will be revised as needed to address special client or project requirements and to keep pace with developing technologies, good laboratory practices and total management of testing services.

All new employees are educated and trained in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions. Our written policy for ethical standards of behavior exists in a separate document entitled "Ethics Policy", Microbac SOP ETH01.

Microbac performs chemical analyses for inorganic and organic constituents in water, solid, and hazardous waste. Microbac's goal's are as follows:

- to protect our clients' interests by providing them with fully documented, legally defensible data useable for sound environmental decisions;
- ensure our client's confidentiality through safeguards related to data reporting by telephone, facsimile, modem, diskette, mail or other means;
- shelter our company's two most important resources, our people and our reputation, by maintaining an environment that fosters excellence and;
- guard against and correct performance shortcomings which could erode data or technical quality

Microbac-OVD is licensed and/or accredited by several state programs. A copy of each license and a list of licensed parameters is maintained in a notebook in the laboratory reception area. Another copy is maintained electronically on the network COMMON drive.

4.0 ORGANIZATION AND RESPONSIBILITY

Microbac Laboratories, Inc. has its corporate headquarters in Pittsburgh, Pennsylvania. The corporate organizational chart for Microbac Laboratories, Inc. is given in Figure 4-1. Microbac's laboratory in Marietta, Ohio (Ohio Valley Division) is a full service environmental laboratory facility for analysis of groundwater, municipal and industrial waste water, soil and solid waste. The Ohio Valley Division's organizational chart is presented in Figure 4-2 along with a laboratory floor plan, which is provided in Figure 4-3. Microbac's Analytical Statement of Qualifications provides the qualifications and experience summaries for key professionals within Microbac. The duties and responsibilities of all key positions at the Ohio Valley facility are provided in Appendix D.

4.1 Responsibilities and Authorities

All employees, including the Managing Director, have the responsibility and authority to identify and report nonconforming work, to initiate actions to prevent QMS departures and nonconformances, to participate in internal audits and corrective action investigations, and to adhere to the QMS procedures.

4.2 Key Personnel

See Appendix D for job descriptions.

4.3 Coverage for Temporary Absence of Key Personnel

Microbac has sufficient staff redundancy and level of experience to cover for extended absence of all personnel described above. The production and report review duties of the Laboratory Director are covered by the Technical Director/ Quality Assurance Officer (QAO). The duties of the QAO are delegated to a senior staff QC chemist with oversight from the Laboratory Director. Responsibilities of departmental supervisors are delegated to the senior chemist or analyst in the department, with oversight from the Laboratory Director. A senior programmer is assigned the duties of the IT (Information Technologies) Manager in his absence. The Managing Director/Laboratory Director will perform the duties of Customer Service Manager(s) in the case of absence. All key personnel have an average of over ten years experience and are cross-trained for multiple positions.

Figure 4-1

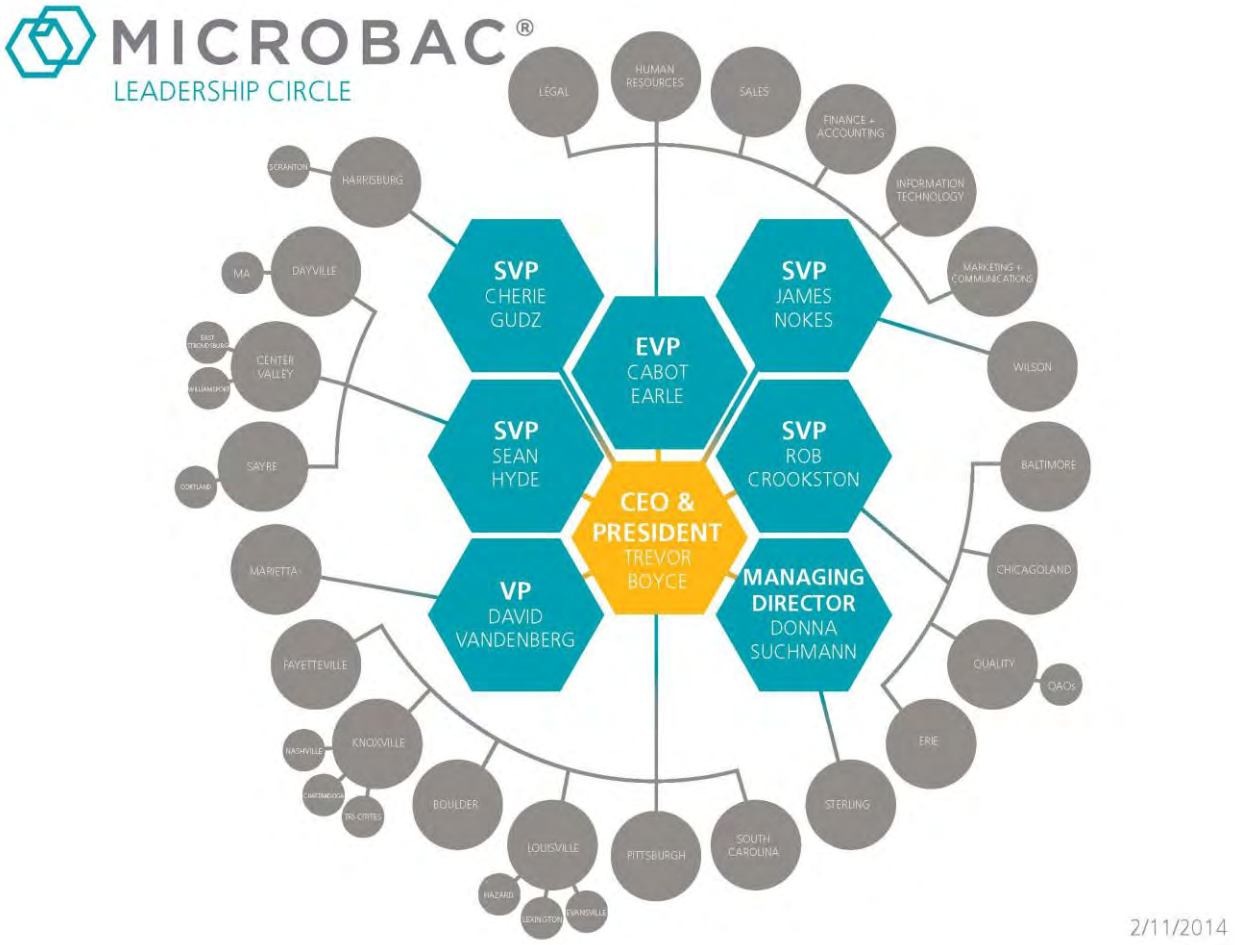

MICROBAC®
LEADERSHIP CIRCLE
LEGAL
HUMAN RESOURCES
SALES
FINANCE + ACCOUNTING
INFORMATION TECHNOLOGY
MARKETING + COMMUNICATIONS
WILSON
BALTIMORE
CHICAGOLAND
QUALITY
QAOs
ERIE
STERLING
SOUTH CAROLINA
PITTSBURGH
LOUISVILLE
HAZARD
LEXINGTON
EVANSVILLE
BOULDER
KNOXVILLE
NASHVILLE
CHATTANOOGA
TRI-CITIES
FAYETTEVILLE
MARIETTA
SAYRE
CORTLAND
CENTER VALLEY
MA
DAYVILLE
SCRANTON
HARRISBURG
SVP CHERIE GUDZ
SVP JAMES NOKES
EVP CABOT EARLE
SVP SEAN HYDE
SVP ROB CROOKSTON
CEO & PRESIDENT TREVOR BOYCE
VP DAVID VANDENBERG
MANAGING DIRECTOR DONNA SUCHMANN
2/11/2014

Figure 4-2

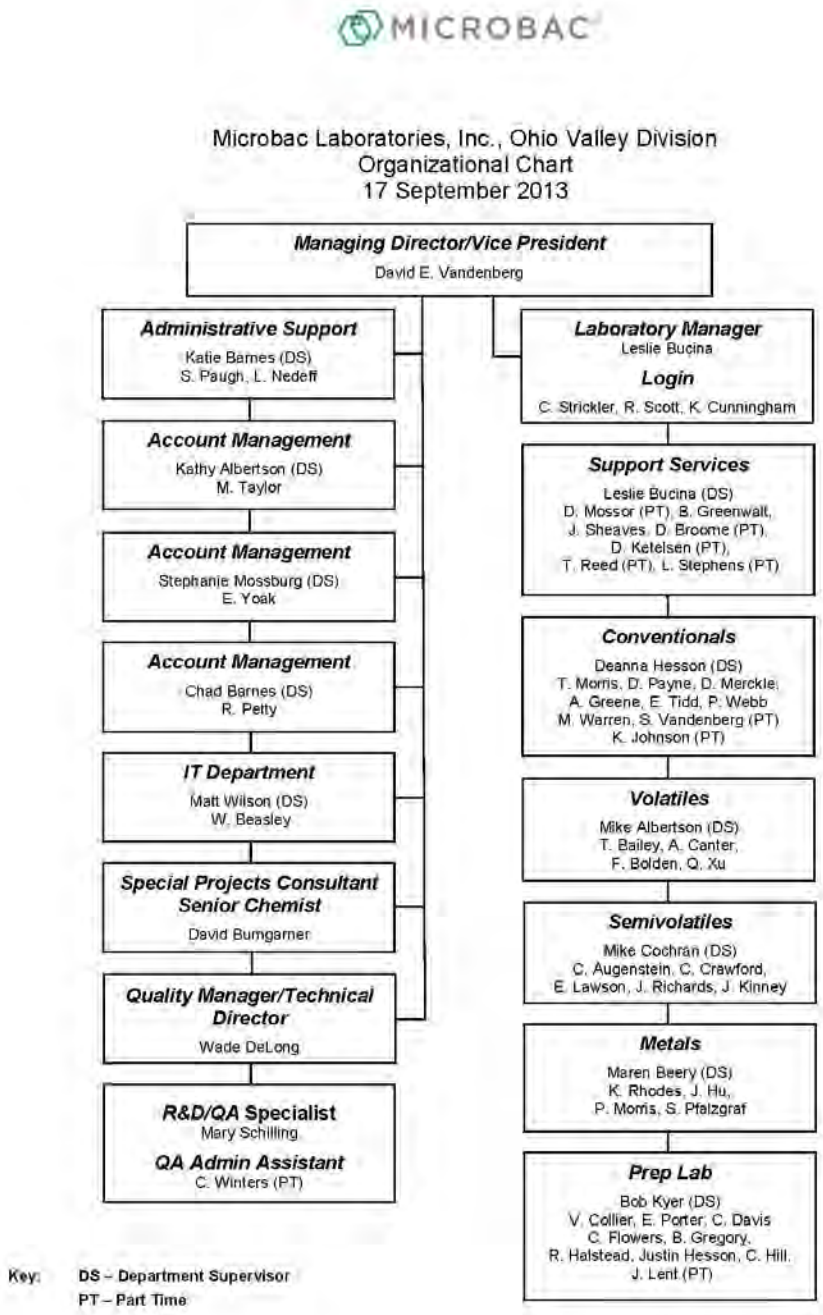
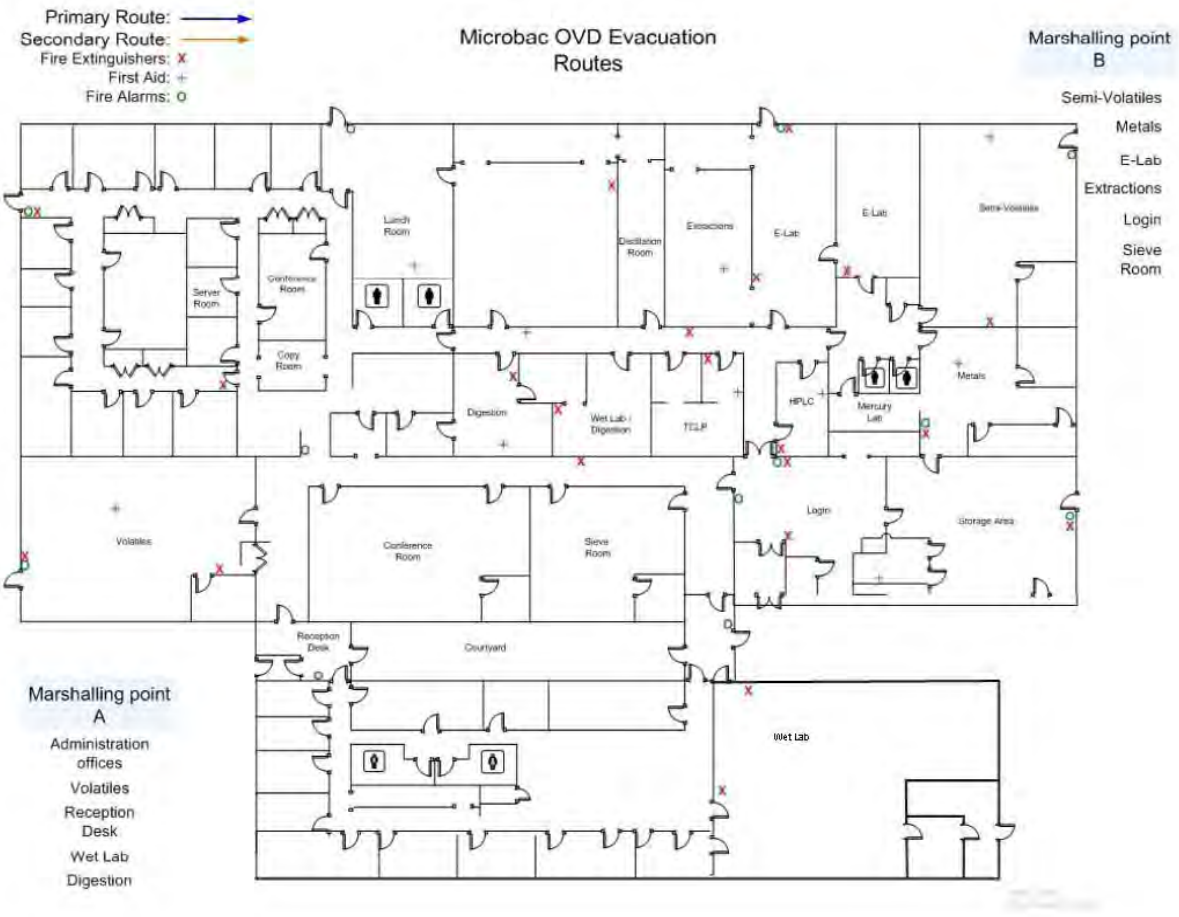

MICROBAC
Microbac Laboratories, Inc., Ohio Valley Division
Organizational Chart
17 September 2013
Managing Director/Vice President
David E. Vandenberg
Administrative Support
Katie Barnes (DS)
S. Paugh, L. Nedeff
Account Management
Kathy Albertson (DS)
M. Taylor
Account Management
Stephanie Mossburg (DS)
E. Yoak
Account Management
Chad Barnes (DS)
R. Petty
IT Department
Matt Wilson (DS)
W. Beasley
Special Projects Consultant
Senior Chemist
David Bumgarner
Quality Manager/Technical Director
Wade DeLong
R&D/QA Specialist
Mary Schilling
QA Admin Assistant
C. Winters (PT)
Laboratory Manager
Leslie Bucina
Login
C. Strickler, R. Scott, K. Cunningham
Support Services
Leslie Bucina (DS)
D. Mossor (PT), B. Greenwalt,
J. Sheaves, D. Broome (PT),
D. Ketelsen (PT),
T. Reed (PT), L. Stephens (PT)
Conventionals
Deanna Hesson (DS)
T. Morris, D. Payne, D. Merckle,
A. Greene, E. Tidd, P. Webb
M. Warren, S. Vandenberg (PT)
K. Johnson (PT)
Volatiles
Mike Albertson (DS)
T. Bailey, A. Canter,
F. Bolden, Q. Xu
Semivolatiles
Mike Cochran (DS)
C. Augenstein, C. Crawford,
E. Lawson, J. Richards, J. Kinney
Metals
Maren Beery (DS)
K. Rhodes, J. Hu,
P. Morris, S. Pfalzgraf
Prep Lab
Bob Kyer (DS)
V. Collier, E. Porter, C. Davis
C. Flowers, B. Gregory,
R. Halstead, Justin Hesson, C. Hill,
J. Lent (PT)
Key: DS – Department Supervisor
PT – Part Time

Figure 4-3
Microbac Laboratories, Inc.
Laboratory Floor Plan



5.0 GENERAL POLICIES AND PROCEDURES

5.1 NELAC Policy Statement

This quality manual, also known as the Laboratory Quality Assurance Plan (LQAP), addresses the elements defined in Section 5.4.2 of the NELAC. A list of National Environmental Laboratory Accreditation Program (NELAP) approved test methods and analytes is presented in Appendix C to this manual. Microbac will comply with all NELAC requirements for all laboratory work covered by our NELAP accreditation.

5.2 Employee Protection from Undue Pressure

It is the policy of this company to protect laboratory employees from undue outside pressures from commercial, financial, or other sources originating from management or clients. This protection is afforded through the implementation of related policies for capacity evaluation, ethics training, and an "open door" policy from upper management. All employees are encouraged to discuss their concerns about such pressures with management.

5.3 Ethics Training

The laboratory requires all new employees to participate in formal training sessions on professional laboratory ethics. Refresher training is also provided to all employees on an annual basis. All employees are educated and trained in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions. All training is documented in accordance with the policy "Ethical Conduct and Data Integrity Agreement". The Ethics Policy is presented in Microbac SOP ETH01. The Microbac Team Manual specifies requirements for ethical behavior and general performance expectations. The Team Manual provides formal policies for disciplinary action and consequences of unethical behavior and other general policy violations.

5.4 Departures from Documented Policies and Procedures

All workload performed under our NELAP certification will be in accordance with this quality manual and all supporting standard operating procedures and policies. In the unlikely event that departures from documented policies and procedures (either technical or non-technical) becomes necessary, only the Managing Director or Technical Director have the authority to approve such deviations.

5.5 Reviewing New Work and Capacity Evaluation

The laboratory adheres to the following mechanism to evaluate new work. The first step is the evaluation of method capability, which includes an initial review of the procedure supplied by the client requesting the work. Management then assesses the laboratory's ability to perform the work with existing apparatus, instrumentation, facilities, and personnel. If management determines that the capability exists, or may be developed, it then evaluates the financial feasibility of performing the new work. The managing director has the sole authority for approval of new work after the above evaluation is completed. All new method development is performed in accordance with Microbac SOP 45 – ***Method Validation Procedures***.

Laboratory capacity is periodically evaluated in conjunction with scheduling and forecasting tools, status updates on outstanding proposals, and the current backlog. The laboratory requires client notification of all sample shipments and clients are advised when large shipments may tax the laboratory ability to meet holding times or delivery dates. It is the policy of laboratory management to evaluate production capacity prior to acceptance of all major delivery orders.

5.6 Complaints

Microbac Laboratories is committed to complete satisfaction of customers and other interested parties. If for any reason a party believes we have not complied with agreed upon specifications or policies, then the following procedures will be implemented:

1. Documentation of Complaint

The appropriate service representative or manager will document in writing the nature of the complaint.

2. Investigation of Complaint

The problem will be investigated to verify the validity of the complaint or concern. If the issue is one of quality control, or breach of policy or procedure, an internal audit will be conducted. If Microbac is found negligent in meeting the written specifications or policies, or if our data is judged to be invalid or unusable for any reason, the problem is referred to the Technical Director/QAO, or other authorized laboratory representative, for a resolution. If the proposed course of action is not acceptable, the problem is referred to the Managing Director. In cases of laboratory negligence the following courses of action are offered:

- Offer to perform services/analyses again at no charge; or
- refund or credit for specific services
- Assist client in dealing with the consequences, i.e. consultation with agency, letter of explanation, re-sampling assistance; and
- seek a resolution that is acceptable to all involved parties.

Written documentation of problem and resolution is maintained in the CAPA Records.

5.7 Document Control and Maintenance

Microbac-OVD has written procedures for controlling both internally generated and external documents that comply with the requirements of ISO 17025:2005, Section 4.3.1. These procedures are presented in detail in Microbac SOP GP-DOC-CONTROL.

5.7.1 Laboratory management shall approve, prior to distribution, all internal and external documents that are part of the management system.

5.7.2 The laboratory shall uniquely identify all quality system documents and include date of issuance, revision number, page numbering, total number of pages, and the issuing authority.

5.7.3 The document control officer, under the oversight of the QAO, shall maintain a master list that uniquely identifies each document, revision status, and record of distribution of all controlled documents. The master list shall be readily available for review.

5.7.4 The document control system and associated master list shall preclude the use of any invalid or obsolete documents.

5.7.5 Authorized versions shall be made available (either electronically or hardcopy) to all laboratory units where documents are essential to effective operations.

5.7.6 All controlled documents are reviewed periodically and revised as necessary to comply with all applicable requirements.

5.7.7 All obsolete or invalid documents are promptly removed to prevent any unintended use.

5.7.8 All obsolete documents are retained in an archive system to meet legal

requirements and to maintain historical continuity.

5.7.9 All changes in documents shall be reviewed by the same function (management personnel) that performed the original review. These personnel shall have access to the pertinent background and information that is needed for review and approval.

5.7.10 Document Integrity

Laboratory management will ensure the integrity of the quality management system as required by ISO 17025:2005, Section 4.2.7. An important element of the annual document reviews will be to assess and eliminate any contradictions within and between internal documents.

5.7.11 Archiving

The QAO is responsible for maintaining an archive copy of each controlled document. Archive copies will be retained for ten years.

5.8 Laboratory Records

The laboratory has written procedures for managing laboratory records and established policies for records retentions that comply with ISO 17025:2005, Section 4.13.1.1. The laboratory has established a policy to archive and retain analytical data, reports, and other records in electronic format, consisting of magnetic tape, CDs, or other equivalent media. Most contracts specify a minimum retention time of five or ten years, however, Microbac will retain all electronically archived data in accordance with contractual and regulatory agency requirements. The disposition or transfer of laboratory records will be negotiated with individual clients upon any change in business status. Additional details are found in Microbac SOP GP-RECORDS.

5.9 Analyst Training and Demonstration of Capability

All analysts must go through a formal training program that includes initial and annual Demonstration(s) of Capability (DOC) for each procedure they perform. These demonstrations are performed in accordance with the requirements of NELAC Section 5.0 Appendix C, and details are presented in Microbac SOP 47 - Employee Training. Alternatively, the laboratory uses the following option to assess Demonstration of Capability when the method is not amenable to Proficiency Testing evaluation, or standard measures of precision and accuracy:

- Four consecutive samples are analyzed with direct observation from another certified analyst, or;

- Results of four analyses are compared to those performed by another certified analyst.

5.10 Protecting Confidentiality and Proprietary Rights

Two factors must be observed regardless of the method chosen to convey information and data to a client. These are client confidentiality and accurate record keeping. The need to preserve the clients' confidentiality must never prevent recording of the date, time, person contacted and subject of the communication. Data capture software allows entire case files/final reports to be captured and put into a file format. This then can be put on disk, or sent by email to a client. This has great value for providing preliminary data to clients on quick turn projects.

5.10.1 Confidentiality Agreements

Protecting the confidentiality of client information and data is of the utmost importance to Microbac. All Microbac employees, as a condition of employment, must sign the Employee Confidential Information and Non-compete Agreement, which was developed to protect the proprietary records and data of Microbac and all of our clients. Upon request from Ohio Environmental Protection Agency (OEPA), Microbac will provide access to OEPA data and documents or information related to Ohio Voluntary Action Program (OVAP).

5.10.2 Client Data

The following Sections 5.10.2.1 – 5.10.2.7 provide instructions for ensuring that, where clients require transmission of test results by telephone, facsimile or other electronic means, confidentiality is preserved.

5.10.2.1 Telephone Communications

In cases where analytical results must be communicated by telephone, a Client Services Specialist (CSS) calls the contact person/client whose name has been provided by the client at telephone number also supplied by the client. Verbal results are not released to any other person unless explicit instructions are received from the relevant client. The date and time of the client contact is recorded in the CSS telephone log.

5.10.2.2 Electronic (Modem) Deliverables

All modem transmissions are done on direct lines to the client and are set up through our modem system using our dedicated lines. The client, where applicable, supplies telephone numbers and passwords.

5.10.2.3 CD ROM Deliverables

CD ROM deliverables are shipped to the client or designated contact person accompanying the hard copy report to which they apply. The client supplies the address and attention name.

5.10.2.4 Email

Where possible, email will be used to transmit information and data to a client. Care must be taken to insure the client receives the email and that the sender at Microbac maintains a record of the transmission. This can be done with our current email system by maintaining a sent file directory.

5.10.2.5 Portal

As data is completed it is uploaded automatically to the Microbac Portal called "MicroView" for clients to view online.

5.10.2.6 SOP's

Care must also be taken to protect client confidentiality in Standard Operating Procedures because they are often read by clients that may be considered a competitor of the client mentioned in the SOP. The utmost care must be taken to not identify specific or specialized procedures as belonging to any one client if possible. If it is necessary to name a client in an SOP then the information naming the client will have to be sanitized or removed before the SOP is given to any other potential or current client for review.

5.11 Identification of Approved Signatories

The Managing Director has the authority to sign contracts, approve new company policies and procedures (SOP's) and certify (sign) laboratory reports. The Technical Director / QAO has the authority to approve laboratory policies and procedures (SOP's) and to certify laboratory reports. The Managing Director may grant authority to other qualified personnel to certify laboratory reports or other official documents for special projects, or in case of emergency.

5.12 Monitoring and Controlling System Time

The system date and time reported by our KOBRA LIMS (Laboratory Information Management System) (and all networked data systems) is synchronized to the data/time of the mail server, which is further synchronized with the internet using network time protocol. Hourly adjustments in time are made automatically, and laboratory staff are not authorized to make manual changes. Unauthorized changes in data system date or times may be considered a violation of the laboratory ethics policy (see also Section 5.3).

5.13 Changes of Ownership/Key Staff

In the event of change in ownership, the laboratory must notify all customers, certifying bodies, and relevant agencies within 30 days of any changes in ownership. This letter will clearly identify the extent of these changes, and discuss policies for ownership, storage, and access to historical data and records. The laboratory must also notify certifying bodies within 30 days or any changes in any key management staff, including the Managing Director or Technical Director(s).

5.14 Method References

The laboratory will include the official method references on laboratory documents including benchsheets, laboratory SOP cover pages, and laboratory analysis reports. These references shall correspond to those listed in the NELAP Scope of Accreditation.

5.15 A2LA Advertising Policy

The laboratory will adhere to the most recent directives established by The American Association for Laboratory Accreditation (A2LA) in regard to display and use of symbols and documents reflecting its accreditation by A2LA. The laboratory will ethically and accurately represent areas of products and services for which this accreditation is applicable, and where used, the laboratory will assure clear exception of products and services outside the scope of that accreditation. Where used, the following restrictions are to be observed:

- The “A2LA” symbol cannot be used by the laboratory for any purpose. Only the “A2LA Accredited” symbol can be used by the laboratory to indicate its accreditation by A2LA. The A2LA Accredited Symbol may be modified for size and/or color, but the integrity of the symbol must be maintained in all respects.

- The laboratory can use the A2LA accredited symbol on business cards, but the location of the symbol is important. The symbol should not be placed near the name of the individual, to in any way suggest accreditation of that individual.

- Only the individual laboratory accredited by A2LA can use the A2LA Accredited symbol; therefore, corporate stationary, websites and marketing materials, which encompass all laboratories under the corporate structure, cannot carry the A2LA Accredited symbol.

As acknowledged by the A2LA directive, not every possible inappropriate use of the A2LA Accredited symbol is addressed in P101-Reference to A2LA Accredited Status-A2LA Advertising Policy; however, all laboratory personnel involved in the promotion of the laboratory's capabilities and credentials are required to review the specific examples, including graphics presented in the Appendix, for unacceptable usage of the A2LA Accredited Symbol. Review and training of staff to insure clear and unambiguous understanding of the proper use of the A2LA Accredited Symbol and ramifications of non-compliance will be conducted annually.

5.16 Purchasing Supplies and Services

The laboratory has developed written policies and procedures in Microbac SOP GP-PURCHASING for the selection and purchasing services and supplies that comply with ISO 17025:2005, Section 4.6.1.

5.17 Uncertainty - Estimating Uncertainty of Measurement

Microbac states on the cover page all laboratory reports that uncertainty data is available when requested by the client. Microbac will produce a separate report in comma separated value (CSV) format that presents the data as a concentration interval about the reported value for each analyte. The lower and upper limits of the interval express the uncertainty estimate for each result, and where applicable are based on the analyte's quality control limits for laboratory control samples (LCS). The expressed uncertainly makes no attempt to address sampling error. Detailed procedures for estimating measurement uncertainty in accordance with ISO 17025:2005, Section 5.4.6.2, are found in SOP GP-UNCERTAINTY.

5.18 Review of Requests, Proposals, and Contracts

Microbac-OVD has established policies, procedures, and a system of record for the review of requests (RFP), tenders (proposals), and contracts. The review

includes any work that is subcontracted, and all contract amendments. The customer is informed of any deviations to contracts before the work begins. Details of the procedures are found in Microbac SOP GP-CONTRACTS.

5.19 Measurement Traceability

The laboratory will adhere to the most recent directives established by A2LA in regard to policy related to measurement traceability, as defined in A2LA document P113-A2LA Policy on Measurement Traceability for Life Sciences Testing Laboratories. Section 8.4 details laboratory policy related to the traceability of calibration standards. The proper procedure and frequency for the calibration of balances, weights, thermometers, pipets, and the spectrophotometers, that are used in the laboratory is detailed in lab SOP K0002: Calibration Techniques.

5.20 Trip Blank hold times

Microbac OVD – Trip Blank Collection Date Policy

The collection date and time of trip blank(s) should be the same as the last sample collected on the chain of custody with which the trip blank(s) is associated. Microbac records the date and time the trip blank(s) are prepared at the laboratory, this date and time should not be used as the collection date and time on the chain of custody. If the prepared date and time are mistakenly entered on the chain of custody for the trip blank, we will edit the date and time of collection of the trip blank to coincide with the last sample listed on the chain of custody. If the collection date and time listed on the chain of custody for the trip blank is within the collection date and time of the first and last sample on the chain of custody we will use the collection date and time as presented for hold time evaluation.

This policy has been established at the recommendation from the MICE interpretation regarding hold times for trip blanks and is consistent with EPA guidance "… a trip blank has the same "life" as a sample with which they are sent. Trip blanks are the same age as the sample set and are used to determine if the sample MAY have been contaminated in transit."

This policy may be superseded by project specific or state specific requirements.

Compliance with this policy will contribute to the production of valid data.

6.0 SAMPLING PROCEDURES

Microbac functions as an environmental analytical facility and as such is not directly responsible for the sampling events. However, Microbac does utilize independent field personnel. Below are sampling guidelines that were developed to outline the primary objective of any external sampling event. The objective is the collection of samples which, when analyzed, will consistently generate accurate, precise and representative data. By developing comprehensive standard sampling procedures and training personnel in the procedures, this objective can be met. These sampling procedures are offered as guidance to field project managers to assure sample integrity. Following the sampling procedures promotes sample integrity and the highest degree of quality control throughout the sampling event.

6.1 General Sampling Guidelines

SOPs should be utilized by all field project managers for potential sample sources. The SOPs should be based on State and USEPA guidance documents. SOPs should be available to and reviewed by all field personnel. Following are general guidelines to be used in the collection of environmental samples:

6.1.1 Sample containers which are prepared in the laboratory may contain measured volumes of preservative. Such containers must not be rinsed prior to filling with sample.

6.1.2 Sample containers for VOA, pH, and TOX must be completely filled with no headspace. All other sample analyte containers must be filled to approximately 95% capacity.

6.1.3 VOA samples must be collected in a manner which minimizes disturbance of the sample and potential for volatilization. Fill vials until a convex meniscus forms then carefully place the septum cap on the vial. Invert the vial and check for the presence of air bubbles.

6.1.4 Field sampling equipment must always be appropriately decontaminated before and after use.

6.1.5 During the collection of all environmental samples, appropriate personal protective gear must be worn. Gloves and safety glasses are the minimum acceptable level of personal protective equipment (PPE) to be worn when samples are collected.

6.1.6 Use pre-cleaned laboratory prepared glassware for the collection of samples whenever possible.

6.2 Microbac Sampling Containers

The measurement of trace constituents in environmental samples demands methods capable of maximum precision and sensitivity. The selection and proper care of laboratory glassware and sample containers is an important part of the quality control program to eliminate errors due to contamination from improper cleaning procedures.

Laboratory supplied containers constructed of materials which are both compatible and non-reactive with the material to be sampled will be used. Microbac uses commercial sample containers which are certified pre-cleaned to EPA standards. These containers are shipped in sealed boxes with custody seals. Glassware is certified cleaned according to Protocol A, B, or C described below. Sample containers used by Microbac follow washing procedures equivalent to these protocols.

Table 6-1
Glassware Cleaning Protocol

Protocol A	Protocol B	Protocol C
Laboratory-grade detergent wash and rinse	Laboratory-grade detergent wash and rinse	Laboratory-grade detergent wash and rinse
Acid, deionized water, and solvent rinse	Multiple deionized water rinses	Acid rinse Multiple deionized water rinses
Oven drying, capping, and packing under quality control conditions	Oven drying, capping, and packing under quality control conditions	Oven drying, capping, and packing under quality control conditions

Table 6-2 lists the container type and volume required for each sample type.

6.3 Sample Preservation

Use of cooling, pH control, and chemicals to retard biological activity or to stabilize the chemical species of a sample is known as preservation. Sample kits prepared by the laboratory include sample containers prepared with the appropriate type and volume of preservative for the analyte of interest. Addition of preservative to samples in the field is not normally required when using the prepared kits. This procedure minimizes the potential for incorrect or inadequate sample preservation. The most common form of preservation is cooling the sample to ≤ 6° C using ice or refrigeration. Other common preservatives used include:

- Hydrochloric acid - dilute 1:1
- Nitric acid - dilute 1:1
- Sulfuric acid - dilute 1:1
- Sodium hydroxide - 50% solution
- Sodium thiosulfate - for dechlorination

Table 6-2 lists the preservation techniques and holding times employed by Microbac for each sample type. Holding time is defined as time elapsed from sample collection date and time to the analysis date and time with the exception of semivolatile extractables. Semivolatile holding times in the table denote designated timing allowed from collection to sample extraction. Hold time for semivolatile analysis from date of extraction is 40 days.

6.4 Sample Documentation

6.4.1 Sample labels

When using laboratory prepared sample kits, pre-labeled and preserved sample containers are provided. Each sample collected by Microbac is clearly labeled using waterproof ink with the following information:

- Client name
- Date and time of collection
- Sample source
- Preservative required
- Name(s) of sampler
- Analyses requested
- Sample identification

6.4.2 Field records

Sampling personnel should maintain complete and accurate records of all field activities. Bound field notebooks and/or field logs specific to the media sampled should be completed during each sampling project. All pertinent information on the sampling event is included in the field record.

6.4.3 Chain-of-Custody

All samples should be accompanied by a completed chain-of-custody form when shipped or hand delivered to the laboratory. Information required on the chain-of-custody includes:

- Sample date and time

- Name(s) of sampler(s)
- Sample ID
- Project name and number
- Analyses requested
- Number of sample containers
- Signature and date of all individuals who have custody of the samples

6.5 Sampling Equipment Decontamination

All field sampling equipment must be appropriately pre-cleaned prior to leaving the base of operations. Use phosphate-free detergent, hot tap water, and analyte free rinse water for this cleaning following the procedure specified for analytes of interest. Cleaned equipment must be wrapped or enclosed to maintain cleanliness during transport to the field for use. Adequate quantities of sampling equipment must be provided for each event to minimize the need for field decontamination.

Decontamination procedures for sampling equipment may include:

- Phosphate-free detergent and tap water wash
- Rinse with tap water

For equipment to be used for trace metals sampling, rinse with 1:1 reagent grade nitric acid solution. **Do not rinse stainless steel sampling equipment with nitric acid**.

- Rinse thoroughly with deionized water
- Rinse with isopropanol or methanol
- Thoroughly rinse with analyte-free water (if available)
- Air dry
- Wrap securely to prevent contamination if equipment is to be stored

Information on specific decontamination procedures are provided in each sampling SOP. Special decontamination procedures will be implemented as necessary, based upon contaminant encountered.

Equipment which is heavily soiled may require steam cleaning and/or high pressure washing. Drilling equipment and other heavy equipment used in field sampling activities will likely require this type of cleaning. If equipment cannot be adequately decontaminated, it will be discarded.

6.6 Field Waste Disposal Practices

Field generated waste must be disposed as required by project specifications and in accordance with applicable local, state, and federal requirements. Wastes commonly generated include drill cuttings, drilling fluids, well development water, well purge water, decontamination fluids, and contaminated personal protective equipment.
Based upon site and project specific requirements, liquid wastes should be containerized for characterization and disposal or discharged directly to an appropriate discharge location. Solid wastes should be containerized and left on-site for disposal or, if appropriate, disposed as general refuse.

Prior to initiating any sampling activity the waste handling requirements should be determined to ensure timely disposal in compliance with regulatory requirements.

6.7 Laboratory Sub-Sampling Procedures

6.7.1 Soil

Microbac employs the following procedures for taking soil subsamples in all methods and departments except for percent moisture determinations and volatile organic analyses:

a) Remove sample bottle contents and place in tray lined with wax paper (metals lab) or aluminum foil (organics extraction lab). Alternatively, remove vertical core section(s) (top to bottom) that is representative of the bottle contents and transfer to the tray.
b) Mix sample with an inert rod or scoop and break up lump. Remove all large stones, sticks, leaves, etc. Do not over mix the sample.
c) Obtain representative sample either by random removal of 3-10 portions of the sample from the pan or by using a “standard” scoop designed to retrieve a linear cross-section of the pan contents.
d) The analyst will not attempt to target an exact weight once a method specified minimum amount is weighed.
e) The remaining sample will be returned to the sample container.

NOTE: These procedures do not apply to samples for volatile organics analysis, except the requirement not to target a specific mass of sample.

6.7.2 Water and Liquid Wastes

Unless otherwise stated in the method SOPs, the analyst will homogenize water and low viscosity liquid wastes by shaking each sample vigorously immediately prior to taking the sample aliquot. Viscous liquids such as oils shall be stirred with a glass rod prior to aliquot removal.

6.7.3 Multiphasic Samples

The laboratory will attempt to identify multiphasic samples prior to login. Our policy is to contact the client to determine whether to attempt homogenization, or to do a phase separation, and log as separate samples. Homogenization may require special procedures such as the use of a blender, however, our recommended approach is to separate the phases. The analyst should notify the project chemist if a multiphasic sample is discovered after receipt.

6.8 Sample Dilutions

The laboratory may dilute samples prior to analysis for the following reasons:

- A previous analysis yielded a result above the upper calibration range for the method.
- The sample contains a target, or non-target analyte, at sufficient concentration to damage sensitive instrumentation.
- Sample matrix interference, confirmed by poor surrogate or spike recoveries, prevents accurate analysis without dilution.

In each case the laboratory will document the sample dilution by reporting the dilution factor, adjusting the reporting and detection limits by that factor. The report narrative will provide additional detail as needed. If dilution analysis is not performed, the laboratory will assign appropriate qualifiers to any results reported above the upper calibration range.

Table 6-2
Sample Containers, Preservation and Hold Times

CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Acidity	100	P, G	Cool, ≤ 6° C	14 Days
Alkalinity	100	P, G	Cool, ≤ 6° C	14 Days
Total Solids	50	P, G	Cool, ≤ 6° C	7 Days
Ash Content @ 750° C	25	P, G	Cool, ≤ 6° C	---
Biochemical Oxygen Demand	500	P, G	Cool, ≤ 6° C	48 Hours
Boron	20	P	Cool, ≤ 6° C	6 Months
Bromide	1000	P, G	Cool, ≤ 6° C	28 Days
BTU	10	P, G	Cool, ≤ 6° C	---
Formaldehyde	20	G	Cool, ≤ 6° C	7 Days
Chloride	25	P, G	Cool, ≤ 6° C	28 Days
Chloride, Total Residual	100	P, G	Cool, ≤ 6° C	6 Hours
Cyanide (midi)	50	P, G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Cyanide, Amenable to Chlorination (midi)	100	P,G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Chemical Oxygen Demand	25	G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Color, Platinum-Cobalt	50	P, G	Cool, ≤ 6° C	48 Hours
Coliform, Fecal	120	P, G	Cool, ≤ 6° C, $Na_2S_2O_3$	8 Hours
Fecal Streptococcus	100	P, G	Cool, ≤ 6° C, $Na_2S_2O_3$	8 Hours
Coliform, Total	100	P, G	Cool, ≤ 6° C, $Na_2S_2O_3$	8 Hours
Specific Conductance	100	P, G	Cool, ≤ 6° C	28 Days
Corrosivity	500	P, G	Cool, ≤ 6° C	---
Corrosivity (pH)	40	P, G	Cool, ≤ 6° C	---
Chromium, Trivalent	200	P, G	Cool, ≤ 6° C	---
Chromium, Hexavalent	150	P, G	Cool, ≤ 6° C	24 Hours
Dissolved Oxygen	300	G	Cool, ≤ 6° C	6 Hours
Fluoride	25	P, G	Cool, ≤ 6° C	28 Days
Ignitability	75	P, G	Cool, ≤ 6° C	---
Fluoride, Total (Distilled/Non-Distilled)	200	P, G	Cool, ≤ 6° C	28 Days
Hardness	100	P, G	Cool, ≤ 6° C, HNO3, pH<2	6 Months
Iodide	400	P, G	Cool, ≤ 6° C	24 Hours
Surfactants (MBAS)	100	P, G	Cool, ≤ 6° C	48 Hours
Coliform Fecal (MPN)	100	P, G	Cool, ≤ 6° C,$Na_2S_2O_3$	8 Hours
Nitrogen, Ammonia (Distilled/Non-Distilled)	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Nitrite	50	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate	75	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate-Nitrite	25	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Organic	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Threshold Odor	1000	G	Cool, ≤ 6° C	48 Hours
Oil and Grease	1000	G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap

Uncontrolled Copy

Table 6-2 (continued)
CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Phenolics, Total	100	Amber, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
Phosphorus, Total	50	P, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
pH Lab	40	P , G	Cool, ≤ 6° C	6 Hours
Orthophosphate	50	P, G	Cool, ≤ 6° C	48 Hours
Reactivity, Cyanide	10	P, G	Cool, ≤ 6° C	---
Reactivity, Sulfide	10	P, G	Cool, ≤ 6° C	---
Sulfite	50	P, G	Cool, ≤ 6° C	24 Hours
Settleable Solids	1000	P, G	Cool, ≤ 6° C	48 Hours
Silica, Dissolved	100	P	Cool, ≤ 6° C	28 Days
Sulfate	100	P, G	Cool, ≤ 6° C	28 Days
Specific Gravity	50	P, G	Cool, ≤ 6° C	---
Total (Organic) Sulfur	10	P, G	Cool, ≤ 6° C	---
Sulfide	200	P, G	Cool, ≤ 6° C, Zinc Acetate, NaOH, pH> 9	7 Days
Total Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days
Turbidity	50	P, G	Cool, ≤ 6° C	48 Hours
Volatile Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Volatile Solids	50	P, G	Cool, ≤ 6° C	7 Days
Volatile Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days

VOLATILE ORGANICS (VOA) - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Gasoline Range Organics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Aromatics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Organics (VOA)	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
VOA – Method 624, Purgeable Halocarbons	40 mL	G, Septa Caps	Cool, ≤ 6° C, $Na_2S_2O_3$	14 Days
VOA – Method 624, Purgeable Aromatic Hydrocarbons	40 mL	G, Septa Caps	Cool, ≤ 6° C, $Na_2S_2O_3$, HCL, pH < 2	14 Days
VOA – Method 624, Acrolein and Acrylonitrile	40 mL	G, Septa Caps	Cool, ≤ 6° C, $Na_2S_2O_3$, HCL, pH 4-5	14 Days

Table 6-2 (continued)
SEMIVOLATILE ORGANICS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Diesel Range Organics	1000 mL	G	Cool, ≤ 6° C	7 Days
Pesticides/PCBs	1000 mL	G	Cool, ≤ 6° C	7 Days
Polyaromatic Hydrocarbons	1000 mL	G	Cool, ≤ 6° C	7 Days
Herbicides	1000 mL	G	Cool, ≤ 6° C	7 Days
Semivolatile Organics	1000 mL	G	Cool, ≤ 6° C	7 Days

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap

METALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
All Metals (26)	500 mL	P, G	HNO_3, pH< 2	6 Months*
Mercury	50 mL	P, G	HNO_3, pH< 2, ≤ 6° C	28 Days

TCLP - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
TCLP Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Semi-Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Pesticides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Herbicides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Metals	100 mL	P, G	Cool, ≤ 6° C	6 Months*

* For (1) TCLP parameter 100 mL required; for full TCLP (2) 1000g
* Mercury is 28 days

Notes:

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. Triple the volumes above for MS/MSD samples
4. Sample hold time is defined as time elapsed from sample collection date and time to sample analysis date and time.
5. TCLP and Semivolatile Organics hold time is defined as time elapsed from sample collection date and time to sample preparation date and time.
6. Refer to the Method specific SOP for further information regarding holding time.

Table 6-2 (continued)

Uncontrolled Copy

SOIL HOLD TIME

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Coliform Fecal	1g	G	Cool, ≤ 6° C	8 Hours
Chromium, Hexavalent	50g	G	Cool, ≤ 6° C	24 Hours
5035	5 g	P, G	Cool, ≤ 6° C	48 Hours
TCLP-VOA	105g	G	Cool, ≤ 6° C	14 Days
TCLP-SV	105g	G	Cool, ≤ 6° C	14 Days
TCLP-Pest/Herb	105g	G	Cool, ≤ 6° C	14 Days
TCLP-Metals	105g	G	Cool, ≤ 6° C	6 Months*
Total Metals (except Hg)	3g	G	Cool, ≤ 6° C	6 Months
Hg	2g	G	Cool, ≤ 6° C	28 Days
TPH	30g	G	Cool, ≤ 6° C	28 Days
Semi-Volatiles	30g	G	Cool, ≤ 6° C	14 Days
Herbicides	50g	G	Cool, ≤ 6° C	14 Days
Volatiles	1g	G	Cool, ≤ 6° C	14 Days
Conventionals (where applicable)	1g – 100g	G	Cool, ≤ 6° C	14 Days
Petroleum Hydrocarbons	30g	G	Cool, ≤ 6° C	14 Days
Percent Moisture	25 g	P, G	Cool, ≤ 6° C	---
Percent Solids	25 g	P, G	Cool, ≤ 6° C	---
Paint Filter Liquids Test	100 g	P, G	Cool, ≤ 6° C	---

* Mercury is 28 days
** All soils stored at 4° C

7.0 SAMPLE CUSTODY

In recognition of the critical nature of sample custody protocols, Microbac Laboratories has implemented stringent standard operating procedures, designed to ensure sample integrity and thorough documentation. This section provides a clear description of sample traceability from sampling kit to final sample disposition.

7.1 Sampling Kits

Microbac Laboratories, at the client's request, will provide sampling kits to the client. Additional information on sampling kits can be found in Microbac SOP LOGIN01.

7.2 Field Custody (Also see Section 6.0)

A vital component of Microbac's quality assurance program is to ensure that a clear and detailed record is kept of all samples and sampling activity. Chain-of-Custody (COC) forms (Figure 7-1) are submitted with all samples. These are kept in the appropriate project files. Water-proof ink is used to label samples as protection against loss of information due to accidental erasure. Microbac's sample custody protocol requires that the following information be recorded.

7.2.1 Date and Time of Sample Collection

7.2.2 Specific description of sample location. This may include a monitoring well number in the case of groundwater sampling. For soil sampling, sample points may be sketched on a site map and confirmed via surveying. For surface water and sediment sampling, the drum number, (if labeled), drum location, suspected contents and phase of drum materials (liquid, solid, or sludge) may be noted, as well as, which layer or layers within the drum were sampled.

7.2.3 Name(s) of sampler(s) will be identified.

7.2.4 A description of weather conditions and general site conditions (disturbed soils, standing water, ongoing activities, etc.) may be provided.

7.2.5 A description of the sampling equipment used, including method for purging monitoring wells may be indicated.

7.2.6 The specific field ID number for a sample may be recorded. The sample sequence number is the order in which a particular sample was taken with respect to all other samples retrieved at the site. The sample sequence number may also be recorded.

7.2.7 Components or constituents of sample to be analyzed are to be noted.

7.2.8 Signature(s) of sampler(s) will be provided.

7.2.9 Types of preservatives and, if necessary, the results of field check (pH, etc.) may be recorded.

7.2.10 Field measurement data (such as VOA reading, pH, specific conductance) may be recorded.

7.3 Sample Transport

Samples are transported to Microbac's laboratory by one of three modes:

7.3.1 Microbac Courier - on many projects, Microbac will have responsibility for preparation of sample bottles, sample kit assembly and delivery of sample kits to the project site. Microbac will also pick up the samples at the project site and transport them to the laboratory. Microbac drivers will follow these established protocols:

- They will carry proper Microbac identification, which they will be prepared to display before entering project sites;
- They will sign the Chain-of-Custody forms when picking up the samples;
- They are responsible for the integrity and security of samples while in their custody;
- They must secure the vehicle at all times when it is necessary to stop and be away from the vehicle for any reason during the transportation of samples to the laboratory.

All samples returned by Microbac personnel are delivered immediately to the Sample custodian in the sample logging station. The Shipping and Receiving entrance is to be used for such deliveries. If delivery is made after hours, the driver will take the cooler temperature, fill out the appropriate form for the sample custodian and store the cooler in a locked sample storage unit.

7.3.2 UPS, FEDEX, or Other Common Carrier - Transporters are directed to the Shipping and Receiving entrance and the samples are delivered to the Sample custodian.

7.3.3 Client Deliveries - Clients may deliver samples in person via the designated Receiving entrance but must be escorted while within the facility.

7.4 Laboratory Custody Procedures

The National Enforcement Investigations Center (NEIC) of EPA defines custody of evidence in the following manner:

- It is in your possession, or;
- It is in your view after being in your possession, or;
- It was in your possession and then you locked or sealed it to prevent tampering, or;
- It is in a secure area.

7.4.1 Normal Custody Procedures

For the purpose of sample custody, Microbac Laboratories maintains that the laboratory in its entirety is a secure area and all samples received and logged into the laboratory remain in the custody of the Sample custodian, Supervisor or Analyst, until time of disposal. Refrigerators, freezers and other designated sample storage areas will be securely maintained or locked. Only the designated Sample custodian or supervisory personnel will have keys to locked sample storage units until removed for sample preparation or analysis. The following minimum custody procedures are followed for all samples:

- All samples are received and inspected in accordance with Section 7.5.
- The sample custodian signs and dates the chain-of-custody form provided by the client.
- The samples are stored in the appropriate storage unit. (The storage unit is also designated in the KOBRA LIMS when the samples are logged in.)
- Several laboratory documents are used to document which laboratory personnel handled the samples, including bench sheets, sample preparation logbooks, and instrument run logbooks.
- The original copy of the chain-of-custody form is included in the laboratory report to the client and a copy is maintained in the laboratory files.

7.4.2 Internal Laboratory Custody (Internal Chain of Custody (ICOC))

- In order to satisfy more stringent chain-of-custody requirements, the following standard operating procedures can be implemented by Microbac Laboratories, Inc. upon the request of the client:

- The work request prepared by the CSS must specify which projects require extended internal laboratory custody. All sample bottles designated for ICOC are identified on the label for easy identification.

- After the samples are logged into the KOBRA LIMS, a unique container number and barcode is generated and printed on the bottle label (Figure 7-4). Each laboratory staff member is also assigned a badge and barcode in order to quickly record all exchanges.

- Samples must remain in secure sample storage units until removed for sample preparation or analysis. All transfers of samples into and out of storage will be posted to the KOBRA LIMS using a standard barcode reader.

- Removal of samples from storage requires a barcode scanning procedure that produces an electronic record of the transaction in the KOBRA LIMS.

- After a sample container has been removed from storage, the analyst is responsible for the custody and integrity of the sample. While the samples are in the custody of the laboratory, they must be locked in the storage units if not in immediate custody of the analyst.

- Samples returned to the storage units must be scanned to document the transfer from analyst back to the custodian.

- The custodian will scan each container at the time of disposal of the sample container. (Transfer of sample extracts and digests are documented on the laboratory preparation benchsheets.)

- A special report from the KOBRA LIMS is used to produce an Internal Sample Custody Form (See Figure 7-3). These records are included in the final report to the client as well as the signed original copy of the field COC form.

7.5 Sample Receipt and Inspection

The sample custodian or designated assistant will receive all incoming samples. The following procedures are outlined in Microbac SOP LOGIN01. The following information is documented on the Sample Receipt Form (Figure 7-2). The custodian will frisk the cooler for radioactivity if required by the project Quality Assurance Project Plans (QAPP). The custodian will open the shipping containers and note the presence/absence of Chain-of-Custody forms and seals, airbills, or bills-of-lading. The sample temperature is then read. The custodian will examine the shipping container to verify the integrity of the

sample(s) and examine the sample documentation and identification to assure it is correct and the proper preservative has been used. The preservatives are checked by the sample custodian. If inspection indicates samples were damaged in transit, the shipping container will be moved to the hood in Log-in, assessments of the damage will be made and the appropriate services representative will be notified. The client will be immediately contacted and determination of the degree of hazard will be made. If damage is minimal and the client requests it, an attempt to salvage the sample(s) will be made if it can be done safely. In the event of damaged hazardous samples, Microbac's Spill Response Team will be notified per the Chemical Hygiene Plan.

Samples received after hours, when the sample custodian is absent, will be placed in the walk-in refrigerator. The person receiving the shipping container will sign for the container, take the cooler temperature if appropriate and place the dated forms on the Custodian's desk. The sample custodian will log in the samples on the next business day. The KOBRA LIMS system will indicate the actual date and time received and the original receipt documentation will be included with the COC form.

The sample custodian will compare the COC forms and labels to verify agreement of information contained therein. If discrepancies are found, they will be documented on the Sample Receipt Form and the services representative will be immediately notified. Written documentation of all problem resolutions will be placed in the project/case file. If there are no problems with the samples received, the sample custodian files the signed COC form and Sample Discrepancy Form in the project/case file. After the sample is logged in, it will be immediately stored under the proper conditions.

Sample labels or other sample documents that appear to be contaminated due to sample breakage or other problems will be dried under a fume hood and be separately sealed in plastic bags, if necessary, prior to being placed in case files. The services representative must also be notified.

7.6 Sample Logging Procedures

The following procedures are outlined in Microbac SOP LOGIN01. The sample custodian is responsible for logging all samples into the Laboratory Information Management System (KOBRA LIMS), signing the COC form, completing the Sample Receipt Form, reporting all problems, inconsistencies, or anything questionable to the services representative, placing all samples in storage, monitoring conditions in sample storage areas, and maintaining records for laboratory COC.

As a new login number is initiated in the KOBRA LIMS, the sample custodian generates and/or enters the following information about the project shipment:

- Microbac Login Number
- Client Project Identification
- Mode of Transportation
- Date and Time Received/Date Due
- Matrix/Sample ID
- Date and Time of Sample Collection
- Storage Location/Container Size/Type/Preservative
- List of Analyses
- Notation of Problems/Special Instructions

In order to maintain sample identity, each sample received will be assigned a unique sample I.D. number. The KOBRA LIMS will assign the unique Microbac Sample Number at the time of log-in, as shown in the following example:

EXAMPLE: L08010001-01

The first nine digits identify a group of related samples and becomes the login number and report number. The two-digit number after the dash identifies a specific sample in the order. These numbers are used by Microbac for continuous identification of the sample from receipt to completion of analysis. In the event of multiple samples for a single analysis, such as 2 vials for volatile organic compounds, letter designations will be added to the end of the identification number. Sample containers will be clearly identified with the appropriate sample number. The sample labeling process is accomplished by the KOBRA LIMS system which prints the required number of computer labels (Figure 7-4). Extract vial numbers and metal digests will correspond to the Microbac sample number from which they originated. These numbers will also be recorded on sample tracking documentation.

7.7 Sample Storage

Samples and extracts will be stored in uniquely identified refrigerators which are in secure areas of the laboratory. The sample custodian or designated assistant will check the temperature of each refrigerator in the log-in area, twice daily (once on weekends and holidays) and maintain a record book. This record book will be reviewed on a monthly basis by the Support Services Supervisor to note any trends or inconsistencies. The acceptable range for sample storage is ≤ 6° C. The sample custodian will notify the Support Services Supervisor of any refrigerator temperature problem which cannot be

corrected by simple thermostat adjustment. A list of emergency repair numbers for the refrigeration units is attached to the walk-in refrigerator's exterior.

7.8 Sample Distribution and Tracking

Both the preparation and the analysis of samples will be documented using special forms (logbooks). Once analysis is complete, the analyst will return the unused sample to the sample custodian area for return to the main cooler or to the Sample Archive Room, whichever is appropriate. Figure 7-3 provides an Microbac Internal Sample Custody form. Samples will be returned to their original storage units after completion of analyses. Samples which have exceeded their regulated holding period will be placed in the Sample Archive Room. They are routinely stored in this area for a minimum of 14 days after the due date for the analytical report. They are then disposed per protocol listed in Microbac's "Waste Management SOP". Extended archive beyond 14 days with refrigeration is available for specific projects or as required by contract.

It is important to note that samples received for analysis of VOA are segregated from other samples. Standards are also segregated from all samples in designated storage units.

7.9 Sample Security

All sample storage refrigerators are equipped with a lock. The units are monitored by the sample custodian during business hours. After business hours, the unit is kept locked and only selected personnel have access (by key) to the sample storage unit. All samples, extracts and digests will be stored in segregated areas.

7.10 Laboratory Building Security

All access doors to the building, with the exception of the main entrance to the reception area and the employee entrance, remain locked. Only select Microbac employees have keys to the access doors to the building. The main entrance and the employee entrance is unlocked only on business days between the hours of 8:00 AM and 5:00 PM. The facility is also equipped with an electronic alarm system and employees are assigned unique pass codes for entry.

7.10.1 Employee Access

All employees must enter the building through the employee entrance. Keys are required except during normal business hours. Employees may exit

through the employee entrance when it is locked without the use of a key, but keys are required for re-entry.

7.10.2 Visitors, Vendors and Deliveries

All visitors to the office or laboratory must enter the main lobby through the main entrance and sign in with the receptionist. Visitors must be escorted at all times and in all areas of the facility. Vendors and delivery personnel are directed to use the door to the Shipping and Receiving area. Access to this door is controlled by the Microbac employee on duty, and upon entrance, the visitor must be escorted at all times.

7.11 Sample Subcontracting/Shipping

For certain projects, it may be necessary for Microbac to subcontract some analyses. Samples to be analyzed will be shipped to the contracted laboratory using the Microbac sample kit. To prevent sample breakage, only EPA-approved sample containers will be used. Freezer packs will be included in each sample kit to serve as a separation divider for the samples and to increase the cooling capacity.

In order to maintain the temperature of samples at ≤ 6° C, wet ice will be supplied by the client when required. Each shipping kit will contain the following:

- Partitions to hold sample bottles;
- Freezer pack(s) (if ice is not used);
- Chain-of-Custody form/Log-out form;

The sample kit will be packed in a hard side cooler. The sample kit will be sealed with Microbac Chain-of-Custody seals.

7.12 Electronic Data Security

Data integrity is insured through KOBRA LIMS multi-level security. Access to the specific user privileges can be individually controlled. Each user has their own user name and password which allows certain privileges. Hard copy data, which contains all of the data regarding a group of samples, are kept in a master file, labeled with the login numbers. Microbac provides electronic data deliverables (EDD) in client specified formats. Both e-mail and CD-ROM are commonly used for electronic transfer of data.

7.13 Automated Field Procedures

Microbac has developed electronic field tools for special projects to assist the sampler and sample custodian in many of the activities described previously in this section. The use of pre-programmed personal data assistants (PDA), barcode technology, and laptop computers enable the field personnel to focus on sample collection, while relying on the electronic tools to handle the laborious paper handling chores. Microbac enters the project sampling locations and the required analytical methods into the KOBRA LIMS, and downloads the information to the PDAs database tables prior to sampling. The sampler uses the barcode enabled PDA and the pre-labeled sample containers to more quickly and accurately collect the appropriate samples. After each sampling event the data from the PDA is transferred to a laptop computer, and the associated files are then uploaded to the KOBRA LIMS. Thus, many of the manual data field and laboratory data handling chores are handled electronically.

Uncontrolled Copy

Figure 7-1
MICROBAC CHAIN-OF-CUSTODY RECORD

COC No. A 15107

158 Starlite Drive
Marietta, OH 45750

Microbac
CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project ID:	
Sampler (print):	Signature:

Program
- ☐ CWA
- ☐ RCRA
- ☐ DOD
- ☐ AFCEE
- ☐ Other ____________

ADDITIONAL REQUIREMENTS

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold															TOTAL # (LAB USE)	

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Remarks:

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page ________ of ________

Figure 7-2
Sample Receipt Form

1000001675

COOLER INSPECTION

Microbac

Received: 09/14/2009 10:02
Delivery Method: UPS
Opened By: Robin Klinger
Comments:

Login(s): L09090265 L09090266 L09090267

Cooler(s)

Cooler #	Temp Gun	Temp	Tracking #	COC #	Comments
0013836	H	18.0	1Z83V9170192071486	091209-01-R	

1	Yes	Were shipping coolers sealed?
2	Yes	Were custody seals intact?
3	NA	Were cooler temperatures in range of 0-6?
4	NA	Was ice present?
5	Yes	Were COC's received/information complete/signed and dated?
6	Yes	Were sample containers and labels intact and match COC?
7	Yes	Were the correct containers and volumes received?
8	Yes	Were correct perservatives used? (water only)
9	Yes	Were pH ranges acceptable? (voa's excluded)
10	NA	Were VOA samples free of headspace (<6mm)?
11	Yes	Were samples received within EPA hold times?

Look closer. Go further. Do more.

Microbac - Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750
Tel: (740)373-4071 Fax: (740)373-4835

Figure 7-3
MICROBAC INTERNAL SAMPLE CUSTODY FORM

Microbac Laboratories Inc.
Internal Chain of Custody Report
Login: L12020203
Account: 2551
Project: 2551.094
Samples: 35
Due Date: 17-FEB-2012

Samplenum	Container ID	Products
L12020203-01	935121	826-SPE

Bottle: 1

Seq.	Purpose	From	To	Date/Time	Accept	Relinquish	pH
1	LOGIN	COOLER	V1	07-FEB-2012 17:09	RLK		<2
2	ANALYZ	V1	ORG4	08-FEB-2012 08:13	MRT	JKT	

Bottle: 2

Seq.	Purpose	From	To	Date/Time	Accept	Relinquish	pH
1	LOGIN	COOLER	V1	07-FEB-2012 17:09	RLK		<2
2	ANALYZ	V1	ORG4	08-FEB-2012 08:13	MRT	JKT	
3	STORE	ORG4	A1	21-FEB-2012 08:09	JKT	MRT	

Samplenum	Container ID	Products
L12020203-02	935122	826-SPE

Bottle: 1

Seq.	Purpose	From	To	Date/Time	Accept	Relinquish	pH
1	LOGIN	COOLER	V1	07-FEB-2012 17:09	RLK		<2
2	ANALYZ	V1	ORG4	08-FEB-2012 08:13	MRT	JKT	
3	STORE	ORG4	A1	21-FEB-2012 08:09	JKT	MRT	

Bottle: 2

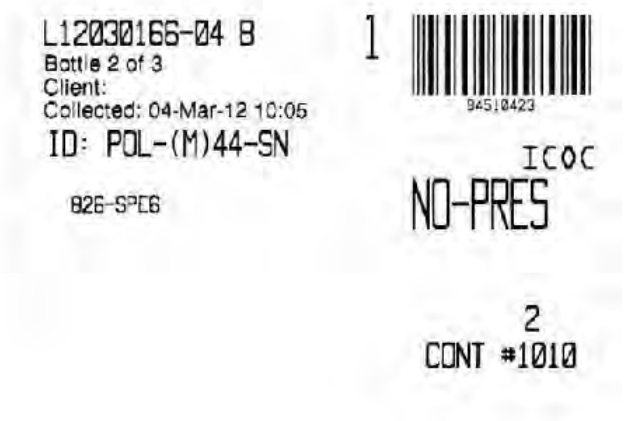
Seq.	Purpose	From	To	Date/Time	Accept	Relinquish	pH
1	LOGIN	COOLER	V1	07-FEB-2012 17:09	RLK		<2
2	ANALYZ	V1	ORG4	08-FEB-2012 08:13	MRT	JKT	
3	STORE	ORG4	A1	21-FEB-2012 08:09	JKT	MRT	

Bottle: 3

Seq.	Purpose	From	To	Date/Time	Accept	Relinquish	pH
1	LOGIN	COOLER	V1	07-FEB-2012 17:09	RLK		<2
2	ANALYZ	V1	ORG4	08-FEB-2012 08:13	MRT	JKT	
3	STORE	ORG4	A1	21-FEB-2012 08:09	JKT	MRT	

A1 - Sample Archive (COLD)
A2 - Sample Archive (AMBIENT)
F1 - Volatiles Freezer in Login
V1 - Volatiles Refrigerator in Login
W1 - Walkin Cooler in Login

Figure 7-4
MICROBAC BOTTLE LABEL



8.0 ANALYTICAL PROCEDURES, STANDARDS, AND REAGENTS

This section provides information about the method selection process and provides general analytical policies and procedures which are common to all methods in use in the laboratory. The process for method selection is described along with our policy on multiple versions of the same method, and addresses method validation and project specific criteria. Procedures are outlined for selection and cleaning of laboratory glassware, the requirements for Class A glassware, and the preparation and documentation for reagents and analytical standards. Also included are procedures for logbooks, proper container labeling, requirements for storage and disposal, and an example of measurement traceability utilizing a final report is given in Section 8.4.13. This information is addressed in more detail for each analytical method in the specific SOPs which are listed in Appendix B.

8.1 Analytical Methods

8.1.1 Selection

Methods which are used for compliance with local, state, US-EPA or other regulatory authorities must meet minimum performance criteria. Methods for which Microbac maintains performance criteria are listed in the SOP list found in Appendix B. Other methods exist that can be used for screening, or for estimating concentrations, or may not have defined performance specifications. Such methods are used for limited applications, or when written approval is granted by the client or agency. In general, analytical methods that are selected for implementation by Microbac must meet one of the following criteria:

- It is a promulgated version of an EPA approved method;
- It is another version of an EPA method that is specified in writing by the client or the project specific QAPP;
- It is a modified EPA method, or a standard method other than EPA which has been specified by the client or in the project specific QAPP. Examples include American Society for Testing and Materials (ASTM), National Institute for Occupational Safety and Health (NIOSH), or a client written method for which an official authorization has been given for use.

8.1.2 Method References

It is Microbac's policy to reference preparatory and analytical methods in appropriate documents including SOPs, preparation logsheets, instrument runlogs, method detection limit (MDL) studies, and analysis reports. The SOP

will reference the official source the current version of each method. Unless directed otherwise, Microbac will use the latest version of a method for which a validation study and SOP exists. It is our goal to implement the latest promulgated version of a method within the deadlines established by the agency or project specific QAPP. It may be necessary to maintain multiple versions of the same method due to project specific QAPPs which are often several years old. Project management tools are used by the client Chemist/Data Specialist (CDS) to assure proper versions are used. Additional information on this process may be found in Microbac SOP 44.

8.1.3 Method Validation

The procedures employed for method validation are found in Microbac SOP 45. The basic requirements include meeting all the reference method criteria for sample preparation, calibration and linearity checks, analytical precision and accuracy, MDL, method blank, surrogates, and the development and approval of the method SOP. Complete records of each validation study are maintained on file.

8.1.4 Project or Client Specific Criteria

The performance criteria established in the tables found in Microbac's analytical SOPs represent laboratory generated statistics often from multiple analysts and instruments. The reporting limits (RL), for example do not necessarily represent the lowest analyte concentrations that can be achieved by the method, nor are they guaranteed for every sample. In other cases, these basic laboratory statistics are superseded by client or project specific requirements for MDL, RL, acceptable control limits, or other special requirements. These situations will require the use of project specific QAPPs and/or SOPs. These are separate documents that are reproduced and distributed in project "kick-off" meetings in which specific project requirements are communicated to the QC and production staff. In such cases, project specific analysis codes are programmed into the KOBRA LIMS which specify the required RLs and control limits.

8.2 Glassware

The measurement of trace constituents in water, demands methods capable of maximum precision and sensitivity. Since the very sensitive analytical systems are subject to errors from improper choice of apparatus, as well as, the contamination effects due to improper cleaning procedures, the selection and proper care of laboratory glassware and sample containers play an important role in our quality control program. Laboratory vessels serve three functions:

storage of reagents and samples, measurement of solution volumes, and confinement of reactions. Borosilicate glass, i.e., "Pyrex" or "Kimax," is the mainstay of the laboratory. The analytical method usually states whether or not borosilicate type glassware is acceptable. A notable example is the analysis of boron, where the digestions are carried out in plastic or Vycor.

8.2.1 Volumetric Glassware

The precision of volumetric work depends in part, on the precision with which volumes of samples and other reagents can be measured. By common usage, accurately calibrated glassware for precise measurement of volume has become known as volumetric glassware. Glassware that meets Federal Specification for Volumetric Glassware is designated as Class A. Except for The National Institute of Standards and Technology (NIST) certified glassware, Class A is the most precise grade and is available in burettes, volumetric flasks, and volumetric pipettes. Class A glassware must be used in the following laboratory procedures:

- Preparation of all primary standards require Class A volumetric flasks and pipettes;
- Preparation and dilution of stock standards require Class A flasks and Class A pipettes;
- All titrimetic standardizations require the use of Class A burette, volumetric flasks and pipettes;
- Class A glassware will be used at all times as specified in the standard analytical method.

8.2.2 Glassware Cleaning Requirements

Table 8-1 summarizes the general cleaning procedures to be used for all laboratory glassware and reusable sample bottles (glass). All glassware must be scrupulously cleaned in order to remove any possibility of contamination of the sample. (**NOTE**: Under no circumstances are sample containers for trace level inorganic or organic analyses ever cleaned and reused).

8.3 Reagents and Solvents

8.3.1 Reagent Water

Reagent water produced at Microbac meets the standards of ASTM Type II Water (for the electrical conductivity and resistivity requirements). Reagent water is produced by an IonPure System maintained by US Filter and consists of a series of 5 filter beds:

- Carbon bed (removes chlorine and organics)
- Cation bed (removes heavy metals)
- Anion bed (removes negatively charged ions)
- Mixed bed (removes remaining negatively and positively charged ions)
- Mixed bed (removes remaining negatively and positively charged ions)

The electrical resistivity is checked daily by means of a remote sensor electrical resistivity meter and recorded by login department personnel. This remote sensor is located in-line, immediately following the filter beds. The finished reagent water must maintain at a minimum a resistivity of 17 MΩ-cm at 25° C. This is an in-house point of reference for monitoring only.

The two mixed bed tanks are both equipped with a 5 range purity light. These tanks are in series so a light monitoring system is utilized as a control to determine when tanks need to be replaced. When the first in the series shows an indicator light change from green to red, US Filter is called and service is requested. The second mixed bed tank continues to service the laboratory during this periods of time. The tanks indicator lights and the electrical resistivity meter are checked twice daily until the tanks are changed. After the tanks are changed, the lines are flushed and the electrical resistivity is rechecked. This system maintains if not exceeds a resistivity of 1.0 MΩ-cm resistivity and 1.0 μS/cm conductivity.

The electrical conductivity is checked daily (Monday – Friday) by means of an electrical conductivity meter and recorded by conventional laboratory personnel. The finished reagent water must have a maximum electrical conductivity of 1.0 μS/cm at 25° C to meet acceptance criteria. This reading is taken on water from the deionized water tap in the Conventionals laboratory. This is the furthest point-of-use tap from the filter beds. Additional UV polishing systems are used in the volatile laboratory to eliminate any trace organics and an ion polishing system is used in the metals laboratory.

8.3.2 Raw Materials

The raw materials which are used in the laboratory must be of a grade which meets or exceeds the method specifications. Inorganic reagents must be American Chemical Society (ACS) reagent grade or better. Acids used for preservatives or digestion for metals methods must be of a grade specified for trace metals. Solvents which are used to prepare standards, reagents and to extract samples must be of the highest grade available for the applicable method. Each lot of solvents or acids must be tested prior to use in the laboratory by using the analysis of a solvent (or reagent) blank which is concentrated to the same level as the sample extracts. Analysis by the method

must confirm the absence of interfering substances before the lot is introduced for client sample analysis. Reagents which are used in the digestion/extraction/analysis process are certified as an ultra pure grade by the manufacturer with certification papers kept on file. All chemicals and reagents are labeled with the date received, date opened and date of expiration.

The laboratory will ensure that all solvents, reagents, standards, and other materials that affect the quality of the analyses, will not be used until they have been inspected for compliance with required method or program specifications. The laboratory will not report data from any analysis, if quality control checks fail to confirm acceptability of solvents, reagents, standards, or materials consumed in the analysis. The laboratory will test and verify all reagents, solvents, and standards prior to use on Department of Defense (DoD) samples.

8.3.3 Preparation Logbooks

The preparation of reagents is done in accordance with requirements and the method specific SOP. The laboratory maintains detailed recipes and preparation records within the KOBRA LIMS, or in hardcopy, for each analytical reagent. Required content of the reagent records are outlined below and an example KOBRA LIMS record is presented as Figure 8-1.

- Reagent ID
- Recipe – lists the preparation details
- Preparer's initials
- Date prepared
- Expiration Date
- Disposal Date
- Concentrations of constituents
- Units
- Reviewer's initials

The preparation date and expiration date must be the same for all reagents prepared fresh daily. The department supervisor is required to perform a periodic review of the logbooks.

8.3.4 Labeling

Labeling of laboratory reagents must contain the following minimum content:

- Reagent name
- Concentration and units
- Initials of preparer

- Date prepared (mm-dd-yy)
- Expiration Date (mm-dd-yy)
- Reagent ID

The reagent ID provides traceability to the KOBRA LIMS records for the preparation described in Section 8.3.3.

8.3.5 Storage

All reagent storage at Microbac is done in such a way that safety is always considered first. All flammable compounds are stored in a flammable cabinet. Volatile reagents are always stored and used in a well-ventilated area. Table 8-2 gives a list of common reagents and solvents used at Microbac and the method with which they are stored. Unless otherwise stated, borosilicate glass or polyethylene bottles are acceptable for storage of reagents used in inorganic analyses.

8.3.6 Waste Disposal

Since all laboratory work with chemicals eventually produces waste, we are aware of our moral and regulatory obligations to utilize sound waste management policies and procedures. Therefore, the disposal of reagent waste at our facility is an issue of great importance and one which has been given much attention. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. The specifics of Microbac's waste management system are included in the Microbac SOP33 – Laboratory Waste Management, a copy of which may be reviewed upon request.

8.4 Analytical Standards

This section describes all of the procedures required to document the preparation and use of calibration and quality control standards. Policies covered include source material selection, receipt, traceability, preparation and labeling of stock, intermediate, and working standards, KOBRA LIMS records, labeling, storage, and disposal. Microbac maintains complete records for standards preparation and traceability electronically within the KOBRA LIMS. Figures 8-2 through 8-5 present example records from the electronic system.

8.4.1 Standard Sources and Preparation

The accuracy of data produced from analytical instrumentation depends primarily on the quality of calibration standards. Microbac uses fully

documented procedures to assure that calibration and quality control standards are prepared to the highest level of accuracy available for a particular analyte. Concentrated stocks are prepared only with the highest purity standards available. Only standards that are certified traceable to NIST and are A2LA, ISO Guide 34 in conjunction with ISO 17025 certified, will be used.

8.4.2 Standard Receipt and Traceability

Once purchased standards are received, they are immediately brought to the area in which they are to be used. At this point, the supervisor or analyst will place the standard in its proper storage area. Volatile standards are stored in a freezer. Semi-volatile standards which are purchased neat are stored at room temperature. Stock standards purchased commercially are stored according to the following:

- Water or methanol based standards are stored at 4° C.
- Methylene chloride or hexane based standards are stored in a freezer.
- Standards of compounds which easily precipitate are stored at room temperature.
- PCB spiking standards are stored at room temperature.

All standards will be stored separately from samples. Safety Data Sheets (SDS) provided with all standards, will be stored in a notebook and will be freely available to any analyst. Other certification sheets will be kept on file within each lab division and stored for future reference. Since much of the important information is provided on the label, (manufacturer, lot number, concentration, purity, formula, compound name and health information), it also serves as a good source of information.

8.4.3 Certificate of Analysis

The laboratory will maintain a system for identifying and filing certificates of analysis (COA) for all purchased standards. The analyst will log the COA into the KOBRA LIMS, which assigns a unique serial COA number. The laboratory will file electronic (pdf) copies of each COA in the COA folder of the network. The filename is in the format <COA#.pdf>. See Figure 8-2.

8.4.4 Primary Standards

Primary standards are used in the classical chemistry laboratory to standardize selected titrants. These chemicals must specify “primary standard grade” to be acceptable for this purpose. The laboratory maintains primary standards for potassium hydrogen phthalate (KHP) and potassium dichromate, which can be

used to standardize a wide variety of acid/base and redox titrants, if necessary. The department supervisor is responsible for the custody of these and other standard reference materials.

8.4.5 Neat Standard Materials

Most calibration and quality control standards are purchased as a concentrated stock solution from an approved vendor, however high purity neat standards may be used in some custom applications. Only materials of a known, certified purity are chosen for this purpose. Certificates of analysis for each of these neat materials are archived in KOBRA LIMS to provide complete documentation and traceability.

8.4.6 Stock Solutions

A record of the preparation of concentrated stock standards will be maintained for each parameter. When a primary standard or concentrated stock standard is prepared, a number is assigned to that standard, along with the date, analyst, compound, lot number, purity net weight, weight adjusted for purity, dilution volume and actual concentration are recorded. When a standard solution is prepared from a neat compound, if the entire sample is used the label is removed from the container and attached to the standards form. When the parent stock is purchased through a commercially prepared source, these standards (i.e., 1000 ppm metal standards for atomic absorption) must have the vendor, concentration, expiration date and lot number of this stock recorded into the standard logbook. Often commercially prepared mixes are purchased.

8.4.7 Intermediate Standards

Once the stock standards are prepared, they are then diluted to prepare intermediate-level standards and/or final working standards using volumetric pipettes and glassware. The choice of the correct apparatus is very important (see Section 8.0). It is in the intermediate stage that more than one parameter is mixed in the right concentration for the final mix. This dilution must also be documented in the same manner as the concentrated stocks. An example KOBRA LIMS record is presented in Figure 8-3.

8.4.8 Working Standards

These are the final dilutions that are used in the calibration or analysis stage of the method. The same procedures and KOBRA LIMS documentation that were described for intermediate standards also apply to the working solutions.

8.4.9 Electronic Records

Electronic records for the preparation, documentation, and traceability of calibration and quality control standards are maintained in the KOBRA LIMS. Each stock, intermediate, and working standard has a unique number that is cross-referenced to the electronic tables. See Figures 8-2 - 8-5.

8.4.10 Labeling

Labeling of calibration and quality control standards must contain the following minimum content:

- Standard name
- Concentration and units
- Initials of preparer
- Date prepared
- Expiration date
- Standard number (unique reference)

Due to the very small size of standard containers employed in gas chromatographic analyses, space may not allow for all of the above information.

8.4.11 Storage

Analytical and quality control standards are stored in the individual laboratories. (See Table 8-3) The Conventionals, Extraction, Semivolatile Organic, and Volatile Organic have refrigerators which are dedicated to reagents and standards. Standards for organic methods are stored in borosilicate glass containers with a Teflon lined cap which have been cleaned and solvent rinsed. Standards used in the Conventionals laboratory are stored in containers compatible with the analyte and solvent i.e., of the same composition as the required sampling container. Metals standards are stored at room temperature and placed in a storage cabinet. Storage of standards used in atomic absorption analyses must be in a polyethylene container, except where noted in the method, i.e., silver. Standards which have expired must be immediately removed from the storage unit and taken to the support services supervisor for proper disposal. Samples and standards must never be stored in the same refrigerator.

8.4.12 Disposal

The disposal of all standards is done in accordance with procedures outlined in the Microbac SOP 33, "Laboratory Waste Management".

8.4.13 The Certificate of Analysis provided with final reports provides information for traceability of each measurement reported. The header for each analysis lists:

- Sample # - which is the laboratory ID
- Client ID – which is the sample name provided by the client on the chain of custody
- Matrix – names the matrix classification of the sample
- Workgroup # - is the LIMS ID for the group of samples included in the analytical batch, the workgroup includes tracking of spiking solutions and reagents used when applicable. The analytical workgroup is also linked in the LIMS to sample preparation workgroups where applicable.
- Collect Date – date of sample collection provided on the chain of custody
- Sample Tag – used to denote separate analyses performed on the particular sample fraction per analytical method
- PrePrep Method – used to track methods like TCLP or water leachate preparations prior to analytical preparations
- Prep Method – lists specific sample preparation methods of extraction or digestion
- Analytical Method – lists specific analytical methods employed
- Analyst – Identifies the analyst who performed the analysis
- Dilution – shows the dilution factor at which the analysis was performed
- Units- the units that apply to the numerical values presented on the associated analysis
- Instrument – indicates the particular instrument used to determine the results being reported
- Prep Date – lists the date analytical sample preparation was performed
- Cal Date – is the date the instrument was calibrated
- Run date – is the date the particular analysis was performed
- File ID – is the raw data file the analytical data for a particular analysis was collected in and stored under

Use of this information can be used to trace the result back to the instrument(s) and analyst(s) who performed the analysis for each the standards and reagents used in each step of the analytical process. The exact configuration of the instrument(s) used are recorded in the instrument maintenance logs. The calibration standards and calibration verification standards, internal standards (if applicable), analytical column (if applicable), the laboratory SOP(s) ID(s) and

version used for that days analytical sequence, the data file name, analytical date(s) and time(s) are recorded in the run log(s). The run log also shows the analysis of calibration and verification standards relevant to the results reported.

8.5 Standardization of Titrating Solutions

Table 8-4 gives a listing of the reagents used as titrants. All solutions are purchased pre-standardized by the manufacturer. The manufacturer's certificate of analysis provides the following information:

- Nominal Concentration (Normality)
- Concentration Limits
- Lot Number
- Primary Standard (Certified traceable to NIST)

As a daily quality control check on the commercial solutions, the laboratory verifies the certified values through the analyses of a laboratory control sample. The standardization is checked monthly using the primary standard. If the results of this analysis indicates a problem, i.e. an out of control situation, then re-standardization against primary standards is required. Alternatively, the out-of-specification solutions are discarded and fresh solutions are obtained as replacements.

TABLE 8-1
LAB GLASSWARE CLEANING PROCEDURES

Analysis/Parameter	Cleaning Procedure (in order specified)
Extractable Organics (including Pesticides and Herbicides)	1-3, 8, 4, 7, 10, 12, 13
Purgeable Organics	Use either disposable glassware or steps 2, 3, 4, 7
Trace Metals: glass plastic	1-6 1-4, 15, 6
Nutrients	1-4, 12
Minerals, COD,BOD, Radiochemistry, Cyanide, Phenols	1-4, 12
Residues	1-4, 9 or 10, 12
MBAS	1-4, 14, 12
Petroleum Hydrocarbons	1-4, 7, 14, 13
Oil & Grease	1-4, 7, 14, 9

Cleaning Procedures:

1. Remove all labels using sponge or acetone.
2. Wash with hot tap water and a brush to scrub inside of glassware, stopcocks, and other small pieces, if possible, using a suitable laboratory-grade detergent.
   - Organics - Liquinox, Alconox or equivalent
   - Inorganic anions - Liquinox or equivalent
   - Inorganic cations - Liquinox, Alconox, Micro or equivalent
   - Bacteriologicals - must pass an inhibitory residue test
3. Rinse thoroughly with hot tap water.
4. Rinse thoroughly with deionized water.
5. Rinse or soak with 10% Nitric Acid.
6. Rinse 3 times with deionized water.
7. Rinse thoroughly with pesticide grade Methanol.
8. Rinse with 25% sulfuric acid.
9. Bake at 105°C for 3-4 hours.
10. Bake at 180°C for 3-4 hours (prior to use as per method).
11. After use, rinse with last solvent used.
12. Store inverted or capped with suitable material or suitable container stopper.
13. Last step (prior to use) must be a rinse with the solvent used in analysis.
14. Rinse thoroughly with acetone.
15. Rinse or soak with 1:1 HCl (Hydrochloric Acid).

TABLE 8-2
REAGENT STORAGE

REAGENT	METHOD OF STORAGE
Hydrochloric Acid (HCl) Sulfuric Acid (H_2SO_4) Nitric Acid (HNO_3) Acetic Acid ($C_2H_4O_2$) Phosphoric Acid (H_3PO_4)	Stored in original containers in a vented cabinet designed for acid storage.
Ammonium Hydroxide (NH_4OH)	Stored in original containers in a vented cabinet designed for base storage.
Acetone (C_3H_6O) Methanol (CH_3OH) Propyl Alcohol (C_3H_8O) Isopropyl Alcohol (C_3H_8O) Pyridine (C_5H_5N) Ethyl Acetone ($C_4H_8O_2$)	Stored in original containers in a vented cabinet designed for flammable materials.
Chloroform ($CHCl_3$) Methylene Chloride (CH_2Cl_2) Ethyl Ether ($C_4H_{10}O$) Hexane (C_6H_{14})	Stored in original containers in a vented cabinet designed for volatile organics storage.
Freon-113	Stored in original containers in a vented corner of the liquid-liquid continuous extraction sample prep area.

TABLE 8-3
STANDARD PREPARATION AND SOURCES

Instrument Group	Concentration	Storage	Preparation from Source	Standard Expiration
		VOLATILE		
Gas Chromatography/ Mass Spectrometry (GC/MS)	primary stock mixture is at variable concentrations	freezer	primary stock mixtures are commercially prepared solution mixes	6 months or manufacturer recommended expiration date
	intermediate standards are at variable concentrations	freezer	intermediate standards are prepared from commercially purchased stock mixes	1 month
	working concentration	used immediately	working standards are prepared from intermediates	prepared fresh daily
		SEMI-VOLATILE		
Gas Chromatography (GC)	primary stock mixture is at variable concentrations	freezer	primary stock mixtures are commercially prepared solution mixes	6 months or manufacturer recommended expiration date
	intermediate standards are at variable concentrations	freezer	intermediate standards are prepared from commercially purchased stock mixes	6 months
	working concentration	used immediately	working standards are prepared from intermediates	prepared fresh daily
Gas Chromatography/ Mass Spectrometry (GC/MS)	primary stock mixture is at variable concentrations	freezer	primary stock mixtures are commercially prepared solution mixes	6 months or manufacturer recommended expiration date
	intermediate standards are at variable concentrations	freezer	intermediate standards are prepared from commercially purchased stock mixes	6 months
	working concentration	freezer	working standards are prepared from intermediates	6 months
High Performance Liquid Chromatography (HPLC)	primary stock mixture is at variable concentrations # 100 ppm	freezer	primary stock mixtures are commercially prepared solution mixes	6 months (8310),1 month (8330) or manufacturer recommended expiration date
	working concentration	freezer	working standards are prepared from commercial stocks	24 hours
Atomic Absorption furnace and cold vapor	primary stock mixture is at variable concentrations ≥ 1000 ppm	room temperature	primary stock mixtures are commercially prepared solution mixes	12 months or manufacturer recommended expiration date
	intermediate standards are at variable concentrations ≥ 10 ppm (high standard)	room temperature	intermediate standards are prepared from commercially purchased stock mixes	6 months
	working concentration	room temperature	working standards are prepared from intermediates	24 hours

TABLE 8-3 (continued)
STANDARD PREPARATION AND SOURCES

Instrument Group	Concentration	Storage	Preparation from Source	Standard Expiration
Inductively Coupled Plasma (ICP)	primary stock mixture is at variable concentrations ≥ 1000 mg/L	room temperature	primary stock mixtures are commercially prepared solution mixes	12 months or manufacturer recommended expiration date
	intermediate standards are at variable concentrations ≥ 10 mg/L (high standard)	room temperature	intermediate standards are prepared from commercially purchased stock mixes	6 months
	Working concentration	room temperature	working standards are prepared from intermediates	prepared fresh daily
Total Organic Carbon (TOC)	primary stock 1000 mg/L solution	room temperature	primary stock is a commercially prepared solution	6 months or manufacturer recommended expiration date
	Working concentration	used immediately	working standards are prepared from intermediate	prepared fresh daily
pH Meters	pH = 4,7,10 Premixed	room temperature	used as received commercial stocks	fresh daily
Turbidimeters	Pre-mixed 4 Gelex Standards NTU 0-1 NTU 1-10 NTU 10-100 NTU 100-1000 scale Check Standard Primary (Formazin)	room temperature	used as received commercial stocks prepared from 4000 NTU Stock	restandardized quarterly manufacturer recommended expiration date
Specific Conductivity Meter	Neat	room temperature.	stock: 14,130 umho/cm working Standard:	6 months
	Working concentration	4° C	1413 umho/cm	24 hours
CONVENTIONAL (SMARTCHEM)				
Ammonia	primary stock = 1000 mg/L	room temperature	used as received commercially prepared	Manufacturer recommended expiration date
	working standards are at variable concentrations	room temperature	working standards are prepared from commercially prepared primary stock	1 month
Chloride	primary stock = 1000 mg/L	room temperature	used as received commercially prepared	manufacturer recommended expiration date
	working standards are at variable concentrations	room temperature	working standards are prepared from commercially prepared primary stock	1 month

TABLE 8-3 (continued)
STANDARD PREPARATION AND SOURCES

Instrument Group	Concentration	Storage	Preparation from Source	Standard Expiration
Nitrate	primary stock = 100 mg/L	room temperature	used as received commercially prepared	manufacturer recommended expiration date
	working standards are at variable concentrations	room temperature	working standards are prepared from commercially prepared primary stock	1 month
Alkalinity	primary stock = 1000 mg/L	room temperature	prepared by Microbac from sodium carbonate	6 months
	working standards are at variable concentrations	room temperature	working standards are prepared from Microbac's prepared primary stock	1 month
Sulfate	primary stock = 1000 mg/L	room temperature	used as received commercially prepared	Manufacturer recommended expiration date
Phosphorus	primary stock = 200 mg/L	room temperature	prepared by Microbac from KH_2PO_4	6 months
	working standards are at variable concentrations	room temperature	working standards are prepared from Microbac's prepared primary stock	1 month
Total Kjeldahl Nitrogen (TKN)	primary stock = 200 mg/L	room temperature	prepared by Microbac from $(NH_4)_2SO_4$	6 months
	working standards are at variable concentrations	room temperature	working standards are prepared from Microbac's prepared primary stock	1 month

Uncontrolled Copy

TABLE 8-4
STANDARDIZATION OF TITRATING SOLUTIONS

Parameter	Titrating Solution	Primary Standard	Frequency of Standardization
Acidity	NaOH	KHP NIST traceable	Prestandardized by the manufacturer checked monthly
Alkalinity	H_2SO_4	Sodium Carbonate Standard Solution NIST traceable	Prestandardized by the manufacturer checked monthly
Hardness	EDTA	Calcium carbonate as $CaCl_2$ NIST traceable	Prestandardized by the manufacturer checked monthly

Note: All titration solutions are purchased pre-standardized.

Figure 8-1
Example – KOBRA LIMS Reagent Record

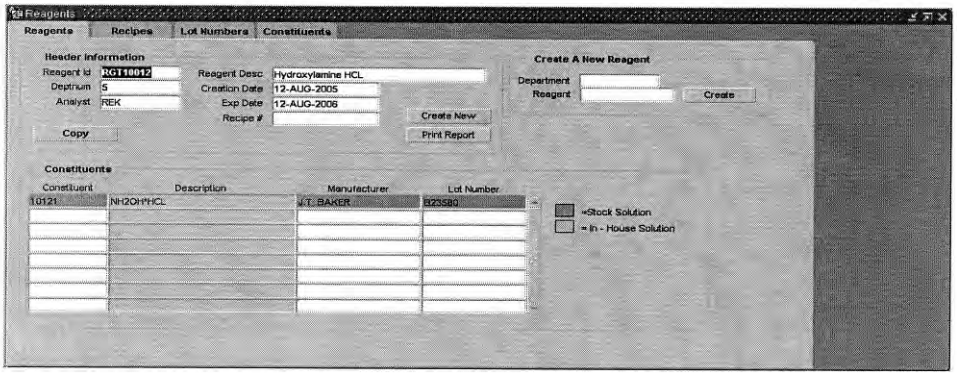


Figure 8-2
Example – KOBRA LIMS Certificate of Analysis Record

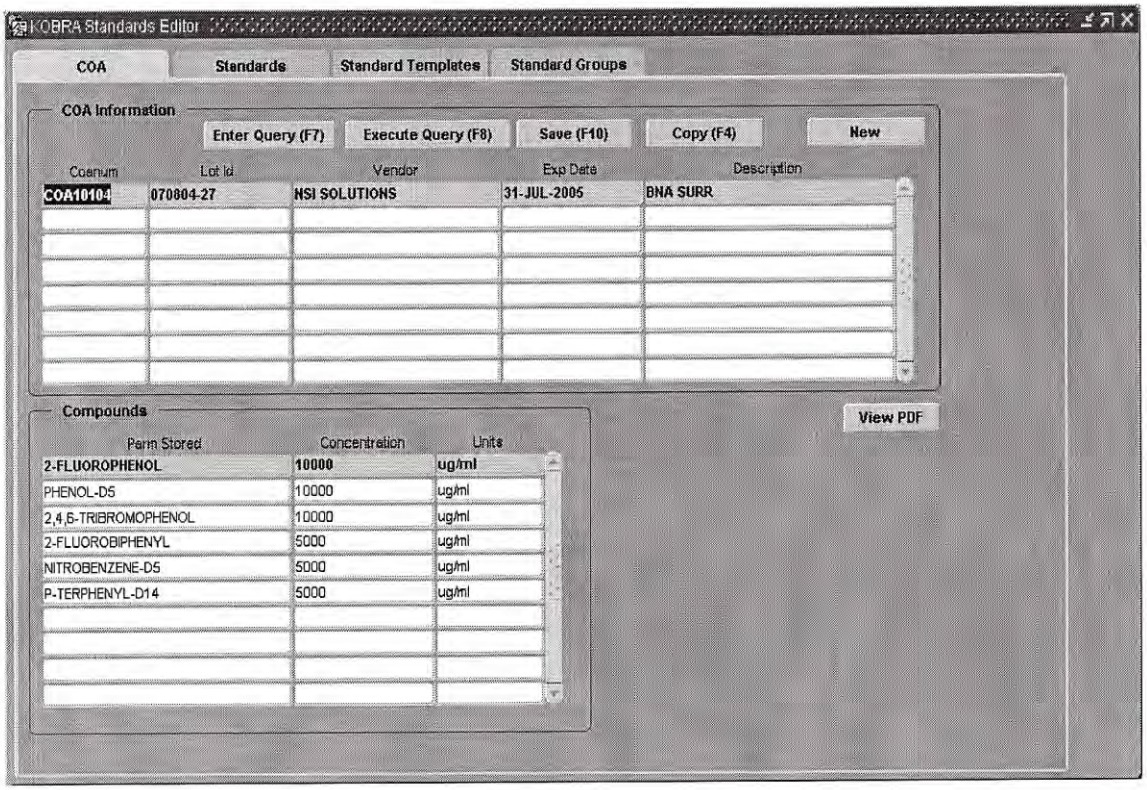


Figure 8-3
Example – KOBRA LIMS Standard Record

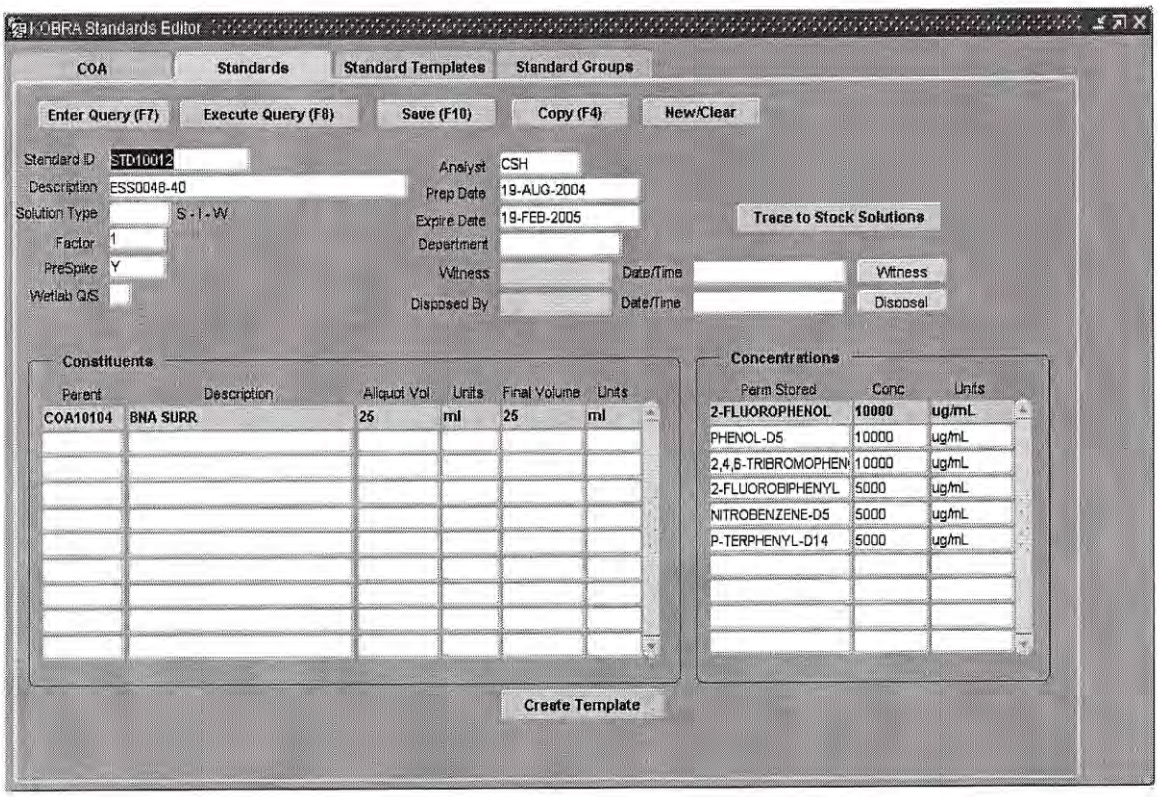


Figure 8-4
Example – KOBRA LIMS Standard Template

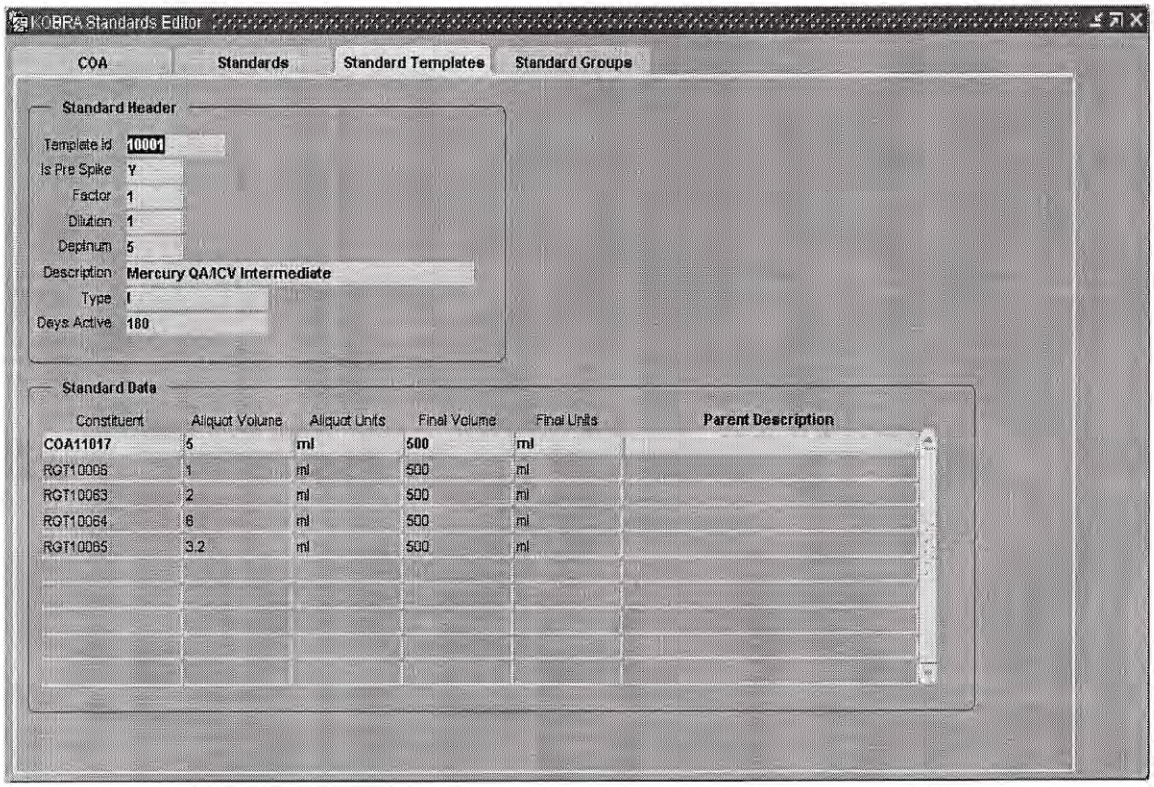
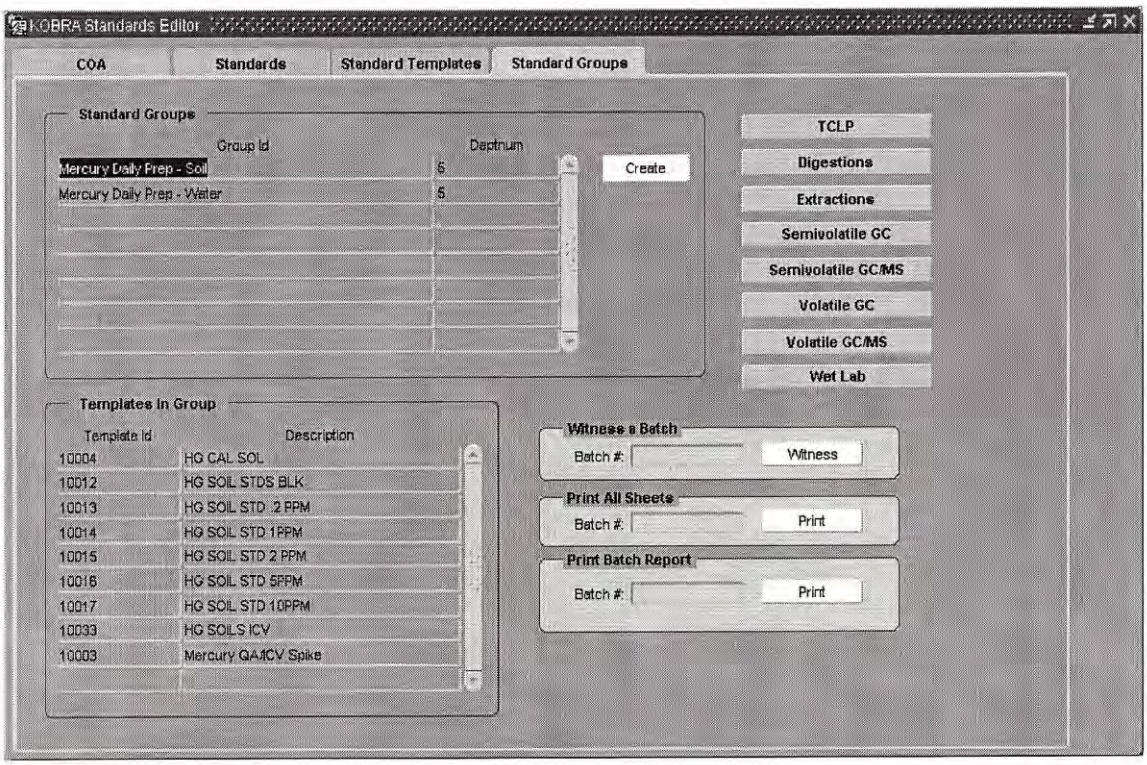


Figure 8-5
Example – KOBRA LIMS Standard Group



9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Instrumentation

Since the modern analytical laboratory depends heavily upon instrumentation, their calibration, operation and maintenance must be a primary concern in the production of satisfactory data. Table 9-1 is a list of the instruments commonly used by Microbac in the analysis of various sample matrices.

Table 9-1

MICROBAC®

Table 1
Microbac – Ohio Valley Division
Equipment List

Location	Instrument Name	Date Acquired	Purchase Condition	Serial Number
Conventionals	Balance - Explorer Pro ED2102	10/07	New	L291112835364P
Conventionals	Balance - AND GR202	01/06	New	14214584
Conventionals	Balance - AND GR202	10/13	New	14241353
Conventionals	CN – Easy Dist Universal Distillation System	12/02	New	483-W001-01
Conventionals	COD Reactor (HACH) I Block Digester	1987	New	930100008493
Conventionals	COD Reactor (HACH) II	03/09	Used	NA
Conventionals	Horizon 3000 XL with Controller	08/13	New	13-1935 13-1353
Conventionals	Horizon 2 with Controller	03/14	Used	05-1232 10-1174
Conventionals	Orion pH/ISE 710A	01/98	New	B08320
Conventionals	Orion pH/ISE Model 710A pH Ion Meter	04/95	New	002576
Conventionals	Orion pH/ISE Model 4Star	08/08	New	B08320
Conventionals	Portable pH Meter	12/12	New	G01544
Conventionals	Shimadzu UV-1201V Spectrophotometer	08/99	New	A108036800041
Conventionals	SmartChem Automated Chemistry Analyzer	04/04	New	W0402048
Conventionals	SmartChem Automated Chemistry Analyzer	07/13	New	W1306317
Conventionals	Speed Vap III	08/13	New	13-0870
Conventionals	Speed Vap II	08/13	Used	05-0287
Conventionals	Tiamio	03/13	New	1855001004249
Conventionals	TOC Shimadzu	12/02	New	39624467
Conventionals	VWR Turbidity Meter	03/01	New	800 1682
Conventionals	Westco Easy Distillation	08/99	New	1061
Conventionals	YSI Model 32 Conductivity Meter	08/93	New	93G07466
Extractions	ASE200 Accelerated Solvent Extractor	05/99 01/03	New New	99040590 02110605
Extractions	Balance – A&D FX1200i	08/09	New	15608655
Extractions	Bomb #1	Prior to 2008	-	110609-M30126
Extractions	Bomb #2	Prior to 2008	-	100908-M20776
Extractions	Bomb #3	03/14	New	032812-M37877
Extractions	Bomb Calorimeter	Prior to 2008	-	2901EB

Page 1 of 4

Table 9-1 (continued)

MICROBAC®

Location	Instrument Name	Date Acquired	Purchase Condition	Serial Number
Extractions	Burrel Wristaction Shaker	2007	New	183070624
Extractions	ETHOS EX Milestone Microwave	2011	New	132778
Extractions	Fisher Scientific Oven	2013	New	613208-639
Extractions	Flash	Prior to 2008	-	70007
Extractions	#1 Glas Col 8 Place, 2 Liter SEP-Funnel Shaker	1996	New	-
Extractions	#2 Glas Col 8 Place, 2 Liter SEP-Funnel Shaker	1996	New	207726
Extractions	#3 Glas Col 8 Place, 2 Liter SEP-Funnel Shaker	2012	Used	002MCRS21000B 7
Extractions	Misonix XL 2020 Ultrasonicator #1	01/96	New	G2664
Extraction	Misonix XL 2020 Ultrasonicator #2	05/12	Used	G1664
Extractions	Misonix XL 2020 Ultrasonicator #3	01/99	New	G3348
Extractions	OA-SYS N-EVAP112	2010	New	51460
Extractions	6 Place Stir Plate	1991	-	07-G31-00032
Extractions	Precision Scientific Gravity Convection Oven	2010	New	9303 007
Extractions	Puck Mill	09/09	New	217510007
Extractions	TurboVap II Workstation	09/96	New	TV9633N7004
Extractions	TurboVap II Workstation	04/97	New	TV9715N7428
Extractions	TurboVap II Workstation	04/97	New	TV9715N7427
Extractions	TurboVap II Workstation	06/05	New	TV0156N12265
Extractions	TurboVap II Workstation	06/05	New	TV0516N12264
Extractions	Water Bath Sonicator	2007	New	Microbac #00130
Extractions	Water Bath Sonicator	2007	New	Microbac #00139
Extractions TCLP	Balance - OHAUS TS600	06/01	New	29515505
Extractions TCLP	Associated Design Tumbler A-1	Prior to 2008	-	1065
Extractions TCLP	Associated Design Tumbler A-2	Prior to 2008	-	1527
Extractions TCLP	Associated Design Tumbler A-5	Prior to 2008	-	1464
Extractions TCLP	Associated Design Tumbler A-4	Prior to 2008	-	1591
Extractions TCLP	Associated Design Tumbler A-3	Prior to 2008	-	-
Extractions TCLP	Associated Design Tumbler A-6	Prior to 2008	-	-
Metals Digest	Balance - AND300	10/13	New	T0340529
Metals Digest	Balance - AND300	07/08	New	14697359

Table 9-1 (continued)

MICROBAC®

Location	Instrument Name	Date Acquired	Purchase Condition	Serial Number
Metals Digest	Balance - AND600	09/06	New	14699618
Metals Digest	Balance - AND GR202	06/05	New	14221258
Metals Digest	Balance - ANDFX-1200iWP	10/08	New	15801769
Metals Digest	Balance - GF-300	05/06	New	14662980
Metals Digest	CEM MARS XPRESS	08/10	New	MD3721
Metals Digest	CEM MARS XPRESS Microwave	01/12	New	MD4470
Metals Digest	CEM MARS XPRESS Microwave	07/05	New	MD7719
Metals	CETAC ASXpress-520 Autosampler (Upgrade Kit)	01/13	New	70845A520
Metals	ES SC-4 DX FAST Autosampler	01/12	New	X4DXS-HS-TSP-16-111002
Metals	ES SC-8 DX FAST Autosampler	07/10	New	X8DX-HS-TSP-16-100602
Metals	ES SC-8 DX FAST Autosampler	02/10	New	X8DX-HS-TSP-16-100102
Metals	Thermo Scientific iCAP 6000	02/08	New	20080504
Metals	Thermo Scientific iCAP 6000	08/08	New	20083006
Metals ICP/MS	Perkin Elmer NexION 300X ICP-MS	01/12	New	SN81XN1110202
Metals ICP	Perkin Elmer Optima 4300 ICP-AES	01/04	New	930S4010403
Metals Mercury	CETAC M-7600 Mercury Analyzer CV-AAS	01/13	Used	1111B12Q76
Semivolatile GC	HP9 6890 Gas Chromatograph – ECD	05/98	New	US00021274
Semivolatile GC	HP14 AG 6890 Gas Chromatograph - FID	11/02	New	US10207074
Semivolatile GC	HP15 AG 6890N Gas Chromatograph – ECD	09/04	New	CN10428037
Semivolatile GC	HP17 6890 Dual/ECD Gas Chromatograph	04/07	New	CN10704083
Semivolatile GC	HP18 Agilent 6890 Gas Chromatograph – FID	09/07	Used	DE00000787
Semivolatile GC	HP18 FID (7890) Gas Chromatograph	01/11	New	CN10481154
Semivolatile HPLC	HPLC3 1100 Liquid Chromatograph	12/97	New	US72102126
Semivolatile HPLC	HPLC4 1100 Liquid Chromatograph	11/99	New	DE91606291
Semivolatile HPLC	HPLC5 1200 Liquid Chromatograph	09/06	New	DEG60555831
Semivolatile HPLC	HPLC6 Shimadzu Prominence XR Liquid Chromatograph	08/10	New	L20454600114
Semivolatile GC/MS	HPMS4 5973 – 6890 Gas Chromatograph/Mass Spec	05/99	New	GC-US00026408 MS-US82322192
Semivolatile GC/MS	HPMS5 5973 – 6890 Gas Chromatograph/Mass Spec	09/96	New	GC-US00004989 MS-US62510073
Semivolatile GC/MS	HPMS7 5973 – 6890 Gas Chromatograph/Mass Spec	04/97	New	GC-US00007949 MS-US70820650

Page 3 of 4

Table 9-1 (continued)

MICROBAC®

Location	Instrument Name	Date Acquired	Purchase Condition	Serial Number
Semivolatile GC/MS	HPMS12 AG 5973 – 6890 Gas Chromatograph/Mass Spec	05/03	New	GC-US10249164 MS-US21869372
Semivolatile GC/MS	HPMS15 AG 5975 – 6890 Gas Chromatograph/Mass Spec	04/07	New	GC-CN10710041 MS-US65135157
Semivolatile IC	IC1 Dionex 500 Ion Chromatograph	12/98	New	98090321
Semivolatile IC	IC2 Ion Chromatograph	08/01	New	01080138
Semivolatile IC	IC3 Ion Chromatograph	01/04	New	13120956
Semivolatile LC/MS	LCMS1 API 4000 Applied Biosystems LC/MS/MS	07/09	New	J0200104
Semivolatile	VWR Ultrasonic Cleaner	10/03	New	01TS37915
Volatiles GC/MS	HPMS6 - Agilent 6890 Gas Chromatograph with 5973 Mass Spec, Varian Archon, Tekmar 3000 Sample Concentrator	12/96	New	GC-Us00006300 MS-US63810266
Volatiles GC/MS	HPMS8 - Agilent 6890 Gas Chromatograph with 5973 Mass Spec, Varian Archon, Tekmar 3000 Sample Concentrator	01/98	New	GC-US0010116 MS-US82321660
Volatiles GC/MS	HPMS9 - Agilent 6890 Gas Chromatograph with 5973 Mass Spec, Tekmar Atomx Concentrator and Autosampler	10/98	New	GC-US00022637 MS-US72821183
Volatiles GC/MS	HPMS11 - Agilent 6890 Gas Chromatograph with 5973 Mass Spec, Varian Archon, Tekmar Velocity Sample Concentrator	08/00	New	GC-US00036002 MS-US94260126
Volatiles GC/MS	HPMS17 - Agilent 7890 Gas Chromatograph with 5977 Mass Spec, Tekmar Atomx Concentrator and Autosampler	10/13	New	GC-CN13293018 MS-US1332L214
Volatiles GC	HP5 - Agilent 6890 Gas Chromatograph PID/FID, Varian Archon, Tekmar 3000 Sample Concentrator	07/92	New	3203A41712
Volatiles GC/MS	HP16 - Agilent 6890 Gas Chromatograph FID/TCD, Tekmar HT3 Autosampler	11/04	New	GC-CN10429033 AS-IT00428012
Volatiles GC	HP19 - Agilent 7890 Gas Chromatograph PID/FID, Varian Archon, Tekmar Velocity Sample Concentrator	01/12	New	GC-CN10932057
Volatiles	Balance - AND EK-200i	05/07	New	P1845012
Volatiles	Balance - OHAUS N02120	06/03	New	1121242247

Page 4 of 4

9.2 General Calibration Requirements

All laboratory instrumentation must be properly calibrated before it is put into official use. Some general policies and guidelines are provided in the following sections, but complete details are found in the Microbac SOP for the particular method. Specific program or project requirements may be provided in the project QAPP or the sampling and analysis plan (SAP) and may supercede the method and Microbac's SOP.

9.2.1 Initial Calibration

The initial calibration (ICAL) must meet the minimum requirements for the method in use. The calibration must contain the minimum number of points and the curve must pass the method criteria. If more than the minimum number of calibration points are analyzed for initial calibration, it is acceptable to eliminate points only at the top or bottom of the range, in order to get the curve to pass ICAL criteria. Points may not be eliminated from the middle portion of curve unless the entire ICAL is repeated.

9.2.2 Second Source Verification

It is the general policy of Microbac to verify the initial calibration with the analysis of a standard from a second source (different vendor). Acceptance criteria for the second source initial calibration verification (ICV) may be found in the specific SOP or the project QAPP or SAP. Any problems with the ICV that cannot be resolved by corrective action must be noted in the report narrative and the data properly qualified.

9.2.3 Continuing Calibration Verification

Continuing calibration verification (CCV) must be performed at the method specified frequency and meet the method SOP, QAPP, or SAP requirements. When required by the method, all samples must be bracketed by passing CCVs. Samples not bracketed by passing CCVs must be reanalyzed. Any problems with the CCV that cannot be resolved by corrective action must be noted in the report narrative and the data properly qualified.

9.3 Gas Chromatography

- ECD (Electron Capture Detector): Pesticides/PCBs/Herbicides
- FID (Flame Ionization Detector):Polynuclear Aromatic Hydrocarbons /DRO/8015/GRO
- PID (Photoionization Detection): Aromatic Volatile Organics

- ELCD (Electrolytic Conductivity Detector): Halogenated Volatile Organics

Initial calibration is at least a 5-point standard curve of all target compounds, (concentrations are in detector linear range) performed on failure of two continuing calibration standards. The lowest calibration standard has a concentration that is equal to or less than the Reporting Limit. Curves are checked by calculating the %RSD for all compounds.

Initial calibration and run sequence:

1. Initial calibration
2. ICV
3. Analyze 10 samples (includes reagent blanks, LCS, spikes and duplicates)
4. Run check standard
5. Continue with steps 3 and 4 for entire run

Daily continuing calibration and run sequence:

1. CCV
2. Analyze 10 samples (includes reagent blanks, LCS, spikes and duplicates)
3. Analyze check standard
4. Continue with analysis steps 2 and 3 for entire run

Samples must be bracketed by passing CCVs. If the CCV does not meet criteria, all affected samples must be rerun. Exceptions will be noted on corrective action forms and approved by management.

9.4 Gas Chromatography/Mass Spectrometry

- Volatile Organic Analysis
- Semivolatile Organic Analysis

Initial calibration is at least at a minimum 5 point standard curve of target compounds (concentrations are in detector linear range) performed on failure of two continuing calibration standards. The lowest calibration standard has a concentration that is equal to, or less than, the Reporting Limit. Method 624 requires a minimum of a 3 point standard curve.

Curves are checked by calculating the %RSD for the calibration check compounds (CCC). The system performance check compounds (SPCC) must meet method specific requirements.

Initial calibration and run sequence:

1. Run BFB or DFTPP. Achieve acceptance criteria based on Key Ion and Abundance Tables 9-2 and 9-3.
2. Initial calibration
3. ICV
4. Analyze samples (including blanks, LCS, spikes and duplicates) within the specified tune time for BFB or DFTPP.

Daily continuing calibration and run sequence:

1. Run BFB or DFTPP. Achieve acceptance criteria based on Key Ion and Abundance Tables 9-2 and 9-3.
2. CCV
3. Check to see if CCV meets criteria. If not, make necessary corrections in GC/MS system and run CCV again.
4. If standard passes, run samples (including blanks, LCS, spikes, and duplicates) for 12 hours from the time BFB or DFTPP was run.
5. At the end of the method specified tune time, repeat steps 1 - 5.

TABLE 9-2
KEY IONS AND ABUNDANCE CRITERIA FOR BFB

Mass	Ion Abundance Criteria
40	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	base peak - 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

TABLE 9-3
KEY IONS AND ABUNDANCE CRITERIA FOR DFTPP

Mass	Ion Abundance Criteria
51	30 to 60% of m/z 198
68	less than 2% of m/z 69
70	less than 2% of m/z 69
127	40 to 60% of m/z 198
197	less than 1% of m/z 198
198	base peak 100% relative abundance
199	5 to 9% of m/z 198
275	10 to 30% of m/z 198
365	greater than 1% of m/z 198
441	present, but less than m/z 443
442	greater than 40% of m/z 198
443	17 to 23% of m/z 442

9.5 HPLC - VWD/FD (PNA and Explosives)

The initial calibration is a 6 point standard curve of all target analytes. The lowest calibration standard has a concentration equal to or less than the Reporting Limit. The curves are checked by calculating the %RSD for all analytes. The %RSD must be ≤ 20%.

Initial Calibration and Run Sequence:

1. Initial Calibration
2. ICV
3. Analyze 10 samples
4. CCV
5. Continue steps 3 & 4 for entire run

Daily Continuing Calibration and Run Sequence:

1. CCV
2. Analyze 10 samples (includes reagent blanks, LCS, spikes and duplicates)
3. Analyze check standard
4. Continue with analysis steps 2 and 3 for entire run

Samples must be bracketed by passing CCVs. If the CCV does not meet criteria, all affected samples must be rerun. Exceptions will be noted on corrective action forms and approved by management.

9.6 IC - Conductivity Detector (Anions)

The initial calibration is a linear or quadratic regression based on six points for all seven analytes plus a blank. The initial calibration verification requires that the response not vary by more than ± 10%. Initial Calibration and Run Sequence:

1. Initial Calibration
2. ICV
3. Analyze 10 samples
4. Analyze one in every ten samples in duplicate
5. CCV
6. Continue steps 3, 4 & 5 for entire run

Daily Continuing Calibration and Run Sequence:

1. CCV - response must not vary more than 10%
2. Analyze 10 samples
3. Analyze one in every 10 samples in duplicate
4. CCV – response must not vary more than 5%
5. Continue steps 2, 3 and 4 for entire run

Samples must be bracketed by passing CCVs. If the CCV does not meet criteria, an initial calibration must be run and all affected samples rerun. Exceptions will be noted on corrective action forms and approved by management.

9.7 Atomic Absorption – Cold Vapor

Initial calibration is a minimum of four standards and a blank and must be within the linear part of the calibration curve. The lowest calibration standard has a concentration that is equal to the Reporting Detection Limit. Calculation is by computer software using linear regression.

Initial Calibration and Run Sequence:

1. Initial Calibration
2. ICV
3. ICB
4. CCV
5. CCB
6. Analyze 10 or less samples, including all batch related QA/QC (LCS, Method Blank, MS/MSD).
7. CCV
8. CCB
9. Continue running samples in groups of 10 or less followed by the CCV and CCB.

Samples must be bracketed by passing CCVs. If the CCV does not meet criteria, an initial calibration must be run and all affected samples must be rerun. Exceptions will be noted on corrective action forms and approved by management.

9.8 ICP-AES, MS

Initial calibration is a minimum of 3 standards and a blank. Calculation is by computer software using linear regression.

Initial Calibration and Run Sequence:

1. Initial calibration
2. ICV
3. ICB
4. Run Interference check samples
5. CCV
6. CCB
7. Analyze 10 or less samples, including all batch related QA/QC (LCS, Method Blank, MS/MSD)
8. CCV
9. CCB
10. Continue running samples in groups of 10 or less followed by the CCV and CCB

Samples must be bracketed by passing CCVs. If the CCV does not meet criteria, all affected samples must be rerun. Exceptions will be noted on corrective action forms and approved by management.

9.9 Support Equipment

9.9.1 pH Meter

The pH meter is recalibrated before each use or after 2 hours of use.

9.9.2 Turbidimeter

The Turbidimeter is calibrated before each use.

9.9.3 Thermometers

Each thermometer in use in the laboratory is identified with a unique I.D. number which is printed on an attached tag. When a thermometer is put into use, it is checked against an NIST certified thermometer and documented in the thermometer calibration logbook. Any variation from the certified thermometer is indicated on the I.D. tag. If no adjustment is necessary, it is indicated by writing “No Correction Necessary” or “NCN” in the book and “NCN” on the tag. If temperature correction is required, it is added to or subtracted from the actual thermometer reading. Microbac records only the corrected temperature. Each thermometer is rechecked annually against the certified thermometer using the procedures outlined in Microbac SOP K0002 and documented in the thermometer logbook.

9.9.4 Mechanical Pipets and Syringes

The laboratory will verify mechanical pipets and syringes employing the procedures found in Microbac SOP K0002. Mechanical pipets are verified daily and gas tight syringes are checked quarterly. The laboratory maintains electronic or hardcopy records of all checks of these devices.

9.9.5 Balances

The most important piece of equipment in any laboratory is the analytical balance. The accuracy of data related to weight-prepared standards can be no better than that of the analytical balance. For this reason, proper care and use of the analytical balance should be of highest priority. The following section describes the standard operating requirements at Microbac for the calibration and care of analytical balances:

- Analytical balances are mounted on a heavy, shock-proof table, constructed of either marble or concrete, and placed in an area of low traffic. The balance level is checked daily and adjusted when necessary.
- Balances must be protected from extreme temperature and humidity change. A beaker of silica gel desiccant must be placed in the balance and changed frequently.
- All spills on the pan or inside the balance must be avoided, and the balance must be kept scrupulously clean at all times.
- Balances will be checked and adjusted at least twice a year under service contract with a professional balance specialist.
- Balances must be checked prior to each day's use applying the criteria specified in Table 9-4. The weights selected must bracket the range of use.

Table 9-4
BALANCE CALIBRATION ACCEPTANCE CRITERIA

Balance Type	Weight (grams)	Criteria
Analytical (0.0001)	0.1	+/-0.0005
	0.2	+/-0.0005
	0.5	+/-0.0005
	1.0	+/-0.0005
	5.0	+/-0.001
	10.0	+/-0.001
	50.0	+/-0.005
	100.0	+/-0.005
Top Loading (0.001)	0.5	+/-0.02
	1.0	+/-0.02
	5.0	+/-0.05
	10.0	+/-0.05
	50.0	+/-0.05
	100.0	+/-0.1
	200.0	+/-0.1
Top Loading (0.01)	1.0	+/-0.05
	5.0	+/-0.05
	10.0	+/-0.05
	50.0	+/-0.05
	100.0	+/-0.1
	200.0	+/-0.1

9.10 Logbooks

Logbooks are kept for each analytical instruments, balances, refrigerators, and incubators in the laboratory. All logbooks are bound controlled documents with numbered pages, logbooks may also be in electronic form.

9.10.1 Balances

Each balance has a logbook to record daily calibrations checks. Maintenance and service records must be entered in the logbook. The daily calibration records must include the date, time, the initials of the operator, and the weights for the standard checks using the procedure described earlier. Each balance is checked with weights which are at or near the weight of samples routinely weighed on each balance. Acceptance criteria are outlined in Table 9-4.

9.10.2 Refrigerators

Each refrigerator has a logbook to record its daily temperature checks. Temperatures must be maintained at ≤ 6° C. Each refrigerator has a calibrated thermometer inserted in a container of polyethylene glycol. This thermometer

is read daily and more often if necessary. The temperature is recorded in the logbook to document the proper temperature and stability of each refrigerator.

9.10.3 Ovens and Incubators

Each oven and incubator has a logbook to record its daily operating temperatures. Each oven and incubator has a calibrated thermometer which is read during its operation. The operating temperatures are recorded in the logbook to document that the proper operating temperature has been utilized.

9.10.4 Instrument Logbooks

Each analytical instrument in the laboratory has a logbook. Daily operational notes, problems, routine maintenance procedures and repairs are kept in the instrument maintenance logbook. Calibrations, QC samples, and sample run information is documented in the instrument runlog.

10.0 PREVENTIVE MAINTENANCE

10.1 Routine Maintenance Activities

All of Microbac's instruments undergo routine maintenance, cleaning, and inspection on a daily, weekly, or monthly basis, according to the manufacturer's recommendation, and/or the requirements of the standard methods employed. Maintenance logs and instrument maintenance checklists are kept, noting problems and the steps taken to correct them. Records are kept on repairs requiring non-Microbac service repairmen. Table 10-1 provides a list of the preventive maintenance for all major laboratory instrumentation. Each instrument has an electronic log, which contains the records of any maintenance or repairs performed.

10.2 Contingency Plan

In the event of a major instrument failure, most of Microbac's instrumentation has a back-up. There are multiple volatile and semivolatile GC/MS's, as well as, multiple ECD's, ELCD's, FID's and PID's. In the inorganic laboratory there are multiple pH meters, spectrophotometers, an AAS and an ICP-MS (for low-level analyses) and ICP-AES's (3). This instrumentation is listed in Section 9.0, Table 9-1. In the event that no backup is available and the sample has holding time left, the sample will be held until repairs have been completed. If the sample is getting close to the end of its holding time, the sample will be subcontracted out to another certified laboratory, or a new sample will be

requested from the client. This decision will be left up to the client. Microbac, under no circumstance, will analyze samples out of holding time or subcontract to another laboratory, without client notification.

TABLE 10-1
LABORATORY INSTRUMENTATION PREVENTIVE MAINTENANCE

EQUIPMENT	ACTIVITY
Gas Chromatographs (GC) Semivolatile Organics	change septum change liners check carrier gas change carrier gas change in-line filters clip column replace ECD replenish Electrolytic Conductivity Detector Solvents change ion exchange resin replace nickel tubing bake out column at completion of sample batch run check system for gas leaks at each column change sylonize injection port liners every liner
Gas Chromatographs (GC) Volatile Organics	check gases change gases clip chromatographic column clean PID Lamp clean FID Lamp change Trap bake out column
Gas Chromatograph/ Mass Spectrometers (GC/MS) Semivolatile Organics	clean mass spectrometer source change septum change liners check carrier gas change carrier gas change in-line filters clip column replace nickel tubing bake out column check system for gas leaks sylonize injection port liners
Gas Chromatograph/ Mass Spectrometers (GC/MS) Volatile Organics	clean mass spectrometer check helium change helium change trap clip column change Purge and Trap ferrules bake out column

TABLE 10-1 (continued)
LABORATORY INSTRUMENTATION PREVENTIVE MAINTENANCE

EQUIPMENT	ACTIVITY
HPLC - VWD/FD	monitor system pressure change guard cartridges reverse column filter samples at 0.45 microns filter eluents at 0.20 microns if needed monitor eluent reservoir content
IC - Conductivity Detector	monitor system pressure monitor baseline detector output change guard column filter samples at 0.45 microns filter eluents at 0.20 micron if needed degas eluent monitor eluent volume monitor waste volume
CV-AAS(mercury)	change tubing check optics check gases check rinse change drying tube
Inductively Coupled Plasma (ICP)	clean nebulizer check torch check gases change tubing check optics
pH Meters	clean probe
TOC Meter	clean detector
Specific Conductivity Meters	clean probe check cell constant replenish/replace probe redetermine cell constant
DO Meter	clean probe check membrane change membrane
Analytical Balance	check pans and compartment check alignment and balance cleaning/calibration/service
Ovens	temperature monitoring
Refrigerators	temperature monitoring
Incubators	temperature monitoring
LC/MS/MS	monitor system pressure change guard cartridges filter samples at 0.45 microns monitor eluent reservoir content check curtain plate for residue buildup

11.0 QUALITY CONTROL CHECKS, ROUTINES TO ASSESS PRECISION, ACCURACY, AND CALCULATION OF METHOD DETECTION LIMIT

This section describes the basic techniques of laboratory quality control and quality assessment normally used to evaluate analytical proficiency. General concepts are defined and discussed as measurement tools for evaluation of data quality. Topics addressed include analytical methods, the concept of batch control, specific types of quality control samples, routines to assess accuracy and precision, statistical control, method detection limits and reporting limits, and internal and external performance evaluation samples. For additional information on related subjects, the reader is referred to Section 9.0 for instrument calibration procedures and to Section 13.0 for corrective action criteria.

11.1 Analytical Methods

An important element in Microbac's Quality Assurance/Quality Control (QA/QC) program is strict adherence to the referenced analytical methods. Whenever possible, Microbac will use only methods that are US-EPA approved and promulgated for analytical investigations. The quality control samples described in Section 11.3 are designed to meet requirements of SW-846, Chapter 1, as well as, the specific method QA/QC criteria. However if program or project A/QC requirements are more stringent, Microbac will comply with the specific project requirements or document the individual variances.

11.2 The Batch Concept

The basic unit for laboratory production and quality control is the **batch**. Samples are most effectively handled and processed in related groups. Examples of batches and their corresponding laboratory identifiers are discussed briefly below. The Sample Delivery Group (SDG) is a group of samples received in one shipment for a specific project and client and become a login batch. They are assigned a unique login number by the KOBRA LIMS at the time of login. The login number also is the unit for the **laboratory reporting batch.**

The **preparation batch** is a group of twenty or less samples of the same matrix type that are prepared for analysis by a unique analytical procedure (SOP). Examples include extraction batches for semivolatile organics and digestion of samples prior to analysis for metals. A preparation batch employs the same analses, reagents, equipment, method (SOP), clean up, and concentration procedures. Each preparation batch has associated with it the appropriate quality control samples required by the method and usually includes the

preparation blank (method blank), the laboratory control sample, matrix spike, sample duplicate or matrix spike duplicate. Each preparation batch is assigned a unique KOBRA LIMS tracking number referred to as **workgroup**.

The **analytical batch** refers to a group of samples analyzed by a specific analytical method, technique, or instrument, and is the analysis prior to data reduction and reporting. The analytical batch can be identical in sample makeup to the preparation batch, but can include parts of preparation batches, or multiple preparation batches. They include samples that are analyzed together within the same time period by the same analyst and/or instrument. They are always associated with the required method instrument quality control samples such as GC/MS tunes, instrument blanks, initial calibrations and/or continuing calibration verifications. A separate workgroup number is assigned to the analytical batch for the purposes of scheduling and tracking.

The **data entry batch** refers to a group of client and laboratory QC samples for which the analytical results are entered and verified in the KOBRA LIMS. A unique identifier referred to as the run ID is assigned to this data entry batch.

11.3 Batch Quality Control Samples

Each preparation and analytical batch is assigned a unique number by the KOBRA LIMS called a workgroup. For analyses such as wet chemical procedures and purge and trap methods for volatile organics, the preparation batch and analytical batch are one and the same. Other methods require a separate extraction or digestion prior to analysis by the instrument and separate preparation workgroups are applicable. The following analytical quality control samples will be associated with each workgroup if the control procedure is applicable to the analysis. Data tabulation and control charting procedures for these quality control samples are described in more detail in Section 11.7. Acceptance criteria and corrective action measures are presented in the specific laboratory SOP.

11.3.1 Method blank

A method blank (MB), or preparation blank (PB), is analyzed with each preparation batch as a check on analytical system contamination. A sample consisting of laboratory reagent water or analyte free laboratory sand is processed through the entire analytical method including all sample preparation procedures such as extraction, digestion, and filtration. As a quality control sample, the results are used in conjunction with other control data to validate overall system performance and data quality. Client samples are associated with the method blank by sharing a common preparation workgroup number.

11.3.2 Laboratory Control Sample

The LCS is used to assess the general performance of the analytical procedure. The LCS is a quality control sample, similar in composition to the method blank, spiked with the analytes of concern at a known concentration, and is processed through the entire analytical procedure. The purpose of these samples is to monitor analysis control and to assess the accuracy of the procedure in the absence of matrix interference. The results of the LCS are charted or tabulated and must meet the criteria established by the project data quality objectives or the laboratory derived statistical control limits. The LCS is normally used to verify system control, but evaluation should take into consideration several factors including the number of spiked analytes, their performance characteristics, and the severity of the non-compliance. NELAC requires that all certified analytes be included in the LCS over a two year period, including all EPA Appendix IX organics, pesticides and Aroclors. When the spike list is extensive, there exists a high probability of having a few analytes outside the acceptance limits. If the spiking concentrations are not specified in the project QAPP, an attempt is made to spike a level near the mid-range for the method. Client samples are associated with a specific LCS by sharing a common preparation workgroup number.

11.3.3 Laboratory Control Sample Duplicate

An Laboratory Control Sample Duplicate (LCSD) is prepared with the LCS for those batches that do not have another measure of precision, such as a sample duplicate, or matrix spike/ matrix spike duplicate (MS/MSD).The LCSD must meet the same acceptance requirements for accuracy (% recovery) as the LCS and the same precision requirements (RPD) as the sample duplicate (or MS/MSD).

11.3.4 Matrix Spikes

MS are analyzed with each batch of samples of a similar matrix. They consist of a field sample that is spiked with the same analytes as the LCS and normally at the same concentration. The MS is used to assess the performance of the method in client's sample matrix. The percent recoveries are tabulated in a summary report and the results are used to assess bias or other matrix effects. The MS analyses are not used to establish laboratory control, but can be used to properly qualify data.

If sufficient sample quantity is available, the laboratory will select samples for matrix spiking based on the following approach:

- All client specified MS, MSD or duplicate samples are logged into the KOBRA LIMS and reported to the client.

- If no client specified samples have been submitted, the laboratory will randomly select a sample for the MS/MSD or duplicates based on sample availability and client approval requirements.

- If there is insufficient sample volume to perform MS/MSD or duplicate analysis the laboratory will perform an LCS and LCS duplicate in order to provide a measure of precision and accuracy for the analytical batch.

11.3.5 Duplicates and Matrix Spike Duplicates

Duplicate samples (DUP) or MSD may be with each batch of samples of a similar matrix to monitor the method precision. The results in relative percent difference (RPD) are tabulated as outlined in Section 11.5. This data may be used to evaluate the precision of the method in real sample matrices. For analytical methods for which spiking is inappropriate, sample duplicates are used to assess precision.

11.3.6 Selecting Samples for MS/MSD/DUP (Batch QC)

The laboratory will implement one of the following procedures for selecting samples for duplicate analysis (DUP), MS, and MSD.

a) If client samples are specified for batch QC in the KOBRA LIMS or in the project QAPP, the analyst must select these sample(s).

b) If there are no client-specified samples, but extra sample aliquots are available for batch QC, the analyst will randomly select sample(s) for the MS/MSD or DUP. This option must not be used if it depletes all remaining sample. There must be enough field sample volume remaining to allow for re-analysis if necessary.

c) If there is insufficient volume of field samples available for DUP/MS/MSD and re-analysis, the analyst will include an LCS duplicate in the batch in lieu of MS, MSD or DUP.

11.4 Quality Assurance Summary Reports

Quality assurance summary reports are prepared for each analytical workgroup. Whenever samples are analyzed, the batch quality control sample results described in Section 11.3 are entered into the laboratory database.

Reports are generated by workgroup for review by department supervisors, QA/QC personnel, and general management. Copies of the quality assurance summary reports are provided to the client upon request. They are included in all laboratory reports designated as Level 2, or higher, as described in Section 12.0.

11.5 Measuring Precision, Accuracy and Method Detection Limits

This section discusses method performance parameters and how they are related to data quality objectives (DQO). Definitions are given for precision, accuracy, representativeness, completeness, comparability (PARCC), MDL and RL. Tables which provide current statistics for methods including MDLs, RLs, precision and accuracy are updated annually and are available in the individual method SOPs (listed in Appendix B). The following definitions that are used establish baseline data quality objectives for all analytical projects. The PARCC parameters and MDL/RL data are a useful template for development of SAP and QAPP for a wide scope of programs.

11.5.1 Precision (% RPD)

The laboratory objective is to equal or exceed the precision data generated by the applicable method validation studies on similar matrices. Relative percent difference (% RPD) as derived from duplicate matrix spike results (or duplicate sample results for metals) will be used to evaluate precision. With each analysis of a duplicate sample, or matrix spike/matrix spike duplicate, the RPD is calculated from the following formula:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2} \right] 100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

Method precision can be evaluated through the analysis of duplicate samples or matrix spike duplicates. For most inorganic methods, the results of duplicate analyses are used to calculate advisory limits, but the upper limit for the RPD is usually set at 20%. Another measure of precision is the method standard deviation. The standard deviation determined from the analysis of laboratory control samples can also be used to estimate the upper control limit for the RPD. The later procedure, which is taken from Method 1020B (Standard

Methods – 18th Edition), is used for organic methods having long lists of analytes. However, for most projects the advisory upper limit for the RPD of organic analytes is set at 40%.

11.5.2 Accuracy (% recovery)

The laboratory objective is to equal or exceed the accuracy data generated by the applicable method validation studies on similar matrices. Percent recovery, as derived from the analysis of LCS will be used to evaluate accuracy. The LCS contains all of the analytes of interest at the concentration of interest, either at the midrange of the method, or as specifically defined in the QAPP.

Accuracy statements (ranges) are derived from statistical analysis of the percent recoveries for the LCS for the applicable media. Criteria are established at three standard deviations from the mean LCS recoveries. The ranges for accuracy are generally used as control limits, however, projects DQO's, laboratory policies, or performance method requirements may dictate exceptions. Recovery is defined mathematically as follows:

For a laboratory control samples:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the measured concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

Calculating recovery for matrix spikes (MS/MSD):

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

Representativeness

Representativeness ensures that a set of data accurately depicts the distinguishing characteristics of the sample source. This is usually achieved through sample collection in accordance with statistically defined bounds of the population mean and variance, and by following existing sampling protocols.

Comparability

To achieve a performance level in terms of accuracy and precision for sample parameters in similar matrices, so that one set of data is comparable to another in terms of quality of measurement on a consistent basis. This assures that all Microbac analyses fall within an acceptable range.

Completeness

Refers to the percentage of measurements made which are judged to be valid. The completeness goals for most methods are 95% on all water analyses and 90% on all soil analyses.

11.6 Statistical Evaluation of Data

11.6.1 Statistical Methods

Microbac has established its statistical approach to quality control based on the following standards:

Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-60014-79-019, March 1979.

Standard Methods for the Examination of Water and Wastewater, 22nd Edition, APHA/AWWA/WEF, Methods 1010, 1020, 1030.

Test Methods for Evaluating Solid Waste - Physical/ Chemical Methods, SW-846, 3rd Edition, Chapter 1.

11.6.2 LCS Control Limits

The laboratory shall establish statistically derived control limits for laboratory control samples. The laboratory will evaluate the data from at least thirty (30) LCS data points to compute the mean and standard deviation(s).

The lower control limit (LCL) shall be established at the mean –3s

The upper control limit (UCL) shall be established at the mean +3s

11.6.3 MS/MSD Control Limits

Microbac will use the LCS control limits to evaluate MS/MSD data, unless the project QAPP specifies otherwise.

11.6.4 Annual Evaluation

The laboratory will also generate an annual LCS summary report from the KOBRA LIMS menu system. The summary report will display the individual LCS results in concentration and percent recovery, computing the mean, standard deviation, and limits at +/-3 sigma. This evaluation will occur concurrently with the annual SOP review. The supervisor and QAO will compare the statistics to the existing default control limits, to determine if these limits are supported by the new statistic. The laboratory will update the KOBRA LIMS QC tables and the appropriate SOP tables as necessary. The laboratory supervisory will review the data for method bias, trends, and shifts, and forward the data package to the QAO. The QAO will review, approve, and /or initiate investigations based on the data.

11.7 General Control Charting Procedure

The laboratory constructs control charts using percent recovery data from LCS. Control Charts are constructed for all inorganic analytes and a representative group of organic analytes from each method. In constructing an initial chart, twenty or more consecutive data points are necessary and the time required to generate such a chart will vary with the frequency of the analysis (daily, weekly, monthly, etc.). This data is charted against the control limits effective during the evaluation period. Control charts are used not only to monitor system control and to evaluate accuracy and precision, but also to check for general trends. Each analyst is trained to interpret and identify problems such as shifts, trends and biases. This method ensures continued evaluation of laboratory and personnel performance. An example Microbac Control Chart is presented in Figure 11-1 of this section.

11.8 Method Detection Limits

The MDL is defined as the minimum concentration of an analyte that can be determined with 99 percent confidence to be greater than zero. All MDL determinations provided by Microbac are made in accordance with Microbac SOP 45, which is derived from the US-EPA procedures outlined in 40 CFR part 136, Appendix B. MDL's are evaluated annually and/or verified quarterly.

Verification consists of analyzing a detectability check standard (DCS), which is a fortified blank spiked at a concentration near the concentration of the laboratory MDL and must not exceed the standard reporting limit. This check confirms that the laboratory MDL, which is derived from multiple instruments and analysts, is routinely achievable on a specific instrument and method over the course of time. In order to be considered acceptable, the MDL verification must give a measurable response that also meets the method's qualitative criteria, or is at least distinguishable from the response in a blank sample. The laboratory will perform an MDL verification immediately after each (initial) MDL study and quarterly thereafter. The laboratory will perform a verification if MDLs must be lowered for any reason.

The laboratory shall evaluate the acceptability of MDLs at the time of the SOP review for the method. This review shall include the status and acceptability of the MDL data for water and soil in the initial (EPA) studies and quarterly verification data, if applicable. Based on these reviews, the laboratory may choose to revise current MDLs, or determine the need for additional studies.

11.9 Quantitation Limits and Reporting Limits

Practical Quantitation Limits (PQL) and Limits of Quantitation (LOQ) are defined as the smallest concentration of analyte that can be reported to a known level of accuracy. The PQL is often defined as 3-10 times the MDL, but Microbac normally estimates the PQL as the concentration of the lowest standard in the initial calibration curve for the method. Laboratory RLs are often used in place of the PQL, since they are generally accepted by the environmental industry. They can be adjusted for specific projects requirements but are independent of instrument variation and statistics. At Microbac, the RL/LOQ is usually at least two times the laboratory observed MDL for that analyte, and is normally equivalent to the concentration of the lowest standard on the calibration curve. For some programs such as DoD, it is necessary to report both the MDL and the RL. The standard laboratory reporting limits may be found in the applicable SOP and the KOBRA LIMS.

11.10 Proficiency Testing Studies (PT)

Microbac currently participates in several government administered and private sector single blind PT studies. For each of these programs, it is our internal policy to evaluate each department's performance and prepare corrective action plans for all unacceptable results. All performance summaries and our corrective action plans are made available to clients and state agencies upon request. See also Section 14.2.

For certified methods for which there are not commercially available proficiency testing samples, Microbac OVD will rely on quality control checks for assuring the quality of testing results. The quality control checks include (but are not limited to) alternate source check standards or comparison of new standard lots to previous standard lots prior to use. These quality control checks samples must be analyzed at least twice per year.

For those accrediting bodies that require the submission of proficiency testing results and, associated CAPAs if necessary, the laboratory will submit as per the stated requirement.

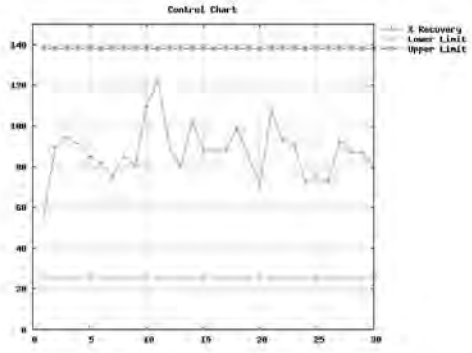
Uncontrolled Copy

Figure 11-1
CONTROL CHART

Control Chart #2013 — Method: 8081 — Matrix: SOLID
TOXAPHENE

Mean: 87.3 — Standard Deviation: 12.7 — Lower Limit: 49.3 — Upper Limit: 125

Seq#	Sample#	Analysis Date	% Recovery	Lower	Upper	Instrument
1	WG443664-04	2013-08-07 01:03:00	56.4	25.0	138	HP15
2	WG441172-03	2013-08-06 15:31:00	89.4	25.0	138	HP15
3	WG440930-03	2013-08-03 20:00:00	94.3	25.0	138	HP15
4	WG438548-03	2013-07-24 15:48:00	91.3	25.0	138	HP15
5	WG438455-03	2013-07-24 01:41:00	85.2	25.0	138	HP15
6	WG437566-03	2013-07-17 01:25:00	81.9	25.0	138	HP15
7	WG437017-03	2013-07-13 07:15:00	75.4	25.0	138	HP15
8	WG436404-06	2013-07-12 17:22:00	84.6	25.0	138	HP15
9	WG436671-03	2013-07-12 05:18:00	80.5	25.0	138	HP15
10	WG436245-06	2013-07-11 15:24:00	110	25.0	138	HP15
11	WG436075-06	2013-07-10 04:34:00	123	25.0	138	HP15
12	WG435329-03	2013-06-27 14:31:00	89.2	25.0	138	HP15
13	WG433744-04	2013-06-14 04:23:00	80.3	25.0	138	HP15
14	WG433976-04	2013-06-11 09:41:00	102	25.0	138	HP15
15	WG432775-06	2013-06-07 05:35:00	88.0	25.0	138	HP15
16	WG432253-03	2013-06-05 20:54:00	87.2	25.0	138	HP15
17	WG432236-03	2013-06-04 22:39:00	86.2	25.0	138	HP15
18	WG432035-03	2013-06-04 17:05:00	96.4	25.0	138	HP15
19	WG431693-04	2013-05-28 14:31:00	85.1	25.0	138	HP15
20	WG431611-06	2013-05-22 15:24:00	71.3	25.0	138	HP15
21	WG430132-03	2013-05-16 19:07:00	107	25.0	138	HP15
22	WG429664-03	2013-05-13 18:12:00	93.1	25.0	138	HP15
23	WG426459-06	2013-04-15 17:44:00	91.2	25.0	138	HP15
24	WG426163-04	2013-04-11 20:15:00	72.4	25.0	138	HP15
25	WG423027-04	2013-03-12 11:55:00	74.9	25.0	138	HP15
26	WG422251-04	2013-03-04 17:28:00	72.8	25.0	138	HP15
27	WG420493-03	2013-02-13 18:38:00	82.5	25.0	138	HP15
28	WG420226-03	2013-02-11 19:22:00	87.1	25.0	138	HP15
29	WG419425-04	2013-01-30 14:24:00	87.5	25.0	138	HP15
30	WG417804-04	2013-01-04 14:32:00	79.3	25.0	138	HP15



12.0 DATA REDUCTION, REVIEW, VERIFICATION, AND REPORTING

This section describes the process of data reduction, data review, data validation, data entry, and editing that begins with the raw data generated at the bench level in the laboratory and culminates with a useful collection of information in the form of summary laboratory reports and data deliverables packages.

12.1 Data Reduction

12.1.1 Data reduction is the conversion of raw data from instrument readings to reportable results. The raw data is converted into reportable values by instrument hardware and software or by other manual procedures suggested in the required reference method.

12.1.2 Policy on Significant Figures

The following policies relating to the use of significant figures for reporting analytical data apply in general to all departments and methods except as noted in the individual method SOPs. All analytical results which are taken from bench sheets, instrument quantitation reports, or other raw data shall be rounded to three (3) significant figures as per Section 12.1.3 and entered without adjustment into the KOBRA LIMS. The KOBRA LIMS shall be programmed to generate the final analytical report with the appropriate number of significant figures based on the project requirements.

- All results for performance evaluation samples will be reported with three (3) significant figures using the rounding rules specified in Section 12.1.3.
- All quality control sample results will be reported with three (3) significant figures.
- All results for surrogate compound recovery will be reported with three (3) significant figures.
- All results for client (real world) samples will be reported with three (3) significant figures (except as noted below).
- Project specific requirements may override these general guidelines and the KOBRA LIMS products and data entry screens will be modified accordingly.

12.1.3 Rules for Rounding

For rounding off numbers to the appropriate level of precision (significant figures), the laboratory staff shall follow these rules:

- If the figure following the digit to be retained is less than five (5), drop it (round down).
- If the figure following the digit to be rounded is greater or equal to five, round up.

Examples:

Three Significant Figures:	Two Significant Figures:	One Significant Figure:
6.014 rounds to 6.01	0.0511 rounds to 0.051	0.015 rounds to 0.02
6.016 rounds to 6.02	0.0519 rounds to 0.052	0.016 rounds to 0.02
6.015 rounds to 6.02	0.0515 rounds to 0.052	1.9 rounds to 2
		25 rounds to 30
		26 rounds to 30
		24 rounds to 20

12.2 Review of Data and Deliverables

The laboratory has established policies and procedures for internal review of data and deliverables (reports) to assure the quality of reported environmental/analytical data.

This is a multi-tiered process involving the sample receiving staff, a detailed analytical review by the primary analyst, a secondary technical review by the department supervisor, or a qualified peer analyst, and an administrative review for completeness by a member the client services staff. These reviews utilize a combination of manual and electronic tools. In addition, the quality manager, or qualified designee, shall perform reviews (audits) on 10 percent of the final laboratory (reports).

Detailed procedures for review of data and deliverables is presented in SOP GP-DATA-REVIEW.

12.3 Data Entry and Verification

The OVD LIMS has been programmed to retrieve data directly from instrumentation using a datafile uploading process. Other data must be manually entered by a data specialist or technician. Data verification is a check on the data that is entered into the KOBRA LIMS. Data is entered by the analyst or a clerical staff assistant and is checked for accuracy by the department supervisor or a designated peer reviewer. Incorrect entries are immediately corrected. EDDs are generated by either the client's representative or Information Technologies (IT) personnel. Electronic files are verified by either the IT staff or the client's representative before being sent to the client by mail, or electronically.

12.4 Report Content and Levels

Microbac provides four levels of laboratory reports in order to match the documentation level required by specific projects. A summary of the four levels of data reporting follows:

Level 1 - Standard Laboratory Report

- Cover page with signature
- Results of analysis
- Reporting units
- Reporting/detection limits
- Analysts and analysis dates
- Method references
- Chain-of-custody form
- Surrogate recovery

Level 2 - Standard Report with QA/QC Summary of method blanks, LCS, MS/MSD, and duplicate results.

Level 3 - A Level 2 report with additional summary forms and no raw data.

Level 4 – CLP - Like report with summary forms and raw data.

Reporting level requirements are logged into the KOBRA LIMS system with the sample set. Level 1 and 2 data packages are assembled upon printout of the final report by the data entry staff. Copies of the raw analytical data for level and data packages are turned in by the analysts upon completion of analysis. Data summary forms are printed from specialized software packages by the analysts in each laboratory. For a list of standard Microbac data qualifiers see Figure 12-1. Customized “Level – X” reports are available for special projects.

12.5 Data Integrity, Storage and Archive

Insuring data integrity is one of Microbac’s main objectives. QA controls, as well as, Good Laboratory Practice are instituted by Microbac to achieve this objective. In the daily flow of data, laboratory personnel initial and date any corrections introduced into the data. Unused areas of the daily bench sheets and instrument logs are crossed out, initialed and dated by the corresponding analyst or technician. Controlled benchsheets and log books are required to maintain data integrity. Each sample delivery group is assigned a unique login number when initially logged into the KOBRA LIMS system. The report master files are stored in order of the login number and can be easily retrieved using

this system. Each login is scanned electronically and stored in the Masterfile up to 18 months then removed to CD-ROM, Blu-Ray, and tape archive storage for a period of five to ten years depending on contractual agreements. OVAP requires notification prior to disposal of any records.

Computer records from the Microbac KOBRA LIMS are maintained on the system up to one year and are then archived on magnetic tape. Two backup copies of the system are maintained on magnetic tape at all times. The backup and archive tapes are stored in a fireproof safe at the laboratory indefinitely. Extraction lab logbooks and bench sheets are maintained chronologically and stored in the laboratory for five years. Inorganics raw data, including analyst notebooks, bench sheets, computer printouts and instrument logs, are stored by parameter in the individual laboratories or in the archive area for a period of five years. GC data is stored as hardcopy in the individual master files. GC/MS data is stored as hardcopy for three-five years in the workorder files and on magnetic tape for at least one year. GC/MS Data stored on magnetic tape can be retrieved by the file numbers assigned to them at the time of analysis.

Figure 12-1

Microbac Laboratories Inc.
List of Valid Qualifiers
February 11, 2014

Qualkey: STD

Qualifier	Description
*	Surrogate or spike compound out of range
+	Correlation coefficient for the MSA is less than 0.995
<	Result is less than the associated numerical value.
>	Result is greater than the associated numerical value.
A	See the report narrative
B	Analyte present in method blank
B1	Target analyte detected in method blank at or above the method reporting limit
B3	Target analyte detected in calibration blank at or above the method reporting limit
B4	The BOD unseeded dilution water blank exceeded 0.2 mg/L
C	Confirmed by GC/MS
CG	Confluent growth
CT1	The cooler temperature at receipt exceeded regulatory guidelines for requested testing.
DL	Surrogate or spike compound was diluted out
E	Estimated concentration due to sample matrix interference
EDL	Elevated sample reporting limits, presence of non-target analytes
EMPC	Estimated Maximum Possible Concentration
F, S	Estimated result below quantitation limit; method of standard additions(MSA)
F,CT1	Estimated value; the analyte concentration was less than the RL/LOQ. The cooler temperature at receipt exceeded regula
FL	Free Liquid
H1	Sample analysis performed past holding time.
I	Semiquantitative result (out of instrument calibration range)
J	Estimated value; the analyte concentration was less than the RL/LOQ.
J,B	Analyte detected in both the method blank and sample above the MDL.
J,CT1	Estimated value; the analyte concentration was less than the RL/LOQ.
J,CT1	Estimated value; the analyte concentration was less than the RL/LOQ. The cooler temperature at receipt exceeded regula
J,P	Estimate; columns don't agree to within 40%
J,S	Estimated concentration; analyzed by method of standard addition (MSA)
L	Sample reporting limits elevated due to matrix interference
L1	The associated blank spike (LCS) recovery was above the laboratory acceptance limits.
L2	The associated blank spike (LCS) recovery was below the laboratory acceptance limits.
M	Matrix effect; the concentration is an estimate due to matrix effect.
N	Tentatively identified compound(TIC)
NA	Not applicable
ND	Not detected at or above the reporting limit (RL/MDL).
ND, CT1	Analyte was not detected. The concentration is below the reported LOD. The cooler temperature at receipt exceeded reg
ND, H1	Not detected; Sample analysis performed past holding time.
ND, L	Not detected; sample reporting limit (RL) elevated due to interference
ND, S	Not detected; analyzed by method of standard addition (MSA)
NF	Not found by library search
NFL	No free liquid
NI	Non-ignitable
NR	Analyte is not required to be analyzed
NS	Not spiked
P	Concentrations >40% difference between the two GC columns
Q	One or more quality control criteria failed. See narrative.
QNS	Quantity of sample not sufficient to perform analysis
RA	Reanalysis confirms reported results
RE	Reanalysis confirms sample matrix interference
S	Analyzed by method of standard addition (MSA)
SMI	Sample matrix interference on surrogate
SP	Reported results are for spike compounds only
TIC	Library Search Compound
TNTC	Too numerous to count
U	Analyte was not detected. The concentration is below the reported MDL.
UJ	Undetected; the MDL and RL are estimated due to quality control discrepancies.
UQ	Undetected; the analyte was analyzed for, but not detected.
W	Post-digestion spike for furnace AA out of control limits
X	Exceeds regulatory limit
X, S	Exceeds regulatory limit; method of standard additions (MSA)
Z	Cannot be resolved from isomer - see below

13.0 CONTROL OF NONCONFORMING WORK

13.1 Nonconforming Work

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred or when any aspect of testing, result generation or reporting does not conform to established procedures or contract requirements of the customer. The procedures detailed in Microbac SOP GP-RCA, "Root Cause Analysis" and Microbac SOP GP-CAPA, "Corrective Action and Preventive Action: Initiating, Tracking and Monitoring", are to be followed when such occurs. The department supervisor and/or the analyst has the ultimate authority and responsibility for the management of nonconforming work and the appropriate actions to be taken. The department supervisor, analyst and/or the QA department have the authority, under their scope of responsibilities, to initiate the necessary procedure; the procedure insures that:

- An evaluation of the significance of the nonconforming work is made.
- Corrective actions are taken immediately, as necessary
- When necessary, the customer is notified and the work is recalled.

Where there is doubt about the compliance of laboratory operations with policies or procedures, with the ISO17025:2005 standard or determination is made that nonconforming work could recur, the QA department will audit the appropriate areas of activity as discussed in Microbac SOP GP-INTAUDIT, Internal Audit Guidelines and the procedures described in Microbac SOP GP-CAPA, "Corrective Action and Preventive Action: Initiating, Tracking and Monitoring" are to be followed.

13.2 Corrective Action

Corrective action may be required as a result of both analytical and non-analytical events. The purpose of the corrective action is not only to provide documentation of the event, but also to correct and prevent recurrence of the nonconformance or departure from the policies and procedures.

The procedures in Microbac SOP GP-RCA, Root Cause Analysis and Microbac SOP GP-CAPA, Corrective Action and Preventative Action: Initiating, Tracking and Monitoring detail and insure the following:

- An investigation is made to determine the root cause(s) of the problem.
- Potential corrective actions are identified and subsequently the most appropriate are implemented.

- Corrective actions are chosen appropriate to the magnitude of the problem.
- The entire process is properly documented.
- Follow-up is made to monitor and determine the effectiveness.

13.3 Preventive Action

Preventive action is the proactive identification of potential problems or areas for improvement. No nonconforming event needs to take place in order to stimulate a preventive action. This practice applies to the quality management system. Needed improvements and potential sources of nonconformance, whether administrative, technical or quality related are identified, selected, implemented, monitored and documented following Microbac SOP GP-CAPA, "Corrective Action and Preventive Action: Initiating, Tracking and Monitoring". Effectiveness of these actions is evaluated during the management review process.

13.4 General Analytical Requirements

Analysts have the primary responsibility for assessing method compliance and evaluating the acceptability of the batch quality control samples. Laboratory criteria that must be met and the resultant corrections are addressed in detail in the individual SOPs. An example of the contents is outlined in Tables 13-1 of the analytical SOPs. Some methods have additional control criteria such as surrogates, ICVs, CCVs etc. A departure occurs if a blank, calibration standard, laboratory control sample, sample replicate, or spike recovery analyses fail to meet the quality control criteria outlined in the SOPs. An investigation to find the cause of the problem is undertaken by the analyst and department supervisor and a nonconforming work report (Figure 13.1) is initiated. This form is used to document the problem, its resolution and to provide the means to inform management (including the QAO) of chronic quality problems and non-compliant systems. Corrective action must be approved by the QAO who will ensure implementation and documentation of the corrective action. More specific discussion of the various laboratory corrections due to nonconformance are discussed in the sections that follow. The special requirements of the OVAP are presented in appropriate section of OVAP SOPs.

13.5 Calibration Requirements

Instrument calibration is fundamental to all quantitative chemical analysis. Each US-EPA or other reference analytical method has specified calibration criteria that must be met in order for the data to be fully useable. Proper initial calibration must be performed in accordance with the method specifications for number, source and level of calibration standards, as well as, meet the linearity requirements. Each initial calibration must be verified at the specified

frequencies and meet acceptance limits. Repeated failure of CCV standards necessitates that the analysis be stopped, the instrument evaluated, and the initial calibrations repeated. Data for problematic analytes that do not pass the method specification must be qualified appropriately in the final laboratory summary reports and narratives.

13.6 Method Blanks

Method blanks are used to evaluate the analytical system for contamination. For most analytes the upper limit for blank contamination is the reporting limit, however, it is our policy to evaluate the source of any contamination greater than two times the MDL.

The criteria used to determine the acceptability of method blanks varies with the particular analyte. Factors such as the level of analyte in the sample and the existence of established regulatory limits, or action limits, must be taken into consideration. No correction other than documentation is required when the levels of analyte in the samples are at least ten times the level in the blank. Laboratory action involving blank contamination would include an investigation documented by a nonconformance report and one of the following actions:

- re-preparation and re-analysis of the affected samples
- B-flagging of the data
- documentation of the problem in the report narrative along with the effects on data quality, if any

13.7 Laboratory Control Samples

The data from the LCS is crucial to assessing the usability of the data from samples in the same batch. Normally, the LCS is approached from the stand point of pass/fail. If the LCS is out-of-control, the samples in the associated batch are re-analyzed, starting from the preparation step. This is the standard laboratory policy for single analyte methods such as wet chemistry or metals. For multi-analyte methods such as volatile and semi-volatile organics, both the number and severity of LCS outliers must be evaluated. A certain amount of professional judgment is required of the laboratory management as to what constitutes an out-of-control LCS. If the number of failing analytes is excessive (i.e. > 10% the analytes spiked), indicating a general system failure, then correction shall include the re-preparation and re-analysis of the entire batch. When the outliers are out high, there is no correction required if the samples do not contain reportable levels of these analytes. Furthermore, if the analytes are out low, but only marginally, this also might not indicate a method problem. The results of the LCS are included in the QC Summary Report and all outliers are

appropriately identified. The report narrative will discuss any problems with non-compliant LCS data, and the proper flags used to qualify the analytical results.

13.8 Matrix Spikes and Duplicates

Evaluating the data from MS, MSD, and duplicate samples is more complex than for method blanks and LCSs. In general, Microbac does not base batch control on the results of these analyses due to matrix effects and the issues of heterogeneity and sampling. The control limits for the LCS will be used to assess the acceptability of the MS/MSD data for the purposes of data flagging only.

13.9 Nonconformance Reports

Nonconformance reports (NCR) are used to document out of control events which consists of but are not limited to the non-fulfillment of a requirement of a customer, quality management system or regulatory agency (Figure 13.1). This report can be generated by any employee when nonconformances are identified. The form describes the event, correction and signifies a return to control if achieved. Further action is required if there is not a return to control and/or the out of control event is recurring. Further actions include but are not limited to corrective action procedures.

13.10 Corrective Action/Preventive Action Reports

Corrective Action/Preventive Action Reports (CAPA's) are used to document nonconformances requiring corrective action, as well as, actions designed to prevent initial occurrences of a nonconformance (Figure 13.2). These reports are typically generated by the analyst or individual who has encountered the nonconforming or potential nonconforming situation. This individual would be responsible for generating the CAPA form by describing the nonconformance issue and recommending or describing the correction taken. After the correction is performed the root cause is determined and the solution(s) implemented, the CAPA form is reviewed and signed by the department supervisor, a member of the QA department and the laboratory managing director. Post closure monitoring is performed and recorded in the monthly corrective action spreadsheet.

Figure 13.1

MICROBAC®

Ohio Valley Division

NONCONFORMANCE REPORT

Date: _____ **Department:** _____ **Initiator:** _____

Prep WG# _____ **Preparation Date:** _____ **Parameter:** _____

I. Description of Nonconformance:

Sample ID(s) _____

_____

☐ Method Blank _____

☐ LCS Failures _____ analytes out _____

☐ Other _____

_____

_____

II. Correction:

☐ Reprep affected samples only _____

☐ Reprep entire workgroup _____

☐ Reanalyze affected samples only _____

☐ Report data with qualifiers _____

☐ Other _____

_____

III. Return to Control?: Not Applicable _____ Yes _____ No _____

Control Verification _____

IV. Further Action Required?: (required if No is checked in III.)

_____

_____

_____

Approvals:

Department Supervisor: _____ Date: _____

QA Department: _____ Date: _____

Last Revised: 08/07/2013 Document Control Form #454

Figure 13.2

Ohio Valley Division

MICROBAC®

CORRECTIVE ACTION/PREVENTIVE ACTION REPORT

Date:_____ **Department:**_____ **Initiator:**_____

I. Description of Nonconformance:

_____

_____

_____

_____

II. Correction (CAR only):

_____

_____

III. Root Cause Analysis Results (optional for PAR):

_____

_____

IV. Action to be Taken:

_____

_____

_____

_____

_____

_____

V. Describe evidence of effectiveness:

_____

_____

_____

_____

_____

Approvals:

Department Supervisor: _____ Date: _____

QA Department: _____ Date: _____

Director/Manager: _____ Date: _____

CAPA 3850

Last revised: 08-07-2013

Document Control Form #455

14.0 PERFORMANCE AND SYSTEMS AUDITS

14.1 External Audits

Microbac actively participates in several Federal and State performance evaluation testing programs and system audits. External systems audits of the Microbac facility occur on a regular basis. These include audits by the West Virginia Department of Natural Resources (WVDNR), the OEPA, and the Florida Department of Health (FLDOH) (NELAC Primary Accreditor). Additionally, audits are conducted every 2 - 3 years by the California Department of Health Services, and the US Army Corps of Engineers.

14.2 Performance Audits and Proficiency Testing

Performance audits are required for numerous state and federal programs. These certifications include the submittal of single blind analytical proficiency testing (PT) samples from NIST certified providers. Microbac formally participates semiannually in the following performance evaluation programs:

- NELAC/WP/DMRQA
- NELAC Soil
- NELAC UST

If problems are identified in any of these programs, the QAO will submit internal PT samples to verify the effectiveness of our corrective actions.

14.3 Internal Audits

The QAO will perform an annual internal audit of the laboratory operations to assure continued adherence to the LQAP, including the referenced methods and SOPs. The QAO may conduct the audit over the course of the year, with assistance from other technical directors or supervisors. The results of the audit shall be documented and the results are presented in a report to the Managing Director. The QAO may perform additional internal audits based on results of internal and external performance feedback. If the laboratory receives a customer complaint, an audit will be used to investigate the appropriate areas. The laboratory will perform corrective actions and notify the client(s) within three business days if any data are affected by findings resulting from an internal or external audit. The laboratory shall have thirty (30) days to close out any internal audit finding.

The procedures for conducting the internal audit are presented in Microbac SOP GP-INTAUDIT

14.4 Management Review Procedures (Managing Director)

Senior Management annually conducts a Quality Systems Review. Each system is reviewed for client responsiveness, overall efficiency, value towards quality and economic value. Human resources and equipment needs are evaluated. With the summation of the review, changes are made (corrective action) as appropriate, equipment is purchased and overall business strategy is adjusted. The procedures for conducting the management reviews in accordance with ISO 17025:2005, Section 4.15.1 are presented in Microbac SOP GP-QMSR. This review includes but is not limited to:

- Laboratory Quality System
- Report Generation/Content (Hard Copy & EDD)
- Laboratory Data QA/QC
- KOBRA LIMS development and improvement
- Sales/Marketing Function/System
- Proposal/Quotation generation
- Project Management/Technical Service
- Production efficiencies
- Volume of work
- Internal audit reports
- Performance evaluation studies
- Client satisfaction
- Client feedback

15.0 QUALITY ASSURANCE REPORTS

The QAO and other laboratory technical directors perform an annual review of the quality system. This review includes all of the following elements:

- Results of internal audits
- Results of external audits
- Results of Proficiency Testing (PT) studies
- Summary of review of Corrective Action Reports
- Reports and deliverables
- Electronic Data Deliverables (EDD)
- Annual revision of the Quality Assurance Manual

15.1 Laboratory Quality Assurance Reports

The QAO prepares a monthly report for the Managing Director and to Microbac's corporate Director of Quality Improvement. The report summarizes laboratory activities for audits, PT studies, corrective action closure, certification changes, training, customer feedback, and other significant events.

The QAO also presents a written annual report to the Managing Director that summarizes the annual system review activities. Any significant quality assurance or quality control problems along with the recommended solutions for the problems will be listed.

15.2 Quality System Review Report

The Quality System Review is conducted annually (see Section 14.4) and a report of this review is issued to the president of the company. This report is included in the Annual Business Plan with the recommendations, observations, and adjustments incorporated in the business plan/strategy for the following year.

15.3 Special Reports

The QAO shall prepare special quality assurance reports as necessary to document special concerns and problems. These reports may indicate the discussion of chronic PT performance problems, evaluation of Corrective Action Reports, results of storage blanks, follow-up from internal and external audits, customer complaints, and any trends determined from periodic evaluation of statistical data.

16.0 SPECIAL PROGRAM REQUIREMENTS

This section identifies the specific requirements of various state and federal programs that have requirements that go beyond what is outlined in this quality manual and the laboratory policies and procedures (SOPs) referenced in Appendix B. It is not the intent to list all of the QA/QC requirements of these programs, since these documents are incorporated by reference. Only the specifications that are more stringent than our standard protocols will be identified.

16.1 Department of Defense

Microbac performs analyses for projects that must comply with the DoD's Quality Systems Manual (QSM). The QSM is based on the NELAC standards,

but has special requirements that are identified in the DoD "gray boxes". The appendices to the QSM summarize the QA/QC requirements for several common methods and list special surrogate and LCS control limits. To assure compliance with the DoD's QSM, Microbac has implemented the following special procedures.

16.1.1 QAPP and Control Limits

The DoD QSM has special QA/QC acceptance limits for the LCS, MS/MSD, and surrogates. The KOBRA project set up specifications shall identify the QAPP and QC-Key i.e. DOD3. The KOBRA QC Table is populated with the correct LCS/surrogate acceptance limits for DoD3. These steps will assure that the correct control limits are used for all QSM projects.

16.1.2 Internal Standards

Methods 8260 and 8270 must compare the internal standard (IS) area to the mid-point of the ICAL curve. Use KOBRA project set-up to indicate this requirement.

16.1.3 Soil Sub-sampling – Method 8330B

Microbac will follow these procedures for drying, sieving, grinding, and multi-increment sub-sampling of soil samples in preparation for SW-846 method 8330B. The laboratory obtained additional guidance for these procedures from "DoD" Guide for Implementing EPA SW-846 Method 8330B", July 7, 2008.
Sample Drying

Remove the entire contents of the 8330 sample container and spread the soil evenly in a shallow pan lined with aluminum foil. Remove all large pebbles, sticks, and other solids too large to pass through a 2 mm screen. Air-dry the soil at room temperature in the soil drying room or other low traffic area of the laboratory.

Sieving Procedure

Gently crush the air-dry soil using a large spoon or glass rod and transfer to a clean 2 mm sieve. Stir, shake, and mix the sample and allow it to pass through the sieve, and collect in a foil-lined pan. Do not use excessive force in this step, as this may damage the screens. The sample is now the portion that passes the 2 mm screen.

Grinding Procedure

Transfer the sieved sample to a mill grinder or a mortar and pestle. Grind entire sample reducing the particle size to 75 micron, or less.

Sub-sampling

Transfer the entire ground sample to a clean shallow pan, spreading the soil to thickness of 1 – 2 cm. Using a small spatula, randomly remove at least thirty increments of approximately 0.33 g each, giving a representative sub-sample of 10 g. Record the weight of the subsample to the nearest 0.01 g. This is the sample aliquot to be taken for extraction via ultrasonic bath.

Triplicate Analysis and MS/MSD (Optional)

For each sample delivery batch of 1-20 samples, process the client-specified sample(s) in triplicate by processing three discrete 10 g sub-samples as per Section 7.4. Individual projects may vary the rate of samples processed in triplicate. Repeat the sub-sampling procedure two more times for each sample specified for MS/MSD.

Grinding Blanks

A grinding blank with each batch of samples will be used to verify the effectiveness of the cleaning procedures. Some projects may require grinding blanks after each sample. The project QAPP will specify the frequency of these blanks, and they will be logged into the LIMS accordingly. The grinding blank(s) must meet the same criteria as the 8330B method blank (no target analytes > one-half the RL).

Grinder Cleaning Procedures

Use the following procedures to clean the mortar and pestle and coffee grinder(s) between samples:

1. Remove bowl/blade from base of grinder
2. Immerse bowl/blade in soapy water and remove all visible soil residue
3. Rinse w/ tap water
4. Rinse w/ DI Water
5. Wipe with paper towel and air dry

16.1.4 Soil Sub-sampling – Metals

Microbac will use the following procedures for the drying, sieving, grinding, and sub-sampling of soil samples in preparation for metals digestion by SW-846 methods 3050 or 3051. This procedure may be extended to other methods if specified in the project QAPP.

Sample Drying

Remove the entire contents of the metals sample container and spread the soil evenly in a shallow pan lined with wax paper. Remove all large pebbles, sticks, and other solids too large to pass through a 2 mm screen. Air-dry the soil room temperature in a secure, low traffic area of the laboratory.

Sieving Procedure

Gently crush the air-dry soil using a large plastic, wood or glass rod and transfer to a clean 2 mm sieve. Stir, shake, and mix the sample and allow it to pass through the sieve, and collect in a foil-lined pan. Do not use excessive force in this step, as this may damage the screens. The sample is now the portion that passes the 2 mm screen.

Grinding Procedure

Transfer the sieved sample to a mortar and pestle. Grind entire sample reducing the particle size to 75 micron, or less.

Sub-sampling

Transfer the entire sample to a clean shallow pan, spreading the soil to thickness of 0.3-0.5 cm. Using a rectangular micro-spatula, remove representative cross-section(s) of the soil giving a sample in the range of 1.0-2.0 g. Record the weight of the sub-sample to the nearest 0.01 g. This is the aliquot to be taken for microwave digestion using SW-846 method 3051A.

16.2 Air Force Center for Environmental Excellence (AFCEE)

Microbac performs project work that must meet the requirements of various documents prepared by the AFCEE. We have provided analytical support to prime contractors under QAPP versions 1.0 – 4.0.02. Each version has special QA/QC requirements, the details of which are identified in the specific QAPP reference. To assure compliance with the AFCEE QAPPs, Microbac has implemented the following special procedures.

16.2.1 QAPP and Control Limits

The AFCEE QAPPs have special QA/QC acceptance limits for the LCS, MS/MSD, and surrogates. The KOBRA project set up specifications shall identify the QAPP and QC-Key, i.e: AF40, AF31, etc.. The KOBRA QC Table is populated with the correct LCS/surrogate acceptance limits for each AFCEE QAPP These steps will assure that the correct control limits are used for all AFCEE projects.

16.2.2 AFCEE Reporting Limits

AFCEE QAPPs specifies that the MDL must be no greater than one-half the project's RL. The KOBRA project review system shall assure use of the special reporting lists created just for AFCEE projects (pkeys). Microbac has/will adjust the RLs accordingly to comply with the projects requirement, but in some cases it may be necessary to repeat MDL studies, if a lower RL is required. Microbac will get client approval of all reporting limits before proceeding with an AFCEE project.

17.0 REFERENCES

17.1 NELAC Standards, June 2003.

17.2 Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, Washington, DC 20460.

17.3 RCRA QAPP Instructions, U.S. EPA Region 5, Revision: April 1998.

17.4 ASTM D-5283-92. Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation.

17.5 "American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)", 1994.

17.6 EPA 2185 – Good Automated Laboratory Practices, 1995.

17.7 ANSI/ISO/IEC 17025 - 2005 General requirements for the competence of calibration and testing laboratories.

17.8 QA/R-2: EPA Requirements for Quality Management Plans, August 1994.

17.9 QA/G-4: Guidance for the Data Quality Objectives Process EPA/600/R-96/055, September, 1994.

17.10 QA/R-5: EPA Requirements for Quality Assurance Project Plans Draft – November 1997.

17.11 QA/G-5: Guidance on Quality Assurance Project Plans EPA/600/R-98/018, February, 1998.

17.12 QA/G-6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November, 1995.

17.13 QA/G-9: Guidance for the Data Quality Assessment: Practical Methods for Data Analysis EPA/600/R-96/084, January, 1998.

17.14 Manual for the Certification of Laboratories Analyzing Drinking Water EPA/570/9-90/008.

17.15 TNI Standards 2009 Environmental Laboratory Sector

17.16 DoD Quality Systems Manual for Environmental Laboratories Version 4.2

APPENDIX A
DEFINITIONS

The following definitions are used in the production of this Comprehensive Quality Assurance Plan.

Analytical Batch:

The basic unit for analytical quality control. The analytical batch is defined as samples which are analyzed (or sampled together) with the same method sequence, the same lots of reagents, and with the same treatment common to all samples. The samples should have been analyzed (or collected) within the same specified time period or in continuous sequential time periods. Samples in each batch should be of similar composition.

Audits:

A systematic check to determine the quality of operation of some function or activity.

1. **Performance Audits:** Quantitative data are independently obtained for comparison with routinely obtained data in a measurement system. Examples of these audits are EPA performance evaluation programs, commercial performance evaluation programs, split sampling programs involving at least two laboratories, and blind spike samples.

2. **Systems Audit:** These are qualitative in nature and consist of an on-site review and evaluation of a laboratory or field operation quality assurance system and physical facilities for sampling, calibration and measurements.

Calibration:

Process by which the correlation between instrument response and actual value of a measured parameter is determined.

1. **Calibration Curve:** A curve which plots the concentration of known analyte standards against the instrument response to the analyte. Also known as a Standard Curve or the ICAL.

2. **Calibration Standard:** Solutions or dilutions of a substance or material with a verifiable accuracy which are used to evaluate the sample property of an unknown sample. In analytical terms, these standards are used to establish a calibration curve or standard instrument response factors.

3. **Continuing Calibration Verification:** Standards that are analyzed during an analytical set to verify the accuracy of the calibration curve.

4. **Internal Standard:** A compound having similar chemical characteristics to the compounds of interest, but which is not normally found in the environment or does not interfere with the compounds of interest. A known and specified concentration of the standard is added to each sample prior to analysis. The concentration in the sample is based on the response of the internal standard relative to that of the calibration standard and the compound in the standard.

Confidence Level:

The statistical probability associated with an interval of precision (or accuracy) values in a QC chart. The values of confidence intervals are generally expressed as percent probability. It is a commonly accepted convention that the result being tested is significant if the calculated probability is greater than 99 percent.

Data Quality Objectives:

A set of specifications that the environmental data should meet in order to be acceptable for its intended use in a program area. DQO's are commonly established for limits of detection and quality of data (precision, accuracy, representativeness and comparability).

1. **Accuracy:** The degree of agreement of a measurement X (or an average of measurements of the same thing), with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the percentage of the reference or true value, 100 (X/T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.

2. **Precision:** A measure of mutual agreement among individual measurements of the same property, usually prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist, depending upon the "prescribed similar conditions."

3. **Representativeness:** Expresses the degree to which data accurately and precisely represents, a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

4. **Comparability:** Expresses the confidence with which one data set can be compared to another.

5. **Completeness:** Completeness refers to the percentage of measurements made which are judged to be valid. The completeness goal is the same for all data uses: that we achieve 95% completeness on our first analysis. Subsequently our goal is to achieve 100% completeness by resampling or doing additional analysis.

Detection Limits:

The smallest concentration/amount of an analyte of interest that can be measured with a stated probability of significance.

1. **Method Detection Limit (MDL):** The smallest concentration of an analyte of interest that can be measured and reported with 99 percent confidence that the concentration is greater than zero. The MDL's are determined from the analysis of a sample in a given matrix containing the analyte at a specified level. Determination of MDL's should be done by procedures determined in Appendix B of 40 CFR, Part 136. Equivalent procedures to determine MDL's may be used.

2. **Detection Limit (DL):** The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

3. **Limit of Detection (LOD):** The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

4. **Limit of Quantitation (LOQ):** The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

5. **Practical Quantitation Limit (PQL):** The smallest concentration of an analyte of interest that can be reported with a specific degree of confidence. PQL's shall be determined in the same way as MDL's by using the procedures outlined in Appendix B of 40 CFR, Part 136. The standard deviation (sd) derived from the procedure will be used to calculate the PQL: PQL = 10sd, which corresponds to an uncertainty of ± 30 percent in the measures value at the 99 percent confidence level.

6. **Instrument Detection Limit (IDL):** The smallest amount of an analyte of interest that generates an instrument response (signal) under prescribed conditions, so that the magnitude of the signal is larger than the absolute uncertainty (error) associated with it.

Environmental Sample:

Means any sample from a natural source or source that reasonably may be expected to contribute pollution to or receive pollution from groundwaters or surface waters of the State. This includes, but is not limited to: receiving waters; waters used to define natural background conditions; soils; sediments; industrial, domestic or municipal discharge effluents; chemical storage or handling facilities; waste disposal facilities or areas; industrial or agricultural chemical handling or application areas; surface water run-off; and facilities for the handling or applying of chemicals for weed or insect control.

Holding Time:

Sample hold time is defined as time elapsed from sample collection date and time to sample extraction or analysis date and time.

Parameter Group:

Is defined as a group of samples that have been preserved in the same manner, prepared by similar protocols and analyzed using instruments of similar technology (also known as analyte group). Examples of parameter groups are:

- Volatiles (EPA methods 601, 602, and 624)
- Pesticides (EPA methods 608, 614, and 622)
- Trace Metals (All metals except mercury)
- Nutrients (Total Kjeldahl Nitrogen, Nitrate-Nitrite, Total Phosphorus)

Performance Evaluation Samples:

A sample submitted for analysis whose composition and concentration are known to the submitter but unknown to the analyst. Also known as a Blind Sample.

Quality Assurance:

A system of activities whose purpose is to provide the producer or user of environmental data the assurance that it meets defined standards of quality with a stated level of confidence.

Quality Assurance Plans (QAP):

An orderly assembly of detailed and specific procedures which delineates how data of a known and accepted quality is produced.

Quality Assurance Project Plans (QAPP):

A QA plan that is written for a specific project outlining specific QA targets and data quality objectives as well as protocols and QC measures needed to meet the project specific objectives.

Quality Control Measures:

1. Blanks:

   An artificial sample of an analytical matrix designed to monitor the introduction of artifact into the system.

   a) Field Quality Control Blanks

- Field Blanks: Blanks of analyte-free water prepared on-site by filling the pre-preserved sample containers with the water, sealing the containers, and completing the documentation. These blanks should be prepared during the middle to end of a sampling event by filling sample containers with water from the equipment decontamination water transport containers. They are to be treated, stored, transported, and analyzed in the same manner as the sample group for which it was intended. These blanks may be submitted for all water parameter groups.

- Equipment Blanks: Blanks of analyte-free water that are prepared on-site by pouring the equipment decontamination water through decontaminated field equipment. Appropriate pre-preserved sample containers for each analyte group should be used, and documentation should be completed. These blanks are to be stored, transported and analyzed with the intended parameter groups. At least one equipment blank is required for each water and solid matrix analytical group, and should be collected at the beginning of the sampling episode. If field decontamination is performed on-site, additional equipment blanks should be submitted for all water and solid matrix analytical groups.

- Trip Blanks: These blanks are required for volatile organic compound (VOC) water samples only. Blanks of volatile organic-free water are prepared by the organization providing the sample containers. These are transported to the site with the empty VOC sample containers, and shipped to the analyzing laboratory in the same containers. They remain unopened for the entire trip. Proper labeling and documentation should be completed. One trip blank for VOC sent per project unless client requests more.

b) Laboratory

- Method Blank: A blank of an analyte-free matrix that is processed (digested, extracted, etc.) and analyzed with a specified sample set.

- Reagent Blank: An aliquot of analyte-free water or solvent that is analyzed with a sample set.

2. Spiked Samples:

These are samples fortified to a known and validated concentration of analyte. Percent recoveries are calculated for each compound in the spike.

a) Field

- An environmental sample fortified to a known and validated concentration in the field. These may be submitted as blind spike (laboratory does not know they are spiked) or as identified field spikes.

b) Laboratory

- LCS (Laboratory Control Sample): Samples of an analyte-free matrix (deionized water, sand, soil, etc.) that are fortified to a known and validated concentration of analyte(s) before sample preparation.

- Sample (Matrix Spikes): Environmental sample selected from a set (not blanks) that is fortified to a known and validated concentration of analyte(s) before sample preparation. The concentration of each analyte in the spiking solution should be approximately 3-5 times the level expected in the sample.

- Surrogate Spikes: A compound having similar chemical characteristics to the compounds of interest, but which is not normally found in environmental samples. Known concentrations of these compounds are added to all samples in the set before sample preparation.

3. Replicate Sample:

Samples that have been collected at the same time from the same source (field replicates) or aliquots of the sample that are prepared and analyzed at the same time (laboratory replicates).

a) Duplicate samples: Samples that are one type of a replicate sample. The analytical results from replicates are used to determine the precision of a system.

If the concentration of analytes in the sample is below detectable limits, Duplicate Spike Samples may be used to determine precision.

b) Blind Replicates (Duplicates): Samples that are replicates that have been collected (field replicate) or prepared (laboratory replicate) and are submitted and analyzed as separate samples (analyst does not know they are replicates).

4) Quality Control Checks:

Standards or samples from an independent source that are analyzed at a specific frequency.

a) Quality Control Check Standards: Standard solutions from a source other than normal calibration standards that are certified and traceable. These standards are used to check the accuracy of a calibration curve.

b) Quality Control Check Samples: (also known as Reference Materials): Samples obtained from an independent source for which the level(s) of analytes have been validated. These samples are prepared and analyzed with a sample set of similar matrix. If these samples have been obtained from the National Institute of Standards and testing (formerly National Bureau of Standards), they are referred to as Standard Reference Materials.

5) Split Samples:

Replicate of the same sample that is given to two independent laboratories for analysis.

Sample Custody:

All record and documentation required to trace a sample from point of origin to disposal after analysis. These records should include, but are not limited to:

a) Field notebooks;
b) Field sample ID tags
c) Laboratory transmittal forms (if applicable);
d) Laboratory sample receipt logs;
e) Sample extraction/preparation logs or worksheets;
f) Analytical (instrument) logs or worksheets;
g) Calibration and quality control data associated with a sample set;
h) Instrument maintenance logs;
i) Sample disposition logs; and
j) Final reports.

Legal Chain of Custody is a special type of sample custody in which all events associated with a specific sample should be documented in writing. In addition to the records described above, chain of custody records should include the following:

a) Sample transmittal forms or tags that have adequate spaces for the dated, original signatures of all individuals who handle the sample (or cleaned sample containers if obtained for a contracted laboratory) from time of collection (or container receipt) to laboratory delivery.

b) Laboratory sample storage logs that identify date, time, and individuals who remove samples from storage.

c) Secure, limited access storage areas.

Sample Matrix:

This means that characteristic of an environmental laboratory sample, associated with its physical and chemical properties, which defines how such a sample is handled when subjected to the intended analytical process. The following samples matrices (major matrix groups), defined below should be used in QA plans whenever specifying data quality objectives:

1. **Reagent Water:** A sample of water which conforms to ASTM TYPE II, III, IV.

2. **Drinking Water:** Includes finished (treated) or raw source water designated as potable water. Such resources may be from surface or ground water.

3. **Surface Water:** Includes fresh or saline waters from streams, canals, rivers, lakes, ponds, bays and estuaries (natural or manmade).

4. **Groundwater:** Includes all waters found below ground in confined or unconfined aquifers.

5. **Wastewater:** Includes any influent or effluent associated with domestic or industrial waste treatment facilities.

6. **Chemical Waste:** Includes sludges and residuals from domestic or industrial wastewater processing, and liquid or solid chemicals no longer used for their intended purpose.

7. **Soil/Sediment:** Surface or subsurface soils and sediments of fresh or salt water origin.

8. **Biological Tissue:** Includes tissue of plant or animal origin. The most common of these are shellfish, finfish, and aquatic plants.

APPENDIX B

STANDARD OPERATING PROCEDURES (SOP)
TABLE OF CONTENTS

Appendix B
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Conventionals	15	Deanna Hesson	Leslie Bucina	03-15-2014	15	K0100	Fecal Coliform (Membrane Filtration) - Standard Methods 9222D-2006
Conventionals	19	Deanna Hesson	Leslie Bucina	11-15-2013	17	K0002	Calibration Techniques
Conventionals	11	Deanna Hesson	Leslie Bucina	03-15-2014	8	K0005	Extraction of Soils and Sludges
Conventionals	10	Deanna Hesson	Leslie Bucina	01-15-2014	11	K4829	ASH - ASTM D482-07/D2974-87
Conventionals	16	Deanna Hesson	Leslie Bucina	03-20-2014	15	K3761	Sulfide (Titrimetric, Iodine) - EPA 376.1/SM 4500-S F-2000 (2011 editorial Revision)
Conventionals	15	Deanna Hesson	Leslie Bucina	01-15-2014	15	K1501	pH (Electrometric) - SW846 9040C, 9045D, EPA Method 150.1, SM4500H+B-2000
Conventionals	3	Deanna Hesson	Leslie Bucina	11-15-2013	4	K0001	Glassware Washing
Conventionals	18	Deanna Hesson	Leslie Bucina	03-15-2014	22	K3512	Total Kjeldahl Nitrogen (Semi-Automated Block Digester) EPA 351.2
Conventionals	10	Deanna Hesson	Leslie Bucina	03-15-2014	20	K7332	Reactivity (Cyanide and Sulfide) - SW846 Section 7.3
Conventionals	10	Deanna Hesson	Leslie Bucina	07-15-2013	11	K2710	Specific Gravity - Standard Method 2710F-2009
Conventionals	16	Deanna Hesson	Leslie Bucina	03-20-2014	21	K3252	Chloride (Automated, Ferricyanide) EPA 325.2, SM4500 Cl-E - 1997 (2011 Editorial Revision)
Conventionals	10	Deanna Hesson	Leslie Bucina	01-15-2014	20	K5310	TOC in Soil - Lloyd Kahn Method
Conventionals	13	Deanna Hesson	Leslie Bucina	01-29-2014	15	K3101	Alkalinity (Titrimetric, pH 4.5) - EPA Method 310.1/SM 2320B-1997 (Editorial Revision 2011)
Conventionals	14	Deanna Hesson	Leslie Bucina	01-15-2014	18	K4251	Methylene Blue Active Substances (MBAS, Surfactants) - SM 5540C - 2000 (2011 Editorial Revision)
Conventionals	8	Deanna Hesson	Leslie Bucina	11-15-2013	15	K3758	Total Sulfur - SW846 5050/EPA 375.4
Conventionals	14	Deanna Hesson	Leslie Bucina	01-15-2013	17	K1302	Hardness, Total (Titrimetric, EDTA) EPA 130.2/SM 2340C-1997
Conventionals	13	Deanna Hesson	Leslie Bucina	07-15-2013	15	K1601	Residue, Filterable (TDS, Gravametric, 180 degrees C) EPA Method 160.1, SM2540 C-1997
Conventionals	13	Deanna Hesson	Leslie Bucina	01-15-2013	15	K1201	Conductance (UMHOS at 25 Degrees Celcius) EPA 120.1, SM2510B-1997
Conventionals	16	Deanna Hesson	Leslie Bucina	01-24-2014	30	K3354	Cyanide, All Forms(Spectrophotometric)EPA335.2/335.4/9010C/9014/SM4500CN-CE-1999/2011 Editorial Revision/TotalEPA335.1/SM9010C/9014/4500CN-CG-1999/2011 Editorial Revision/Amenable to Chlorination/SM4500CN-I-1999/2011 Editorial Revision/Weak-Dissociable
Conventionals	18	Deanna Hesson	Leslie Bucina	03-15-2014	23	K3654	Total Phosphorus: (Semi-Automated Block Digestor/Automated) - EPA Method 365.4
Conventionals	11	Deanna Hesson	Leslie Bucina	07-15-2013	16	K1801	Turbidity (Nephelometric)-EPA 180.1, SM2130 B-2001
Conventionals	18	Deanna Hesson	Leslie Bucina	03-15-2014	15	K0101	Total Coliform (Membrane Filtration) - Standard Methods 9222B-2006
Conventionals	20	Deanna Hesson	Leslie Bucina	01-24-2014	23	K2186	Chromium, Hexavalent (Colorimetric) Standard Method 3500-Cr B-2009 (2011 Editorial Revision)
Conventionals	12	Deanna Hesson	Leslie Bucina	01-15-2014	15	K3771	Sulfite (Titrimetric) - Method SM 4500-S03 B-2000, EPA 377.1

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Conventionals	22	Deanna Hesson	Leslie Bucina	01-24-2014	21	K4051	Biochemical Oxygen Demand - SM 5210 B-2001(2011 Editorial Revision)
Conventionals	15	Deanna Hesson	Leslie Bucina	01-15-2014	21	K4105	Chemical Oxygen Demand (COD) - EPA 410.4 (MOD)/SM 5220 D-1997 (2011 Editorial Revision)/Hach 8000
Conventionals	20	Deanna Hesson	Leslie Bucina	03-15-2014	27	K3501	Nitrogen, Ammonia (Automated Phenate) - EPA 350.1/350.2 and SM 4500-NH3 B,G-1997 (2011 Editorial Revision)
Conventionals	11	Deanna Hesson	Leslie Bucina	09-15-2012	15	K3052	Acidity (Titrametric) EPA Method 305.2/SM2310B(4d)
Conventionals	11	Deanna Hesson	Leslie Bucina	07-15-2013	12	K1604	Volatile Solids (Total/Dissolved/Suspended) Gravemetric, Ignition 550 degrees C - EPA Method 160.4/SM 2540E, G-1997
Conventionals	7	Deanna Hesson	Leslie Bucina	01-15-2014	17	K3500	Ferrous Iron - SM3500 Fe B-1997 (2011 Editorial Revision)
Conventionals	2	Deanna Hesson	Leslie Bucina	07-15-2013	17	K5468	Gross Calorific Value of Waste Materials (BTU) - ASTM D5468
Conventionals	11	Deanna Hesson	Leslie Bucina	09-15-2012	15	K3051	Acidity (Titrimetric, H2O2) EPA Method 305.1/SM2310B(4a)
Conventionals	16	Deanna Hesson	Leslie Bucina	03-20-2014	22	K4151	Organic Carbon, Total (Oxidation) - EPA 415.1/SW846 9060A/SM5310C-2000 (2011 editorial Revision)
Conventionals	12	Deanna Hesson	Leslie Bucina	07-15-2013	20	K5050	Halides, Total (TX) - SW 846 Method 5050
Conventionals	10	Deanna Hesson	Leslie Bucina	01-15-2014	10	K9095	Paint Filter Liquids Test - SW846 Method 9095B
Conventionals	10	Deanna Hesson	Leslie Bucina	03-20-2014	13	K3601	Dissolved Oxygen and Winkler Calibration, EPA Method 360.1, SM 4500-O C,G, 2001 (2011 editorial Revision)
Conventionals	13	Deanna Hesson	Leslie Bucina	07-15-2013	14	K1603	Residue, Total (TS, Gravemetric, 103 - 105 Degrees C) EPA Method 160.3, SM2540 B-1997
Conventionals	16	Deanna Hesson	Leslie Bucina	03-15-2014	14	K0103	Most Probable Number of Fecal Coliforms - Standard Methods 9221 B,C,E-2006
Conventionals	16	Deanna Hesson	Leslie Bucina	01-15-2014	17	K3541	Nitrogen, Nitrite (Spectrophotometric) EPA 354.1/SM 4500-NO2B-2000 (2011 Editorial Revision)
Conventionals	13	Deanna Hesson	Leslie Bucina	01-15-2014	21	K9030	Sulfide (Acid Soluble/Acid Insoluble) - SW846 9030B/9034
Conventionals	8	Deanna Hesson	Leslie Bucina	01-15-2014	20	K1664	n-Hexane Extractable Material and Silica Gel Treated n-Hexane Extractable Material, Methods 1664A and 1664B (Automated Solid Phase)
Conventionals	20	Deanna Hesson	Leslie Bucina	01-15-2014	23	K3532	Nitrogen, Nitrate-Nitrite (Automated, Cadmium Reduction) - EPA 353.2/SM4500NO3 F - 2000 (2011 Editorial Revision)
Conventionals	15	Deanna Hesson	Leslie Bucina	03-15-2014	18	K3402	Fluoride (Potentiometric, Ion Selective Electrode) - SM 4500-F C-1997 (2011 Editorial Revision)
Conventionals	10	Deanna Hesson	Leslie Bucina	08-15-2013	10	K1605	Settleable Solids (Volumetric, IMHOFF Cone) - EPA Method 160.5/SM 2540F
Conventionals	15	Deanna Hesson	Leslie Bucina	01-24-2014	15	K1602	Residue, Non-Filterable (TSS, Gravemetric, 103 Degrees C) EPA Method 160.2/SM2540 D-1997 (2011 Editorial Revision)
Conventionals	18	Deanna Hesson	Leslie Bucina	01-15-2014	21	K4201	Phenolics, Total Recoverable (Spectrophotometric, Manual 4-AAP/Easy-Distillation) EPA Method 420.1

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Conventionals	16	Deanna Hesson	Leslie Bucina	12-15-2013	21	K3102	Alkalinity (Automated Methyl Orange) EPA Method 310.2
Conventionals	16	Deanna Hesson	Leslie Bucina	03-20-2014	17	K9075	Total Residual Chlorine - Standard Methods 4500-Cl G, 2000 (2011 editorial Revision)
Conventionals	18	Deanna Hesson	Leslie Bucina	03-20-2014	20	K3754	Sulfate, Total (Turbidimetric) - EPA Method 375.4, Method 4500-SO4 2 E, 1997 (2011 Editorial Revision)
Conventionals	15	Deanna Hesson	Leslie Bucina	01-15-2014	18	K3653	Orthophosphate (Colorimetric, Ascorbic Acid, Single Reagent) EPA 365.2/SM 4500-P E-1999 (2011 Editorial Revision)
Conventionals	13	Deanna Hesson	Leslie Bucina	01-24-2014	14	K1102	Color (Colorimetric-Platinum-Cobalt) - Standard Method 2120 B-2001 (2011 editorial Revision)
Conventionals	3	Deanna Hesson	Leslie Bucina	03-15-2014	15	K0105	Escherichia Coli (E. Coli) m-Colblue-24 (Hach Method 10029)
Conventionals	2	Deanna Hesson	Leslie Bucina	01-15-2014	18	K9016	Free Cyanide (Microdiffusion)
Conventionals	1	Deanna Hesson	Leslie Bucina	03-15-2014	19	KAVS	Analytical Method for Determination of Acid Volatile Sulfide (AVS) in Sediment
Conventionals	00		Leslie Bucina				1_KT Placeholder-Fluoroborate by ISE
Digestions	13	Bob Kyer	Leslie Bucina	02-27-2014	11	K0003	Percent Solids (Percent Moisture) - ASTM D2216-90
Digestions	13	Robert Kyer	Leslie Bucina	05-15-2013	18	ME406	Microwave Digestion of Sediments, Sludges, Soils and Oils (3051A)
Digestions	16	Maren Beery	Leslie Bucina	02-15-2014	33	ME404	Mercury (7470A, 245.1)
Digestions	12	Maren Beery	Leslie Bucina	02-15-2014	34	ME405	Mercury - SW-846 Method 7471
Digestions	9	Maren Beery	Leslie Bucina	03-15-2014	8	ME201	Cleaning Procedure for Preparation of Glassware for the Analysis of Trace Metals
Digestions	15	Bob Kyer	Leslie Bucina	12-15-2013	17	ME407	Microwave Digestion - Aqueous - SW846-3015A
Digestions	00	Bob Kyer	Leslie Bucina	07-15-2013	14	ME408	Acid Digesting of Waters for Treatment for Acid-Extractable Metals Inductively Coupled Plasma Spectroscopy (Standard Method 3030C)
Digestions	00	Bob Kyer	Leslie Bucina	06-15-2013	14	ME301	Sieving Procedure for Soil Samples Prior to Digestion and Percent Solids Determinations
Digestions	00		Leslie Bucina	Processing		Method 80-3.2.2	Method 80-3.2.2 Digestion
Digestions	00	Bob Kyer	Leslie Bucina	08-22-2013	14	ME401A	Preparation of Dissolved Metals in Waters for Direct Analysis by Inductively Coupled Plasma Spectroscopy (EPA 200.7)
Extractions	16	Bob Kyer	Leslie Bucina	11-15-2013	17	K1010	Flash Point (Pensky-Martens Closed Cup Method) - SW846 1010A
Extractions	11	Mike Cochran	Leslie Bucina	03-15-2014	14	EXTNT02	Solid Extraction of Nitroaromatics and Nitroamines - Method 8330
Extractions	16	Bob Kyer	Leslie Bucina	02-15-2014	13	EXB01	Continuous Liquid-Liquid Extraction of Semivolatiles (Base/Neutral/Acid (BNA)) - Method 3520C
Extractions	19	Mike Cochran	Leslie Bucina	03-15-2014	17	EXD01	Extraction of Diesel Range Organics
Extractions	14	Bob Kyer	Leslie Bucina	02-15-2014	9	EXB03	Waste Dilution for Semivolatiles - Method 3580A
Extractions	14	Bob Kyer	Leslie Bucina	10-31-2013	15	EXP01	Separatory Funnel Liquid-Liquid Extractions for Pesticides and PCBs - SW846 Methods 3510, 3500, and CFR Method 608

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Extractions	12	Mike Cochran	Leslie Bucina	03-15-2014	12	EXTNT01	Liquid Extraction of Nitroaromatics and Nitroamines - SW846 Method 8330
Extractions	17	Bob Kyer	Leslie Bucina	02-15-2014	13	EXA01	Separatory Funnel Liquid-Liquid Extraction for Polynuclear Aromatic Hydrocarbons - Method 3510C/3520C
Extractions	9	Bob Kyer	Leslie Bucina	12-15-2013	19	ASE01	Accelerated Solvent Extractor - SW846 Method 3545A
Extractions	16	Bob Kyer	Leslie Bucina	02-15-2014	13	EXB02	Sonication Extractions for Semivolatiles - Method 3550
Extractions	11	Chris Hill	Leslie Bucina	04-15-2013	10	EXP04	Preparation of Spiking Solutions - Methods 8081 and 8082
Extractions	10	Chris Hill	Leslie Bucina	04-15-2013	10	EXP03	Waste Dilution for PCB's in Oil - SW-846 Method 3580
Extractions	10	Chris Hill	Leslie Bucina	04-15-2013	9	EXP08	Extraction of PCB Wipes
Extractions	9	Bob Kyer	Leslie Bucina	07-15-2013	9	K3987	ASTM Shake Test - ASTM-D3987-06
Extractions	7	Mike Cochran	Leslie Bucina	03-15-2014	16	EXPRO	Extraction of Petroleum Range Organics
Extractions	14	Bob Kyer	Leslie Bucina	02-15-2014	12	EXA02	Sonication Extractions for Polyaromatic Hydrocarbons (PAH) - Method 3550
Extractions	13	Bob Kyer	Leslie Bucina	02-15-2014	13	EXB08	Separatory Funnel Liquid-Liquid Extractions for Semivolatiles (BNA) - Method 3510C
Extractions	10	Robert Kyer	Leslie Bucina	10-23-2013	33	TCLP01	Toxicity Characteristic Leaching Procedure - SW 846 Method 1311/1312
Extractions	5	Robert Kyer	Leslie Bucina	06-15-2013	13	EXWET01	California Waste Extraction Test (WET) Procedures
Extractions	4	Robert Kyer	Leslie Bucina	06-15-2013	6	SOP37	Extraction Glassware Washing
Extractions	16	Bob Kyer	Leslie Bucina	09-25-2013	15	EXP02	Sonication Extractions for Pesticides and PCBs - SW846 Methods 3500 and 3550
Extractions	12	Bob Kyer	Leslie Bucina	02-15-2014	8	EXA03	Preparation of a Polyaromatic Hydrocarbons (PAH) Surrogate and Spiking Solution
Extractions	20	Bob Kyer	Leslie Bucina	09-05-2013	20	EXH01	Extraction of Chlorinated Herbicides - SW-846 Method 8151A
Extractions	00	Bob Kyer	Leslie Bucina	02-15-2014	15	EXMW01	Microwave Extraction SW846 Method 3546
Extractions	2	Bob Kyer	Leslie Bucina	11-15-2013	15	EXHEMS	Extraction Procedure n-hexane Extractable Material (HEM) for Soils
General	4	Wade DeLong	Leslie Bucina	09-26-2013	18	SOP43	Writing and Maintaining Standard Operating Procedures
General	8	Sheri Pfalzgraf	Leslie Bucina	10-15-2013	15	SOP36	Emergency Response
General	3	Matt Wilson	Leslie Bucina	01-15-2014	11	MISEDD01	Development and Maintenance of Electronic Data Deliverables (EDD)
General	16	Wade DeLong	Leslie Bucina	11-12-2013	25	LOGIN01	Sample Receiving and Login
General	10	Charissa Winters	Leslie Bucina	06-15-2013	21	MISDATA01	Data Entry, Data Review, and Reporting
General	9	Stephanie Mossburg	Leslie Bucina	09-15-2013	9	SOP44	Project Management, Technical Service and Subcontracting
General	7	Wade DeLong	Leslie Bucina	02-15-2014	17	SOP47	Employee Training
General	4	Wade DeLong	Leslie Bucina	07-15-2013	12	SOP42	Waste Minimization Plan - Microbac Laboratory
General	8	Wade DeLong	Leslie Bucina	12-15-2013	16	SOP45	Method Validation Procedures
General	5	Matt Wilson	Leslie Bucina	10-15-2013	15	DU01	Inorganic and Organic Data Upload Procedure

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
General	15	Sheri Pfalzgraf	Leslie Bucina	03-15-2014	31	CHP04	Chemical Hygiene Plan - Ohio Valley Division
General	3	Stephanie Mossburg	Leslie Bucina	12-15-2012	5	CRC01	Ensuring Confidentiality of Results
General	14	Wade DeLong	Leslie Bucina	05-24-2013	49	SOP33	Laboratory Waste Management
General	00	Matt Wilson	Leslie Bucina	10-15-2013	9	MISBUR	Backup and Recovery
General	3	Mike Albertson/Mike Cochran	Leslie Bucina	10-15-2013	20	SOP41	Manual Integration of Chromatographic Peaks
General	4	Wade DeLong	Leslie Bucina	01-15-2014	9	ETH01	Ethics Policy - Ohio Valley Laboratory
General	1	Wade DeLong	Leslie Bucina	09-26-2013	7	GP-QMSR	Quality Management System Review
General	1	Wade DeLong	Leslie Bucina	10-15-2013	6	GP-UNCERTAINTY	Estimation of Uncertainty of Measurement
General	1	Stephanie Mossburg	Leslie Bucina	09-15-2013	11	GP-CONTRACTS	Review of Contracts
General	00	Wade DeLong	Leslie Bucina	09-15-2013	7	GP-RCA	Root Cause Analysis
General	2	Wade DeLong	Leslie Bucina	09-15-2013	11	GP-PURCHASING	Purchasing Services and Supplies
General	3	Charissa Winters	Leslie Bucina	10-29-2013	11	GP-DOC-CONTROL	Document Control
General	1	Wade DeLong	Leslie Bucina	09-15-2013	9	GP-CAPA	Corrective Action and Preventive Action: Initiating, Tracking, and Monitoring
General	1	Wade DeLong	Leslie Bucina	09-15-2013	9	GP-RECORDS	Managing Records
General	1	Wade DeLong	Leslie Bucina	09-15-2013	8	GP-INTAUDIT	Internal Audit Guidelines
General	16	Wade DeLong	David Vandenberg	10-31-2013	203	LQAP	Laboratory Quality Assurance Plan - Microbac Laboratories, Inc. - Ohio Valley Division
General	00	Matt Wilson	Leslie Bucina	01-15-2014	11	IT-Software	Software Version Control
General	1	Wade DeLong	Leslie Bucina	04-15-2013	10	GP-TEMP-SSU	Temperature Control Systems for Sample Storage Units
General	00	Matt Wilson	Leslie Bucina	04-15-2013	5	IT-Hardware	LIMS Hardware Design and Maintenance
General	00	Wade DeLong	Leslie Bucina	07-15-2013	15	GP-Review of Analytical Data	Review of Analytical Data
General	00	Matt Wilson	Leslie Bucina	10-15-2013	15	IT-GENPP	General IT Policies and Procedures
General	00		Leslie Bucina				1_KT Placeholder - Permit Management
Metals	14	Maren Beery	Leslie Bucina	10-30-2013	50	ME600E	Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy SW-846 6010/ EPA 200.7
Metals	5	Maren Beery	Leslie Bucina	10-30-2013	52	ME600G	Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy SW-846 Method 6010/EPA 200.7

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Metals	00	Maren Beery	Leslie Bucina	06-17-2013	46	ME700A	Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer (SW-846 6020/EPA Method 200.8)
OVAP	00	Maren Beery	Leslie Bucina	11-15-2010	14	OVAP ME401	OVAP Acid Digesting of Waters for Total Recoverable or Dissolved Inductively Coupled Plasma Spectroscopy (3005A)
OVAP	00	Chad Barnes	Leslie Bucina	11-15-2010	13	OVAP EXA01	OVAP Separatory Funnel Liquid-Liquid Extraction for Polynuclear Aromatic Hydrocarbons - Method 3510C/3520C
OVAP	00	Deanna Hesson	Leslie Bucina	12-15-2010	19	OVAP K2186	OVAP Chromium, Hexavalent - Standard Method 3500-Cr D
OVAP	00	Mike Albertson / Mike Cochran	Leslie Bucina	11-15-2010	20	OVAP SOP41	OVAP Manual Integration of Chromatographic Peaks
OVAP	00	Maren Beery	Leslie Bucina	11-15-2010	16	OVAP ME407	OVAP Microwave Digestion - Aqueous - SW846 - 3015
OVAP	00	Maren Beery	Leslie Bucina	11-15-2010	16	OVAP ME406	OVAP Microwave Digestion of Sediments and Soils (3051)
OVAP	1	Maren Beery	Leslie Bucina	02-21-2013	30	OVAP ME405	OVAP Mercury SW-846 Method 7471A
OVAP	00	Chad Barnes	Leslie Bucina	12-15-2010	15	OVAP EXB02	OVAP Sonication Extractions for Semivolatiles - Method 3550B
OVAP	00	Chad Barnes	Leslie Bucina	12-15-2010	15	OVAP EXP02	OVAP Sonication Extractions for Pesticides and PCB's - SW846 Method 3550B
OVAP	00	Chad Barnes	Leslie Bucina	12-15-2010	13	OVAP EXA02	OVAP Sonication Extrations for Polyaromatic Hydrocarbons - Method 3550B
OVAP	1	Maren Beery	Leslie Bucina	02-21-2013	31	OVAP ME404	OVAP Mercury (7470A)
OVAP	00	Maren Beery	Leslie Bucina	12-15-2010	37	OVAP ME600G	OVAP Thermo iCap 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy SW-846 Method 6010B
OVAP	00	Maren Berry	Leslie Bucina	01-15-2011	41	OVAP ME600E	OVAP Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy SW-846 - Method 6010B
OVAP	00	Chad Barnes	Leslie Bucina	12-15-2010	15	OVAP EXB01	OVAP Continuous Liquid-Liquid Extraction Semivolatiles (BNA) - Method 3520C
OVAP	00	Mike Albertson	Leslie Bucina	12-15-2010	16	OVAP PAT01	OVAP Purge and Trap for Volatile Organics - SW846 Methods 5030B and 5035A
OVAP	00	Chad Barnes	Leslie Bucina	12-15-2010	14	OVAP EXB08	OVAP Separatory Funnel Liquid-Liquid Extractions for Semivolatiles (BNA) - Method 3510C
OVAP	00	Chad Barnes	Leslie Bucina	01-15-2011	17	OVAP EXH01	OVAP Extraction of Chlorinated Herbicides - SW-846 Method 8151A
OVAP	00	Chad Barnes	Leslie Bucina	01-15-2011	20	OVAP ASE01	OVAP Accelerated Solvent Extractor - SW846 Method 3545
OVAP	1	Maren Beery	Leslie Bucina	11-15-2012	39	OVAP ME700	OVAP Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer - SW-846 6020
OVAP	00	Mike Cochran	Leslie Bucina	02-15-2011	24	OVAP GCS02	OVAP Analysis of Middle and Heavy Distillates - Method 8015B
OVAP	00	Mike Albertson	Leslie Bucina	02-15-2011	29	OVAP GCV05	OVAP Light Distillates - SW846 8015B
OVAP	00	Chad Barnes	Leslie Bucina	02-15-2011	14	OVAP EXP01	OVAP Separatory Funnel Liquid-Liquid Extractions for Pesticides and PCB's SW846 Method 3510C and 3500C
OVAP	00	Chad Barnes	Leslie Bucina	02-15-2011	17	OVAP EXD01	OVAP Extraction of Middle and Heavy Distillates SW-846 Method 3550B and 3510

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
OVAP	1	Deanna Hesson	Leslie Bucina	03-29-2013	30	OVAP K3354	OVAP Total Cyanide (Spectrophotometric) - EPA 335.2 (CLP-M)
OVAP	00	Mike Cochran	Leslie Bucina	03-15-2011	26	OVAP GCS10	OVAP Organic Analysis of PCB's - Method 8082
OVAP	00	Mike Albertson	Leslie Bucina	03-15-2011	47	OVAP MSV01	OVAP Analysis of Volatile Organic Compounds by Method 8260A and 8260B
OVAP	00	Mike Cochran	Leslie Bucina	03-15-2011	25	OVAP GCS04	OVAP Organic Analysis of Chlorinated Herbicides - Method 8151A
OVAP	00	Mike Cochran	Leslie Bucina	03-15-2011	28	OVAP GCS09	OVAP Organic Analysis of Pesticides - Method 8081A
OVAP	00	Mike Cochran	Leslie Bucina	03-15-2011	39	OVAP MSS01	OVAP Organic Analytes - Method 8270C
OVAP	00	Mike Cochran	Leslie Bucina	04-15-2011	31	OVAP MSS03	OVAP Low Level PAH's - Method 8270
OVAP	00		Leslie Bucina	00	39	OVAP ME700A	PLACE HOLDER - OVAP Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer (SW-846 6020)
OVAP	00	Chad Barnes	Leslie Bucina	Processing		OVAP EXMW01	PLACE HOLDER - OVAP Microwave Extraction SW846 Method 3546
Semivolatiles	12	Mike Cochran	Leslie Bucina	04-15-2013	24	GCS07	1,2 Dibromoethane (EDB) and 1,2 Dibromo-3-chloropropane (DBCP) Method SW-846 Method 8011
Semivolatiles	15	Mike Cochran	Leslie Bucina	11-15-2013	30	GCS09	Organic Analysis of Pesticides - Method 8081
Semivolatiles	7	Mike Cochran	Leslie Bucina	02-20-2014	31	GCS05	The Analysis of TCEQ Methods for Total Petroleum Hydrocarbons - Methods T1005/T1006
Semivolatiles	12	Mike Cochran	Leslie Bucina	10-15-2013	25	GCS03	Analysis of Petroleum Range Organics - Method FLA-PRO
Semivolatiles	4	Mike Cochran	Leslie Bucina	12-19-2013	25	HPLC04	Formaldehyde, Acetaldehyde and Propionaldehyde - Method 8315A
Semivolatiles	15	Mike Cochran	Leslie Bucina	03-15-2014	24	IC01	Determination of Inorganic Anions By Ion Chromatography - Method 9056/300.0
Semivolatiles	15	Mike Cochran	Leslie Bucina	05-16-2013	28	GCS02	Analysis of Diesel Range Organics - Method 8015
Semivolatiles	12	Mike Cochran	Leslie Bucina	03-20-2014	31	GCS08	Organic Analysis of Pesticides and PCBs - Method 608
Semivolatiles	6	Mike Cochran	Leslie Bucina	11-15-2013	30	HPLC06	Perchlorate - Method 6850/331.0
Semivolatiles	17	Mike Cochran	Leslie Bucina	07-15-2013	29	HPLC02	Nitroaromatics and Nitramines by High Performance Liquid Chromatography - Method 8330
Semivolatiles	6	Mike Cochran	Leslie Bucina	12-15-2013	22	HPLC03	Organic Analysis of Metabolic Acids - Method 830MBA
Semivolatiles	22	Mike Cochran	Leslie Bucina	10-30-2013	63	MSS01	Organic Analytes - Method 8270
Semivolatiles	11	Mike Cochran	Leslie Bucina	10-30-2013	33	MSS03	Low Level PAHs - Method 8270
Semivolatiles	8	Mike Cochran	Leslie Bucina	10-15-2013	23	GCS12	Alcohol - Method 8015
Semivolatiles	14	Mike Cochran	Leslie Bucina	10-15-2013	29	GCS10	Organic Analysis of PCBs - Method 8082
Semivolatiles	14	Mike Cochran	Leslie Bucina	12-15-2013	27	GCS04	Organic Analysis of Chlorinated Herbicides - Method 8151A
Semivolatiles	4	Mike Cochran	Leslie Bucina	03-15-2014	24	HPLC07	Hexamethylphosphoramide - Method XMPA-LCMS

Appendix B (continued)
SOP Table of Contents

Microbac

SOP By Department Listing

Department	Rev.	Author	Approved By	Approval Date	# Pages	Name	Title
Semivolatiles	00	Mike Cochran	Leslie Bucina	02-15-2014	31	GCS13	Method for the Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions, Washington State Department of Ecology
Semivolatiles	00	Mike Cochran	Leslie Bucina	04-15-2013	23	HPLC09	Benzotriazole and Tolyltriazole Method TTA-LCMS
Semivolatiles	00	Mike Cochran	Leslie Bucina	04-15-2013	24	MSS05	Measurement of Triclosan in Extracts Prepared form Swabs
Semivolatiles	2	Mike Cochran	Leslie Bucina	11-05-2012	44	MA-EPH	Massachusetts Department of Environmental Protection method for the Determination of Extractable Petroleum Hydrocarbons (EPH)
Semivolatiles	1	Mike Cochran	Leslie Bucina	12-15-2013	22	GCS11A	Analysis of Glycols Method 8015B Modified (Glycols)
Semivolatiles	00	Mike Cochran	Leslie Bucina	01-15-2014	25	OK-DRO	Oklahoma Analysis of Diesel Range Organics Method 8015 (Modified)
Semivolatiles	00	Mike Cochran	Leslie Bucina	03-15-2014	22	HPLC12	Acetate and Formate
Volatiles	16	Mike Albertson	Leslie Bucina	11-11-2013	17	PAT01	Purge and Trap for Volatile Organics - SW-846 Methods 5030 and 5035
Volatiles	5	Mike Albertson	Leslie Bucina	07-15-2013	7	VGW01	Cleaning Glassware Used in Organic Analyses
Volatiles	19	Mike Albertson	Leslie Bucina	11-11-2013	63	MSV01	Volatile Organic Analytes by Methods 8260 and SM6200B
Volatiles	16	Mike Albertson	Leslie Bucina	07-15-2013	27	RSK01	Dissolved Gases in Ground Water - EPA RSKSOP-175
Volatiles	14	Mike Albertson	Leslie Bucina	10-25-2013	33	GCV05	Gasoline Range Organics - SW846 8015
Volatiles	12	Mike Albertson	Leslie Bucina	11-11-2013	36	MSV10	Anaylses of Volatile Organic Analytes by Method 624
Volatiles	00	Mike Albertson	Leslie Bucina	03-15-2014	32	GCV08	Determination of Volatile Petroleum Hydrocarbons (VPH)
Volatiles	1	Mike Albertson	Leslie Bucina	09-25-2013	36	MA-VPH	Massachusetts Department of Environmental Protection Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)
Volatiles	00	Mike Albertson	Leslie Bucina	02-15-2014	29	OK-GRO	Oklahoma Analysis of Gasoline Range Organics SW846 8015

APPENDIX C

NELAP ACCREDITED ANALYTES

State of Florida
Department of Health, Bureau of Public Health Laboratories
This is to certify that

E87551

MICROBAC LABORATORIES, INC., OHIO VALLEY DIVISION
158 STARLITE DRIVE
MARIETTA, OH 45750

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - GROUP III UNREGULATED CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2013 Expiration Date: June 30, 2014

Victor Johnson, Director
Division of Emergency Preparedness and Community Support
DH Form 1697, 7/04
NON-TRANSFERABLE E87551-27-07/01/2013
Supersedes all previously issued certificates

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code:** OH00218 (740) 373-4071

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acetylene	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012
Carbon dioxide	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012
Ethane	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012
Ethylene	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012
Methane	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012
Perchlorate	EPA 331.0	Secondary Inorganic Contaminants	NELAP	11/15/2012
Perfluorobutane sulfonate (PFBS, perfluorobutane sulfonic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Perfluoroheptanoate (PFHpA, perfluoroheptanoic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Perfluorohexane sulfonate (PFHxS, perfluorohexane sulfonic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Perfluorononanoate (PFNA, perfluorononanoic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Perfluorooctane sulfonate (PFOS, perfluorooctane sulfonic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Perfluorooctanoate (PFOA, perfluoroactanoic acid)	EPA 537	Group III Unregulated Contaminants	NELAP	11/15/2012
Propane	RSK-175	Group II Unregulated Contaminants	NELAP	11/15/2012

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1,2-Tetrachloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/16/2003
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/30/2006
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trimethylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	5/7/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	5/7/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dibromoethane (EDB, Ethylene dibromide)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/16/2003
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	5/16/2003
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	3/30/2006
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	3/1/2011
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	3/30/2006
1,3-Butadiene	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/16/2003
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichloropropane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	3/1/2011
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	3/30/2006
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/16/2003
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	OVL MSS01/GC-MS	Extractable Organics	NELAP	7/30/2010
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	3/30/2006
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1-Methylnaphthalene	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,2-Dichloropropane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	3/30/2006
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	3/30/2006
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	5/16/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	3/30/2006
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	3/30/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	5/16/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	3/30/2006
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	3/30/2006
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	3/30/2006
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	5/16/2003
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	5/16/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorotoluene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	SM 6200 B	Volatile Organics	NELAP	11/15/2012
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	3/30/2006
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	3/30/2006
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	5/16/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	7/30/2010
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	3/30/2006
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	3/30/2006
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	3/30/2006
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	3/30/2006
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	5/16/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	5/16/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	3/30/2006
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	5/16/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Chlorotoluene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	3/30/2006
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	3/30/2006
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	3/30/2006
a,a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
Acenaphthene	EPA 625	Extractable Organics	NELAP	5/16/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	5/16/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetic acid	OVL HPLC03/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetone	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	3/30/2006
Acetylene	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	5/16/2003
Acidity, as CaCO3	SM 2310 B	General Chemistry	NELAP	4/17/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	5/16/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	5/16/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	5/16/2003
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	5/16/2003
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	3/30/2006
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Aluminum	EPA 200.7	Metals	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 7 of 32

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	5/16/2003
Amenable cyanide	EPA 9010/9014	General Chemistry	NELAP	5/7/2008
Amenable cyanide	SM 4500-CN- G	General Chemistry	NELAP	4/17/2007
Ammonia as N	EPA 350.1	General Chemistry	NELAP	5/16/2003
Ammonia as N	SM 4500-NH3 G	General Chemistry	NELAP	11/15/2012
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	5/16/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.7	Metals	NELAP	5/16/2003
Antimony	EPA 200.8	Metals	NELAP	5/16/2003
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Aramite	EPA 8270	Extractable Organics	NELAP	3/30/2006
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1262 (PCB-1262)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	3/27/2013
Aroclor-1268 (PCB-1268)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	11/15/2012
Arsenic	EPA 200.7	Metals	NELAP	5/16/2003
Arsenic	EPA 200.8	Metals	NELAP	5/16/2003
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Atrazine	OVL MSS01/GC-MS	Extractable Organics	NELAP	7/30/2010
Barium	EPA 200.7	Metals	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 8 of 32

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Barium	EPA 200.8	Metals	NELAP	3/30/2006
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	3/30/2006
Benzaldehyde	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Benzene	EPA 624	Volatile Organics	NELAP	5/16/2003
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Benzidine	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	5/16/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	5/16/2003
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	5/16/2003
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	5/16/2003
Biphenyl	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	5/16/2003
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	5/16/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	5/16/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 9 of 32

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Boron	EPA 200.7	Metals	NELAP	5/16/2003
Boron	EPA 6010	Metals	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	5/16/2003
Bromide	EPA 9056	General Chemistry	NELAP	5/7/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	5/16/2003
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Bromoform	EPA 624	Volatile Organics	NELAP	5/16/2003
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	5/16/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyric acid (Butanoic acid)	OVL HPLC03/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Cadmium	EPA 200.7	Metals	NELAP	5/16/2003
Cadmium	EPA 200.8	Metals	NELAP	3/30/2006
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	3/30/2006
Calcium	EPA 200.7	Metals	NELAP	5/16/2003
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Caprolactam	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Carbazole	EPA 8270	Extractable Organics	NELAP	3/30/2006
Carbon dioxide	RSK-175	Volatile Organics	NELAP	3/30/2006
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	5/16/2003
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	5/16/2003
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	5/16/2003
Chemical oxygen demand	HACH 8000	General Chemistry	NELAP	5/16/2003
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 10 of 32

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chloride	EPA 300.0	General Chemistry	NELAP	5/16/2003
Chloride	EPA 325.2	General Chemistry	NELAP	5/16/2003
Chloride	EPA 9056	General Chemistry	NELAP	5/7/2008
Chloride	SM 4500-Cl⁻ E	General Chemistry	NELAP	7/30/2010
Chlorobenzene	EPA 624	Volatile Organics	NELAP	5/16/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Chloroethane	EPA 624	Volatile Organics	NELAP	5/16/2003
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Chloroform	EPA 624	Volatile Organics	NELAP	5/16/2003
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Chloroprene	EPA 8260	Volatile Organics	NELAP	3/30/2006
Chromium	EPA 200.7	Metals	NELAP	5/16/2003
Chromium	EPA 200.8	Metals	NELAP	3/30/2006
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	3/30/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	3/30/2006
Chromium VI	SM 3500-Cr B (20th/21st Ed.)/UV-VIS	General Chemistry	NELAP	5/7/2008
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	Metals	NELAP	5/16/2003
Chrysene	EPA 625	Extractable Organics	NELAP	5/16/2003
Chrysene	EPA 8270	Extractable Organics	NELAP	3/30/2006
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/16/2003
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Cobalt	EPA 200.7	Metals	NELAP	5/16/2003
Cobalt	EPA 200.8	Metals	NELAP	3/30/2006
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	3/30/2006
Color	EPA 110.2	General Chemistry	NELAP	5/16/2003
Color	SM 2120 B	General Chemistry	NELAP	4/17/2007

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Conductivity	EPA 120.1	General Chemistry	NELAP	5/16/2003
Conductivity	SM 2510 B	General Chemistry	NELAP	11/15/2012
Copper	EPA 200.7	Metals	NELAP	5/16/2003
Copper	EPA 200.8	Metals	NELAP	3/30/2006
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	3/30/2006
Cyanide	EPA 335.2	General Chemistry	NELAP	5/16/2003
Cyanide	SM 4500-CN E	General Chemistry	NELAP	4/17/2007
Cyclohexane	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Cyclohexanone	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	5/16/2003
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	5/16/2003
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromochloromethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Dibromofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorodifluoromethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	3/30/2006
Diesel range organics (DRO)	TX1005	Extractable Organics	NELAP	3/30/2006
Diethyl ether	EPA 8260	Volatile Organics	NELAP	3/30/2006
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	5/16/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-isopropylether (DIPE)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	5/16/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl sulfide	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dimethyldisulfide	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	5/16/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	5/16/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	3/30/2006
Diphenylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
EPH Aliphatic C19-C36	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
EPH Aliphatic C9-C18	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
EPH Aromatic C11-C22 Unadjusted	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
Ethane	RSK-175	Volatile Organics	NELAP	3/30/2006
Ethanol	EPA 8015	Volatile Organics	NELAP	5/7/2008
Ethanol	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	3/30/2006
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	3/30/2006
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	3/30/2006
Ethylbenzene	EPA 624	Volatile Organics	NELAP	5/16/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Ethylene	RSK-175	Volatile Organics	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	3/27/2013
Ethyl-t-butylether (ETBE)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Ferrous iron	SM 3500-Fe B (20th/21st Ed.)/UV-VIS	Metals	NELAP	7/30/2010
Fluoranthene	EPA 625	Extractable Organics	NELAP	5/16/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	5/16/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	5/16/2003
Fluoride	EPA 340.2	General Chemistry	NELAP	5/16/2003
Fluoride	EPA 9056	General Chemistry	NELAP	5/7/2008
Fluoride	SM 4500 F-C	General Chemistry	NELAP	5/16/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	5/7/2008
Free cyanide	EPA 9016	General Chemistry	NELAP	11/15/2012
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	3/30/2006
Hardness	EPA 130.2	General Chemistry	NELAP	5/16/2003
Hardness	SM 2340 B	General Chemistry	NELAP	5/16/2003
Hardness	SM 2340 C	General Chemistry	NELAP	4/17/2007
Hardness (calc.)	EPA 200.7	Metals	NELAP	7/30/2010
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	5/16/2003
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	5/16/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	3/30/2006
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	3/30/2006
Hexamethylphosphoramide (HMPA)	OVL HPLC07/HPLC-MS-MS	Extractable Organics	NELAP	7/30/2010
Hexamethylphosphoramide (HMPA)	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	5/16/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Iron	EPA 200.7	Metals	NELAP	5/16/2003
Iron	EPA 6010	Metals	NELAP	7/1/2003
Isobutane	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Isophorone	EPA 625	Extractable Organics	NELAP	5/16/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isoprene	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	5/7/2008
Isopropyl ether	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Isosafrole	EPA 8270	Extractable Organics	NELAP	3/30/2006
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	5/16/2003
Lactic acid	OVL HPLC03/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Lead	EPA 200.7	Metals	NELAP	5/16/2003
Lead	EPA 200.8	Metals	NELAP	5/16/2003
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lithium	EPA 200.7	Metals	NELAP	3/30/2006
Lithium	EPA 6010	Metals	NELAP	7/1/2003
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	7/30/2010
m+p-Xylenes	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Magnesium	EPA 200.7	Metals	NELAP	5/16/2003
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Malathion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Manganese	EPA 200.7	Metals	NELAP	5/16/2003
Manganese	EPA 200.8	Metals	NELAP	3/30/2006
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	3/30/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mercury	EPA 245.1	Metals	NELAP	5/16/2003
Mercury	EPA 7470	Metals	NELAP	5/16/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	3/30/2006
Methane	RSK-175	Volatile Organics	NELAP	3/30/2006
Methanol	EPA 8015	Volatile Organics	NELAP	5/7/2008
Methapyrilene	EPA 8270	Extractable Organics	NELAP	3/30/2006
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl acetate	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	5/16/2003
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl bromide (Bromomethane)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	5/16/2003
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Methyl isobutyl ketone (Hexone)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	3/30/2006
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	3/30/2006
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Methyl tert-butyl ether (MTBE)	OVL MSV10/GC-MS	Volatile Organics	NELAP	7/30/2010
Methyl tert-butyl ether (MTBE)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Methylcyclohexane	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Methylene chloride	EPA 624	Volatile Organics	NELAP	5/16/2003
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Molybdenum	EPA 200.7	Metals	NELAP	5/16/2003
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Naphthalene	EPA 625	Extractable Organics	NELAP	5/16/2003
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Naphthalene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
n-Butane	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	3/30/2006
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
n-Butylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Nickel	EPA 200.7	Metals	NELAP	5/16/2003
Nickel	EPA 200.8	Metals	NELAP	3/30/2006
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	3/30/2006
Nitrate	EPA 9056	General Chemistry	NELAP	5/7/2008
Nitrate	SM 4500-NO3 F	General Chemistry	NELAP	7/30/2010
Nitrate as N	EPA 300.0	General Chemistry	NELAP	5/16/2003
Nitrate as N	EPA 353.2	General Chemistry	NELAP	5/16/2003
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	5/16/2003
Nitrite	EPA 9056	General Chemistry	NELAP	5/7/2008
Nitrite	SM 4500-NO2-B	General Chemistry	NELAP	4/17/2007
Nitrite as N	EPA 300.0	General Chemistry	NELAP	5/16/2003
Nitrite as N	EPA 354.1	General Chemistry	NELAP	5/16/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	5/16/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	3/30/2006
Nitrocellulose	OVL KNITRO-C-W/UV-VIS	General Chemistry	NELAP	5/7/2008
Nitroglycerin	EPA 8330	Extractable Organics	NELAP	6/13/2007
Nitroglycerin	OVL HPLC02/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Nitroguanidine	OVL HPLC05/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	3/30/2006
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	5/16/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	3/30/2006
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	5/16/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	5/16/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/30/2010
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
n-Propylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	3/30/2006
Oil & Grease	EPA 1664A	General Chemistry	NELAP	5/16/2003
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	5/16/2003
Orthophosphate as P	EPA 365.2	General Chemistry	NELAP	5/16/2003
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	4/17/2007
o-Toluidine	EPA 8270	Extractable Organics	NELAP	3/30/2006
o-Xylene	EPA 8260	Volatile Organics	NELAP	3/30/2006
o-Xylene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Pentachlorobenzene	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Pentachloroethane	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	3/30/2006
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	5/16/2003
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentaerythritoltetranitrate (PETN)	EPA 8330	Extractable Organics	NELAP	6/13/2007
Pentamethylphosphoramide (PMPA)	OVL HPLC07/HPLC-MS-MS	Extractable Organics	NELAP	7/30/2010
Perchlorate	EPA 6850	General Chemistry	NELAP	7/30/2010
pH	EPA 150.1	General Chemistry	NELAP	5/16/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	4/17/2007
Phenacetin	EPA 8270	Extractable Organics	NELAP	3/30/2006
Phenanthrene	EPA 625	Extractable Organics	NELAP	5/16/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	5/16/2003
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Phosphorus, total	EPA 200.7	Metals	NELAP	7/30/2010
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	5/16/2003
Phosphorus, total	EPA 6010	Metals	NELAP	7/30/2010
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
p-Isopropyltoluene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Potassium	EPA 200.7	Metals	NELAP	5/16/2003
Potassium	EPA 6010	Metals	NELAP	7/1/2003
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	3/30/2006
Propane	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
Propionic acid (Propanoic acid)	OVL HPLC03/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Propylene	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
Propylene glycol	EPA 8015	Volatile Organics	NELAP	11/15/2012
Propyne	OVL RSK01/GC-FID	Volatile Organics	NELAP	5/7/2008
Pyrene	EPA 625	Extractable Organics	NELAP	5/16/2003
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyruvic acid	OVL HPLC03/HPLC-UV	Extractable Organics	NELAP	5/7/2008
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	3/30/2006
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	5/16/2003
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	4/17/2007
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	5/16/2003
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	4/17/2007
Residue-settleable	EPA 160.5	General Chemistry	NELAP	5/16/2003
Residue-settleable	SM 2540 F	General Chemistry	NELAP	4/17/2007
Residue-total	EPA 160.3	General Chemistry	NELAP	5/16/2003
Residue-total	SM 2540 B	General Chemistry	NELAP	4/17/2007
Residue-volatile	EPA 160.4	General Chemistry	NELAP	5/16/2003
Safrole	EPA 8270	Extractable Organics	NELAP	3/30/2006
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
sec-Butylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Selenium	EPA 200.7	Metals	NELAP	5/16/2003
Selenium	EPA 200.8	Metals	NELAP	5/16/2003
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	5/16/2003
Silica as SiO2	EPA 6010	Metals	NELAP	7/1/2003
Silica-dissolved	EPA 200.7	General Chemistry	NELAP	5/16/2003
Silicon	EPA 200.7	Metals	NELAP	5/16/2003
Silicon	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 200.7	Metals	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Silver	EPA 200.8	Metals	NELAP	3/30/2006
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	3/30/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	5/16/2003
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Strontium	EPA 200.7	Metals	NELAP	3/30/2006
Strontium	EPA 6010	Metals	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Styrene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Sulfate	EPA 300.0	General Chemistry	NELAP	5/16/2003
Sulfate	EPA 375.4	General Chemistry	NELAP	5/16/2003
Sulfate	EPA 9056	General Chemistry	NELAP	5/7/2008
Sulfide	EPA 376.1	General Chemistry	NELAP	5/16/2003
Sulfide	SM 4500-S F	General Chemistry	NELAP	4/17/2007
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	4/17/2007
T-amylmethylether (TAME)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/1/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
tert-Butylbenzene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	5/16/2003
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Tetrachlorvinphos (Stirophos, Gardona)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Tetraethyl pyrophosphate (TEPP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Tetrahydrofuran (THF)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Tetramethylphosphoramide (TMPA)	OVL HPLC07/HPLC-MS-MS	Extractable Organics	NELAP	7/30/2010
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	3/30/2006
Thallium	EPA 200.7	Metals	NELAP	5/16/2003
Thallium	EPA 200.8	Metals	NELAP	5/16/2003
Thallium	EPA 6010	Metals	NELAP	7/1/2003
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Tin	EPA 200.7	Metals	NELAP	5/16/2003
Tin	EPA 6010	Metals	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Titanium	EPA 200.7	Metals	NELAP	5/16/2003
Titanium	EPA 6010	Metals	NELAP	3/30/2006
Toluene	EPA 624	Volatile Organics	NELAP	5/16/2003
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Toluene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	5/7/2008
Total organic carbon	EPA 415.1	General Chemistry	NELAP	5/16/2003
Total organic carbon	EPA 9060	General Chemistry	NELAP	5/7/2008
Total organic carbon	SM 5310 C	General Chemistry	NELAP	4/17/2007
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	3/22/2011
Total Petroleum Hydrocarbons (TPH)	FL-PRO	General Chemistry	NELAP	7/30/2010
Total Petroleum Hydrocarbons (TPH)	TX1006	Extractable Organics	NELAP	5/7/2008
Total phenolics	EPA 420.1	General Chemistry	NELAP	5/16/2003
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/16/2003
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,2-Dichloroethylene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/16/2003
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropene	SM 6200 B	Volatile Organics	NELAP	11/15/2012
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	3/30/2006
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	5/16/2003
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	5/16/2003
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Trimethylphosphoramide (TriMPA)	OVL HPLC07/HPLC-MS-MS	Extractable Organics	NELAP	7/30/2010
Turbidity	EPA 180.1	General Chemistry	NELAP	5/16/2003
Uranium	EPA 200.8	Metals	NELAP	7/30/2010
Uranium	EPA 6020	Metals	NELAP	7/30/2010
Vanadium	EPA 200.7	Metals	NELAP	5/16/2003
Vanadium	EPA 200.8	Metals	NELAP	3/30/2006
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	EPA 6020	Metals	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Uncontrolled Copy

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl acetate	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Vinyl chloride	EPA 624	Volatile Organics	NELAP	5/16/2003
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	SM 6200 B	Volatile Organics	NELAP	11/15/2012
VPH Aliphatic C5-C8	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
VPH Aliphatic C9-C12	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
VPH Aromatic C9-C10	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
Weak acid dissociable cyanide	SM 4500 CN-I	General Chemistry	NELAP	5/7/2008
Xylene (total)	EPA 624	Volatile Organics	NELAP	5/16/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	SM 6200 B	Volatile Organics	NELAP	11/15/2012
Zinc	EPA 200.7	Metals	NELAP	5/16/2003
Zinc	EPA 200.8	Metals	NELAP	3/30/2006
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	3/30/2006
Zirconium	OVL ME600/ICP-AES	Metals	NELAP	7/30/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	3/1/2011
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	5/16/2003
1,3-Butadiene	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	3/1/2011
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	5/16/2003
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/16/2003
1,4-Dioxane (1,4-Diethyleneoxide)	OVL MSS01/GC-MS	Extractable Organics	NELAP	7/30/2010
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	5/16/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	5/16/2003
1-Methylnaphthalene	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/16/2003
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	5/16/2003
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	5/16/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	5/16/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	5/16/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	5/16/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/16/2003
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/16/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/16/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/16/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	5/16/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	5/16/2003
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	7/30/2010
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	5/16/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/16/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	5/16/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	5/16/2003
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/16/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	3/30/2006
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/16/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	5/16/2003
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	5/16/2003
a,a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Acetone	EPA 8260	Volatile Organics	NELAP	5/16/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/16/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	5/16/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/16/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	3/30/2006
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aluminum	EPA 6010	Metals	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Page 25 of 32

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aniline	EPA 8270	Extractable Organics	NELAP	5/16/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Antimony	EPA 6010	Metals	NELAP	5/16/2003
Antimony	EPA 6020	Metals	NELAP	5/16/2003
Aramite	EPA 8270	Extractable Organics	NELAP	5/16/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Aroclor-1262 (PCB-1262)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	11/15/2012
Aroclor-1268 (PCB-1268)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	11/15/2012
Arsenic	EPA 6010	Metals	NELAP	5/16/2003
Arsenic	EPA 6020	Metals	NELAP	5/16/2003
Atrazine	OVL MSS01/GC-MS	Extractable Organics	NELAP	7/30/2010
Barium	EPA 6010	Metals	NELAP	5/16/2003
Barium	EPA 6020	Metals	NELAP	3/30/2006
Benzaldehyde	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Benzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Benzidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	3/1/2011
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	5/16/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	5/16/2003
Beryllium	EPA 6010	Metals	NELAP	5/16/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Biphenyl	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	5/16/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	5/16/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	5/16/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Page 26 of 32

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Boron	EPA 6010	Metals	NELAP	5/16/2003
Bromide	EPA 9056	General Chemistry	NELAP	5/16/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Bromoform	EPA 8260	Volatile Organics	NELAP	5/16/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Cadmium	EPA 6010	Metals	NELAP	5/16/2003
Cadmium	EPA 6020	Metals	NELAP	3/30/2006
Calcium	EPA 6010	Metals	NELAP	5/16/2003
Caprolactam	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Carbazole	EPA 8270	Extractable Organics	NELAP	5/16/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/16/2003
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/16/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Chloride	EPA 9056	General Chemistry	NELAP	5/16/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Chloroform	EPA 8260	Volatile Organics	NELAP	5/16/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Chromium	EPA 6010	Metals	NELAP	5/16/2003
Chromium	EPA 6020	Metals	NELAP	3/30/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	3/30/2006
Chrysene	EPA 8270	Extractable Organics	NELAP	5/16/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/16/2003
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Cobalt	EPA 6010	Metals	NELAP	5/16/2003
Cobalt	EPA 6020	Metals	NELAP	3/30/2006
Copper	EPA 6010	Metals	NELAP	5/16/2003
Copper	EPA 6020	Metals	NELAP	3/30/2006
Cyclohexane	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Cyclohexanone	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	5/16/2003
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Dibromofluoromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	11/5/2004
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/16/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Di-isopropylether (DIPE)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Dimethyl sulfide	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Dimethyldisulfide	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	5/16/2003
Diphenylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
EPH Aliphatic C19-C36	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
EPH Aliphatic C9-C18	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
EPH Aromatic C11-C22 Unadjusted	MADEP-EPH (MA-EPH)	Volatile Organics	NELAP	11/15/2012
Ethanol	EPA 8015	Volatile Organics	NELAP	5/16/2003
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	3/30/2006
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/16/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	5/16/2003
Ethyl-t-butylether (ETBE)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Fluoride	EPA 9056	General Chemistry	NELAP	5/16/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	5/16/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	11/5/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Hexamethylphosphoramide (HMPA)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Ignitability	EPA 1010	General Chemistry	NELAP	5/16/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Iron	EPA 6010	Metals	NELAP	5/16/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	5/16/2003
Isoprene	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	5/16/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Isosafrole	EPA 8270	Extractable Organics	NELAP	5/16/2003
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Lead	EPA 6010	Metals	NELAP	5/16/2003
Lead	EPA 6020	Metals	NELAP	5/16/2003
Lithium	EPA 6010	Metals	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Magnesium	EPA 6010	Metals	NELAP	5/16/2003
Malathion	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Manganese	EPA 6010	Metals	NELAP	5/16/2003
Manganese	EPA 6020	Metals	NELAP	3/30/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Mercury	EPA 7471	Metals	NELAP	2/6/2004
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/16/2003
Methanol	EPA 8015	Volatile Organics	NELAP	5/16/2003
Methapyrilene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Methyl acetate	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/16/2003
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	5/16/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Methylcyclohexane	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/16/2003
Molybdenum	EPA 6010	Metals	NELAP	5/16/2003
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	3/30/2006
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
n-Hexane	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
Nickel	EPA 6010	Metals	NELAP	5/16/2003
Nickel	EPA 6020	Metals	NELAP	3/30/2006
Nitrate	EPA 9056	General Chemistry	NELAP	5/16/2003
Nitrite	EPA 9056	General Chemistry	NELAP	5/16/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	5/16/2003
Nitrocellulose	OVL KNITRO-C-S/UV-VIS	General Chemistry	NELAP	5/7/2008
Nitroglycerin	EPA 8330	Extractable Organics	NELAP	6/13/2007
Nitroguanidine	OVL HPLC05/HPLC-UV	Extractable Organics	NELAP	5/7/2008
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	5/16/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	5/16/2003
Oil & Grease	EPA 9071	General Chemistry	NELAP	11/15/2012
o-Toluidine	EPA 8270	Extractable Organics	NELAP	5/16/2003
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	5/16/2003
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Pentachloroethane	OVL MSS01/GC-MS	Extractable Organics	NELAP	5/7/2008
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
Pentaerythritoltetranitrate (PETN)	EPA 8330	Extractable Organics	NELAP	6/13/2007
Perchlorate	EPA 6850	General Chemistry	NELAP	7/30/2010
pH	EPA 9040	General Chemistry	NELAP	2/6/2004
pH	EPA 9045	General Chemistry	NELAP	2/6/2004
Phenacetin	EPA 8270	Extractable Organics	NELAP	5/16/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Phenol	EPA 8270	Extractable Organics	NELAP	5/16/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/1/2011
Phosphorus, total	EPA 6010	Metals	NELAP	7/30/2010
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Potassium	EPA 6010	Metals	NELAP	5/16/2003
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	5/16/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	3/30/2006
Propylene glycol	EPA 8015	Volatile Organics	NELAP	11/15/2012

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pyrene	EPA 8270	Extractable Organics	NELAP	5/16/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	5/16/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	5/16/2003
Reactive cyanide	Sec. 7.3 SW-846	General Chemistry	NELAP	5/16/2003
Reactive sulfide	Sec. 7.3 SW-846	General Chemistry	NELAP	5/16/2003
Safrole	EPA 8270	Extractable Organics	NELAP	5/16/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Selenium	EPA 6010	Metals	NELAP	5/16/2003
Selenium	EPA 6020	Metals	NELAP	11/7/2003
Silver	EPA 6010	Metals	NELAP	3/1/2011
Silver	EPA 6020	Metals	NELAP	3/30/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Sodium	EPA 6010	Metals	NELAP	5/16/2003
Strontium	EPA 6010	Metals	NELAP	5/16/2003
Styrene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Sulfate	EPA 9056	General Chemistry	NELAP	5/16/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	5/16/2003
Sulfotepp	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	5/16/2003
T-amylmethylether (TAME)	OVL MSV01/GC-MS	Volatile Organics	NELAP	5/7/2008
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/16/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Tetrachlorvinphos (Stirophos, Gardona)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Tetraethyl pyrophosphate (TEPP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/30/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	5/16/2003
Thallium	EPA 6010	Metals	NELAP	5/16/2003
Thallium	EPA 6020	Metals	NELAP	5/16/2003
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Tin	EPA 6010	Metals	NELAP	5/16/2003
Titanium	EPA 6010	Volatile Organics	NELAP	3/30/2006
Toluene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	7/30/2010
Total organic carbon	Lloyd Khan	General Chemistry	NELAP	5/7/2008
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	5/16/2003
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	3/30/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87551-27, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87551 **EPA Lab Code: OH00218** **(740) 373-4071**

E87551
Microbac Laboratories, Inc., Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total Petroleum Hydrocarbons (TPH)	TX1006	Extractable Organics	NELAP	3/30/2006
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/16/2003
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	5/16/2003
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/16/2003
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/16/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/16/2003
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/16/2003
Uranium	EPA 6020	Metals	NELAP	7/30/2010
Vanadium	EPA 6010	Metals	NELAP	5/16/2003
Vanadium	EPA 6020	Metals	NELAP	3/30/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/16/2003
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/16/2003
VPH Aliphatic C5-C8	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
VPH Aliphatic C9-C12	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
VPH Aromatic C9-C10	MADEP-VPH (MA-VPH)	Volatile Organics	NELAP	11/15/2012
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/16/2003
Zinc	EPA 6010	Metals	NELAP	5/16/2003
Zinc	EPA 6020	Metals	NELAP	3/30/2006
Zirconium	OVL ME600/ICP-AES	Metals	NELAP	7/30/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

APPENDIX D

KEY PERSONNEL
JOB DESCRIPTIONS

Job Description
Ohio Valley Laboratory

POSITION: Managing Director/Laboratory Director
CLASSIFICATION: Exempt
REPORTS TO: President/CEO

Position Summary and Interrelationships:

The Managing Director reports to the President of Microbac Laboratories, Inc. and manages the business development and technical staff presented in the organization charts of this section (Figures 1 and 2). The Managing Director has primary responsibility for assuring compliance with ISO/IEC 17025:2005. The Technical Director/QAO, Senior Chemist – QA/QC, and all departmental supervisors will assist in the implementation of the specific requirements of this standard. The Managing Director also serves as the Laboratory Director, and has general responsibility for business development and operations. Included are strategic planning, resource allocation and profit and loss for the analytical division as a whole. It is the Managing Director's responsibility to obtain and develop both financial and personnel resources in an effort to maintain quality services and to match resources with the market demands. Additional responsibilities include proposal development and review, production management. Other duties include oversight of the laboratory safety and waste management programs, certifying that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited, monitoring standards of performance in quality control and quality assurance, and ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. The Managing Director has authority to:

Responsibilities and Authorities:

1. Reviews and approves quality policies, the quality manual, and laboratory standard operating procedures (SOP).

2. Maintains SOP documents file in hard copy and computer format and distributes revisions to the various laboratory departments.

3. Reviews laboratory quality assurance data, laboratory records for precision and accuracy, statistical control limits, and method detection limits, limits of detection (LOD), and limits of quantitation (LOQ).

4. Reviews statistical warning and control limits and analytical control charts.

5. Performs internal system and technical audits and recommends corrective action plans for areas of deficiency.

6. Introduces internal “blind” performance evaluation samples into the LIMS and reports results of studies to management.

7. Prepares monthly quality assurance reports to management.

8. Communicates any quality concerns to the Technical Director and Managing Director.

9. Communicates the QA program objectives and requirements to the laboratory staff and clients.

10. Schedules and orders proficiency testing (PT) samples and prepares reports for auditing or certifying agencies.

11. Reviews PT data and scores, communicates unacceptable analytical performance to the Managing Director, and prepares corrective action plans for auditing or certifying agencies.

12. Implements system for maintaining calibration records of balances, thermometers, etc.

13. Maintains all records of laboratory certification

14. Assists clients and field operations in the preparation of specific QA Project Plans (QAPPs).

15. Assists in the development of technical training procedures and performs staff training.

16. Reviews and approves laboratory and analyst demonstrations of capability.

17. Performs other duties as directed by the Managing Director.

18. Performs audits of sub-contractor laboratories as necessary.

19. Develops and maintains a system of quality control standards, which are independent of calibration standards.

20. Assists the Managing Director in development and maintaining the health and safety program and a waste disposal system.

21. Approve policies and procedures.

22. Implementation and maintenance of the quality management system.

23. Review and approve contracts and tenders.

24. Approve certificates-of-analysis (laboratory report).

25. Evaluating staff training effectiveness.

26. Stopping nonconforming work.

27. Resuming nonconforming work.

28. Quality Planning (Management Review).

Education/Experience Requirements:

1. BS/BA Degree - Chemistry Minor (20 semester hours) and ten years of laboratory experience.

or 2. BS/BA Degree – Chemistry Major (30 semester hours) and five years of laboratory experience.

or 3. MS/PhD Degree – Chemistry Major and three years of laboratory experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Technical Director
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Technical Director reports to the Managing Director and is responsible for the following departments and programs: QA/QC, IT, Support Services, and methods development. The Technical Director directs the activities of the Quality Assurance Officer (QAO), the Senior Chemist for Methods Development, the Computer Programmers and IT support staff, and the Support Services Supervisor.

Responsibilities and Authorities:

1. Directs/coordinates the QA/QC program with the assistance of the QAO.

2. Directs/coordinates the development and maintenance of the Laboratory Information Management System (LIMS), the network fileservers and work stations, and all hardware software applications.

3. Evaluates/coordinates the development of all new laboratory technology and new analytical methods with assistance of the Senior Chemist.

4. Directs/coordinates the activities of the Support Services department including sample login, sample custody, building maintenance, shipping/receiving, and waste management.

5. Reviews laboratory quality assurance data, laboratory records for precision and accuracy, statistical control limits, and method detection limits, limits of detection (LOD), and limits of quantitation (LOQ).

6. Performs internal system and technical audits and recommends corrective action plans for areas of deficiency.

7. Communicates any quality concerns to the Technical Director and Managing Director.

8. Communicates the QA program objectives and requirements to the laboratory staff and clients.

9. Assists clients and field operations in the preparation of specific QA Project Plans (QAPPs).

10. Assists in the development of technical training procedures and performs staff training.

11. Develops and maintains a system of quality control standards, which are independent of calibration standards.

12. Approves technical policies and procedures

13. Implementation and maintenance of the quality management system

14. Review and approve contracts and tenders

15. Approves certificates-of-analysis (laboratory reports)

16. Evaluates staff training effectiveness

17. Stops nonconforming work

18. Resumes nonconforming work

19. Performs internal audits

20. Performs other duties as directed by the Managing Director.

Education/Experience Requirements:

1. BS/BA Degree - Chemistry Minor (20 semester hours) and ten years of laboratory experience.

or 2. BS/BA Degree – Chemistry Major (30 semester hours) and five years of laboratory experience.

or 3. MS/PhD Degree – Chemistry Major and three years of laboratory experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Quality Assurance Officer
CLASSIFICATION: Exempt
REPORTS TO: Technical Director

Position Summary and Interrelationships:

The Quality Assurance Officer (QAO) reports to the Managing Director and functions independently of the laboratory operations. The QAO supervises the QA Administrative Assistant and coordination with the Document Control Officer. The QAO is responsible for development, implementation, and oversight of the laboratory Quality Assurance Program. It is the QAO's responsibility to assess and ensure the quality of all work performed in the laboratory.

Responsibilities and Authorities:

1. Reviews and approves quality policies, the quality manual, and laboratory standard operating procedures (SOP).

2. Maintains SOP documents file in hard copy and computer format and distributes revisions to the various laboratory departments.

3. Reviews laboratory quality assurance data, laboratory records for precision and accuracy, statistical control limits, and method detection limits, limits of detection (LOD), and limits of quantitation (LOQ).

4. Reviews statistical warning and control limits and analytical control charts.

5. Performs internal system and technical audits and recommends corrective action plans for areas of deficiency.

6. Introduces internal "blind" performance evaluation samples into the LIMS and reports results of studies to management.

7. Prepares monthly quality assurance reports to management.

8. Communicates any quality concerns to the Technical Director and Managing Director.

9. Communicates the QA program objectives and requirements to the laboratory staff and clients.

10. Schedules and orders proficiency testing (PT) samples and prepares reports for auditing or certifying agencies.

11. Reviews PT data and scores, communicates unacceptable analytical performance to the Managing Director, and prepares corrective action plans for auditing or certifying agencies.

12. Implements system for maintaining calibration records of balances, thermometers, etc.

13. Maintains all records of laboratory certification.

14. Assists clients and field operations in the preparation of specific QA Project Plans (QAPPs).

15. Assists in the development of technical training procedures and performs staff training.

16. Reviews and approves laboratory and analyst demonstrations of capability.

17. Performs other duties as directed by the Managing Director.

18. Performs audits of sub-contractor laboratories as necessary.

19. Develops and maintains a system of quality control standards, which are independent of calibration standards.

20. Assists the Managing Director in development and maintaining the health and safety program and a waste disposal system.

21. Approve policies and procedures.

22. Implementation and maintenance of the quality management system.

23. Evaluating staff training effectiveness.

24. Has stop work authority.

25. Monitors the laboratory's corrective action processes.

Education/Experience Requirements:

1. BS/BA Degree - Chemistry Minor (20 semester hours) and ten years of laboratory experience.

or 2. BS/BA Degree – Chemistry Major (30 semester hours) and five years of laboratory experience.

or 3. MS/PhD Degree – Chemistry Major and three years of laboratory experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Conventionals Laboratory Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Conventionals/Wet Chemistry Supervisor reports to the Managing Director. The Conventionals/Wet Chemistry Supervisor is responsible for coordinating the production and quality control of all work involving preparation and analysis of samples by wet chemical methods in accordance with company policies and Standard Operating Procedures (SOP). General duties include implementation of laboratory safety program in the Wet Chemistry department, implementation of the laboratory quality control program for wet chemical analysis in accordance with company SOPs and prudent laboratory practices, scheduling production of routine and non-routine work, supervising all technicians/analysts involved in sample preparation, standard preparation, and instrumental analyses.

Responsibilities and Authorities:

1. Schedules and reviews work of all analysts and lab assistants
2. Implements the laboratory Chemical Hygiene Plan and all laboratory SOPs
3. Schedules instrument maintenance
4. Performs employee orientation, training, and performance reviews
5. Performs QC review of all departmental data
6. Maintains inventory of supplies and chemicals
7. Performs general review of departmental data deliverables (Level 4)
8. Takes corrective action on all out-of-control analyses and submits reports to the QC Supervisor and Laboratory Manager
9. Evaluates staffing and instrumentation requirements and makes recommendations

to management.

10. Approves policies and procedures
11. Implementation and maintenance of the quality management system
12. Approves departmental data and reports
13. Evaluating staff training effectiveness

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of wet lab experience.

or 2. BS/BA Degree with chemistry major and three years of wet lab experience.

or 3. BS/BA Degree with Chemistry minor and four years of wet lab experience.

or 4. AS/BA Degree in science and 8-10 years of wet lab experience.

Job Description
Ohio Valley Laboratory

POSITION: Metals Laboratory Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Metals Laboratory Supervisor reports to the Managing Director. The Metals Laboratory Supervisor is responsible for coordinating the production and quality control of all work involving digestion and analysis of samples by AA and ICP methods in accordance with company policies and Standard Operating Procedures (SOP). General duties include implementation of laboratory safety program in the metals department, implementation of the laboratory quality control program for metals analyzed in accordance with company SOPs and prudent laboratory practices, scheduling, production of routine and non-routine work, supervising all technicians/analysts involved in standard preparation, sample digestion and instrumental analyses. Other duties include the scheduling of routine maintenance and emergency service for departmental instrumentation, preparing daily work schedules for full and part-time employees, monitoring departmental backlogs, turnaround time, priority analysis deadlines, reviewing and approving data generated by the metals department.

Responsibilities and Authorities:

1. Schedules and reviews work of all analysts and lab assistants;

2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, and all laboratory SOPs;

3. Schedules instrument maintenance;

4. Performs employee orientation, training, and performance reviews;

5. Performs QC review of all departmental data;

6. Maintains inventory of supplies and chemicals;

7. Performs general review of departmental data deliverables (Level 4);

8. Takes corrective action on all out-of-control analyses and submits reports to the QC Supervisor and Laboratory Manager;

9. Evaluates staffing and instrumentation requirements and makes recommendations to management;

10. Approve policies and procedures

11. Implementation and maintenance of the quality management system

12. Approve departmental reports and data packages.

13. Evaluating staff training effectiveness

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of environmental laboratory experience

or 2. BS/BA Degree with chemistry major and four years of environmental laboratory experience

or 3. BS/BA Degree with Chemistry minor and five years of environmental laboratory experience

or 4. AS/BA Degree in science and eight to ten years of environmental laboratory experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Semivolatile Organics Laboratory Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Semivolatile Organics Supervisor reports to the Managing Director. The Semivolatile Organics Supervisor is responsible for coordinating the production and quality control of all work involving instrumental analysis of samples by HPLC, IC, GC/FID, GC/ECD, and GC/MS employing semivolatile methods in accordance with company policies and Standard Operating Procedures (SOP). General duties include implementation of laboratory safety program in the department, implementation of the laboratory quality control program for semivolatile organics analyzed in accordance with company SOPs and prudent laboratory practices. Other duties include scheduling production of routine and non-routine work, supervising technicians/analysts involved in standard preparation and instrumental analyses, scheduling routine maintenance and emergency service for departmental instrumentation, preparing daily work schedules of full and part-time employees, monitoring departmental backlogs, turnaround time, and priority analysis deadlines and reviewing and approving data generated by the department.

Responsibilities and Authorities:

1. Schedules and reviews work of all analysts and lab assistants;

2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, and all laboratory SOPs;

3. Schedules instrument maintenance;

4. Performs employee orientation, training, and performance reviews;

5. Performs QC review of all departmental data;

6. Maintains inventory of supplies and chemicals;

7. Performs general review of departmental data deliverables (Level 4);

8. Takes corrective action on all out-of-control analyses and submits reports to the QC Supervisor and Laboratory Manager;

9. Evaluates staffing and instrumentation requirements and makes recommendations to management;

10. Approve policies and procedures;

11. Implementation and maintenance of the quality management system;

12. Approves departmental data and reports;

13. Evaluating staff training effectiveness.

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of GC/MS experience.

or 2. BS/BA Degree with chemistry major and three years of GC/MS experience.

or 3. BS/BA Degree with Chemistry minor and four years of GC/MS experience.

or 4. AS/BA Degree in science and 8-10 years of GC/MS experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Volatile Organics Laboratory Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Volatile Organics Supervisor reports to the Managing Director. The Volatile Organics Supervisor is responsible for coordinating the production and quality control of all work involving preparation and analysis of samples for volatile organics analysis (VOA) by GC/PID/FID/TCD and GC/MS methods in accordance with company policies, client QAPPs, and Standard Operating Procedures (SOP). Specific duties include, but are not limited to, implementation of laboratory safety program in the department, implementation of the laboratory quality control program, scheduling production of routine and non-routine work, supervising all technicians/analysts, instrumental analyses, scheduling routine maintenance and emergency service for departmental instrumentation, preparing daily work schedules for employees, monitoring departmental backlogs, turnaround time, and priority analysis deadlines and reviewing and approving data generated by the department.

Responsibilities and Authorities:

1. Schedules and reviews work of all analysts and lab assistants
2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, and all laboratory SOPs
3. Schedules instrument maintenance
4. Performs employee orientation, training, and performance reviews
5. Performs QC review of all departmental data
6. Maintains inventory of supplies and chemicals
7. Performs general review of departmental data deliverables (Level 4)

8. Takes corrective action on all out-of-control analyses and submits reports to the QC Supervisor and Laboratory Manager

9. Evaluates staffing and instrumentation requirements and makes recommendations to management

10. Approve policies and procedures

11. Implementation and maintenance of the quality management system

12. Approves departmental reports and data packages

13. Evaluating staff training effectiveness

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of GC/MS experience.

or 2. BS/BA Degree with chemistry major and three years of GC/MS experience.

or 3. BS/BA Degree with Chemistry minor and four years of GC/MS experience.

or 4. AS/BA Degree in science and 8-10 years of related experience.

Job Description
Ohio Valley Laboratory

POSITION: Extraction Laboratory Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Organic Sample Preparation Supervisor reports to the Managing Director. The Organic Sample Preparation Supervisor is responsible for all organic extractions. He ensures that all samples for organics analysis are prepared in accordance with the appropriate USEPA methods, that appropriate QC samples are prepared, and that all samples are extracted within the appropriate regulatory holding times. Other responsibilities include preparation of samples for extractable organic analysis, methods development for unusual matrices and preparation of standards, sample cleanup as required, TCLP extraction procedures, ordering of supplies and standards for extraction laboratory, and maintenance of extraction lab documentation.

Responsibilities and Authorities:

1. Schedules and reviews work of all analysts and lab assistants;
2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, and all laboratory SOPs;
3. Schedules instrument maintenance;
4. Performs employee orientation, training, and performance reviews;
5. Performs QC review of all departmental data;
6. Maintains inventory of supplies and chemicals;
7. Performs general review of departmental data deliverables (Level 4);
8. Takes corrective action on all out-of-control analyses and submits reports to the QC Supervisor and Laboratory Manager;
9. Evaluates staffing and instrumentation requirements and makes recommendations

to management;

10. Approve policies and procedures;
11. Implementation and maintenance of the quality management system;
12. Approves departmental data and reports;
13. Evaluating staff training effectiveness.

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of related experience.

or 2. BS/BA Degree with chemistry major and three years of related experience.

or 3. BS/BA Degree with Chemistry minor and four years of related experience.

or 4. AS/BA Degree in science and 8-10 years of related experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Support Services Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Technical Director

Position Summary and Interrelationships:

The Support Services Supervisor reports to the Technical Director. The Support Services Supervisor is responsible for the non-analytical operations of the laboratory. General duties include coordinating and supervising all activities associated with login, sample containers and kit preparation, sample disposal, and the Microbac courier services. Other responsibilities include supervision of shipping/receiving, peer review of login, logging, temperature blanks and coordination of the laboratory waste management system.

Responsibilities and Authorities:

1. Schedules or coordinates, and reviews as necessary, the work customer service representatives, sample custodians, and support service assistants.

2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, Waste Management Plan, and all other departmental standard operating procedures.

3. Assists in the development and regular revision of pertinent standard operating procedures.

4. Performs departmental employee orientation, training, and performance reviews.

5. Schedules Support Services vehicle maintenance.

6. Coordinates or schedules Microbac sample courier service.

7. Maintains inventory of supplies for sample kits.

8. Supervises the laboratory waste management system, including the proper disposal of all sample residuals in accordance with company policies and pertinent government regulations.

9. Assists in the maintenance of the physical facility through the efforts of departmental assistants or outside contractors.

10. Assists in proposal development, writing quotations, customer service activities, and selected project and account management.

11. Performs other duties as required in special projects or emergencies.

10. Approve policies and procedures

11. Implementation and maintenance of the quality management system

12. Evaluating staff training effectiveness

Education/Experience Requirements:

1.0 AS/BA Degree in science and 3-5 years of related experience.

or 2. 5 -10 years of laboratory experience.

Uncontrolled Copy

Job Description
Ohio Valley Laboratory

POSITION: Chemists/Data Specialist
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Chemist/Data Specialist reports to the Managing Director. The Chemist/Data Specialists (CDS) perform important functions at the beginning and end of the process chain. At project inception, they critically review all data quality objectives, EDD specifications, and regulatory/program requirements and customize instructions to the laboratory and MIS staff accordingly. Method builds, new regulatory/field applications and other non-routine requests are researched and processed by the CDS, with the support of senior management. All finalized data packages, including electronic data deliverables, are routed back to the CDS for a final review of completeness, accuracy and assured compliance to client specification. Post delivery, the CDS fields all technical questions associated with the data package, its validation and integration. Depending on the scale and scope of a project, the CDS may be assigned complete project management responsibilities. As project manager, they coordinate all facets of supporting services and are the client's primary contact for the duration of the project. In this role, the CDS initiates electronic reporting in a variety of media and configurations, including portal uploads and electronic data verifications.

Responsibilities and Authorities:

1. All technical review for proposals & new awards (methods, reporting limits, compound lists, QC). Creates "drop-in" technical narratives and tables for proposals based on technical review of RFP/QAP. Supports sales channels through clarification of discrepancies between lab capabilities and project specifications at proposal stage.

2. All workorders, kit requests and project set up in LIMS (p-keys, account level pricing, etc)

3. All interface with MIS for EDD development and progress tracking through final EDD design.

4. All interface with sampling crew as required

5. All subcontracting arrangements

6. Communication of client requirements to lab as well as communication of lab issues to the client

7. Review of all folders from log-in, resolution of anomalies with client and submittal of sample receipt acknowledgements.

8. Expediting work to insure turn around times (TAT) (no penalties, capture all premiums)

9. All forms of data review (complete, correct and compliant with contract provisions) including EDD

10. Interface with validators and auditors

11. Participation in on-going process improvement initiatives, and subsequent cross-training. Occasional research in methods development and changing regulations to support future product line expansions.

12. Some travel required as technical sales support (including some overnights) and for client-required project kick-offs.

13. Accept new work within the capacity guidelines of the Managing Director

14. Identify nonconforming work and potential preventative actions

15. Process client complaints

Education/Experience Requirements:

1. BS/BA Degree – Chemistry Minor (20 semester hours)

or 2. BS/BA Degree – Natural Science Major
1 year of related lab experience
8 hours college chemistry

Job Description
Ohio Valley Laboratory

POSITION: Client Services Supervisor
CLASSIFICATION: Exempt
REPORTS TO: Managing Director

Position Summary and Interrelationships:

The Client Services Supervisor reports to the Managing Director. This position supervises, coordinates, and reviews the tasks of the Client Services Specialists (CSS) and related departmental activities. In addition to supervision, the position has the same duties, technical requirements, responsibilities, and authorities as Chemists/Data Specialists.

Responsibilities and Authorities:

1. Coordinates, schedules and reviews work of Client Service Representatives

2. Implements the laboratory Chemical Hygiene Plan, Comprehensive QA Plan, and all departmental SOPs

3. Performs employee orientation, training, and performance reviews

4. Evaluates staffing requirements and makes recommendations to management

5. Approve policies and procedures

6. Implementation and maintenance of the quality management system

7. Evaluating staff training effectiveness

8. Performs technical review for proposals & new awards (methods, reporting limits, compound lists, QC). Creates “drop-in” technical narratives and tables for proposals based on technical review of RFP/QAP. Supports sales channels through clarification of discrepancies between lab capabilities and project specifications at proposal stage.

9. Performs workorders, kit requests and project set up in LIMS (p-keys, account level pricing, etc)

10. Interfaces with MIS for EDD development and progress tracking through final EDD design.

11. Interfaces with sampling crew as required

12. Subcontracting arrangements

13. Communication of client requirements to lab as well as communication of lab issues to the client

14. Review folders from log-in, resolution of anomalies with client and submittal of sample receipt acknowledgements.

15. Expediting work to insure turn around times (TAT) (no penalties, capture all premiums)

16. Performing data review (complete, correct and compliant with contract provisions) including EDD

17. Interfaces with validators and auditors

18. Participation in on-going process improvement initiatives, and subsequent cross-training. Occasional research in methods development and changing regulations to support future product line expansions.

19. Some travel required as technical sales support (including some overnights) and for client-required project kick-offs.

20. Accept new work within the capacity guidelines of the Managing Director

21. Identify nonconforming work and potential preventative actions

22. Process client complaints

Education/Experience Requirements:

1. Advanced Degree in chemistry and two years of experience.
or 2. BS/BA Degree with chemistry major and three years of experience.
or 3. BS/BA Degree with Chemistry minor and four years of experience.
or 4. AS/BA Degree in science and 8-10 years of related experience.

Job Description
Ohio Valley Laboratory

POSITION: Client Services Specialist
CLASSIFICATION: Non-Exempt
REPORTS TO: Client Services Supervisor

Position Summary and Interrelationships

Client Services Specialist (CSS) have two primary roles. They function as project/account managers on routine and repeat events with an assigned list of clients. They also assist Chemists/Data Specialists in the logistical coordination of major projects and provide administrative support for those projects. Chemists/Data Specialists provide technical/chemistry support to the CSS's on an "as needed" basis.

Responsibilities and Authorities

1. Primary liaison with select list of clients. Gathers and transmits to the laboratory all essential order entry information via work orders and account/project setup in LIMS. Selects and issues orders to subcontracting labs as required. Performs all routine customer service functions for assigned clients (phones, e-mails, etc) and provides additional coverage as assigned backup to other CSS's.

2. Reviews in-process master folders following sample receipt from log-in. Reconciles any anomalies with client. Enters field data where required. Sends sample receipt confirmations to clients as required by contracts. Approves release of samples to the lab through a LIMS change in status.

3. Reviews deliverables post production as final check of case completeness, correctness and compliance with specifications. Deliverables include required EDD's, approval invoices and subcontracted reports.

4. Performs invoicing, transmittal and AR functions with accuracy and on-time.

5. Assists in the development and implementation of process changes to maximize efficiency, conserve resources and promote optimal customer care as directed by Client Services Supervisor.

6. Requires strong attention to detail, ability to work under pressure, excellent oral and written communication skills, computer aptitude, and superior interpersonal

skills to maintain established standards for client care and interaction with co-workers.

7. Work hours will have limited flexibility to insure coverage. Routine hours for this position have been established as 8-5. On occasion, the position may require you to stay later than 5:00 p.m. as the workload dictates.

Education/Experience Requirements:

1. AS Degree in Natural Science

or 2. High School Diploma
2 years related lab experience

ATTACHMENT C
MWH EDD FORMAT

MWH ELECTRONIC DATA DELIVERABLE REQUIREMENTS
(rev. March 2011)

Field Name	Format	Description	Constraints	Comments
AFIID	C5 (Valid Value List)	USAF Installation Code	Required	
LABSAMPID	C20	Lab Sample Identifier	Required	
LOCID	C15	Location Name	Required	
MATRIX	C2 (Valid Value List)	Sampling Matrix	Required	
SBD	N8,2	Sample Beginning Depth	Required	
SED	N8,2	Sample Ending Depth	Required	
LOGDATE	D11 (DD-MMM-YYYY)	Sample Date	Required	
LOGTIME	C4 (HHMM)	Sample Time	Required	
LABCODE	C4 (Valid Value List)	USAF Lab Identifier	Required	
SACODE	C2 (Valid Value List)	Sample Type	Required	
SAMPNO	N2,0	Sample Number	Required	
ANMCODE	C7 (Valid Value List)	Analytical Method Code	Required	
EXMCODE	C7 (Valid Value List)	Extraction Method Code	Required	
EXTDATE	D11 (DD-MMM-YYYY)	Extraction Date	Conditional	
EXTTIME	C4 (HHMM)	Extraction Time	Conditional	
ANADATE	D11 (DD-MMM-YYYY)	Analysis Date	Required	
ANATIME	C4 (HHMM)	Analysis Time	Required	
LABLOTCTL	C10	Laboratory Preparation Batch ID	Required	
PARLABEL	C12 (Valid Value List)	Parameter Label	Required	
PARVAL	N16,6	Measured Concentration	Required	
UNITS	C10 (Valid Value List)	Units of Measure	Required	
PARVQ	C2 (Valid Value List)	Parameter Value Qualifier	Required	
BASIS	C1 (Valid Value List)	Wet or Dry Basis (for soil data)	Required	
DILUTION	N16,6	Dilution Factor	Required	
LOGCODE	C4 (Valid Value List)	Logging Company Code	Required	Should equal "MWSL"
SMCODE	C2 (Valid Value List)	Sampling Method Code	Required	
FLDSAMPID	C30	Field Sample ID	Required	
COCID	C12	Chain of Custody ID	Optional	
COOLER	C10	Field Cooler ID	Optional	
ABLOT	C8 (DDMMYYNN)	Ambient Blank Lot ID	Optional	
EBLOT	C8 (DDMMYYNN)	Equipment Blank Lot ID	Optional	
TBLOT	C8 (DDMMYYNN)	Trip Blank Lot ID	Optional	
PARUN	N12,4	Uncertainty	Conditional	
PRECISION	N1	Primary Value Precision	Required	
EXPECTED	N16,6	Expected Value (for spiked samples)	Conditional	
EVPREC	N1	Expected Value Precision	Conditional	
MDL	N16,6	Method Detection Limit	Conditional	
RL	N16,6	Reporting Limit	Conditional	
LCHMETH	C7 (Valid Value List)	Leachate Method	Required	
RUN_NUMBER	N2	Run Number	Required	
LCHDATE	D11 (DD-MMM-YYYY)	Leachate Date	Conditional	
LCHTIME	C4 (HHMM)	Leachate Time	Conditional	
LCHLOT	C10	Leachate Lot	Conditional	
ANALOT	C10	Analytical Lot	Required	
PRCCODE	C3 (Valid Value List)	Analyte Type	Required	
CALREFID	C10	Calibration Reference	Optional	
VQ_1C	C2 (Valid Value List)	PARVQ of first Column	Optional	
VAL_1C	N16,6	Result of first Column	Optional	
FCVALPREC	N1	Precision of first Column	Optional	
VQ_CONFIRM	C2 (Valid Value List)	PARVQ of Confirmation Column	Optional	
VAL_CONFIRM	N16,6	Result of Confirmation Column	Optional	
CNFVALPREC	N1	Precision of Confirmation Column	Optional	
LAB_DQT	C5 (Valid Value List)	Type of Data Qualifier System	Optional	To be provided by MWH project chemist
LAB_QC_FLAG	C6 (Valid Value List)	Laboratory Flags	Conditional	Previously referred to as QAPP_FLAG
REC_DATE	D11 (DD-MMM-YYYY)	Date Sample Received in Lab	Required	
COMPNAME	C50	Compound Name	Required	Not an ERPIMS field

MWH ELECTRONIC DATA DELIVERABLE REQUIREMENTS
(rev. March 2011)

Field Name	Format	Description	Constraints	Comments
CASNUMBER	C10	Chemical Abstract Service No.	Optional	Not an ERPIMS field
SPIKE_ADDED	N16,6	Concentration Spiked	Conditional	
PRIME_FLAG	C6	Validation Qualifiers	Leave Null	Previously referred to as EPA_FLAGS
LOWER_ACCURACY	N14,2	Minimum Precision Control Limit	Conditional	Previously referred to as LOW_LIMIT
UPPER_ACCURACY	N14,2	Maximum Precision Control Limit	Conditional	Previously referred to as HIGH_LIMIT
UPPER_RPD	N14,2	Maximum RPD control limit	Conditional	Previously referred to as RPD
PERCENT_RECOVERY	N14,2	Percent Recovery	Conditional	New field requirement

Notes:

An Excel file is preferred, however, a single delimited text file (.csv) is acceptable. If a csv file is used the delimiters may be commas with quote text qualifiers ("VAL1","VAL2") or tabs.

It is required that all fields be provided in the order listed above, a place-holder must be provided for any null entries.

The latest ERPIMS DLH (Data Loading Handbook) version must be used to obtain valid values.

The format column lists the data type ([N]umber, [C]haracter, [D]ate) followed by the number of allowed characters. For number data types the number of decimal places is indicated by the number following the comma. Additional format constraints are listed within parenthesis.

The PARVAL field is the actual concentration (not percent recovery) unless the PRCCODE = STD (surrogate results).

The LOGDATE and LOGTIME fields are requred, for LABQC samples use the earlier of the EXMDATE/TIME or ANMDATE/TIME.

Conditional Constraints:

FLDSAMPID should repeat the LABSAMPID for LABQC samples.

EXTDATE, EXTTIME are required unless the EXMCODE = "NONE"

LCHDATE, LCHTIME and LCHLOT are required unless the LCHMETH = "NONE"

PERCENT RECOVERY is required whenever the SACODE = MS,SD,BS, or BD. It should not be populated when the PRCCODE = STD (surrogates).

PARUN is required only when PRCCODE = RN (Radionuclides); it should not be populated in any other instance.

MDL and RL are required for all results unless the PRCCODE = MI,PM,BAC or STD. Do not populate MDL and RL for TICs (PARVQ = TI). When QSM is used RL = LOQ and MDL = LOD.

EXPECTED is required when SACODE <> 'N'. EXPECTED is required for records with a PRCCODE = STD regardless of SACODE. EXPECTED is reported in the same units as PARVAL.

EVPREC should be populated each time an EXPECTED value is required.

LAB_QC_FLAG is required when a flag is needed.

SPIKE ADDED, LOWER AND UPPER ACCURACY are required whenever the SACODE = MS,SD,BS, or BD. It is also required whenever the PRCCODE = STD.

UPPER_RPD is required for all records with a SACODE of LR,SD,BD,FR,or FD unless the PRCCODE = STD.

ATTACHMENT D
SAMPLE LDC REPORT

Report# #######

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: Southeast Idaho Mine Sites

Report Date: November 1, 2008

Matrix: Water

Parameters: Metals by ICPMS SW-846 Method 6020A

Validation Level: EPA Level IV

Laboratory: Microbac

Sample Delivery Group (SDG): 44433

Sample Identification	Collection Date	Laboratory Identification
TSB-GJ-08-10	9/15/08	44433-01
TSB-GJ-08-20	9/15/08	44433-02
TSB-GJ-08-30	9/15/08	44433-03
TSB-GJ-08-40	9/15/08	44433-04
TSB-GJ-08-10MS	9/15/08	44433-05
TSB-GJ-08-10MSD	9/15/08	44433-06

Introduction

This data review covers 6 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA SW 846 Method noted below:

- Method 6020A ICPMS: Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, and Zinc, and Zirconium.

This review follows the specific guidance in the QAPP Addendum (MWH 2009) to the project SAP (April 2004) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following are not data qualifiers but are provide for the purpose of evaluating the laboratory's performance:

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

1 Holding Time
2 Sample Preservation (including receipt temperature)
3 Sample Custody
4 Missing Deliverable
5 ICPMS Tune
6 Initial Calibration
7 Initial Calibration Verification
8 Continuing Calibration Verification
9 Low-Level Calibration Check Sample
10 Calibration Blank
11 Laboratory or Preparation Blank
12 ICPMS or ICP Interference Check Standard
13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
14 Laboratory Control Sample Precision
15 Laboratory Duplicate Precision
16 Matrix Spike or Matrix Spike Duplicate Recovery
17 Matrix Spike/Matrix Spike Duplicate Precision
18 ICPMS or ICP Serial Dilution
19 ICPMS Internal Standard
20 Field Replicate Precision
21 Equipment Rinsate Blank
22 Linear Range Exceeded
23 Other reason

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections preformed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements were met: 6 months for water and soil (note NIST soil standard reference samples are valid for up to 3 years).

All samples were received intact with proper preservation (pH < 2 for water).

II. ICP-MS Tune Analysis

ICP MS Tuning was performed by the laboratory. All isotopes in the tuning solution mass resolution were within 0.1 amu.

The percent relative standard deviations (%RSD) of all isotopes in the tuning solution were less than or equal to 5.0%.

III. Calibration

An initial calibration was performed each day of analysis. The frequency and analysis criteria (90-110%) of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

The low-level initial calibration verification (LLICV) and low-level continuing calibration verifications (LLCCVs) standard frequency and limits (70-130%) were met. Limit for cobalt, manganese and zinc are 50 -150%. Only undetected data, or values < 2 x RL are qualified or impacted.

IV. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L 8.0 ug/L	All samples in SDG 44433

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
	Mercury	0.1 ug/L	

Sample concentrations were compared to concentrations detected in the ICB/CCB/PBs per the National Functional Guidelines (and associated field results between the MDL and RL were flagged as U at the detected values). No samples were qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-08-10	Mercury	0.2 ug/L	0.2U ug/L
TSB-GJ-08-20	Thallium Tungsten	0.40 ug/L 0.70 ug/L	0.40U ug/L 0.70U ug/L
TSB-GJ-08-30	Lithium	10.0 ug/L	10.0U ug/L

Sample "RINSATE 1" (from SDG 4444120137) was identified as a rinsate. No metal contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE 1	6/11/08	Calcium Iron Magnesium Manganese Silicon Sodium Strontium	131 ug/L 154 ug/L 17.9 ug/L 0.84 ug/L 38.6 ug/L 39.2 ug/L 1.5 ug/L	All samples in SDG 44433

Sample concentrations were compared to concentrations detected in the field blanks. No samples were qualified.

V. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

ICP interference check samples were reviewed for each analyte as applicable. Percent recovery (%R) of the ICSAB were within the QC limits of 80-120%.

VI. Laboratory Control Sample (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 80-120% with the following exceptions:

Spike ID (Associated Samples)	Analyte	LCS (%R) (Limits)	Flag	A or P
TSB-GJ-08-10LCS (All samples in SDG 44433)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (80-120) 72.5 (80-120) 65.4 (80-120) 68.4 (80-120) - - - -	J- (all detects) UJ (all non-detects)	A

All samples in the batch for the analytes having %Rs outside control limits were qualified as summarized above.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Relative percent differences (RPDs) were within the acceptance criteria of $\leq$ 20% for water or $\leq$ 35% for soil. For low level results, <5 x RL, a difference of $\pm$ 1 x RL is allowed for water and $\pm$ 2 x RL for soils.

VIII. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 75-125% and relative percent differences (RPD) were within 20% limits with the following exceptions (qualification applies only if the spike value X 4 > sample result):

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Sulfur Phosphorus	140.1 (75-125) 134.8 (75-125)	135.4 (75-125) -	- -	J+ (all detects) J+ (all detects)	A
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (75-125) 72.5 (75-125) 65.4 (75-125) 68.4 (75-125) - - - -	39.4 (75-125) 60.9 (75-125) 44.6 (75-125) 56.0 (75-125) 69.8 (75-125) 71.1 (75-125) 60.6 (75-125) 62.2 (75-125)	- - - - - - - -	J- (all detects) UJ (all non-detects)	A
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Niobium	40.6 (75-125)	29.7 (75-125)	-	J- (all detects) R (all non-detects)	A

Aluminum, calcium, iron, magnesium, manganese, strontium, and titanium results were outside the QC limits; results were not qualified since the original sample (TSB-GJ-08-10) was greater than 4X the spike amount.

A post-digestion spike was analyzed for any spike recovery outlier when the spike x 4 is greater than the sample result. For spike % R < 30% and post digest spike $\geq$ 75%, data

are qualified ‘J’ or ‘UJ’. For spike %R < 30% and post digest was < 75% or not reported, the ‘R’ matrix effect is verified for undetected data. The matrix spike qualifier ‘J’ is verified with consideration of significant low bias. No additional qualification is required for the post digest spike as data are already qualified for the matrix spike. If the post digest spike is not correlated (high or low) to the matrix spike, the difference is noted. If a post digest spike is not reported, the serial dilution may be used for further evaluation (see following section).

IX. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria of ±10% difference for values greater than 50 times the lower limit of quantitation (i.e., the method detection limits [MDLs]) were met, with the following exceptions:

Sodium and Uranium results were outside the QC limits; data were not qualified since the concentration was less than 50 times the MDLs.

X. ICP-MS Internal Standards

All internal standard percent recoveries (%R) were within 70-130% or a 2x dilution was run with acceptable recoveries with the following exceptions:

Sample	Internal Standard	%R (Limits)	Analyte	Flag	A or P
TSB-GJ-08-20	Scandium-45	127.557 (70-130)	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A
TSB-GJ-08-30	Scandium-45	129.653 (70-130)	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A

XI. Field Replicates

Field replicate samples were collected in triplicate. Control limit(s) were not established in the SAP since the average of the replicate samples is used as the final value for the field location. Results of field replicate samples or other project samples were not qualified based on the precision of field replicate samples.

XII(a). Sample Result Verification

All sample result verifications were acceptable.

XII(b). Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

Metals - Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Flag	A or P	Reason
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Sulfur Phosphorus	J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Niobium	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-20 TSB-GJ-08-30	Silicon Strontium	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	A	Internal standards (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Iron	J (all detects)	A	ICP serial dilution (%D)

Metals - Laboratory Blank Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Modified Final Concentration	A or P
44433	TSB-GJ-08-10	Mercury	0.2U ug/L	A
44433	TSB-GJ-08-20	Thallium Tungsten	0.40U ug/L 0.70U ug/L	A
44433	TSB-GJ-08-30	Lithium	10.0U ug/L	A

Report# #######

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: Southeast Idaho Mine Sites

Report Date: November 1, 2008

Matrix: Water

Parameters: Metals by ICP SW-846 Method 6010B

Validation Level: EPA Level IV

Laboratory: Microbac

Sample Delivery Group (SDG): 44433

Sample Identification	Collection Date	Laboratory Identification
TSB-GJ-08-10	9/15/08	44433-01
TSB-GJ-08-20	9/15/08	44433-02
TSB-GJ-08-30	9/15/08	44433-03
TSB-GJ-08-40	9/15/08	44433-04
TSB-GJ-08-10MS	9/15/08	44433-05
TSB-GJ-08-10MSD	9/15/08	44433-06

Introduction

This data review covers 6 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA SW 846 Method noted below:

- Method 6010B ICP: Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Niobium, Palladium, Phosphorus, Platinum, Potassium, Selenium, Silicon, Silver, Sodium, Strontium, Sulfur, Thallium, Tin, Titanium, Tungsten, Uranium, Vanadium, and Zinc, and Zirconium.

This review follows the specific guidance in the QAPP Addendum (MWH 2009) to the project SAP (April 2004) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following are not data qualifiers but are provide for the purpose of evaluating the laboratory's performance:

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

1 Holding Time
2 Sample Preservation (including receipt temperature)
3 Sample Custody
4 Missing Deliverable
5 ICPMS Tune
6 Initial Calibration
7 Initial Calibration Verification
8 Continuing Calibration Verification
9 Low-Level Calibration Check Sample
10 Calibration Blank
11 Laboratory or Preparation Blank
12 ICPMS or ICP Interference Check Standard
13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
14 Laboratory Control Sample Precision
15 Laboratory Duplicate Precision
16 Matrix Spike or Matrix Spike Duplicate Recovery
17 Matrix Spike/Matrix Spike Duplicate Precision
18 ICPMS or ICP Serial Dilution
19 ICPMS Internal Standard
20 Field Replicate Precision
21 Equipment Rinsate Blank
22 Linear Range Exceeded
23 Other reason

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections preformed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements were met: 6 months for water and soil (note NIST soil standard reference samples are valid for up to 3 years).

All samples were received intact with proper preservation (pH < 2 for water).

II. Calibration

An initial calibration was performed each day of analysis. The frequency and analysis criteria (90-110%) of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

The low-level initial calibration verification (LLICV) and low-level continuing calibration verifications (LLCCVs) standard frequency and limits (70-130%) were met. Limit for antimony, lead and thallium are 50 -150%. Only undetected data, or values < 2 x RL are qualified or impacted.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Antimony Thallium Tungsten Vanadium Lithium Mercury	1.3 ug/L 1.1 ug/L 1.4 ug/L 2.7 ug/L 8.0 ug/L 0.1 ug/L	All samples in SDG 44433

Sample concentrations were compared to concentrations detected in the ICB/CCB/PBs per the National Functional Guidelines (and associated field results between the MDL and RL were flagged as U at the detected values). No samples were qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-08-10	Mercury	0.2 ug/L	0.2U ug/L
TSB-GJ-08-20	Thallium Tungsten	0.40 ug/L 0.70 ug/L	0.40U ug/L 0.70U ug/L
TSB-GJ-08-30	Lithium	10.0 ug/L	10.0U ug/L

Sample "RINSATE 1" (from SDG 4444120137) was identified as a rinsate. No metal contaminants were found in this blank with the following exceptions:

Rinsate ID	Sampling Date	Analyte	Concentration	Associated Samples
RINSATE 1	6/11/08	Calcium Iron Magnesium Manganese Silicon Sodium Strontium	131 ug/L 154 ug/L 17.9 ug/L 0.84 ug/L 38.6 ug/L 39.2 ug/L 1.5 ug/L	All samples in SDG 44433

Sample concentrations were compared to concentrations detected in the field blanks. No samples were qualified.

IV. ICP Interference Check Sample (ICS) Analysis

The frequency of analysis was met.

ICP interference check samples were reviewed for each analyte as applicable. Percent recovery (%R) of the ICSAB were within the QC limits of 80-120%.

V. Laboratory Control Sample (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 80-120% with the following exceptions:

Spike ID (Associated Samples)	Analyte	LCS (%R) (Limits)	Flag	A or P
TSB-GJ-08-10LCS (All samples in SDG 44433)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (80-120) 72.5 (80-120) 65.4 (80-120) 68.4 (80-120) - - - -	J- (all detects) UJ (all non-detects)	A

All samples in the batch for the analytes having %Rs outside control limits were qualified as summarized above.

VI. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Relative percent differences (RPDs) were within the acceptance criteria of $\leq$ 20% and $\leq$ 35% for soils. For low level results, < 5 x RL, a difference of $\pm$ 1 x RL is allowed for water and $\pm$ 2 x RL for soils.

VII. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 75-125% and relative percent differences (RPD) were within 20% limits (35% soils) with the following exceptions (qualification applies only if the spike value X 4 > sample result):

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Sulfur Phosphorus	140.1 (75-125) 134.8 (75-125)	135.4 (75-125) -	- -	J+ (all detects) J+ (all detects)	A
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	55.2 (75-125) 72.5 (75-125) 65.4 (75-125) 68.4 (75-125) - - - -	39.4 (75-125) 60.9 (75-125) 44.6 (75-125) 56.0 (75-125) 69.8 (75-125) 71.1 (75-125) 60.6 (75-125) 62.2 (75-125)	- - - - - - - -	J- (all detects) UJ (all non-detects)	A
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Niobium	40.6 (75-125)	29.7 (75-125)	-	J- (all detects) R (all non-detects)	A

Aluminum, calcium, iron, magnesium, manganese, strontium, and titanium results were outside the QC limits; results were not qualified since the original sample (TSB-GJ-08-10) was greater than 4X the spike amount.

A post-digestion spike was analyzed for any spike recovery outlier when the spike x 4 is greater than the sample result. For spike % R < 30% and post digest spike $\geq$ 75%, data are qualified 'J' or 'UJ'. For spike %R < 30% and post digest was < 75% or not reported, the 'R' matrix effect is verified for undetected data. The matrix spike qualifier 'J' is verified with consideration of significant low bias. No additional qualification is required for the post digest spike as data are already qualified for the matrix spike. If the post digest spike is not correlated (high or low) to the matrix spike, the difference is noted. If a post digest spike is not reported, the serial dilution may be used for further evaluation (see following section).

VIII. ICP Serial Dilution

ICP serial dilution analysis was performed by the laboratory. The analysis criteria of $\pm$10% difference for values greater than 50 times the lower limit of quantitation (i.e., the method detection limits [MDLs]) were met, with the following exceptions:

Sodium and Uranium results were outside the QC limits; data were not qualified since the concentration was less than 50 times the MDLs.

IX. Field Replicates

Field replicate samples were collected in triplicate. Control limit(s) were not established in the SAP since the average of the replicate samples is used as the final value for the field location. Results of field replicate samples or other project samples were not qualified based on the precision of field replicate samples.

X(a). Sample Result Verification

All sample result verifications were acceptable.

X(b). Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

Metals - Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Flag	A or P	Reason
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Sulfur Phosphorus	J+ (all detects) J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Antimony Copper Silicon Vanadium Lithium Nickel Tungsten Zinc	J- (all detects) UJ (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Niobium	J- (all detects) R (all non-detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Iron	J (all detects)	A	ICP serial dilution (%D)

Metals - Laboratory Blank Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Modified Final Concentration	A or P
44433	TSB-GJ-08-10	Mercury	0.2U ug/L	A
44433	TSB-GJ-08-20	Thallium Tungsten	0.40U ug/L 0.70U ug/L	A
44433	TSB-GJ-08-30	Lithium	10.0U ug/L	A

Report# #######

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: Southeast Idaho Mine Sites

Report Date: November 1, 2008

Matrix: Water

Parameters: Mercury by CVAA EPA Method 7470A

Validation Level: EPA Level IV

Laboratory: Microbac

Sample Delivery Group (SDG): 44433

Sample Identification	Collection Date	Laboratory Identification
TSB-GJ-08-10	5/12/08	44433-01
TSB-GJ-08-20	5/12/08	44433-02
TSB-GJ-08-30	5/12/08	44433-03
TSB-GJ-08-40	5/12/08	44433-04
TSB-GJ-08-10MS	5/12/08	44433-05

Introduction

This data review covers 6 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA Method noted below:

- Method 7470A: Mercury.

This review follows the specific guidance in the QAPP Addendum (MWH 2009) to the project SAP (April 2004) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following are not data qualifiers but are provide for the purpose of evaluating the laboratory's performance:

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

The following “Reason Codes” will be applied as applicable to the validated data:

1 Holding Time
2 Sample Preservation (including receipt temperature)
3 Sample Custody
4 Missing Deliverable
5 ICPMS Tune
6 Initial Calibration
7 Initial Calibration Verification
8 Continuing Calibration Verification
9 Low-Level Calibration Check Sample
10 Calibration Blank
11 Laboratory or Preparation Blank
12 ICPMS or ICP Interference Check Standard
13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
14 Laboratory Control Sample Precision
15 Laboratory Duplicate Precision
16 Matrix Spike or Matrix Spike Duplicate Recovery
17 Matrix Spike/Matrix Spike Duplicate Precision
18 ICPMS or ICP Serial Dilution
19 ICPMS Internal Standard
20 Field Replicate Precision
21 Equipment Rinsate Blank
22 Linear Range Exceeded
23 Other reason

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections preformed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements (28 days) were met.

All samples were received intact with proper preservation (pH < 2 for water).

II. Calibration

An initial calibration was performed each day of analysis. The blank plus 4 standard curve produced a correlation coefficient of > 0.995. The frequency and analysis criteria (80-120%) of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

The low-level initial calibration verification (LLICV) and low-level continuing calibration verifications (LLCCVs) standard frequency and limits (70-130%) were met.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Mercury	0.1 ug/L	All samples in SDG 44433

Sample concentrations were compared to concentrations detected in the ICB/CCB/PBs per the National Functional Guidelines (and associated field results between the MDL and RL were flagged as U at the detected values). No samples were qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-08-10	Mercury	0.2 ug/L	0.2U ug/L

Sample "RINSATE 1" (from SDG 4444120137) was identified as a rinsate. No metal contaminants were found in this blank. Association of results in rinsates samples to field samples and impact of concentrations detected in rinsate samples to field sample results are not addressed in this report, but will be assessed as part of a separate data usability assessment.

IV. Laboratory Control Sample (LCS)

Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 80-120% with the following exceptions:

Spike ID (Associated Samples)	Analyte	LCS (%R) (Limits)	Flag	A or P
TSB-GJ-08-10LCS (All samples in SDG 44433)	Mercury	125.2 (80-120) -	J+ (all detects) UJ (all non-detects)	A

All samples in the batch for the analytes having %Rs outside control limits were qualified as summarized above.

V. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Relative percent differences (RPDs) were within the acceptance criteria of $\leq$ 20% for water or $\leq$ 35% for soil. For low level results, <5 x RL, a difference of $\pm$ 1 x RL is allowed for water and $\pm$ 2 x RL for soils.

VI. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 75-125% and relative percent differences (RPD) were within 20% limits with the following exceptions (qualification applies only if the spike value times 4 > sample result):

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Mercury	140.1 (75-125)	135.4 (75-125)	-	J+ (all detects)	A

VII. Field Replicates

Field replicate samples were collected in triplicate. Control limit(s) were not established in the SAP since the average of the replicate samples is used as the final value for the field location. Results of field replicate samples or other project samples were not qualified

based on the precision of field replicate samples.

VIII(a). Sample Result Verification

All sample result verifications were acceptable.

VIII(b). Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

Metals - Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Flag	A or P	Reason
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Mercury	J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Mercury	J+ (all detects)	A	Laboratory control sample (%R)

Metals - Laboratory Blank Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Modified Final Concentration	A or P
44433	TSB-GJ-08-10	Mercury	0.2U ug/L	A

Report# #######

Data Validation Company Name
Data Validation Report

Project/Site Name: Southeast Idaho Mine Sites.

Report Date: November 1, 2008

Matrix: Water

Parameters: Major Anions by Ion Chromatography (IC) EPA Method 300.0

Validation Level: EPA Level IV

Laboratory: Laboratory Name

Sample Delivery Group (SDG): 44433

Sample Identification	Collection Date	Laboratory Identification
TSB-GJ-08-10	9/15/08	44433-01
TSB-GJ-08-20	9/15/08	44433-02
TSB-GJ-08-30	9/15/08	44433-03
TSB-GJ-08-40	9/15/08	44433-04
TSB-GJ-08-10MS	9/15/08	44433-05

Introduction

This data review covers 6 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA Method noted below:

- Method 300.0: Chloride.

This review follows the specific guidance in the QAPP Addendum (MWH 2009) to the project SAP (April 2004) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following are not data qualifiers but are provide for the purpose of evaluating the laboratory's performance:

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

1 Holding Time
2 Sample Preservation (including receipt temperature)
3 Sample Custody
4 Missing Deliverable
5 ICPMS Tune
6 Initial Calibration
7 Initial Calibration Verification
8 Continuing Calibration Verification
9 Low-Level Calibration Check Sample
10 Calibration Blank
11 Laboratory or Preparation Blank
12 ICPMS or ICP Interference Check Standard
13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
14 Laboratory Control Sample Precision
15 Laboratory Duplicate Precision
16 Matrix Spike or Matrix Spike Duplicate Recovery
17 Matrix Spike/Matrix Spike Duplicate Precision
18 ICPMS or ICP Serial Dilution
19 ICPMS Internal Standard
20 Field Replicate Precision
21 Equipment Rinsate Blank
22 Linear Range Exceeded
23 Other reason

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections preformed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements (28 days) were met.

All samples were received intact (no preservation required).

II. Calibration

An initial calibration was performed each day of analysis. The blank plus 5 standard curve produced a correlation coefficient of > 0.995. The frequency and analysis criteria (90-110%) of the initial calibration verification (ICV) and continuing calibration verification (CCV) were met.

III. Blanks

Method blanks were reviewed for each matrix as applicable. No contaminant concentrations were found in the initial, continuing and preparation blanks with the following exceptions:

Method Blank ID	Analyte	Maximum Concentration	Associated Samples
ICB/CCB	Chloride	0.2 mg/L	All samples in SDG 44433

Sample concentrations were compared to concentrations detected in the ICB/CCB/PBs per the National Functional Guidelines (and associated field results between the MDL and RL were flagged as U at the detected values). No samples were qualified with the following exceptions:

Sample	Analyte	Reported Concentration	Modified Final Concentration
TSB-GJ-08-10	Chloride	0.21 mg/L	0.21U mg/L

Sample "RINSATE 1" (from SDG 4444120137) was identified as a rinsate. Major anions were not found in this blank, so associated field samples did not require qualification.

IV. Laboratory Control Sample (LCS)

Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 80-120% with the following exceptions:

Spike ID (Associated Samples)	Analyte	LCS (%R) (Limits)	Flag	A or P
TSB-GJ-08-10LCS (All samples in SDG 44433)	Chloride	125.2 (80-120) -	J+ (all detects) UJ (all non-detects)	A

All samples in the batch for the analytes having %Rs outside control limits were qualified as summarized above.

V. Duplicate Sample Analysis

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Relative percent differences (RPDs) were within the acceptance criteria of $\leq$ 20% for water or $\leq$ 35% for soil. For low level results, <5 x RL, a difference of $\pm$ 1 x RL is allowed for water and $\pm$ 2 x RL for soils.

VI. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within 75-125% and relative percent differences (RPD) were within 20% limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	RPD (Limits)	Flag	A or P
TSB-GJ-08-10MS/MSD (All samples in SDG 44433)	Chloride	140.1 (75-125)	135.4 (75-125)	-	J+ (all detects)	A

VII. Field Replicates

Field replicate samples were collected in triplicate. Control limit(s) were not established in the SAP since the average of the replicate samples is used as the final value for the field location. Results of field replicate samples or other project samples were not qualified based on the precision of field replicate samples.

VIII(a). Sample Result Verification

All sample result verifications were acceptable.

VIII(b). Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

Metals - Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Flag	A or P	Reason
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Chloride	J+ (all detects)	A	Matrix spike/Matrix spike duplicates (%R)
44433	TSB-GJ-08-10 TSB-GJ-08-20 TSB-GJ-08-30 TSB-GJ-08-40	Chloride	J+ (all detects)	A	Laboratory control sample (%R)

Metals - Laboratory Blank Data Qualification Summary - SDG 44433

SDG	Sample	Analyte	Modified Final Concentration	A or P
44433	TSB-GJ-08-10	Chloride	0.21 mg/L	A

Report# #######

Data Validation Company Name
Data Validation Report

Project/Site Name: Southeast Idaho Mine Sites

Report Date: November 1, 2008

Matrix: Water

Parameters: Gross Alpha and Gross Beta by Method 900.0
Gas Flow Proportional Counting System (GFPC)

Validation Level: EPA Level IV

Laboratory: Laboratory Name

Sample Delivery Group (SDG): L55555

Field Sample Identification	Date Collected	Laboratory Sample Identification
091207GWMST049-1-U	9/15/2008	L55555-01
091207GWMST049-2-U	9/15/2008	L55555-03
091207GWMST049-3-U	9/15/2008	L55555-05
091207GWMST049-B-U	9/15/2008	L55555-07
091207GWMST049-EQ-U	9/15/2008	L55555-09

Introduction

This data review covers 5 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA Method noted below:

- Method 900.0: Gross Alpha and Gross Beta.

This review follows the specific guidance in the QAPP Addendum (MWH 2009) to the project SAP (April 2004) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.

UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

The following are not data qualifiers but are provide for the purpose of evaluating the laboratory's performance:

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

1 Holding Time
2 Sample Preservation (including receipt temperature)

3 Sample Custody
4 Missing Deliverable
5 - not applicable to this method -
6 Initial Calibration
7 (not applicable to this method)
8 Continuing Calibration Verification
9 - not applicable to this method -
10 - not applicable to this method -
11 Laboratory or Preparation Blank
12 - not applicable to this method -
13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
14 Laboratory Control Sample Precision
15 Laboratory Duplicate Precision
16 Matrix Spike or Matrix Spike Duplicate Recovery
17 Matrix Spike/Matrix Spike Duplicate Precision
18 - not applicable to this method –
19 - not applicable to this method -
20 Field Replicate Precision
21 Equipment Rinsate Blank
22 Linear Range Exceeded
23 Other reason
24 Result is less than the MDC
25 Result is less than two times the error

I. Chain-of-Custody Procedure, Sample Preservation, and Holding Time

_X__ Signatures on chain(s) and all samples accounted for
_X__ Gross alpha and beta: preserved with nitric acid to pH < 2; 6-month holding time (if preserved)

A total of three groundwater samples and two field blank samples were collected on September 15, 2008 in preserved containers. Samples were shipped and arrived at the laboratory on September 16, 2008. Sample chain-of-custody and laboratory receipt documentation appear intact. The samples were analyzed within the 6-month holding time on September 26, 2008, 11 days from collection to analysis.

II. Instrument Calibration

_X__ Confirm dates of calibration, detectors IDs, geometry, counting times, number of counts for each standard, measured activity for all standards, identity and true value of all standards
_X__ Confirm matrix used in geometry standard
_X__ Evidence of decay correction of standard prior to calculation of efficiencies, as appropriate
_X__ Calibration points including efficiencies for each detector
_X__ Background checks performed at the time of initial calibration
_X__ Self absorption curves for each detector, covering an appropriate range of residue masses
_X__ Alpha-beta cross talk values and voltage plateaus (if needed)
_X__ Certificates for NBS- or NIST-traceable standards
_X__ Review standard preparation and dilution logs for accuracy

Initial calibration data were within all required criteria.

III. Calibration Verification

_X__ Tolerance chart or statistical control chart of the appropriate efficiencies and background activities (at least 20 points) with ± 2 sigma error (warning) and ± 3 sigma error (failure) limits
_X__ Routine (daily, weekly, monthly) background checks for each detector
_X__ Daily efficiency checks for each detector
_X__ Evidence of decay correction of standard prior to calculation of efficiencies, as appropriate
_X__ Confirm detector IDs and geometries used in analysis
_X__ Check if sample residues are within the range of the self absorption curve

Calibration verification data were within all required criteria.

IV. Target Compound Identification and Quantitation

_X___ Confirm all samples less than MDC are qualified not detected (U)
_X___ Less than two times the uncertainty (2 sigma error) were reported by the laboratory as not detected

Sample results that were reported as values less than the MDC were qualified as not detected at the MDC (flagged U). Sample results that were less than two times the error were qualified as not detected at the MDC (flagged U) or qualified as not detected at the reported concentration (flagged U). The following results were qualified:

Field Sample Identification	Laboratory Sample ID	Parameter	Result (pCi/L)	MDC (pCi/L)	2*Error	Data Validation Result	Reason Code
091207GWMST049-1-U	L55555-01	Gross Alpha	3.6	2.0	3.8	3.6 UJ	25
091207GWMST049-1-U	L55555-01	Gross Beta	0.24	4.0	2.4	4.0 UJ	24

V. Blanks

_X___ Confirm method blank results < MDC
If method blank result is ≥ MDC:
If sample result is > 5X the concentration of the method blank, then no action.
If sample result is ≤ 5X the concentration of the method blank, then U at detected value.

Gross Alpha and Gross Beta were not detected in the method blank above their MDCs.

VI. Laboratory Duplicates

_X___ Confirm laboratory duplicate analyzed for each batch or for every 20 samples
_X___ Confirm that the DER ≤ 1.42 or RPD ≤ 20
Where the duplicate (or replicate) error ratio (DER) =

$$\frac{\left|([\text{Sample}] - [\text{Duplicate}])\right|}{([2\ \text{sigma error}^2_{\text{Sample}}] + [2\ \text{sigma error}^2_{\text{Duplicate}}])^{1/2}}$$

_____ If DER > 1.42, qualify sample J
_____ If RPD > 20 and result(s) ≥ 5x MDC, then qualify J. If result(s) < 5x MDC, then calculate absolute difference. If absolute difference is within ± MDC (water) or ± 2x MDC (soil), then no action; if > ± MDC (water) or ± 2x MDC (soil), then qualify sample J.

All DERs are less than 1.42 (or RPDs less than 20) for Gross Alpha and Gross Beta.

VII. Laboratory Control Samples

_X___ Must be analyzed for each batch or for every 20 samples
_X___ Compare %R for Gross Alpha with lab control limits (75-125%)
_X___ Compare %R for Gross Beta with lab control limits (75-125%)

Recoveries for Gross Alpha and Gross Beta in each laboratory control sample were within control limits.

VIII. Matrix Spike Samples

_X___ Must be analyzed for each batch or for every 20 samples
_X___ Compare %R for Gross Alpha with lab control limits (65-135%)
_X___ Compare %R for Gross Beta with lab control limits (65-135%)

Recoveries for Gross Alpha and Gross Beta in each matrix spike sample were within control limits.

IX. Equipment and Water Blank Samples

One equipment blank and one water blank were collected on September 15, 2008 for Gross Alpha and Gross Beta analysis. Gross Alpha and Gross Beta were not detected in any of the field blank samples.

X. Overall Assessment of Data

With the following exceptions, all quality control data associated with the field samples were within control limits. All other field results are usable as reported by the laboratory.

Summary of Qualified Data:

Field Sample Identification	Laboratory Sample ID	Parameter	Result (pCi/L)	Data Validation Result	Reason Code
091207GWMST049-1-U	L55555-01	Gross Alpha	3.6	3.6 UJ	25
091207GWMST049-1-U	L55555-01	Gross Beta	0.24	4.0 UJ	24

APPENDIX C

BALLARD, HENRY, AND ENOCH VALLEY MINES
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY

HEALTH AND SAFETY PLAN

DRAFT
Revision 2

March 2015

TABLE OF CONTENTS

1.0 INTRODUCTION 1-1
2.0 SITE HEALTH AND SAFETY ANALYSIS 2-1
2.1 POTENTIAL CHEMICALS HAZARDS 2-1
2.2 RADIOLOGICAL HAZARDS 2-3
2.3 PHYSICAL HAZARDS 2-3
2.4 BIOLOGICAL HAZARDS 2-6
3.0 ASSIGNMENT OF RESPONSIBILITIES 3-1
3.1 VICE PRESIDENT-IN-CHARGE 3-1
3.2 PROJECT MANAGER 3-1
3.3 PROGRAM SAFETY OFFICER 3-1
3.4 ON-SITE SAFETY OFFICER 3-2
3.5 FIELD TEAM LEADER 3-4
3.6 FIELD PERSONNEL 3-4
3.7 SITE VISITORS 3-4
4.0 PERSONNEL TRAINING 4-1
4.1 INITIAL SITE-SPECIFIC HEALTH AND SAFETY TRAINING 4-1
4.2 TAILGATE SAFETY MEETINGS 4-3
4.3 BLOODBORNE PATHOGENS AND FIRST AID/CPR 4-3
4.4 DOCUMENTATION OF TRAINING 4-3
5.0 MEDICAL SURVEILLANCE 5-1
6.0 PERSONAL PROTECTIVE EQUIPMENT 6-1
6.1 PERSONAL PROTECTIVE EQUIPMENT 6-1
6.2 LEVEL D PERSONAL PROTECTIVE EQUIPMENT 6-2
6.3 PPE STORAGE 6-4
7.0 HAZARD ASSESSMENT 7-1
7.1 HEAT STRESS 7-1
7.2 COLD STRESS 7-2
7.2.1 Frostbite Monitoring 7-2
7.2.2 Hypothermia Monitoring 7-3
8.0 SITE CONTROL 8-1
8.1 MULTIPLE-EMPLOYER JOB SETTING 8-1
8.2 SITE SECURITY 8-1
8.3 COMMUNICATION SYSTEMS 8-2
9.0 DECONTAMINATION PROCEDURES 9-1
9.1 GENERAL DECONTAMINATION PROCEDURES 9-1
9.2 DECONTAMINATION WASTE HANDLING AND DISPOSAL 9-1
10.0 EMERGENCY RESPONSE PLANNING 10-1
11.0 HEALTH AND SAFETY DOCUMENTATION 11-1
12.0 GENERAL SITE SAFETY 12-1
12.1 GENERAL HEALTH AND SAFETY PROGRAM ELEMENTS 12-1
12.1.1 Hazard Communication Program 12-1
12.1.2 Sanitation 12-2
12.1.3 Illumination 12-2

12.1.4 Standard Emergency Hand Signals 12-2
12.1.5 Fire Protection 12-3
12.1.6 Earthquake and Disaster Preparedness 12-6
12.2 COMMON PHYSICAL HAZARDS AND CONTROLS 12-6
12.2.1 Slip/Trip/Fall 12-7
12.2.2 Heavy Lifting 12-7
12.2.3 Motor Vehicle Hazards 12-7
12.2.4 Sharp Edges and Pinch Points 12-7
12.3 SEVERE WEATHER 12-7
12.3.1 Lightning 12-8
12.3.2 Tornadoes 12-8
12.3.3 Winter Storms 12-9
12.4 ENGINEERED CONTROLS 12-9
12.5 ADMINISTRATIVE CONTROLS 12-9

LIST OF TABLES

Table 2-1 Occupational Exposure Limits and Toxicological Properties for Potential Chemical Exposure Hazards
Table 2-2 Ten Essentials for Wilderness Travel
Table 3-1 Project Health and Safety Program Contact List

LIST OF FIGURES

Figure 6-1 Sample Protective Equipment Ensembles

LIST OF APPENDICES

Appendix A Activity Hazard Analysis
Appendix B MWH EH&S Procedure No. 811, Drilling Safety
Appendix C MSDSs
Appendix D Portable Generator Hazards
Appendix E Emergency Contacts
Appendix F Personal Acknowledgement Form
Appendix G Monsanto Contractor/Guest ES&H Site Guidelines
Appendix H Pre-Job Risk Analysis Worksheet
Appendix I Degerstrom Ore Haulroad Travel Requirements
Appendix J OSHA Job Safety and Health Protection Poster
Appendix K Occupational Incident Report Forms & Procedures

ACRONYMS AND ABBREVIATIONS

ACGIH	American Congress of Governmental Industrial Hygienists
ANSI	American National Standards Institute
CFR	Code of Federal Regulations
CPR	Cardiopulmonary Resuscitation
EPA	Environmental Protection Agency
ft	Feet
FTL	Field Team Leader
HASP	Health and Safety Plan
IDLH	Immediately Dangerous to Life or Health
IMASC	Idaho Mining Association Selenium Committee
JRA	Pre-Job Risk Analysis
MSDS	Material Safety Data Sheet
MSHA	Mine Safety and Health Administration
NFPA	National Fire Protection Association
NIOSH	National Institute for Occupational Safety and Health
NPS	National Parks Service
OSHA	Occupational Safety and Health Administration
OSO	On-Site Safety Officer
PEL	Permissible Exposure Limit
PPE	Personal Protective Equipment
PSO	Project Safety Officer
REL	Recommended Exposure Limit
RI/FS	Remedial Investigation and Feasibility Study
SOP	Standard Operating Procedure
TLV	Threshold Limit Value
TWA	Time-Weighted Average
yds	Yards

1.0 INTRODUCTION

This Health and Safety Plan (HASP) has been prepared to establish the responsibilities, requirements, and procedures for protecting MWH personnel during the Enoch Valley, Henry, and Ballard Mines Remedial Investigation and Feasibility Study (RI/FS). This HASP has been prepared to provide assigned field personnel with a safe working environment as the investigation proceeds. Specifically, the HASP has been developed to minimize the potential for job-related injuries and illnesses, and to prevent job-related injuries and illnesses from occurring. Where this HASP addresses safe practices for various specific construction activities, this information is provided solely as directives or guidelines for protecting MWH employees and establishing minimum requirements for MWH subcontractors. Any questions over implementation of this plan should be addressed to the Project Safety Officer or the MWH Regional EH&S Manager.

A fundamental principle of industrial safety and loss prevention is that most accidents that cause injury, illness, or property damage are preventable. Investigations of the causes of industrial accidents and illnesses have demonstrated that most injuries or illnesses are the result of unsafe acts or conditions. Thus, minimizing industrial accidents and illnesses can be accomplished by recognizing, evaluating, and controlling unsafe acts and conditions.

Employees are required to employ safe work practices and comply with applicable MWH requirements, as well as the applicable requirements of the agencies responsible for regulating industrial health and safety, including the OSHA and MSHA. Furthermore, P4 Production employs its own practice of training every contractor that works on the Soda Springs Phosphate Mine site to ensure that everyone on the site is knowledgeable and aware of the hazards that exist. This process and the applicable documents are discussed where applicable in this HASP.

2.0 SITE HEALTH AND SAFETY ANALYSIS

This section of the HASP identifies activity-specific hazards that may be encountered at project sites during the course of planned field activities, as well as methods that will be employed to control exposure to these hazards. The field activities discussed herein include the potential collection of soil, sediment, vegetation, surface and groundwater samples, well installation supervision, and direct push well installation supervision. Activity locations will generally be either on or adjacent to Ballard, Henry, or Enoch Valley Mines. Exceptions may include regional investigations at some distance from the Mines.

Some of the selected sample sites are readily accessible by vehicle; others, however, are not directly accessible by vehicle, and may require travel on foot. Health and safety issues that are associated with these sites requiring extensive foot travel will be addressed herein.

Specific activities that may be involved in the investigation are listed in Appendix A, *Activity Hazard Analysis*. Appendix A details the hazards that may be associated with these activities, and recommended controls to minimize risk to MWH and subcontractor field personnel. Appendix B, *MWH EH&S Procedure No. 811, Drilling Safety,* addresses, in detail, hazards and safety concerns related to well installation and drilling supervision. Recognized specific chemical, radiological, physical, and biological hazards that may be encountered during field activities are also discussed in the following paragraphs.

Although phosphate ore bodies are known to have slightly elevated concentrations of uranium-238 (^{238}U), radiation has not been identified as a hazard that is likely to be associated with this project and monitoring for ionizing radiation will not be undertaken during the investigation. Additional safety practices more generally applicable to field activities are summarized in the subsequent sections of this plan.

2.1 POTENTIAL CHEMICALS HAZARDS

The hazards that may be associated with chemical contaminants can be assessed through comparison of measured or estimated personnel exposures to established occupational exposure limits. Permissible exposure limits (PELs) are established by the Occupational Safety and Health Administration (OSHA), while recommended exposure limits (RELs) are established by the National Institute for Occupational Safety and Health (NIOSH). Immediately dangerous to life and health (IDLH) concentrations are also established by NIOSH. Threshold limit values (TLVs) and time-weighted averages (TWAs) are established by the American Conference of Governmental Industrial Hygienists (ACGIH). PELs may be expressed as an 8-hour TWA or as a ceiling limit. Ceiling limits may not be exceeded at any time, and are enforceable by law. RELs are published guidelines that set employee exposure limits for airborne contaminants. RELs are expressed as a TWA or ceiling limit. The ACGIH TLV/TWA is the airborne concentration of a substance to which nearly any worker may be repeatedly exposed 8 hours per day, 40 hours per week, without experiencing adverse health effects. For some substances, the overall exposure to a substance is aggravated by contact with skin, mucous membranes, or the

eyes. Other substances have a ceiling value that may not be exceeded during any part of the workday. An IDLH concentration is the maximum airborne concentration of a substance that one could escape within 30 minutes without impairing symptoms or irreversible health effects. It is not anticipated that MWH personnel will be exposed to airborne contaminants above regulatory limits if prudent work practices, including dust control, are employed during the sampling event(s).

Extensive sampling has been undertaken in many areas of the Southeast Idaho Phosphate Resource Area to date by MWH and P4 Production, the IMA companies, and regulatory agencies. Based on area history, the events that prompted this investigation, and sampling to date, naturally-occurring selenium, cadmium, molybdenum, and other trace elements in the phosphate ore body could potentially pose a hazard to sub-populations. Empirical evidence and evaluation of existing data indicate that there has been, and is no, reasonable possibility for health hazards to local residents. Such residents (who are assumed to consume exposed fish, game, and cattle) have high degrees of exposure—speaking relatively, rather than absolutely. Exposures to employees implementing the scope of work covered by this plan are substantially less and thus, there is no reasonable possibility for employee exposure to safety or health hazards associated with these substances. Table 2-1 presents occupational exposure limits and toxicological information for selenium and for other metals that could be present at the sampling sites.

In addition to the naturally occurring elements discussed above, field personnel will use a variety of chemical standards in the calibration of their field instruments. These include solutions for calibrating: conductivity (potassium chloride), pH-4, pH-10, oxidation-reduction potential or ORP (potassium chloride, potassium ferrocyanide trihydrate, potassium ferricyanide), and a calibration check (Quick Cal™) solution (potassium dihydrogen phosphate, potassium chloride, sodium hydroxide, potassium ferrocyanide trihydrate, potassium ferricyanide). These chemicals are generally non-toxic and non-corrosive, but nitrile gloves and safety glasses should still be worn when handling. All MSDSs can be found in Appendix C.

Field Personnel also are in contact with small (but still dangerous) amounts of nitric acid used for sample preservation in water samples. Nitrile gloves and safety glasses should ALWAYS be used, and extreme care should always be taken when opening sample bottles; at high altitudes, bottles can become pressurized and acid can be released violently when opened. Nitric acid is an extremely corrosive oxidizer and will cause severe burns to skin and even clothing. Fumes are toxic and employees should therefore avoid breathing in fumes directly.

Field measurements of nitrite and ferrous iron in groundwater samples require the use of chemical reagent packets. Nitrite reagents are composed of a variety of compounds, predominantly potassium phosphate, and ferrous iron reagents are composed of 1,10 – phenanthroline and sodium bicarbonate. Nitrile gloves and safety glasses must be donned while using either reagent.

When field equipment must be used that is not station-dedicated (i.e. the use of trowels and scissors during soil and vegetation sampling), equipment must be decontaminated with a water-Alconox™ detergent solution between uses. No safety hazards are anticipated when handling the

solution, but when concentrated Alconox™ powder is handled, nitrile gloves and safety glasses should be used and caution should be taken.

During well installation, there is the possibility of hydraulic fluid/oil leaks or spills from drill rigs. MWH personnel must ensure that drilling contractors are capable of containing the release in such an event. Drillers often use liquid "drilling detergent" to increase the viscosity of the drilling fluid (typically water). Drillers typically use Dawn™ liquid dishwashing soap. While this is a relatively safe household item, safety glasses should be worn in the case of splatter.

2.2 RADIOLOGICAL HAZARDS

Phosphate ore in the southeast Idaho Phosphate Resource Area has slightly elevated levels of naturally occurring radionuclides, i.e., isotopes that produce ionizing radiation. Most notable is the isotope ^{238}U. Natural background in soils varies significantly throughout the United States but generally ranges from 2 to 10 picocuries per gram (pCi/g), whereas phosphate ore contains 23 to 28 pCi/g of ^{238}U. In addition, the ^{238}U found in ore is in equilibrium with all of its decay daughters, including radium, radon, and polonium (i.e., all of the decay daughters would be expected to range from 23 to 28 picocuries per gram in the ore as well). This level of radiological activity does not pose a threat to persons on a mine site. However, certain common sense precautions are worth noting. These precautions are similar to those used to prevent exposure to other trace elements in media being sampled, i.e., avoid inhalation and ingestion. The precautions include:

- Avoid breathing dusts;
- Do not eat and drink in dusty areas;
- Use proper hygiene techniques such as washing your hands prior to eating;
- Change clothing daily if clothing becomes soiled with ore. Note that clothing can be washed normally to remove the ore; and,
- If using a respirator, ensure that you keep the respirator clean.

2.3 PHYSICAL HAZARDS

Physical hazards associated with active mine sites as well as those associated with persons working out-of-doors in a mountainous terrain probably pose the greatest threat to field personnel on this project. The physical hazards that may be encountered will vary according to the type of site being investigated. Active mines pose hazards that are specific to mine operations, including the operation of heavy equipment, blasting, and the possibility of mine wall collapse. Large mine haul trucks may travel at relatively high rates of speed and pose a unique traffic threat to anyone traveling on mine haul roads. Railroad trains are also used at mines to haul ore to processing plants and pose railroad crossing hazards. Field personnel working on or around mine traffic areas should be especially mindful of mine-related traffic and mine-specific traffic rules.

Mine wall collapse could also occur at inactive mine sites. At some of the older mines, driving or walking near a high wall may pose a significant hazard as well. Field personnel are required to wear hardhats and safety glasses whenever they are in a mine pit, near a high wall, or any other situation in which an overhead hazard may exist. Field personnel must be alert and aware of their surroundings. Therefore, prior to entering an inactive mine site, MWH personnel must be

cautious and aware of any potential instability that could lead to rock slides, the collapse of high walls, and other physical hazards. MWH personnel will also be acquainted with the signs of instability that could lead to rock slides, the collapse of high walls, and similar hazards.

At other sites, hazards associated with collecting samples are likely to be minimal. Traveling by foot through, or collecting samples in, pasture land, cropland, or forest, possible hazards may be largely limited to slipping, tripping, and falling. It is important that proper footwear is always worn, and that care and common sense is always used while walking to and from stations. Safety glasses are also required while collecting soil and vegetation samples due to the possibility of dust or other debris blowing into the eye. While hiking to and from a sample location, it is also a good idea to wear eye protection to protect from tree branches and other eye-level hazards. Stream sampling can pose risks associated with loss of balance, including head and limb injuries, scrapes, hypothermia, and drowning. Personnel will need to use good judgment to determine whether it is safe to enter a body of water. In particular, while sampling from streams or rivers, field personnel should be aware of hazards that may be created by high or turbulent water, including being swept out by the current. Additionally, field personnel should be aware of hazards that may be created by stepping in deep, soft sediments, or floating debris.

Travel to remote areas engenders additional hazards specific to the method of travel. Hazards associated with foot travel include muscle strains and sprains, dehydration, heat or cold stress, and slips, trips, and falls. If a sample location cannot be reached by vehicle, and a significant hike is required, precautions must to be taken to ensure that field personnel are properly protected from the weather, whether it is heat stress or cold stress. Hazards associated with vehicle transport include flat tires, getting the vehicle stuck, and other miscellaneous vehicle malfunctions. It is essential that both the vehicle operator and vehicle passengers are cautious and observant of on/off road obstacles and dangers. Operator and passengers should also carry a 2-way radio and cell phone or satellite phone (if available) with them at all times, and know how to change a flat tire on the vehicle.

Travel in remote areas can also pose hazards that are best countered by acquiring a knowledge of wilderness safety, awareness of methods for handling contact with animals, and by safely operating equipment. Specific hazards associated with travel in remote areas are listed in Appendix A, *Activity Hazard Analysis*, together with recommended practices and procedures to minimize risks to MWH and subcontractor field personnel. Table 2-2, *Ten Essentials for Wilderness Travel* lists the ten essential items that field personnel should always have with them when working in remote areas.

Overseeing well installation presents a unique set of physical hazards due mainly to the heavy machinery used. Hearing protection is essential as well as hard hats and safety glasses to protect from flying debris caused by drilling. Long hours in one location on hot, sunny days present a challenge in staying cool while donning required PPE. Sunscreen, light colored clothing, and replenishing fluids are essential. Taking refuge inside of a vehicle may be acceptable if the drilling supervision task is not compromised. Furthermore, hands should be kept away from all moving parts and care should be taken when walking around drilling sites to avoid slips, trips, and falls.

TABLE 2-1
OCCUPATIONAL EXPOSURE LIMITS AND TOXICOLOGICAL PROPERTIES FOR POTENTIAL CHEMICAL EXPOSURE HAZARDS

Contaminant	OSHA PEL (mg/m^3)	NIOSH REL (mg/m^3)	ACGIH TLV (mg/m^3)	ACGIH/OSHA STEL	OSHA/NIOSH IDLH (mg/m^3)	IP (eV)	Route of Exposure	Symptoms of Exposure
Cadmium	0.005 (see 29 CFR 1910.1027)	Ca (lowest feasible)	0.01 & 0.002 (respirable fraction)	NA	50	NA	INH, ING	Pulmonary edema, dyspnea, cough, chest tightness, pain, headache; chills, muscle aches; nausea, vomiting, diarrhea; emphysema, mild anemia
Fluoride, as F	2.5	2.5	2.5	NA	500	NA	INH, ING, CON	Eye irritation, respiratory system, nausea, abdominal pain, diarrhea, excessive saliva, thirst, and sweat.
Molybdenum (soluble compounds as Mo)	5	NA	0.5 (respirable fraction)	NA	NE	NA	INH, ING	In animals: irritant to eyes, nose and throat; anorexia; diarrhea; weight loss; listlessness; liver and kidney damage.
Nickel (soluble compounds as Ni)	1	0.015 (Ca)	0.1	NA	10 (Ca)	NA	INH, ING, CON	Headache, vertigo; nausea, vomiting, epigastric pain, substernal pain; cough, hyperpnea; cyanosis; weakness; leukocytosis, pneuitis; delirium, convulsion.
Selenium	0.2	0.2	0.2	NA	NA	NA	INH, ING, CON, ABS	Irritant to eye, nose and throat; visual disturbance; headache; chills, fever; dyspnea, bronchitis; metallic taste, garlic breath, gastro-intestinal disturbance; dermatitis; and skin, eye burns.
Vanadium (as Vanadium pentoxide)	0.5	0.05	0.05	0.5 (ceiling value)	1	NA	INH, ING, CON	Irritant to eyes; green tongue, metallic taste, eczema; cough; fine rales, wheezing, bronchitis, dyspnea; irritant to the throat
Zinc	NA	NA	NA	NA	NA	NA	INH	Sweet, metallic taste; dry throat, cough; chills, fever; tight chest, dyspnea, rales, reduced pulmonary function; headache, blurred vision; muscle cramps, low back pain; nausea, vomiting; fatigue, lassitude and malaise

ACGIH-American Conference of Governmental Industrial Hygienists
Ca-NIOSH considered carcinogen
CFR-Code of Federal Regulations
CON-Skin or mucous membrane contact
eV-Electron volts
IDLH-Immediately dangerous to life or health
ING-Ingestion
INH-Inhalation
IP-Ionization potential
NA-Not applicable or available
NE-Not established
NIOSH-National Institute of Occupational Safety and Health
OSHA-Occupational Safety and Health Administration
PEL-Permissible exposure limit
REL-Recommended exposure limit
STEL-Short term exposure limit
TLV-Threshold limit values

Table 2-2 Ten Essentials for Wilderness Travel		
To Find Your Way	**For Your Protection**	**For Emergencies**
Map of the area **Compass and/or GPS(with sufficient backup battery)** Flashlight or headlamp	Sunglasses Extra food and water Extra clothing	Waterproof matches Candle, fuel tablets, or other long–burning fire starter Pocket knife First aid kit

Low-flow well sampling also presents unique physical hazards. Compressed nitrogen gas is often used to provide air pressure and the large storage cylinders can be difficult to manage. Cylinders are to be stored in a secured upright position at all times, in temperatures not exceeding 52° C (125 ° F), and valves should be closed whenever not in use (even if empty). Cylinders should not be dropped, rolled, or dragged, and should be transported using a suitable hand truck. Additional information can be found in Appendix C in the Nitrogen MSDS.

Portable gasoline powered generators may also be used to power air compressors to provide air pressure during low-flow well sampling. Portable generators should only be used in wide open spaces; carbon monoxide poisoning from the toxic engine exhaust is deadly. Gasoline for the generator must only be stored in flammable liquid safety cabinets and should only be used to refuel the generator after it has been turned off and allowed to cool. Gasoline spilled on hot engine parts could ignite. Additional safety information can be found in Appendix D.

2.4 BIOLOGICAL HAZARDS

Biological hazards that could potentially be encountered at during the RI/FS may include snakes, spiders, ticks, fleas, big game and other wildlife, aggressive livestock, poisonous/irritating plants such as poison oak and poison ivy, and micro–organisms such as the hantavirus.

Field personnel should be aware of their surroundings and avoid contact with snakes and insects. Snakes, spiders, and fleas typically occupy cool, dark, moist areas. The possibility of an encounter most frequently arises when reaching into dark, covered places. Suggestions for controlling the risks associated with snakes and insects include using a long stick to break apart webs or to remove soil cover from sheltered areas. A flashlight should also be used to inspect dark cavities before reaching into them.

Big game animals (deer, elk, and moose) are often seen from vehicles while traveling to and from sites, but are easily scared away and are rarely seen on foot. If big game is encountered while on foot, the National Parks Service (NPS) recommends staying at least 75 ft (25 yds) away. While generally docile, if game feel threatened, they will charge and attack; especially females with young. Other more dangerous wildlife (but notably more uncommon) may include wolves, bear, mountain lions, and coyotes. The NPS recommends staying at least 300 ft (100 yds) away if one of these is encountered.

Aggressive livestock are another (rare but legitimate) biological safety concern. It may be

necessary to enter an active pasture to access a sample location. Livestock are generally very skittish and can easily be frightened, but personnel should still be cautious, maintain a distance of 75ft (25 yds), and avoid turning their back on the animals. A pasture or enclosure with a male cow (bull) should never be entered. Adult males are generally separated from the females and juveniles and are kept by themselves. They are easily distinguished and are very volatile; they should not be approached.

As a matter of courtesy, field personnel should obtain permission from private landowners prior to entering private lands and immediately close any gates opened in order to access sampling stations and when leaving such stations to make sure that livestock do not inadvertently escape.

Poisonous plants such as poison ivy and poison oak grow wild in dark, moist areas, and at the base of or around seedling and adult trees. Some individuals are prone to skin rashes on contact with the oil from certain plants. A visual site inspection and identification of possible poisonous plants should be completed prior to each shift so that assigned personnel are aware of the potential for exposures.

The Center for Disease Control in Atlanta, Georgia has established a hotline for inquiries regarding the hantavirus, at (877)-232-3322. Hantavirus has resulted in several deaths in the western part of the United States. While there may not have been any outbreaks or notices of the virus at a given project site, field personnel should be aware of the exposure route for the hantavirus and potential control methods. The hantavirus is transmitted through atmospheric dispersion of dried rodent excreta. The disease associated with the hantavirus begins with one or more symptoms that may include fever, muscle aches, headache, and cough. The disease progresses rapidly to a severe lung disease that often requires intensive care and treatment.

3.0 ASSIGNMENT OF RESPONSIBILITIES

Implementation of the Health and Safety Plan will be accomplished through an integrated effort of the following personnel:

TABLE 3-1
PROJECT HEALTH AND SAFETY CONTACTS

Company or Agency	Contact	Title	Telephone
P4 Production	Rachel Roskelly	Program Manager	208-547-1248
MWH	Vance Drain	Project Manager	801-617-3250
	Emily Yeager	MWH Program Safety Officer/Field Team Lead	801-349-6276
	Ken Moosman	Database Manager	801-617-3238
	Betty VanPelt	Project Chemist	801-617-3243

The roles of the key individuals from the preceding table are discussed further in the following paragraphs. Individuals involved in the project will follow this plan and will sign the *Personal Acknowledgement Form* in Appendix F.

3.1 VICE PRESIDENT-IN-CHARGE

As the senior management representative for the program, the Vice President-in-Charge is responsible for defining program objectives, allocating resources, establishing the management organization, and evaluating program outcomes. The Vice President-in-Charge, working through the assigned MWH Program Manager, is ultimately responsible for:

- Providing the facilities, equipment, and budget needed to perform work safely;
- Ensuring adequate personnel and schedule for safe operations;
- Ascertaining appropriate review and distribution of health and safety documents;
- Supporting the efforts of program and field personnel; and,
- Applying appropriate disciplinary action for unsafe acts or practices.

3.2 PROJECT MANAGER

The Project Manager has overall responsibility for the safe performance of project activities. If a health and safety issue develops in the course of performing the contract that requires consultation with the client, the Project Manager is responsible for contacting the appropriate client representative and obtaining agreement on necessary actions, and for providing project personnel with suitable guidance.

3.3 PROGRAM SAFETY OFFICER

The PSO shall:

- Work with the OSO to implement the requirements of this Health and Safety Plan.
- Be available for consultation with the Project Manager, FTL, and the OSO during the course of field work covered by this Health and Safety Plan.
- If needed, conduct periodic inspections of field activities to evaluate the effectiveness of the health and safety program and compliance with the Health and Safety Plan.
- Be responsible for the preparation of and any subsequent amendment to the Health and Safety Plan.
- Consult with the Project Manager as necessary prior to approving changes to the Health and Safety Plan.
- Coordinate modifications to the Health and Safety Plan with the OSO.
- Prepare the materials to be used for project-specific health and safety training.

The PSO is also responsible for the following:

- Ensuring that health and safety documentation conforms to applicable federal, state, and local health and safety requirements.
- Ensuring that medical monitoring, incident reporting, and health and safety recordkeeping conform to applicable federal, state, and local requirements.
- Overseeing project-specific employee training and medical surveillance.

3.4 ON-SITE SAFETY OFFICER

The MWH OSO, which will be determined on a project to project basis, has the responsibility and authority to halt or modify any activity or to remove personnel from the site if he or she considers conditions to be unsafe. Additionally, the OSO shall:

- Be responsible for implementing the requirements of this Health and Safety Plan.
- Maintain current certification in cardiopulmonary resuscitation (CPR) and first aid.
- Ensure that field personnel understand and comply with safety requirements, as outlined in this Health and Safety Plan.
- Ensure that a copy of the Health and Safety Plan accompanies each sampling team.
- Coordinate with the PSO, FTL, and Project Manager to address any unanticipated conditions that develop during the course of field activities.

- Coordinate with any subcontractor-designated OSO to resolve unsafe behavior and unsafe conditions posed by subcontractor personnel.

- Be responsible for dismissing subcontractor personnel when resolution of unsafe acts and conditions cannot be reached.

- Obtain approval of amendments to the Health and Safety Plan from the PSO before implementing any deviations from stipulated health and safety procedures.

- Provide the field personnel with the JRA worksheets and review them daily during tailgate safety meetings.

- Conduct and document daily safety debriefing meetings and inform team leader of any accidents or near hit/misses.

- Be responsible for controlling the entries to and exits from sampling locations.

- Monitor on-site hazards and conditions.

- Monitor field personnel for signs of thermal stress and fatigue.

- Enforce the buddy system.

- Enforce procedures for personnel and equipment decontamination, as specified in this Health and Safety Plan.

- Know emergency procedures and evacuation routes, as well as the telephone numbers of the nearest ambulance service, local hospital, poison control center, fire department, and police department.

- Verify the route to emergency medical facilities, and ensure that route information is posted.

- Serve as the primary MWH contact during any on-site emergency.

- Direct responses to emergencies as outlined by emergency response plans (see Appendix E).

- Participate in accident/incident and near miss investigations.

- Ensure that personal protective equipment (PPE) specified for use in this Health and Safety Plan is available and is being used by project personnel.

- Ensure that equipment used for assessing health hazards is calibrated and maintained in good working order.

- Periodically inspect protective clothing, as well as equipment used for assessing health hazards, for defects and signs of wear.

- Inspect and maintain first-aid kits and other emergency supplies.

- Confirm with the PSO or the responsible subcontractor official the ability of each individual assigned to field activities to perform site work, and maintain a file of current training and medical surveillance certificates.

- Enforce written medical restrictions for field personnel, as necessary.

3.5 FIELD TEAM LEADER

The FTL, which will be determined on a project to project basis, is responsible for assembling and managing the field personnel and field equipment during the sampling event. He should thereby assist the OSO in looking out for unsafe equipment and procedures. The FTL is also responsible for managing the sampling schedule and determining what team members sample what location or perform what task. There may be an occasional task or sampling location is more difficult to undertake, or reach, than others. It is therefore be the responsibility of the FTL to delegate tasks responsibly, keeping safety in mind. The FTL is responsible for sending, via email, a daily field report to the P4 Production Program Manager. Thus it is the responsibility of the FTL to discuss with the OSO any incidents so that the FTL can include this in his/her daily report.

3.6 FIELD PERSONNEL

Field personnel are also responsible for understanding and complying with the requirements of this Health and Safety Plan, and are required to sign an acknowledgment to that effect. Field personnel are also responsible for bringing perceived unsafe conditions, accidents, or near hits/misses to the attention of the FTL and OSO during each daily safety debriefing meeting, or sooner if conditions warrant. During the daily tailgate safety meetings, any subcontractor personnel who will be providing services shall inform the rest of the field team of any additional hazards posed by their procedures or the operation of their equipment.

3.7 SITE VISITORS

Visitors, including MWH and subcontractor management or staff, regulatory agency personnel, or client personnel, may be present at the project site during field activities. Visitors can most likely be provided with a general viewing area at a safe location that will not contribute any cross contamination. The OSO can provide a brief overview of the field activities to any site visitors.

If unannounced visitors request access to a project site, the OSO shall inform the appropriate client representative to obtain permission or denial of access.

4.0 PERSONNEL TRAINING

Individuals assigned by MWH to complete field work in the Southeast Idaho Phosphate Resource Area will, as appropriate, be required to meet the general site worker standards of this Plan including the training requirements described below.

4.1 INITIAL SITE-SPECIFIC HEALTH AND SAFETY TRAINING

Site-specific health and safety training will be provided by P4 to MWH employees charged with completing the field assignments as part of this investigation. This site-specific safety training attempts to encompass all hazards that may be encountered at any of the P4 mines. This training also contains a vehicle safety checklist that must be passed before driving on the premises is allowed. This training document is attached herein as Appendix G, *Monsanto Contractor/Guest ES&H Site Guidelines*. P4 also requires contractors to complete a pre-Job Risk Analysis (JRA) worksheet for each individual job the contractor performs. JRAs are meant to bring to light the potential hazards and risks associated with each job. A blank JRA worksheet is attached herein as Appendix H. Furthermore, N.A. Degerstrom Inc., the contracted on-site mining operator, requires that all users of the haul roads at the site be trained in haul road safety. A copy of the Degerstrom training document is attached herein as Appendix I, *Degerstrom Ore Haul Road Travel Requirements*.

P_4 Production's three Sites, associated surface water features that drain these Sites, and applicable background locations, form the study area for the project for which this health and safety plan covers. A specific map of the project area will be located in the task-specific Sampling and Analysis Plans (SAPs).

On the basis of available and extensive information, MWH has demonstrated that the work being conducted under this plan does not involve the reasonable possibility for employee exposure to safety or health hazards attributable to hazardous substances. Furthermore, the waste rock, which is the source of contamination of interest, does not meet the four-prong definition of a hazardous substance per 29 CFR 1910.120(a)(3). Therefore, the training requirements of 40 CFR §1910.120 are not applicable to field personnel involved in the 2009 sampling efforts; however, it is recommended that all MWH personnel are HAZWOPER trained.

It is the responsibility of the OSO to review each area subject to sampling, prior to allowing a field team to enter the area, to ensure that none of the proposed sampling areas fall within the above-listed locations or activities. If a proposed sampling location or activity is determined by the OSO (or other informed person) to fall within one of the above-listed locations or activities, the OSO has the authority and responsibility to stop the proposed sampling activity until it is ascertained that the field personnel entering the area meet the requirements of 40 CFR §1910.120, including the appropriate level of HAZWOPER training.

Notwithstanding the above statements, MWH expects that all field personnel are familiar with, understand, and follow the health and safety requirements and guidance provided in this manual. Training will be conducted prior to job start-up, and as needed thereafter. The PSO, OSO, and/or

the Program Manager(s) will conduct the initial site-specific training to ensure that field personnel have a thorough understanding of this Health and Safety Plan, applicable standard operating procedures (SOPs), and the chemical, physical, and biological hazards that may be associated with the investigation. This training will be repeated for new field personnel tasked with field assignments connected with the investigation as well, prior to starting field work.

Topics that will be addressed in the initial site-specific health and safety training will include the following:

- Names of personnel and others responsible for project safety and health.
- Employee rights and responsibilities under OSHA.
- The Health and Safety Plan, including the medical surveillance program.
- The acute and chronic effects of exposure to hazardous substances that may be encountered during field activities; the potential routes of exposure and symptoms of exposure for these substances; the PELs and IDLH concentrations assigned to these substances; and the level of personal exposure that can be anticipated.
- Likely physical hazards such as slipping, tripping, or falling; noise; electrocution; being struck; or being caught in or between moving equipment.
- Site control measures, including procedures for chemical handling, spill containment, decontamination, fire prevention, and any SOPs prepared specifically for the project.
- Hazard communication (per the requirements of 40 CFR § 1910.1200).
- PPE and the action levels for upgrading PPE and for evacuating work sites.
- Engineered controls such as dust suppression techniques adopted for this project.
- Emergency procedures and equipment.
- Any procedures adopted for air monitoring, including the functions, limitations, use, and maintenance of monitoring equipment.
- Proper use of heavy equipment and machinery, as applicable.
- Personal cleanliness and restrictions on eating, drinking, and smoking at the job site.
- Heat and cold stress prevention, monitoring, and treatment.
- Contractor injury and illness prevention programs, if applicable.

Field Personnel will also be instructed in the use of the buddy system. The buddy system will be used whenever field personnel are collecting samples from any location in the project study area.

The buddy system is a method of organizing work groups so that an individual is always available to provide his or her partner with assistance in an emergency; to monitor his or her partner for signs of chemical or physical exposure, to periodically check that his or her partner's PPE is functioning properly, and to notify emergency response personnel if an emergency occurs. The buddy system usually requires that two or more people maintain visual contact while working. However, the buddy system can employ radio or cell phone contact if site conditions are such that a person could otherwise work alone. In order to deviate from the buddy system, the PSO will require an explanation of the specific task to be completed, along with a procedure for assuring that a single person work party is safe.

Note that activities that will be conducted during the investigation WILL require the MSHA 24-hour training specified under 30 CFR 48.21 through 48.30. The OSO will maintain documentation that each site worker has successfully completed the initial site-specific training and any additional safety training furnished by a mine operator at an active mine site.

4.2 TAILGATE SAFETY MEETINGS

Tailgate safety meetings will be conducted by the OSO each day field activities take place. This meeting will review the JRAs for the applicable jobs, cover site activities, changes in site conditions, other activity-specific health and safety issues, and include a review of pertinent topics detailed in the initial site-specific health and safety training. Field personnel will also be informed of the availability of emergency assistance, as well as the most probable route of evacuation from a site, should an evacuation become necessary.

A daily safety debriefing will also be conducted at the conclusion of every work day; at which any unsafe working conditions or equipment and any incidents or near hits/misses must be discussed and documented. The OSO must inform the FTL of any such incidents.

If MWH personnel are working directly with a contractor (i.e. a well driller), the MWH contractor supervisor is to engage in this same tailgate safety meeting process and report to the OSO or PSO any safety concerns and incidents or near hits/misses.

4.3 BLOODBORNE PATHOGENS AND FIRST AID/CPR

Personnel assigned to conduct field work for this project do not conduct first aid or CPR as a primary job function. Rather, selected field personnel (e.g., the OSO) have been trained in first aid and CPR for application in an emergency only. Acting in the capacity of a designated emergency first aid provider is not mandatory, and anyone who is uncomfortable with the possibility of being so designated should notify the OSO.

4.4 DOCUMENTATION OF TRAINING

Written documentation verifying compliance with the training requirements of this section must be submitted to the MWH PSO or OSO prior to the beginning of field work or site access. Documentation of each worker's current training credentials will be kept by the OSO for review by authorized agency personnel.

5.0 MEDICAL SURVEILLANCE

Personnel who will be completing field assignments in support of the investigation on locations or involved in activities in which the requirements of 29 CFR § 1910.120 govern (see discussion in Section 4.1 of this Plan), must be participating in a medical surveillance program consistent with the requirements of that regulation. These requirements mandate that field personnel receive medical examinations prior to participating in hazardous waste site activities; annually; upon termination; following occupational exposure or injury; and additionally as needed, on a case-by-case basis.

The medical surveillance program required of each company that allows personnel to conduct field work at hazardous waste sites must be overseen by a licensed physician who is certified in occupational medicine by the American Board of Preventive Medicine, or, who by training and experience, is Board-eligible. When applicable, the MWH PSO will maintain copies of the physician's written authorization statements that employees conducting hazardous waste site operations are fit for hazardous waste site duty and are able to wear respiratory protection. No one shall be permitted to participate in hazardous waste site operations subject to these requirements until a copy of their medical certification is received by the MWH PSO. Copies of the physician's authorization for field personnel will be available to the field personnel upon request. Medical and exposure records will be retained for the length of the employee's employment, plus 30 years.

Field personnel will receive additional medical monitoring upon notifying the OSO, PSO, Program Manager, or other authorized MWH personnel of symptoms consistent with over-exposure to site contaminants, or if the employee is injured or exposed to contaminants at concentrations in excess of a PEL during emergency response operations. Further medical examinations may be required before an employee returns to work after a serious illness or injury. Such examinations may be necessary to assure the employee's continued ability to carry out assigned duties. The need for these examinations will be determined by the MWH PSO, in cooperation with the occupational health physician representing the company. An injury or illness incurred by one of the field personnel, whether on or off the job, shall be reported to the PSO or OSO immediately. Such injury or illness may also require work restrictions when/after the employee returns to work. If the injury or illness required seeing a physician, either the attending physician or the physician giving the employment physical will be involved in deciding when the employee can return to work, and if any work restrictions will apply.

6.0 PERSONAL PROTECTIVE EQUIPMENT

6.1 PERSONAL PROTECTIVE EQUIPMENT

The Environmental Protection Agency (EPA) designations of Levels D, C, B and A for PPE are used to describe the general PPE ensembles that may be employed during hazardous waste site operations. These ensembles are depicted in Figure 6-1. Based on site contaminant information and established exposure limits, Level D has been selected as the level of protection appropriate for field personnel during this investigation. Field personnel will also adhere to the requirements of individual mine operators when collecting samples at active and inactive mine sites. These requirements may include the use of hardhats, eye protection and steel-toed boots. It is the responsibility of the OSO and the OSO representing any subcontract personnel to ascertain the appropriate/required level of PPE for each mine site, communicate such findings to the field personnel, and to ensure that the field personnel are provided with the PPE in a timely manner.

PPE that will be employed for project field tasks and procedures is identified in the Activity Hazard Analysis in Appendix A. For activities undertaken during this project, personnel will incorporate the following into the standard Level D ensemble:

Coveralls:	Personnel will wear a work uniform that includes long pants and a short-sleeved t-shirt at a minimum.
Gloves:	Nitrile gloves are required when handling samples, reagents, decon solutions, and calibration standards.
Hardhat:	Personnel will wear a hardhat when entering active and inactive mine pits to protect against rock falls, when they are visible from the haul road, and when supervising well installations.
Safety Glasses:	Personnel will wear safety glasses when entering active and inactive mine pits and when visible from the haul road. They will also be worn during sample collection, equipment decontamination, and when supervising well installations.
Ear Plugs:	Personnel will use ear plugs or ear muffs with sufficient muffling abilities while drill rigs are in operation.

Once on site, the OSO and the OSO representing any subcontractor personnel will evaluate work conditions and adjust the level of PPE as necessary to properly protect field personnel and meet the local mine requirements. When specifying a PPE ensemble, the following will be evaluated:

- The local mine requirements;
- The anticipated site hazards that were used to select the initial PPE ensemble;
- The limitations of each piece of PPE;
- Work duration;

- The effect of temperature extremes on the PPE ensemble;
- PPE maintenance, storage, decontamination, and disposal requirements;
- Inspections of PPE completed prior to, during, and after use;
- Personnel training in PPE use and the need for fit-testing;
- Procedures for donning and doffing; and
- Evaluation of the effectiveness of the current PPE program.

Adjustments to the PPE ensemble will be communicated to field personnel via amendment of this Health and Safety Plan or during the tailgate safety meeting.

6.2 LEVEL D PERSONAL PROTECTIVE EQUIPMENT

Level D protection may be used when the following conditions are met:

- Substances that pose inhalation hazards are not present above individual or combined PELs.

- Oxygen is present at a minimum concentration of 19.5 percent.

- Toxic organic compounds are not present in the air space at concentrations that exceed normal background concentrations or specified action levels requiring use of respiratory protection.

- Work functions preclude splashes, immersion in, unexpected inhalation of, or direct contact with hazardous concentrations of harmful chemicals.

Level D protective equipment shall consist, at minimum, of the following:

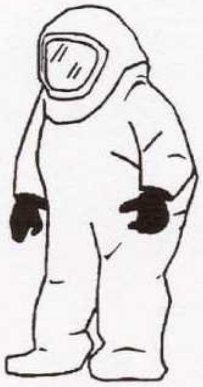
- Dedicated work uniforms with long pants and short-sleeve shirt.

- Steel-toed and shank leather, PVC, or rubber safety shoes or boots meeting the specifications of American National Standards Institute (ANSI) Z41.

- Safety glasses, goggles, face shield, or other approved eye protection.

- Hardhat, unless specifically stated otherwise.

The dedicated work uniforms may include chemical-resistant coveralls or standard Tyvek coveralls, or standard cotton or cotton blend work uniforms. Approved eye protection must meet the specifications of ANSI Z87.1. The use of contact lenses is discouraged, but not prohibited, during Level D operations. However, safety glasses or goggles that fit over prescription lenses or prescription safety glasses or goggles are recommended. Approved hardhats must meet the specifications of ANSI Z89.1.



Level A Protection
Totally encapsulating vapor-tight suit with full-facepiece SCBA or supplied-air respirator.

Level B Protection
Totally encapsulating suit does not have to be vapor tight. Same level of respiratory protection.

Level C Protection
Full-face canister air purifying respirator. Chemical protective suit with full body coverage.

Level D Protection
Basic work uniform, i.e. longsleeve coveralls, gloves, hardhat, boot, faceshield or goggles.

SAMPLE PROTECTIVE EQUIPMENT ENSEMBLES
FIGURE 6-1

6.3 PPE STORAGE

PPE must be stored properly to prevent damage or malfunction due to exposure to dust, moisture, sunlight, damaging chemicals, extreme temperatures, and impact. Potentially contaminated PPE should be stored separately from new PPE and street clothing. Field personnel should always review the manufacturer's instructions for care and maintenance of PPE. PPE storage will be provided in the field by the OSO. However, each individual is responsible for ensuring that his or her issued PPE is protected from extremes of temperature, and is stored in a manner that prevents the PPE from becoming damaged or disfigured.

7.0 HAZARD ASSESSMENT

Because of the relatively low exposures that are expected during the investigation, no vapor or dust monitoring will be undertaken. However, personnel monitoring for heat and cold stress will be performed. Heat and cold stress will be monitored qualitatively, as described in the following paragraphs.

7.1 HEAT STRESS

The stress of working in a hot environment can cause a variety of illnesses, including heat exhaustion or heat stroke; the latter can be fatal. The use of PPE can increase heat stress significantly, although heat stress can overcome people wearing regular, permeable work clothing, as well. To reduce or prevent heat stress, frequent rest periods and the intake of salts and liquids to conserve and replace body fluids may be necessary.

Personnel should recognize the symptoms of heat stress, and take appropriate action on recognition. Some of the symptoms that indicate heat exhaustion are:

- Clammy skin
- Lightheadedness
- Slurred speech
- Rapid pulse
- Weakness, fatigue
- Confusion
- Fainting
- Nausea (vomiting)

If these symptoms are noted, the following steps should be taken:

- Remove the victim to a cool and uncontaminated area;
- Remove protective clothing; and
- Give water to drink, if conscious.

Symptoms that indicate heat stroke include:

- Staggering gait
- Hot skin, temperature rise (yet may feel chilled)
- Incoherent, delirious
- Mental confusion
- Convulsions
- Unconsciousness

If these symptoms are noted, the following steps should be taken:

- Remove victim to a cool, uncontaminated area;
- Cool the victim, whole body, with water, compresses and/or rapid fanning;
- Give water to drink, if conscious; and
- Transport the victim to the designated medical facility for further cooling and monitoring of body functions.

HEAT STROKE IS A MEDICAL EMERGENCY!

7.2 COLD STRESS

On days of low temperature, high wind, and humidity, anyone can suffer from the cold. Severe exposure to cold can be life threatening. Several factors increase the harmful effects of cold: being very young or very old, wearing wet clothing, having wounds or fractures, smoking, drinking alcoholic beverages, fatigue, emotional stress, and certain diseases and medications.

Cold weather injuries may be local or systemic. Local cold weather injuries include chilblains (chronic injury of the skin and peripheral capillary circulation) and frostbite. Frostbite occurs in three progressive stages: frostnip, superficial frostbite, and deep frostbite. Systemic cold injuries associated with hypothermia affect the entire body system. Hypothermia is caused by exposure to cold and is aggravated by moisture, cold winds, fatigue, hunger, and inadequate clothing or shelter.

Precautionary measures that will be taken to prevent or mitigate cold stress will include:

- Providing field shelters or wind screens.
- Monitoring temperature and wind speed to determine appropriate safety measures.
- Adjusting work schedule based on weather conditions and temperature.
- Providing insulated clothing for field workers.
- Adhering strictly to the buddy system so that workers can monitor for symptoms of cold stress in their co-workers.

7.2.1 Frostbite Monitoring

Frostbite is a potentially crippling condition that can occur when inadequately protected skin or body parts are exposed to freezing weather. Team members should continually be alert for signs of frostbite in co-workers, and bring any occurrences to the attention of the OSO. A cold feeling, pain, and numbness precede the onset of frostbite. Frostbite usually appears as gray or white waxy spots on skin. Areas most susceptible to frostbite are the nose, ears, and cheeks.

The following steps should be taken to avoid frostbite:

- Dress warmly;
- Wear layers of clothes;
- Keep boots and gloves loose-fitting;
- Stay dry;
- Carry extra clothing;
- Avoid touching cold metal with bare hands; and
- Avoid spilling cold fuel, alcohol, or other liquids that freeze below 32°F on your body or clothing.

If a person is frostbitten, get them to a hospital as soon as possible. If transport to a hospital is not immediately available, get the person to a warm shelter and immediately perform the following:

- Cover exposed areas with additional clothing;
- Wrap the person in blankets or a sleeping bag;
- Give the person warm, non-alcoholic drinks;
- Undress the frozen part and submerge the frozen part in a tub of warm water (102° F to 105°F), or put the frostbitten person in a large tub of warm water, if available, and stir the water;
- Warm with skin to skin contact, such as placing warm hands on frozen nose or ears, (but do not rub); and
- Get the person to a hospital as soon as possible.

Do not rub the frozen part; do not give the person liquor; do not allow the person to walk on thawed feet; do not let the person smoke; do not break any blisters that may form; do not let the thawed part freeze again; and do not warm the frozen part in front of a source of dry heat, such as an open fire or oven.

7.2.2 Hypothermia Monitoring

Hypothermia is a lowering of the body's temperature due to exposure to cool or cold temperatures. Field personnel should be continually alert for signs of hypothermia in co-workers, and bring any signs of hypothermia to the attention of the OSO. Most cases of hypothermia occur at temperatures between 30°F and 50°F. Hypothermia is a medical emergency: if not properly treated, hypothermia can cause death. Safety equipment for hypothermia should include a synthetic sleeping bag and a hypothermia thermometer. Personnel suffering from hypothermia should be transported to a hospital as soon as possible, even if they appear to be recovering.

To prevent hypothermia:

- Eat well prior to exposure;
- Dress warmly; and
- Avoid becoming wet through sweating, rain or snow, or falling in water.

Early signs of hypothermia may include violent shivering, slurred speech, a loss of coordination, confusion and an inability to answer simple questions, unusually irritable or strange behavior, or a tendency to drop or lose clothing or equipment. As hypothermia progresses into more serious stages, the victim typically develops trouble seeing clearly, becomes sleepy and numb, and begins to move with difficulty. Eventually, the victim will lapse into unconsciousness if not properly cared for.

The following actions should be taken to treat a hypothermia victim:

- Get the victim to a warm, dry shelter as soon as possible;
- Remove any wet or cold garments and dry the person thoroughly; and
- Wrap the victim in blankets, sleeping bags, or dry clothing to prevent more heat loss.

If a warm area is not available, build a shelter and put the victim in the warmest, driest area available. Remove any wet or cold garments, and have one or more persons remove their clothing and lay next to the victim, providing skin to skin contact. Then, wrap the victim and

rescuers in dry, warm blankets, sleeping bags, or clothing. When the victim becomes conscious, place warm objects along the victim's sides to warm vital areas. When the victim is able to swallow easily, provide warm, sweetened drinks and food, preferably candy or sweets. Do not give the victim alcohol or allow the victim to smoke, do not rub the victim's skin, and keep checking the victim and providing additional assistance as needed.

8.0 SITE CONTROL

Site control is an important part of a field health and safety program. The purposes of site control are to minimize potential worker exposures, protect the public from site hazards, and prevent vandalism of site facilities. Site control procedures that will be implemented during the RI/FS, which are discussed in this section, consist of site security controls and communication systems.

Under no conditions are deviations from safe work practices to be tolerated by anyone on site. If any deviation continues after a reminder of proper procedures or a reasonable warning, the MWH OSO will be informed of the circumstances. The MWH OSO will attempt to correct the unsafe behavior or unsafe condition. Should this attempt fail, the MWH OSO shall halt site activities and dismiss the non-cooperative personnel.

8.1 MULTIPLE-EMPLOYER JOB SETTING

Enforcing safe work practices at a multiple-employer job site presents many challenges. Under OSHA, each employer is required to provide a safe and healthful working environment for its employees (see OSHA poster in Appendix J). Most hazardous waste sites require several contractors to work simultaneously on different project tasks. In this situation, the activities of one company could create hazards for the employees of another company. It is not possible to anticipate every hazard associated with activities at a multiple-employer job site in a Health and Safety Plan. The OSO must discuss particular safety and health issues that may be associated with each day's activities at the daily tailgate safety meeting.

8.2 SITE SECURITY

Existing site controls that are likely to be encountered during the course of the investigation will vary from no controls to strict property perimeter controls. When possible, client personnel will be requested to investigate any suspicious activities at the field sites. In some cases, an independent security watch may be needed. Security at the sites will be the responsibility of the client during periods of inactivity, including weekends. To maintain security at the sites during working hours, the OSO will:

- Control site entrances and exits as necessary through the installation of appropriate safety barricades, signs, and/or signal lights.
- Establish a personnel identification system.
- Be responsible for enforcing entry and exit requirements.
- Utilize temporary fencing to control site access, where feasible.
- Post warning signs around the perimeter of the work area, should the use of temporary fencing not be feasible.

To maintain security during nonworking hours, the OSO will secure the site prior to leaving at the end of a working day and equipment and supplies will be secured or stored in locked facilities.

8.3 COMMUNICATION SYSTEMS

Two general types of communication systems should be available for workers assigned to field projects. One system will ensure adequate communication between field personnel, and the other will ensure the ability to contact personnel and emergency assistance off site. Internal communication will be used to:

- Alert team members to emergencies.
- Pass along safety information, such as weather conditions that could affect heat stress, cold stress or general safety.
- Maintain site control.
- Facilitate site work by being able to call the appropriate party for information without having to decontaminate the work party and equipment and secure the site.

Verbal communication can be impeded by background noise and limitations imposed by PPE. It is therefore vital that pre-arranged signals of communication be arranged prior to the initiation of site activities, particularly when heavy equipment may be operating in the vicinity. Common types of internal communication devices include:

- Radios;
- Noisemakers such as compressed air horns, megaphones, sirens, and whistles; or,
- Hand and arm signals.

External communication systems between on-site and off-field personnel are necessary to:

- Coordinate emergency response efforts,
- Report to upper management about site activities, and
- Maintain contact with essential off-field personnel.

The primary means of external communication are telephones, radios, facsimile machines, and computer networks.

9.0 DECONTAMINATION PROCEDURES

Decontamination procedures are implemented to prevent cross-contamination of samples, to control possible migration of site contaminants to clean areas, and to prevent personnel exposure to chemicals or pathogens that may contaminate clothing or protective gear. Personnel conducting field activities must decontaminate upon the completion of these activities**.** Equipment must also be decontaminated before it is moved. Any material that is generated by decontamination procedures will be labeled and stored until final disposal arrangements are made.

9.1 GENERAL DECONTAMINATION PROCEDURES

Decontamination procedures shall be supervised by the OSO. The type of solution to be used for equipment decontamination is specified in the task-specific SAP and Field Sampling Plan (FSP). Personnel decontamination will be accomplished using ordinary soap and water or an alcohol based hand sanitizer. Personnel will be required to wash or sanitize their hands, and optionally their faces, before eating or drinking, unless specific procedures are in place to ensure that a drink can be taken without the possibility of contamination. Personnel may also be required to wash or sanitize their hands, and optionally their faces, before leaving the work site. Decontamination solutions will be changed daily at a minimum.

The following decontamination procedures and guidelines shall be implemented:

- Disposable protective clothing will be used when possible to eliminate the need for decontaminating clothing.
- Decontamination procedures will be designed to prevent or minimize direct contact with waste materials.
- Disposable protective clothing and contaminated material will be collected in plastic sacks and disposed of appropriately.

9.2 DECONTAMINATION WASTE HANDLING AND DISPOSAL

Wastes generated as a result of site activities will be handled in accordance with applicable environmental regulations. Investigation-derived wastes and contaminated site materials will be handled and disposed of in accordance with the provisions of the FSP or client specifications. Unless specifically stated, personnel are to treat decontamination wastes as part of the investigation derived wastes.

10.0 EMERGENCY RESPONSE PLANNING

The objective of this Health and Safety Plan is to minimize exposure to chemical, biological, and physical hazards, and to prevent work-related illnesses and injuries. Emergency response planning is included as part of this plan to provide procedures for responding to emergencies that may occur. This section contains information on how to deal with emergencies. It is not the purpose of this Health and Safety Plan, however, to provide guidance for emergency response as part of field operations. Field personnel are instructed to assess emergencies and make the appropriate notification to emergency responders. **Under no circumstances are field personnel to take emergency response actions for which they are not properly trained.**

The MWH OSO will serve as the primary MWH contact during any on-site emergency. The OSO will be responsible for making the appropriate notifications, directing responses to emergencies until relieved by a qualified Emergency Medical Technician (EMT) or equivalently trained professional. As part of his or her duties, the OSO will be required to know basic first aid emergency procedures and evacuation routes, as well as the telephone numbers of the nearest ambulance service, local hospital, poison control center, fire department, and police department. The OSO will also be responsible for verifying the route to emergency medical facilities, and ensuring that route information is posted and available to field personnel. Emergency telephone numbers and maps showing the locations of the hospitals and emergency clinics capable of providing emergency service for field personnel are provided in Appendix E. Telephone numbers for the Poison Control Center, local Police and Sheriff's Departments, local Fire Departments, including the emergency rescue squad, the Office of Emergency Services, MWH management, and client contacts also are included. Copies of the hospital route maps provided in Appendix E will be kept in site support vehicles and field personnel will become familiar with the routes and the travel times involved.

The OSO shall immediately notify the Program Manager of the following:

- Any required site evacuation;
- Any fatality or injury to one or more field personnel that requires medical attention; and
- Any physical hazard creating the potential for death or permanent injury.

Vehicles that can be used to transport injured personnel from work sites will be available during working hours. A system will also be available on site for communicating with off-site personnel. On-site communication systems may include cell phones, two-way radios, or other suitable devices. Additionally, first aid supplies and potable water will be available at every site for emergency use.

Cell phones may not always be operable in remote areas. Thus, when entering remote areas field personnel will file their anticipated itinerary with the FTL. The itinerary will include where the field team expects to be, the travel route, and the expected time of return. The field team will notify the FTL upon their return. If the sampling team leaders do not hear from a field team by an agreed-upon time, the FTL will initiate search and rescue operations. In addition, the FTL will

attempt to call the P4 Program Manager daily to report on the daily activities, any health and safety issues encountered, and the expected itinerary for the next day.

Prior to the start of work, project personnel will be acquainted with established emergency response procedures and equipment. Furthermore the OSO will be certified to render first aid and CPR prior to commencement of field activities. The buddy system will be used when working in remote areas, near significant waterways, or in active or inactive mine pits. The buddy system will not be required while collecting samples in agricultural areas when cell phone contact is available.

Accidents, safety-related incidents, and safety-related near misses will be documented and reported to the MWH EH&S Department, PSO, OSO and P4 and MWH Program Managers on a daily basis at a minimum.

See Appendix K for incident reporting forms and procedures.

11.0 HEALTH AND SAFETY DOCUMENTATION

Examples of some of the forms that may be employed for documenting compliance with the MWH health and safety program and this Health and Safety Plan are presented in the appendices. The MWH PSO or designated OSO will maintain and update these documents. Appropriate regulatory agency personnel shall be granted access to these records if requested.

Unanticipated field conditions may occasionally require temporary modification of this Health and Safety Plan. Client notification and approval procedures will depend on field conditions and nature of the modification. Any upgrade to PPE will be reported in an updated JRA worksheet. Minor changes to the Health and Safety Plan to accommodate on-site conditions can be implemented by the OSO upon review and approval of the PSO; such changes might include minor revisions to decontamination or site control procedures. Permanent changes to this HASP must be approved by the MWH Project Manager Project Manager as listed in Table 3-1.

12.0 GENERAL SITE SAFETY

The health and safety program contained in this portion of each MWH Health and Safety Plan has been developed in accordance with relevant occupational safety and health regulations and requirements, and applies to field sites and workplaces. Because this section is intended to be applicable to a wide range of sites and conditions, there may be information in this section that applies to certain areas of the country only.

The following practices are expressly forbidden during site work:

- Smoking, eating, drinking, chewing tobacco, or applying cosmetics while taking samples or while near exposed samples.
- Contact with potentially contaminated substances; walking through puddles or pools of liquid; kneeling on the ground; or leaning, sitting, or placing equipment on contaminated soil.
- Performance of tasks without a buddy; personnel will be required to use the buddy system unless specifically exempted elsewhere in the Health and Safety Plan.

Personnel must keep the following guidelines in mind when performing field activities:

- Hazard assessment is a continuous process. Personnel must be aware of their surroundings and the chemical and physical hazards that are present.
- Field personnel will be aware of the physical characteristics of each site, including site access, the location of overhead power lines and underground utilities, wind direction, and the location of communication devices and safety equipment.

12.1 GENERAL HEALTH AND SAFETY PROGRAM ELEMENTS

12.1.1 Hazard Communication Program

MWH has a written Hazard Communication Program. This program appears as MWH H & S Policies Procedure No. 1000. A copy of this will be available to field personnel. As necessary, the hazard communication program of subcontractors also will be solicited for use as a project reference. MWH and subcontractor field personnel will be made aware of the MWH Hazard Communication Program and have access to MSDSs for any chemicals brought to field sites. MWH will be responsible for supplying MSDSs and each field team will have them in their possession when in the field.

12.1.2 Sanitation

Work breaks, eating, drinking, and paperwork tasks will be performed in the field vehicle or other suitable location away from sampling location. Field personnel will wash or otherwise sanitize their hands prior to eating or drinking.

The OSO is responsible for ensuring that an adequate supply of water is available at the site. During times of heavy labor and hot temperatures, it is recommended that approximately one liter of water per hour be ingested. Sport-type beverages also may be provided for field personnel. It is to be assumed that there is no potable water in the field. When decontamination procedures interfere with the ability of field personnel to obtain sufficient drinking water, personnel may drink water without prior personnel decontamination under the following stipulations:

- Water is dispensed from a cooler with a pull-lever pouring spout. Push-button pouring spouts are unacceptable, as dirty fingers can easily contaminate the pouring spout.
- Minimum three-inch tall disposable drinking cups must be used and discarded after each use.
- Drinking cups must be dispensed out of a plastic or metal dispenser attached to the cooler, allowing the bottom of the cup to be grabbed without touching the top rim.
- Bottled water or sports drinks with removable caps, taking care not to touch the drinking surface with potentially contaminated hands.

12.1.3 Illumination

All site work will be done during daylight hours, with the exception of driving to and from the work site.

12.1.4 Standard Emergency Hand Signals

Team members should be familiar with the following emergency hand signals:

- Hand gripping throat: “Respirator problems, can’t breathe.”
- Grip team member’s wrist or place both hands around waist: “Leave site immediately; no debate!”
- Thumbs up: “OK, I’m all right; I understand.”
- Thumbs down: “No, negative.”
- Hands on face: “Put on respirator.”

12.1.5 Fire Protection

Field activities performed during the summer at this location could potentially result in a fire at the site. Cigarette smoking is forbidden at any sampling locations and should be done with care otherwise (extinguished and disposed of properly). Driving off road should be avoided if possible, as vehicles driving over tall stands of brush and grasses can easily spark a fire.

Electrical wiring will be free from frayed ends and sections, and hook-ups will be checked for loose fittings. Portable power tools will be connected to a ground fault circuit interrupter, and care will be taken to ensure that electrical connections do not exceed the maximum load capacity for any one circuit.

12.1.5.1 Wildfires

Wide open areas of natural brush present the danger of wildfires. Many project sites have structures that can provide enough of a fire break to prevent a wildfire from endangering field personnel, but such a structure does not provide absolute protection. The MWH OSO will therefore check regularly with the local fire department during the most common wildfire months of July through November. Should a wildfire threaten a work site, the MWH OSO will watch for changing conditions and evacuate and secure each active site, in accordance with local fire department instructions.

12.1.5.2 Fire or Explosion Response Action

The actions listed below are in a general chronological sequence. Conditions and common sense may dictate changes in the sequence of actions and the addition, elimination, or modification of specific steps.

Immediate Action. Upon detecting a fire/explosion, employees will notify the fire department and determine whether or not the fire is small enough to extinguish readily with immediately available portable extinguishers or water, or if other fire-fighting methods are necessary. Non-essential personnel will be directed away from the area of the fire. If it is judged that a fire is small enough to fight with available extinguishing media, employees will attempt to extinguish the fire provided that:

- They have been properly trained on the use of the specific fire extinguisher to be used.
- They are able to approach the fire from the upwind side, or opposite to the direction of the fire's progress.
- The correct extinguisher is readily available.
- No known complicating factors are present, such as likelihood of rapid spread, imminent risk of explosion, or gross contamination.

Personnel leaving a fire/explosion area will notify the fire department and will account for employees in that area as soon as possible. The OSO or designee will perform a head count of sampling team members.

Notification. The MWH OSO will be notified as soon as possible of the location, size, and nature of the fire/explosion. A member of the MWH management team will notify appropriate agency personnel in the event of a fire or explosion resulting in a release of a hazardous material to the environment. As conditions dictate, the OSO will declare an emergency, initiate the remedial procedures, request assistance from the fire department, and make the necessary on-site and off-site notifications. If assistance from the fire department is required, an escort appointed by the OSO will direct responders' vehicles over clean roads to the extent possible to limit contamination. Note: National Fire Protection Association (NFPA) guidelines call for notifying the fire department, even for small fires, to ensure proper handling.

Rescue. If personnel are unable to evacuate themselves from a fire/explosion area for any reason, their rescue will be the first priority of responders. The FTL and/or OSO will determine whether on-site resources are sufficient to proceed, or if rescue must be delayed until outside responders arrive. **Field personnel are not to take any actions which place themselves or other in danger and/or for which they have not been properly trained.**

Fire-Fighting Procedures. Planned fire-fighting procedures are described below. These apply to small fires that the project personnel are able to control.

Fire During Working Hours: In the event that a fire occurs during working hours, the following measures will be taken to put out the fire provided that the person is properly trained to do so. These measures are sequential, that is, if the first measure does not succeed in containing the fire, the next measure will be initiated.

- Use fire extinguishers.
- Confirm that request for assistance from the fire department has been made.
- Utilize earth moving equipment, foam unit, and water truck, as appropriate. Brush fires will be extinguished with water.

Fire During Non-Working Hours: In the event of a fire during non-working hours, existing alarms, site security (if applicable), or whomever from the project team is notified, will notify the MWH OSO or PSO. Additional actions will be consistent with procedures established for a fire during working hours.

Response Coordination. Upon arrival of outside responders from the fire department, the OSO will coordinate with the leader of the outside responders to direct fire-fighting activities; however, the control of the scene is now the responsibility of the leader of the outside responders.

Protection of Personnel. The primary methods of protecting personnel from fire conditions will be by distance and remaining upwind. Based on the conditions, the OSO will determine appropriate distances and the selection of personal protective equipment for field personnel.

Decontamination. At the conclusion of fire fighting activities, the OSO will:

- Determine to the extent practicable the nature of the contaminants encountered during the incident.
- Arrange for outside responders' fire response equipment, and on-site equipment as necessary, to be processed through decontamination, using methods appropriate for the contaminants involved.
- Equipment not easily decontaminated shall be labeled and isolated for further action, such as determining specific contaminants by wipe sampling or awaiting the delivery of specific decontamination media and supplies.

Fire Extinguisher Information. The four classes of fire, along with their constituents, are as follows:

Class A - Wood, cloth, paper, rubber, many plastics, ordinary combustible materials.

Class B - Flammable liquids, gases, and greases.

Class C - Energized electrical equipment.

Class D - Combustible metals such as magnesium, titanium, sodium, and potassium.

Examples of proper extinguishing agents are as follows:

Class A - Water
Water with one percent AFFF Foam (wet water)
Water with five percent AFFF or Fluoroprotein Foam
ABC Dry Chemical
Halon 1211

Class B - ABC Dry Chemical
Purple K
Halon 1211
Carbon Dioxide
Water with six percent AFFF Foam

Class C - ABC Dry Chemical
Halon 1211
Carbon Dioxide

Class D - Metal-X Dry Chemical

No attempt should be made to extinguish a large fire. Large fires should be handled by the fire department. The complete area of the fire should be determined. If human life appears to be in danger, or the spread of the fire appears to be rapidly progressing, move personnel further upwind and away from the fire. Do not attempt to extinguish even a small fire if you have not been properly trained to do so.

Use of Fire Extinguishers. Inspect the fire extinguisher on a monthly basis to ensure that the unit is adequately charged with extinguishing media. Do not store a fire extinguisher on its side. To use the extinguisher, follow the acronym PASS for instructions listed below:

1. **P**ull the pin on the top of the unit.

2. **A**im at the base of the fire.

3. **S**queeze the handle on the top of the unit.

4. **S**weep the extinguishing media along the base of the fire until the fire is out. Ensure that the fire is fully cooled before assuming it is completely extinguished.

12.1.6 Earthquake and Disaster Preparedness

If an earthquake or natural disaster occurs during working hours and the magnitude is such that field personnel may be in danger, the MWH OSO will initiate the site evacuation procedure. This action is to be taken only if in the judgment of project personnel and/or OSO the earthquake is large enough to have potentially caused damage to any of the structures or equipment being used on the site. If the earthquake or disaster occurs during non-working hours, the OSO will determine whether safe entry onto sampling locations can be made, or if an inspection is needed first. If at any time the inspection team feels that they need the assistance of the fire department, the inspection shall cease until the fire department is able to assist. The inspection will be conducted using the buddy system. The team will look at structures, equipment, and any chemical storage areas for signs of cracks or deterioration. When assessing areas known to contain chemicals, appropriate air monitoring equipment will be used to ensure that leaks are detected quickly and without injury to the inspection team. When inspecting areas where chemical releases could have occurred as a result of a breach of containment, Level B PPE is recommended.

12.2 COMMON PHYSICAL HAZARDS AND CONTROLS

This section provides information concerning the common physical hazards associated with site investigations and recommended controls to minimize risk to field personnel. Section 2.0 and Appendix A list the physical hazards specific to this project.

12.2.1 Slip/Trip/Fall

Field personnel are to be vigilant in providing clear footing, clearly identifying obstructions, holes, or other tripping hazards and maintaining an awareness of uneven terrain and slippery surfaces. It is necessary that shoes providing more elaborate tread be worn to minimize slip, trip and fall hazards.

12.2.2 Heavy Lifting

During manual lifting tasks, personnel will remember to lift with the force of the load suspended on their legs and not their backs. They are to maintain a straight back and hold the object close to the body. Mechanical lifting devices or the help of a fellow field team member should be sought when the object is too heavy for one person to lift.

12.2.3 Motor Vehicle Hazards

Motor vehicle accidents can occur any time people drive. Field personnel are required to employ defensive driving techniques, and obey site speed limits and vehicle safety requirements. Accidents are to be reported to the MWH OSO. Working in an active mine area poses unique hazards to personnel whether in a vehicle or as a pedestrian. Mine equipment, especially haul trucks, are often large, may operate at relatively high rates of speed, often have limited visibility, and cannot stop or maneuver like over-the-road vehicles. Unique mine driving rules often apply within a local mine that are different from public road rules. Haul trucks within a mine area often have the right-of-way in every instance. Therefore, it is extremely important that while walking or driving within an active mine area, to be exceptionally alert to all traffic around you. When in doubt, yield the right-of-way. **Field personnel are required to wear seatbelts at all times when in a moving vehicle and the driver must refrain from using his/her cell phone.**

12.2.4 Sharp Edges and Pinch Points

During the course of this site investigation, it is feasible that personnel will encounter sharp edges and pinch points. Sharp objects may include site debris, field tools, equipment, or other objects. Pinch points are places where the hands may be caught between objects or moving parts. When danger of cuts to the hands or other body parts is probable, employees will either arrange paths where personnel may walk without encountering sharp edges, or will ensure during the tailgate safety meeting that areas with known sharp edges are brought to the attention of field personnel. Heavy work gloves shall be used in conjunction with any chemical resistant gloves in circumstances where handling sharp objects are required.

12.3 SEVERE WEATHER

While each project site will be subject to varying types of weather conditions, this section provides general information and controls on several types of severe weather.

12.3.1 Lightning

If a lightning storm is suspected or observed, site activities must be stopped, and site equipment must be evaluated for its potential for acting as a lightning rod. Personnel should wait indoors for the storm or lightning event to end. If the strike of lightning occurs and personnel are out in the field, the response should be to disband from one another and lay low to the ground by dropping to your knees and bending forward with your hands wrapped around your knees, away from any poles or trees.

Persons struck by lightning will receive a severe electrical shock and may be burned, but they carry no electrical charge and can be handled safely. Someone who appears to have been killed by lightning often can be revived by prompt action. Those unconscious but breathing probably will recover spontaneously. First aid and CPR should be administered as appropriate until medical assistance arrives. Realize that victims who appear to be only stunned or otherwise unhurt also need attention. Check for burns, especially at fingers and toes and next to metal buckles, jewelry, or personal items that the victim is wearing. Remember to treat for shock.

12.3.2 Tornadoes

Tornadoes usually develop from thunderstorms and normally occur at the trailing edge of the storm. Most tornadoes occur in the months of April, May, June, and July in the late afternoon and early evening hours.

When storms are predicted for the project areas it is necessary to monitor weather conditions on a radio. A tornado watch is issued when favorable conditions exist for the development of a tornado. A tornado warning is issued by the local weather service office whenever a tornado has actually been sighted or is strongly indicated by radar.

If a tornado warning is issued, seek shelter immediately. If there are permanent buildings located on site, enter one immediately and move toward interior hallways or small rooms on the lowest floor.

If a tornado warning is issued and you are in a vehicle or a site trailer, leave and go to the nearest building. If there are no buildings nearby, go in the nearest ditch, ravine, or culvert, with your hands shielding your head.

If a tornado is sighted or a warning issued while you are in open country, lie flat in a ditch or depression. Hold onto objects secured to the ground, such as a bush or fence post, if possible.

Once a tornado has passed the site, field personnel are to assemble at the designated assembly area to determine if anyone is missing or injured. Administer first aid and seek medical attention as needed.

12.3.3 Winter Storms

When snow or ice storms are predicted for the project area, field personnel should monitor radio reported weather conditions. A winter storm watch is issued when a storm has formed and is approaching the area. A winter storm warning is issued when a storm is imminent and immediate action is to be taken.

When a storm watch is issued, monitor weather conditions and prepare to halt site activities. Notify the Project Manager or FTL of the situation. Seek shelter at site buildings or leave the site and seek warm shelter.

If you are caught in a severe winter storm while traveling, seek warm shelter if road conditions prevent safe travel. If you are stranded in a vehicle during a winter storm:

- Stay in the vehicle. Disorientation comes quickly in blowing and drifting snow.
- Wait for help.
- Keep a window open an inch or so to avoid carbon monoxide poisoning.
- Run the engine and heater sparingly.
- Keep watch—don't let everyone sleep at the same time.
- Exercise occasionally.

12.4 ENGINEERED CONTROLS

Where economically and practically feasible, engineered controls may be selected to reduce exposure of field personnel to health or safety hazards. The OSO and FTL should always be on the lookout for potential engineered controls that may be implemented.

12.5 ADMINISTRATIVE CONTROLS

When engineered controls are not feasible, administrative controls in the form of work practices will be implemented to minimize risk to personnel from site hazards. Work practices that may be instituted include removing non-essential personnel from sampling areas and work rotation to control exposures to extreme thermal stress.

APPENDIX A

ACTIVITY HAZARD ANALYSIS

Tasks	Hazards	Controls	PPE Required
Sampling at Operating Mine Sites (including sites undergoing reclamation)	• Cuts and scrapes	• Follow procedures of mine operator. Report injuries to buddy or to person designated by mine operator for first aid if necessary. • Come to work alert and ready—make sure that general awareness of surroundings is part of job planning and execution. • Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground.	Minimum: hard-hat, safety glasses, boots, long pants, and cotton shirt; heavy work gloves for handling sharp objects. Additional PPE as specified by the mine operator.
	• Heat or cold stress	• Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0).	
	• Slips/trips/falls	• Maintain general awareness of surroundings.	
	• Being struck by heavy equipment or caught between equipment and a stationary object	• Receive site-specific hazard training. • Be alert to the direction of traffic flow. • Maintain eye contact with heavy equipment operators and give them the right-of-way. • Never stand between operating vehicles and nearby stationary objects. • Ask the mine operator where the blind spots for each piece of equipment are located—DO NOT STAND IN BLIND SPOTS.	

Tasks	Hazards	Controls	PPE Required
Sampling at Operating Mine Sites (continued)	• High wall collapse	• Receive site-specific hazard training. • Perform work under escort of mine employee. • Do not stand between high wall and heavy equipment—make sure you have an escape route. • Know the mine emergency signals and evacuation procedures.	
Sampling at Inactive Mine Sites	• Cuts and scrapes	• Report injuries to buddy for first aid if necessary. • Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. • Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground.	Minimum: hard-hat, boots, long pants, and cotton shirt; heavy work gloves for handling sharp objects.
	• Slips/trips/falls	• Do not walk at the edge of sharp drop-offs. Maintain special care on scree slopes or while working in other areas with unstable footing. Maintain general awareness of surroundings. • Be aware of the possibility of abandoned underground mine portals.	
	• Dislodged rocks	• Avoid areas below people who may dislodge rocks while working or walking on slopes. Cry “ROCK” after dislodging a rock when other people are below.	

Tasks	Hazards	Controls	PPE Required
Sampling at Inactive Mine Sites (continued)	• Deteriorated roads	• Receive site-specific hazard training. • Exercise care while traveling by vehicle.	
	• High wall collapse or rock-fall	• Receive site-specific hazard training. • Know signs of instability. Carefully examine the surroundings to determine if entry is safe. • Be aware of the most efficient evacuation route. • Do not walk on top of high walls. • Avoid working downslope of rock slides.	
	• Heat or cold stress	• Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0).	
	• Drinking water from mine pits	• Water in mine pits is of unknown quality, and WILL NOT be used for drinking water. Water purification with iodine, filters, or boiling will not remove potentially toxic metals.	
Travel in Remote Areas	• General	• Always carry ten essentials for wilderness travel (see Table 2-3).	Heavy work gloves for handling sharp objects.
	• Slips/trips/falls	• Maintain general awareness of surroundings.	

Tasks	Hazards	Controls	PPE Required
Travel in Remote Areas (continued)	• Cuts and scrapes	• Report injuries to buddy for first aid. Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. • Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground.	
	• Safe drinking water	• Contact National Forest officials in advance regarding any water quality advisories. • Bring sufficient water. Assume that you will need one gallon of drinking water per person per day.	
	• Severe weather	• Bring proper rain gear and warm clothes. • Listen to weather forecasts before entering remote areas. If severe weather is likely, postpone sampling. • In case of lightning, avoid high ground and open areas. • In the event of rain, monitor for hypothermia. • In the event of snow, monitor for frostbite and hypothermia. In the event of a blizzard that reduces visibility, stay put in an emergency shelter. Do not risk disorientation.	

Tasks	Hazards	Controls	PPE Required
Travel in Remote Areas (continued)	• Getting lost	• Provide the Program Manager or designee with itineraries, including travel routes and the expected date and time of return. Check in once per day, if possible, when in remote areas. Always check in with the Program Manager or designee before and after sampling. The Program Manager or designee will contact search and rescue if field personnel do not return or call in by the specified time. • Bring emergency shelter. • If lost, stay put. You are easier to find this way.	
	• Heat or cold stress	• Monitor for heat or cold stress as outlined in the Health and Safety Plan (see Section 7.0).	
	• Muscle strains	• Know your limits, and do not overextend yourself.	
	• Poisonous plants and animals	• Be able to recognize poisonous plants and animals and avoid them. • If bitten by a snake or spider, apply cold compresses. Get to a hospital as quickly as possible.	
	• Wildlife	• Avoid, if possible, and leave the area. • Make yourself look large by raising arms and shouting. • Slowly back away, without turning your back to the animal.	

Tasks	Hazards	Controls	PPE Required
General Work Practices	• First aid injuries	• Report injuries to buddy for first aid. • Seek additional medical attention, if necessary. • Notify the PSO.	Minimum: hard-hat, safety glasses, boots, long pants, and cotton shirt. Additional: heavy work gloves and hearing protection, as necessary.
	• Slips/trips/falls	• Practice good housekeeping, and remove or reduce slip/trip/fall hazards. • Maintain general awareness of surroundings.	
	• Cuts/scrapes	• Report injuries to buddy for first aid. • Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. • Wear heavy work gloves when handling sharp objects and point sharp objects towards the ground.	
	• Heat or cold stress	• Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0).	
	• Muscle strain	• Alternate activities as needed to give muscles rest.	
	• Slips/trips/falls	• Practice good housekeeping to remove or reduce slip/trip/fall hazards.	
	• Hearing loss.	• Use hearing protection when operating loud equipment.	

Tasks	Hazards	Controls	PPE Required
General Work Practices (continued)	• Electrocution.	• Use GFCI on portable power equipment.	
	• Power equipment	• See manufacturers instructions for the use of hand and portable power tools.	

APPENDIX B

MWH EH&S PROCEDURE NO. 811, DRILLING SAFETY

811 DRILLING SAFETY

I. PURPOSE

This procedure provides information on the hazards, regulatory requirements, and safe work practices for using drilling equipment and performing drilling operations. This procedure address all forms of drilling, including cable tool, rotary, geo-probe, rotosonic, and hollow-stem auger drilling used for most geotechnical, investigation, groundwater, and subsurface exploration drilling projects.

II. REGULATORY REVIEW

There are no specific Occupational Safety and Health Administration (OSHA) regulations for drilling activities; however, general guidelines may be found in 29 CFR 1926.800, *Underground Construction,* section (q). In addition, some of the OSHA standards that are applicable to drilling operations include:

29 CFR 1926, Subpart E, *Personal Protective and Lifesaving Equipment*
29 CFR 1926.65, *Hazardous Waste Operations and Emergency Response*
29 CFR 1926.251, *Rigging Equipment for Material Handling*
29 CFR 1926.307, *Mechanical Power-transmission Apparatus.*
29 CFR 1926.550, *Cranes and Derricks*
29 CFR 1926, Subpart Z, *Toxic and Hazardous Substances*

Some state OSHA plans may have additional requirements for drilling. Also, the client may have specific permits or operation practices that will be applicable to MWH drilling operations. Contact the Regional ES&H Manager for additional information.

Additional information may be obtained from the following non-regulatory references:

- ATEC Associates, Inc. 1991. *Drilling Safety: Working in the Danger Zone.*
- Diamond Core Drill Manufacturers Association (DCDMA) and the National Drilling Contractors Association (NDCA). *Drilling Safety Guide.* National Drilling Federation.
- Driscoll, Fletcher. 1989. *Groundwater and Wells.*
- West Hazmat Drilling. 1991. *Workplace Injury & Illness Prevention Program.*
- U S Army Corps of Engineers, EM385-1-1, *Safety and Health Requirements* Chapter 16.

III. ROLES AND RESPONSIBILITIES

A. ES&H Director

The ES&H Director is responsible for establishing a program that provides for the protection of MWH employees and subcontractors and that meets the applicable regulatory requirements for the use of, and work adjacent to, drilling equipment.

B. Regional ES&H Manager

The Regional ES&H Manager is responsible for:

- Ensuring that this procedure is implemented within his or her region.
- Ensuring assessments are completed to verify compliance with regulatory and procedure requirements.

C. ES&H Representative

The ES&H Representative is responsible for the following:

- Evaluating work sites and activities to ensure drilling operations are conducted in compliance with the requirements of this procedure and regulatory standards.
- Verifying that equipment inspections are completed and that equipment used is in serviceable condition.
- Verifying that employees are following safe work practices for work with, and adjacent to, drilling operations.

D. Employee

Each employee is responsible for the following:

- Understanding the hazards and following the required safe work practices for drilling operations.
- Using the personal protective equipment (PPE) specified for the task being performed.

IV. DEFINITIONS

Annulus: The space between the drill string (length of connected drill pipe) or casing and the wall of the borehole or outer casing.

Auger Drilling: A general type of drilling in which the borehole is drilled by rotating augers (either hollow-stem or solid-stem), and cuttings are removed by being pushed up the "flights" (corkscrew-like flanges) of the augers. (Note: the term "flight" is often used to indicate the lengths of hollow-stem or solid-stem augers, as well as the flanges on the augers.) Hollow-stem augers allow drillers to send split-spoon samplers down through the center of the augers, thereby eliminating the need to pull out the augers first. Solid-stem augers are used for smaller-diameter holes and for drilling through formations where more concentrated downward force is necessary.

Bit: The cutting tool attached to the bottom of the drill string. Used in rotary drilling.

Blowout: An uncontrolled escape of drilling fluid, gas, oil, or water from the well caused by the formation pressure being greater than the hydrostatic head of the fluid in the borehole. Also, an uncontrolled escape of grout from the borehole or well caused by malfunctioning pressure grouting apparatus.

Cone Penetrometer Testing (CPT): This was originally a method of performing geotechnical evaluations of subsurface soils, now additionally used to obtain rough estimates of aquifer properties. The CPT rig uses a direct-push method to advance a cone equipped with electronic sensors. As the cone is pushed downward, measurements are collected by the sensor and are recorded on the CPT aboveground equipment. Some CPT rigs are also capable of collecting soil gas, soil and groundwater samples at shallow depths (usually less than 50 feet).

Cuttings: Formation particles obtained from a borehole during the drilling process.

Direct-Push Soil Gas/Soil/Groundwater Sampling: A method of sampling either soil gas, soil, or groundwater by advancing a small sampling probe. The probe is hydraulically pushed downward, generating virtually no cuttings and eliminating waste disposal concerns. Direct-push sampling is applicable where no permanent wells are desired, and/or where sampling is required at relatively shallow depths (usually less than 50 feet). Direct-push equipment may be truck-mounted (on a vehicle), or mounted on a modified hand truck.

Drill Collar: A length of extremely heavy steel tube. It is placed in the drill string immediately above the bit to minimize bending caused by the weight of the drill pipe.

Drill Pipe: A special pipe used to transmit rotation from the rotating mechanism to the bit. The pipe also transmits weight to the bit and conveys air or fluid, which removes cuttings from the borehole and cools the bit.

Drilling Fluid or Mud: A water-based or air-based fluid used in the well drilling operation to remove cuttings from the borehole, to clean and cool the bit, to reduce friction between the drill string and the sides of the borehole, and to seal the borehole.

Grouting: The operation by which grout is placed between the casing and the sides of the borehole to a predetermined height above the bottom of the well. This secures the casing in place and excludes water and other fluids from the borehole. A pressure grouting operation injects grout from the surface under high pressure, in order to move grout laterally in the subsurface and ensure an adequate seal.

Kelly: A hollow steel bar or pipe that is the main section of drill string to which the power is directly transmitted from the rotary table to rotate the drill pipe and bit. The cross section of the kelly is either square, hexagonal, or grooved. The kelly works up and down through drive bushings in the rotary table.

Limited Access Drill Rig: A type of drill rig, usually equipped with solid-stem augers, which allows drilling in tight spaces or in areas with low overhead clearance (less than 12 feet). Limited access drill rigs may be mounted on a small lawnmower-like vehicle, or on a modified hand truck.

Rotary Drilling: A general type of drilling in which the borehole is drilled by rotating a bit, and cuttings are removed by continuous circulation of a drilling fluid (e.g., mud, water, air, foam) as the bit penetrates the formation. The bit is attached to the lower end of a string of drill pipe, which transmits the rotating action from the rig to the bit.

Rotary Table: A mechanical or hydraulic assembly that transmits rotational torque to the kelly, which is connected to the drill pipe and the bit. The rotary table has a hole in the center through which the kelly passes.

Split-Spoon Sampler: A thick-walled steel tube split lengthwise used to collect soil samples. The sampler is commonly lined with metal sample sleeves and is pounded or pushed downhole by the drill rig to collect samples.

V. PLANNING

A. Training

Only trained and experienced personnel who are familiar with the use, limitations, and maintenance requirements of the equipment are permitted

to operate the drill rig. The drill rig must be operated in accordance with the manufacturer's instructions and recommendations.

The drill crew shall be familiar with the hazards associated with the drilling operations, personal protective equipment requirements, location of emergency stops, site-specific safety requirements, and the content of this procedure.

Additional training requirements may be required based on the location of the drilling activities. This training may include:

- Hazardous Waste Operations training if work is conducted at a hazardous waste site.
- Waste management training, where waste streams (e.g., drill cuttings, purge water, decontamination water, contaminated personal protective equipment) will be generated.

Site-specific training requirements will be outlined in the project-specific HASP.

B. Medical Surveillance

There are no medical surveillance requirements specific to drilling activities. However, drilling at hazardous waste sites that involve toxicological hazards may require medical surveillance.

Medical surveillance requirements will be addressed in the Project HASP.

MWH drilling subcontractors are responsible for ensuring that their employees receive medical surveillance as required.

C. Safety Equipment

MWH will provide required PPE and safety equipment for its employees and operations. Subcontractors are responsible for providing all PPE and safety equipment necessary for safe operation. Safety equipment will be provided by the subcontractor as delineated in the subcontract and referenced documents. The minimum safety equipment for drilling activities includes safety-toed boots, hard hats, and safety glasses with side shields. Other safety equipment that may be required for drilling operations includes:

- Hearing protection when working in proximity to drilling machinery.
- Body protection (e.g., gloves and protective coveralls) when chemical hazards exist.

- Detection equipment shall be provided if the exact location of underground utilities cannot be determined.

- Air monitoring instruments shall be provided if the potential for a hazardous atmosphere exists in the drilling location.

- High-visibility warning vests are to be worn by all workers exposed to vehicle traffic.

- Fall protection equipment is required if working from unprotected platforms or surfaces greater that 6 feet above the lower level.

D. Subcontractor Selection and Oversight

The *Subcontractor Safety Program Review Criteria—Drilling Operations*, Attachment A, provides the minimum criteria for subcontractor drilling safety procedures. These criteria will be used by the ES&H Representative or ES&H Staff to review subcontractor programs and procedures

Responsibilities for environment, safety and health are expressly defined in the subcontract terms and conditions, and MWH's ES&H practices in the field are determined based on these defined responsibilities. MWH employees shall not direct the means and methods of the subcontractor's operations or direct the details of corrective actions except when MWH employees are responsible for the work activity by contract.

E. Planning Activities

Depending on the contract with the client, the subcontract with a drilling subcontractor, and the physical location of the drilling operation, MWH, may be responsible for some of the following functions to support drilling operations. The responsible party for these items should be clearly defined in the contract and subcontract requirements and included in the Project ES&H Plan.

1. Utility Location

The location of underground utilities, such as electric, fuel, water, cable, telephone, and sewer (either in service or abandoned), and underground installations such as foundations, underground storage tanks, piping, and any other structures need to be identified before drilling is permitted. Utility companies and/or installation owners shall be contacted to provide exact locations of their equipment or structures. Some states have a one-call phone number for locating underground utilities. Most utilities and call

centers require a minimum 48-hour-notice (excluding Saturdays, Sundays, and holidays) to identify utilities before drilling work can begin.

If underground utilities cannot be positively located, or where drilling is performed in areas known or suspected to contain buried objects (e.g. drums, tanks, or cylinders), the area will be surveyed with the aid of audio and radio frequency transmitters and receivers, ground penetrating radar, ultrasonic testing, metal detectors, or other means necessary to ensure safe drilling operations.

Overhead electrical transmission and communication lines also need to be identified. If any portion of the drill rig will be within: 10 feet of electrical lines up to 50kV or 10 feet plus 4 inches for every 10 kV over 50 kV, the utility or other facility operating the system will be contacted to have the lines de-energized.

Access to the work location will also be evaluated. Where rigs must travel under energized electrical lines, clearance will be 4 feet for voltages up to 50kV; 10 feet for voltages over 50kV and up to 345kV; and 16 feet for voltages from 345kV up to 750kV.

2. Permits

The following permits and notifications may be required, depending on state, local, and client requirements. The Regional ES&H Manager should be contracted for assistance in determining applicability.

- Well driller license/certification or Professional Geologist requirements: MWH subcontractors will be required to submit licenses or certifications before subcontract award, where applicable.

- Well installation or abandonment notification: Submittal of a well log or inventory may be required after installation or abandonment.

- A groundwater withdrawal permit may be required for large water withdrawals in some states.

- A "drilling permit" may be required at certain client facilities.

3. Waste Management

Drill cuttings and purge water from uncontaminated soil or ground water shall be appropriately.

When drilling is conducted at hazardous waste sites, the Regional ES&H Manager or designee may be consulted on the proper evaluation, disposal, and decontamination procedures involving potential hazardous waste.

- All waste generated shall be evaluated for appropriate disposal and handled in accordance with the appropriate waste management procedure.

- If drilling involves hazardous waste, MWH decontamination procedures shall be followed. No potentially contaminated equipment shall be permitted to leave the work site.

4. Drilling at Ordnance Explosives (OE) or Unexploded Ordnance (UXO) Sites

If the project site is suspected of OE contamination, the requirements of the ES&H procedure for *Unexploded Ordnance, Open Firing Ranges, and Chemical Warfare Agents,* shall be followed. The following procedures will be implemented, at a minimum:

- Drilling operations on OE sites will not be conducted until a complete plan for the site is prepared and approved by MWH ES&H, and the UXO Safety Officer. OE/UXO avoidance must be conducted during drilling operations on known or suspect OE sites.

- The UXO team will identify and clearly mark the boundaries of a clear approach path for the drilling crews, vehicles, and equipment to enter the site. This path will be, at a minimum, twice the width of the widest vehicle. No personnel will be allowed outside any marked boundary.

- If OE is encountered on the ground surface, the UXO team will clearly mark the area where it is found, report it to the proper authorities, and divert the approach path around it.

- The UXO team will conduct an access survey using the appropriate geophysical instrument over the approach path for avoidance of OE that may be in the subsurface. If a magnetic anomaly is encountered, it will be assumed to be OE and the

approach path will be diverted around the anomaly. Only UXO personnel will operate the appropriate geophysical instrument and identify OE.

- An incremental geophysical survey of the drill hole location(s) will be initially accomplished by the UXO team using a hand auger to install a pilot hole. If OE is encountered or an anomaly cannot be positively identified as inert material, Hazardous Toxic Radiological Waste sampling personnel will select a new drill hole location.

- Once a drilling site has been surface cleared and a pilot hole established as described above, the drilling crew or subcontractor will be notified that the site is available for subsurface drilling.

VI. PROCEDURE

A. Safe Work Practices

Only authorized or licensed personnel, based on applicable state or local requirements, shall be permitted to operate drill rigs.

When moving large equipment in a confined area, spotters shall be used. The spotter and equipment operator must use standard hand signals for communication. Spotters shall never place themselves between equipment and fixed structures or equipment.

Drill site work areas shall be demarcated to deter unauthorized individuals from entering the work area.

Personnel not involved in equipment operation shall remain clear of drill rigs.

Personnel shall stay clear of the rotating augers and other rotating components of drill rigs at all times. Stand to the side while tripping and tailing rods and augers. Never stand under the rod/auger or between the rig and service truck while tripping rods or augers.

Stay as clear as possible of all hoisting operations. Loads shall not be hoisted over personnel. Never work around or under drilling rods or augers being hoisted.

Keep footwear and work area free of mud and drilling fluids. Maintain 3 points of contact when mounting and dismounting a drill rig. Do not climb the drill rig mast without the use of fall protection.

Augers shall not be stored standing up and shall be secured from rolling.

Hand tools shall be inspected before use. Wrench jaws must be periodically inspected and replaced when necessary.

Understand and be aware of all pinch points including breakout wrenches, pull down cables, and pulling jacks. These points should be color-coded. Experienced employees must show new employees these pinch points on the first day of work.

Good housekeeping shall be maintained at all times. Litter will be properly stored, and hand tools and other hardware will be properly secured on the drill rig. Before moving a drill rig, a check shall be made for loose tools and hardware.

All work areas, platforms, walkways, scaffolding, and other accessways should be maintained free of materials, debris, obstructions, and substances, such as ice, grease, or oil.

Drill rods and augers should be placed on dunage and secured to prevent movement. Always use a sling or strap while manually handling rods and augers.

Be aware of your footing to prevent slips, avoid stepping between rods and augers to prevent crushed ankles from their movement.

Do not wear loose-fitting clothing or other items, such as rings or watches, that could get caught in moving parts. Individuals with long hair should have it restrained.

Personnel shall not smoke around drilling operations.

Personnel shall wear the appropriate PPE. Minimum protection includes safety-toed boots, hard hats, safety glasses, and hearing protection.

A daily safety briefing shall be conducted with all work site personnel to discuss the work planned for the day and the ES&H requirements.

Unattended boreholes shall be covered or protected to avoid the possibility of animals or people accidentally falling into them.

Wellheads on roads and parking lots should be flush-mounted.

B. Site Preparation

Verify that underground utilities and structures have been located and marked, and overhead utilities de-energized as required.

As applicable, the drilling site shall be prepared, cleared, and leveled, particularly on steep slopes or areas that are covered with dry dead grass and weeds. Care should be taken in constructing pads if extensive cutting into existing slopes or surfaces is required to level the area. Areas where extensive fill is required should be avoided. Compaction is recommended if significant amounts of fill are needed; the ground must be capable of supporting the impact imposed by the drill rig and associated equipment. Clean fill or gravel can be brought in to cover areas with surface contamination.

Before drilling equipment is mobilized to the drilling pad, the travel route shall be surveyed for overhead and terrain hazards. Access roads shall be designed, constructed, and maintained to safely accommodate the movement of the drill rig and other equipment.

Material Safety Data Sheets (MSDSs) shall be available for all drilling fluids, grout, bentonite, or other substances used in the drilling process.

C. Drill Rig Requirements

All self-propelled drill rigs shall be equipped with the following safety features:

- Seatbelts,
- Multipurpose dry chemical fire extinguisher rated at not less that 2A:10B:C
- Multidirectional alarm
- Operator's Manual
- Horn
- Lights
- Other warning devices specified by the manufacturer

Self-propelled off-road drill rigs will be equipped with roll-over protective structures (ROPS) meeting Society of Automotive Engineers requirements.

The drilling equipment shall be equipped with two easily accessible emergency shutdown devices.

Control levers on the drill rig shall be clearly labeled indicating the function and direction of the movement.

All machine guards shall be in place while the rig is in operation.

Where drill rigs are equipped with a platform, the platform shall be constructed from material strong enough to support the weight of the load that will be placed on the platform. The platforms shall be accessed using a ladder or steps. Platforms over 6 feet above ground surface shall be equipped with a guardrail system that includes a toeboard.

The drill rig and associated equipment shall be inspected each day before use by a qualified mechanic or an operator knowledgeable of the specific equipment. Inspections and tests will be conducted in accordance with the manufacturer's recommendations. A written record of the inspections shall be kept on the equipment or in a project file. Consideration shall be given to the following items in the performance of equipment inspections.

- Missing nuts, bolts, pins, loose fittings and couplings.
- Cracked paint, frayed cables and hoses, evidence of fluid leakage on equipment or ground, and loose tracks and pads.
- Fluid levels in the battery, hydraulic system, brake system, and cooling systems; engine lubrication; and fuel supply. CAUTION: Never use your hands to check for hydraulic leaks. An open flame shall not be used to check fluid levels or look for leaks.
- Condition of glass in cab clean and not broken, gauges checked for proper function and readings; test of brake lights, horn, backup alarm, steering, and other controls.
- All emergency shutdown and warning systems to ensure that they are working correctly.

When deficiencies that affect the safe operation of equipment are identified, the equipment will be immediately taken out of service until unsafe conditions are corrected. A "DO NOT OPERATE" tag indicating that the equipment is not to be operated will be placed on the operator controls. When required or necessary, lockout procedures will be used. When corrections are made, the equipment will be re-tested for safe use before being returned to service.

D. Equipment Travel and Set-up

Safe clearances from overhead electrical transmission lines shall be maintained.

The operator shall ensure ground is stable and that grades, especially side traverses, are within the operating limits of the vehicle.

The mast shall be lowered and rig placed in appropriate configuration for travel. Drilling equipment shall not be transported for even a short distance with the mast up.

The drill rig must be leveled and stabilized with leveling jacks. Cribbing shall be used as necessary. Outriggers shall be extended per the manufacturer's specifications. Cribbing materials should be made from materials that are capable of supporting the weight of the rig. Care should be taken in muddy, soggy soils, or partially frozen areas. In addition to cribbing, guy wires should be used as required by the manufacturer to improve stability if the rig is located on wet, partially frozen ground, or in areas with loose, caving soil, or in an area subject to frequent gusty winds.

Prior to raising the mast (derrick) the operator shall look up for overhead obstructions. The drill rig operator shall verify that all personnel are cleared from the area immediately to the rear and the side of the mast.

Unsecured equipment shall be removed from the mast and cables; mud lines and cat line rope must be secured before raising the mast.

Before starting drill operations, the mast shall be secured and locked in accordance with the manufacturer's recommendations.

E. Equipment Operation

The drill rig shall be provided with a "kill" switch that, when activated, will shut down the rig. The switch should be clearly identified and tested daily to confirm operational status. All drilling crew members should be made aware of the location and purpose of this switch.

The rope, wire rope, or cable on the drill rig should never be wrapped around any part of the body.

The drill rig should not be operated during severe inclement weather, such as lightning storms, high winds, or severe rain. The mast should be lowered during these conditions.

Before starting, the operator shall verify that all gear boxes are in neutral; all hoist levers are disengaged; hydraulic levers are in the correct non-actuating positions; and the cathead rope is not on the cathead.

The operator shall verbally alert workers and visually verify that workers are clear from the dangerous parts of equipment before starting the equipment.

Drill crew members shall not wear loose clothing or clothing with loose ends, straps, drawstrings, belts, or otherwise unfastened parts that might catch on rotating or translating components of the drill rig. Rings and jewelry shall not be worn during a work shift.

The drill rig shall always be operated from the control panel. The operator must never leave the control panel while the drill is in operation. Only one person should operate the machine. If the operator must leave the area of the controls, the operator must shift the transmission controlling the rotary drive into neutral and place the feed control level in neutral. The drill rig shall be shut down before he operator leaves the vicinity of the drill.

Pressurized lines, such as airlines, mud hose, etc. shall be equipped with safety-type couplings and secured with wire or chain at each coupling to prevent whipping in the event of failure. Lines and safety connection shall be inspected daily. Pressurized lines shall not be disconnected until shut off and bled to reduce the pressure.

Drilling fluid discharges shall be channeled away from the work area to prevent the accumulation of water. Mud pits and drainage channels should be safely sloped and located to provide minimum interference with the work. Where necessary, suitable barricades or temporary fencing should be provided to protect the work area and reduce the possibility of injury to persons.

Where compressed air drilling is conducted, the exhaust shall be directed into an approved dust collection system. The cuttings shall be directed to the side away from employees. Workers shall be required to use protective clothing and respiratory protection, when required.

1. Tool Handling – Hoist Line

All wire ropes and rigging hardware shall be thoroughly inspected before use. Defective equipment shall not be used. Shop fabricated rigging or hooks without latches are not permitted. Where a chain sling is used, it shall be an alloy chain and shall be properly labeled.

Hoist and rigging hardware shall be used only for their designated intent and shall not be loaded beyond their rated capacity. Steps shall be taken to prevent two-blocking of hoist.

The tool handling hoist shall only be used for vertical lifting of tools. The tool hoist must not be used to pull on objects away from the drill rig, unless specifically designed for that purpose.

Drill rods shall be neither run nor rotated through rod slipping devices: no more than 1 foot of drill column shall be hoisted above the top of the drill mast. Drill rod joints shall not be made up, tightened, or loosened while a rod-slipping device supports the rod column.

Chuck jaws shall not be used to brake a string of drill rods while being lowered into the hole. A cat line or hoisting cable or plug should be used for braking before tightening the chuck.

- Drilling rods shall not be lowered into the hole with a pipe wrench.

- When stuck tools or similar loads cannot be raised with a hoist, disconnect the hoist line and connect the stuck tool directly to the feed mechanism of the drill. Do this only when the rope is secured with a wrench or dog collar. Do not use hydraulic leveling jacks for added pull to the hoist line or feed mechanism of the drill.

Loads shall not be hoisted over the head, body, or feet of any person. Loads shall not be left suspended when the hoist is unattended. Work is not permitted under a suspended load.

Hoist lines shall not be used to ride up the mast of a drill rig.

When wire rope hoist lines are used, the following precautions shall be followed:

- Wire rope must be properly matched with each sheave. (If too large, the rope will pinch; if too small, the sheave will groove. Once a sheave is grooved, it will pinch and damage the larger rope.)

- Most sheaves on rigs are stationary and designed for a single-part line. Never increase the number of sheaves, winch lines, or part lines unless approved by the drill manufacturer.

- Minimize the shock to wire rope. Pull loads smoothly and steadily, especially in cold weather. Never use frozen ropes.

- Protect wire rope from sharp corners and edges. Replace faulty guides and rollers.

- When handling wire rope, always wear gloves. Do not guide rope onto hoist drums with your hands. Replace the wire rope

according to manufacturer's specifications. When new rope is installed, first lift a light load to allow the rope to adjust.

2. Auger Drilling

Use an auger guide to facilitate the starting of a straight hole through hard ground or pavement, as applicable. Apply an adequate amount of down pressure before rotation to seat the auger head below the ground surface.

The operator and tool handler shall establish a system of responsibility for the series of activities required for auger drilling, such as connecting and disconnecting auger sections, and inserting and removing auger fork.

The operator shall verify that the tool handler and others are clear from the auger column and that the auger fork is removed before starting rotation.

The manufacturer's recommendations must be followed for securing the auger to the power coupling. Workers shall not touch the coupling or the auger with their hands, a wrench, or any other tools during rotation.

Only tight-fitting pins designed for the auger shall be used to secure auger flights. The use of mismatched augers shall be avoided.

Augers shall be cleaned only when the rotating mechanism is in neutral and the auger stopped. Long-handled shovels may be used to move auger cuttings away from the auger.

Workers shall not place their hands or fingers under the bottom of auger section when hoisting the auger over the top auger section in the ground or over hard surfaces such as drill rig platform.

Workers shall, stay clear of the rotating auger and other rotating components of the drill rig. Never reach behind or around a rotating auger for any reason.

3. Cathead Operations

The cathead shall be inspected before use. Inspection shall be made with the engine off. The cathead shall be kept clean and free of rust, oil, and grease. When 1/8-inch or greater rope groves form, the cathead should be replaced. In wet or icy conditions, a cathead cannot be used.

Always use a clean, dry rope. An oily rope may grab on the cathead. Never use a rope that is longer than necessary.

If the rope grabs or tangles, alert personnel to back away and stay clear. If tools are suspended, carefully shutdown the drill and back away. Once you have resolved the situation, the drill may be restarted and the tools lowered to safety.

Hoist lines shall be positioned to prevent contact with the cathead rope.

The following precautions shall be used to prevent cathead incidents:

- The operator should be on a level surface with firm footing.
- Do not wear loose clothing or gloves with loose straps or cuffs.
- Never wrap the rope around your hand, wrist, arm or other body parts. Never stand on the end of the cathead rope.
- Maintain a distance of 18 inches clearance between operating hand and drum.
- Be aware, the rope advances with each hammer blow.
- Do not leave a cathead unattended with the rope wrapped on the drum.

4. Rotary and Core Drilling

Water swivels, hoist plugs, rod chuck jaws etc. shall be inspected before use. Defective equipment shall not be used.

Only the operator of the drill rig shall brake or set a manual chuck so that rotation of the chuck will not occur before removing the wrench from the chuck.

Chuck jaws shall not be used to brake drill rods while lowering rods into hole. Drill rods shall not be held or lowered into the hole with pipe wrenches. If a string of drill rods are accidentally or inadvertently released into the hole, no attempt shall be made to grab the falling rods with hands or a wrench.

When drill rods are hoisted from the hole, they shall be cleaned to facilitate safe handling. The hand should not be used to clean drilling fluids from drill rods.

Drill rods shall never be lifted and leaned unsecured against the mast. Drill rods shall be secured to the upper ends of the drill rod sections for safe vertical storage or shall be laid horizontally.

In the event of a plugged bit or other circulation blockage, the pressure in the piping and hose between the pump and the obstruction should be relieved or bled down before breaking the first tool joint.

The spinning chain is very powerful and must be treated with respect. Spinning chains must have a rope tail. Communication between the driller and tool handler is required for safe operation of the spinning chain.

If freezing weather is expected, all air and water lines should be drained when not in use.

F. Drill Rig Maintenance

Components found to be in defective condition, either during inspections or during rig operation, should be repaired immediately.

Rig maintenance shall only be performed after appropriate lockout/tagout procedures have been implemented.

The cathead should be kept clean and free of rust, oil, and grease. The cathead should be cleaned with a wire brush if it becomes rusty.

Drilling operations may require repair or disentanglement of wire rope on the mast while it is raised. Fall protection shall be used when personnel are exposed to a fall of 6 feet or greater.

Augers should be cleaned only when the drill rig is in neutral and the augers have stopped rotating. Hands or feet should not be used to move cuttings away from the auger.

G. Self-Assessment Checklist

The *ES&H Self-Assessment Checklist – Drilling* provides a method of verifying compliance with established regulations, safe work practices, and industry standards pertaining to drilling operations. The MWH ES&H Representative or ES&H staff may use this checklist when MWH

employees and/or MWH Subcontractors are performing drilling operations or are exposed to the hazards of activities involving drilling.

VII. ATTACHMENTS

Attachment A: *Subcontractor Safety Program Criteria – Drilling Operations*
Attachment B: *ES&H Self-Assessment Checklist – Drilling Operations*

Drilling Operations
ES&H Procedure – 811
Attachment A

Subcontractor Safety Program Criteria – Drilling Operations

MWH

ATTACHMENT A

Subcontractor Safety Program Criteria – Drilling Operations

The following criteria are not intended to be all-inclusive, but are provided as a tool to facilitate review of subcontractor safety procedures. Subcontractors are expected to address the following items, at a minimum, in their safety procedures.

Minimum Acceptable Criteria for Subcontractor Drilling Safety Procedures:

1. Provide name and qualifications of the drilling "competent person" responsible for drilling (years and type of experience, training background, etc.).
2. Describe drill rig and equipment inspection criteria or procedures (frequency of inspections, visual vs. written inspections, items that are inspected).
3. Describe methods of identifying underground utilities (contacting utility companies, detection equipment).
4. Describe methods of avoiding contact with overhead power lines (de-energizing and grounding, insulating, safe clearance distances).
5. Describe methods to identify hazardous atmospheres and controls used to eliminate (detection equipment and controls).
6. Describe leveling and stabilizing methods for drill rig (drilling pad, jacks, cribbing, guy wires).
7. Verify that rig equipment is in good operational condition (including "kill" switch, cathead, ropes, pressurized hoses and lines, operator controls, machine guards, and drilling tools).
8. Describe procedures for operating in inclement weather, including lightning, high winds, and severe rain storms.
9. Describe other safe work practices for equipment operation (drill rig, equipment, tools, rig transportation, rig travel).
10. Describe on-the-job maintenance procedures, including lockout/tagout.
11. Describe safe work practices for other activities to be performed during this project (use of ladders, fall protection, electrical power tools, personal protective equipment, etc.).
12. Describe methods for disposal of non-hazardous drill cuttings and purge water (including accumulation, transport, and disposal).
13. If hazardous waste project, provide documentation of hazardous waste worker training and medical surveillance records for all project personnel (40-hour or 24-hour training, 8-hour refresher training) and describe methods of hazardous waste management (including accumulation, transport, and disposal).
14. Submit a copy of drilling license/certification and drill rig permit.
15. Describe methods and responsibilities for submittal of notifications and logs.
16. Complete the Waste Subcontractor Qualification form for each proposed transport and disposal facility.
17. Describe procedures for drilling site cleanup upon job completion.

<u>If drilling in areas with known or potential Ordance Explosives (OE)/Unexploded Ordnance (UXO) hazards:</u>

18. Provide documentation of UXO qualifications, hazardous waste worker training, and medical surveillance records for all project personnel (40-hour or 24-hour training, 8-hour refresher training).
19. Describe procedures for OE avoidance, identification and marking the boundaries of a clear approach path and work site for the sampling crews, vehicles, and equipment to enter the site.
20. Describe the procedures for drilling and monitoring and the process for encountered OE.

Drilling Operations
ES&H Procedure – 811
Attachment B

ES&H Self-Assessment Checklist – Drilling Operations

MWH

ATTACHMENT B

Self-Assessment Checklist – Drilling Operations

This checklist shall be used by MWH personnel only and may be completed at the frequency specified in the Project HASP or by the ES&H Representative.

This checklist is to be used at locations where (1) MWH employees and/or (2) MWH subcontractors are working on projects involving drilling operations.

During evaluation of MWH subcontractors, the ES&H Representative may consult with subcontractors when completing this checklist, but shall not direct the means and methods of operations nor direct the details of corrective actions unless provided for by contract requirements. Subcontractors shall determine how to correct deficiencies.

If deficiencies are identified that are considered imminent danger (possibility of serious injury or death) hazards, they shall be corrected immediately or all exposed MWH and MWH subcontract personnel shall be removed from the hazard until corrected.

Completed checklists shall be maintained in project ES&H Files.

Project Name: _____ Project No.:_____

Location: _____ PM: _____

Evaluator: _____ Title: _____ Date: _____

This specific checklist has been completed to:

☐ Evaluate MWH employee compliance with requirements

☐ Evaluate an MWH subcontractor's compliance with requirements

Check "Yes" if an assessment item is complete/correct.

Check "No" if an item is incomplete/deficient, section 2 must be completed for all items checked "No."

Subcontractor deficiencies shall be brought to the immediate attention of the subcontractor.

Check "N/A" if an item is not applicable
Check "N/O" if an item is applicable, but was not observed during the assessment

ATTACHMENT B

Self-Assessment Checklist – Drilling Operations

	Yes	No	N/A	N/O
Section 1				
SAFE WORK PRACTICES				
1. Only authorized, licensed operators meeting local requirements.	☐	☐	☐	☐
2. Spotters used for equipment in constricted areas.	☐	☐	☐	☐
3. Drill site work area demarcation in place.	☐	☐	☐	☐
4. Personnel cleared during rig startup.	☐	☐	☐	☐
5. Personnel clear of rotating parts.	☐	☐	☐	☐
6. Personnel not positioned under hoisted loads.	☐	☐	☐	☐
7. Augers and drill strings stored properly.	☐	☐	☐	☐
8. Good Housekeeping maintained on rig and work site.	☐	☐	☐	☐
9. Loose clothing and jewelry removed.	☐	☐	☐	☐
10. Smoking is prohibited around drilling operation.	☐	☐	☐	☐
11. Personnel wearing appropriate personal protective equipment (PPE).	☐	☐	☐	☐
12. Daily safety meeting completed.	☐	☐	☐	☐
13. Traffic control in place, where required.	☐	☐	☐	☐
Site Preparation				
14. Underground utilities & structures identified.	☐	☐	☐	☐
15. Drill site prepared for rig.	☐	☐	☐	☐
16. Travel routes surveyed for obstruction.	☐	☐	☐	☐
17. Required MSDSs on site.	☐	☐	☐	☐
Drill Rig Requirements				
18. Drill rig equipped with emergency stop controls.	☐	☐	☐	☐
19. Drill rig inspected daily, deficiencies corrected.	☐	☐	☐	☐
20. All machine guards are in place.	☐	☐	☐	☐
21. Platforms are properly constructed and accessible.	☐	☐	☐	☐
Equipment Travel and Set-up				
22. Safe clearance from overhead electrical maintained.	☐	☐	☐	☐
23. Mast lowered and equipment secure during travel.	☐	☐	☐	☐
24. Drill rig level and stabilized, proper cribbing used.	☐	☐	☐	☐
25. Mast secured before drilling.	☐	☐	☐	☐
Equipment Operation				
26. Personnel remain clear of cables and ropes.	☐	☐	☐	☐
27. Drilling operations suspended in inclement weather.	☐	☐	☐	☐
28. All gearboxes in neutral before starting.	☐	☐	☐	☐
29. Drill rig is always operated from control panel. Operator never leaves control panel while drill is in operation.	☐	☐	☐	☐
30. Pressurized lines are equipped with safety cables.	☐	☐	☐	☐
31. Drilling discharges channeled away from work area.	☐	☐	☐	☐
32. Compressed-air exhaust is controlled.	☐	☐	☐	☐
33. Wire ropes are inspected before use.	☐	☐	☐	☐
34. Hoist and hardware used only for designed purpose.	☐	☐	☐	☐
35. No more than one foot of drill column hoisted above mast.	☐	☐	☐	☐
36. Chuck jaws are not used as drill rod brake.	☐	☐	☐	☐
37. Wire ropes are properly matched to sleeves and are protected from sharp edges.	☐	☐	☐	☐
38. Operators are not guiding wire ropes with hands.	☐	☐	☐	☐
39. Auger guides are used to start auger in hard surfaces.	☐	☐	☐	☐

ATTACHMENT B

Self-Assessment Checklist – Drilling Operations

	Yes	No	N/A	N/O
40. Operator and tool handler have established responsibilities.	☐	☐	☐	☐
41. Augers are attached to power coupling per Manufacturer's instructions.	☐	☐	☐	☐
42. Auger flights are connected only by pins designed for such use.	☐	☐	☐	☐
43. Workers remain clear of auger and other rotating parts.	☐	☐	☐	☐
44. Cathead inspected before use.	☐	☐	☐	☐
45. Clean dry rope is used on cathead.	☐	☐	☐	☐
46. Proper actions are followed for cathead operations.	☐	☐	☐	☐
47. Drill rods are not leaned unsecured against mast.	☐	☐	☐	☐
48. Prior to disconnection plugged core drill rods, pressure source is isolated and pressure is bled off.	☐	☐	☐	☐
Drill Rig Maintenance				
49. Defects identified during inspection or operation are corrected before continuing operation.	☐	☐	☐	☐
50. Fall protection is used while climbing mast and for other unprotected elevated work.	☐	☐	☐	☐
51. Augers are cleaned only when drill is in neutral and auger has stopped.	☐	☐	☐	☐

ATTACHMENT B

Self-Assessment Checklist – Drilling Operations

SECTION 2

Complete this section for all items checked "No" in Sections 1. Deficient items must be corrected in a timely manner.

Item #	Corrective Action Planned/Taken	Date Corrected

Evaluator:_____ Project Manager:_____

APPENDIX C

MATERIAL SAFETY DATA SHEETS (MSDS)

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00901

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Alconox Detergent
Catalog Number: 2088000

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00901
Chemical Name: Not applicable
CAS No.: Not applicable
Chemical Formula: Not applicable
Chemical Family: Surfactants
Hazard: No effects anticipated.
Date of MSDS Preparation:
Day: 11
Month: December
Year: 2001

2. COMPOSITION / INFORMATION ON INGREDIENTS

Alkyl aryl sulphonates, lauryl alcohol sulfates, phosphates and carbonates
CAS No.: Not applicable
TSCA CAS Number: Not applicable
Percent Range: 100.0
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: No effects anticipated.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: White powder
Odor: None

HMIS:
Health: 0
Flammability: 0
Reactivity: 0

Protective Equipment: X - See protective equipment, Section 8.

NFPA:

Health: 0

Flammability: 0

Reactivity: 0

Symbol: Not applicable

Potential Health Effects:

Eye Contact: No effects are anticipated

Skin Contact: No effects are anticipated

Skin Absorption: No effects anticipated

Target Organs: Not applicable

Ingestion: None reported

Target Organs: None reported

Inhalation: No effects anticipated

Target Organs: Not applicable

Medical Conditions Aggravated: None reported

Chronic Effects: None reported

Cancer / Reproductive Toxicity Information:

This product does NOT contain any OSHA listed carcinogens.

This product does NOT contain any IARC listed chemicals.

This product does NOT contain any NTP listed chemicals.

Additional Cancer / Reproductive Toxicity Information: None reported

Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Flush eyes with water.

Skin Contact (First Aid): Wash skin with plenty of water.

Ingestion (First Aid): Give large quantities of water. Call physician immediately.

Inhalation: None required.

5. FIRE FIGHTING MEASURES

Flammable Properties: Material will not burn.

Flash Point: Not applicable

Method: Not applicable

Flammability Limits:

Lower Explosion Limits: Not applicable

Upper Explosion Limits: Not applicable

Autoignition Temperature: Not determined

Hazardous Combustion Products: None reported

Fire / Explosion Hazards: None reported

Static Discharge: None reported.

Mechanical Impact: None reported

Extinguishing Media: Use media appropriate to surrounding fire conditions

Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:

Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.
Containment Technique: Stop spilled material from being released to the environment.
Clean-up Technique: Sweep up material. Place material in a plastic bag. Mark bag 'Non-hazardous trash', and dispose of as normal refuse. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Keep container tightly closed when not in use.
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: Not applicable
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes Wash thoroughly after handling.
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: White powder
Physical State: Solid
Molecular Weight: Not applicable
Odor: None
pH: Not determined
Vapor Pressure: Not applicable
Vapor Density (air = 1): Not applicable
Boiling Point: Not applicable
Melting Point: Not determined
Specific Gravity (water = 1): Not determined
Evaporation Rate (water = 1): Not applicable
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not applicable
Solubility:
Water: Soluble
Acid: Not determined
Other: Not determined
Metal Corrosivity:
Steel: Not determined
Aluminum: Not determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Excess moisture
Reactivity / Incompatibility: None reported
Hazardous Decomposition: Heating to decomposition releases toxic fumes of carbon monoxide and carbon dioxide.
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: None reported
LC50: None reported
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: None reported
Mutation Data: None reported
Reproductive Effects Data: None reported
Ingredient Toxicological Data: --
No toxicological data available for the ingredients of this product.

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: --
No ecological data available for the ingredients of this product.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Place material in a plastic bag. Mark bag 'Non-hazardous trash', and dispose of as normal refuse.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
DOT Hazard Class: NA
DOT Subsidiary Risk: NA
DOT ID Number: NA
DOT Packing Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
ICAO Hazard Class: NA
ICAO Subsidiary Risk: NA
ICAO ID Number: NA
ICAO Packing Group: NA

I.M.O.:

I.M.O. Proper Shipping Name: Not Currently Regulated

--

I.M.O. Hazard Class: NA

I.M.O. Subsidiary Risk: NA

I.M.O. ID Number: NA

I.M.O. Packing Group: NA

Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit Hazard Class: 9 UN Number 3316.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product does not meet the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): This product is not hazardous under 29 CFR.1910.1200 and therefore is not covered by Title III under SARA.

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

C.P.S.C.: Not applicable

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): None

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

TSCA CAS Number: Not applicable

16. OTHER INFORMATION

Intended Use: Surfactant

References: Vendor Information. Technical Judgment.

Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2007

GFS CHEMICALS, INC.
P.O. Box 245 Powell, OH 43065
740-881-5501(Tel.) 740-881-5989(Fax)
1-800-424-9300(Chemtrec 24Hr. Info.)

MATERIAL SAFETY DATA SHEET

2174

CONDUCTIVITY STANDARD SOLUTION

CHEMICAL NAME & SYNONYMS	DOT CLASS	SARA TITLE 313
Conductivity Standard Solution	NR	No
Potassium Chloride, Solution	**TSCA listed** - Yes	

FORMULA	REPORTABLE QUANTITY	F.W.	CAS#
KCl <5% - Not a hazardous material.	N/A	74.55	7447-40-7
H_2O >95%	N/A	18.02	7732-18-5

PHYSICAL DATA
Boiling point 100°C; Density 1.0; Conductivity @25°C 1,413 uS/cm (uMHO). Completely miscible with water.

APPEARANCE & ODOR
Clear, colorless solution. Odorless.

REACTIVITY & CONDITIONS TO AVOID
None.

FIRE HAZARDS
None.

EXTINGUISHER	FLASHPOINT	LEL	UEL
Fight surrounding fire.	N/A	N/A	N/A

HEALTH HAZARDS
Toxicity of this solution is very low (this is a very weak salt solution). Any salt solution may be irritating to eyes and mucous membranes. LD/TD: not found. OSHA PEL/ACGIH TLV not established. Not considered to be a carcinogen.

SPECIAL PRECAUTIONS
Always use good laboratory practices.

FIRST AID
Wash with water. If irritation develops, get medical attention.

SPILLS & LEAKS
Wash up with water. Disposal to sanitary drain.

CATALOG #	PREPARED BY	DATE
2174	MDM	April 1, 2004

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00024

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Ferrous Iron Reagent
Catalog Number: 2514025

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00024
Chemical Name: Not applicable
CAS No.: Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Hazard: May cause irritation.
Date of MSDS Preparation:
Day: 05
Month: October
Year: 2007

2. COMPOSITION / INFORMATION ON INGREDIENTS

1, 10-Phenanthroline
CAS No.: 5144-89-8
TSCA CAS Number: 66-71-7
Percent Range: 1.0 - 10.0
Percent Range Units: weight / weight
LD50: Oral Rat LD_{50} = 132 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: May cause irritation.

Sodium Bicarbonate
CAS No.: 144-55-8
TSCA CAS Number: 144-55-8
Percent Range: 90.0 - 100.0
Percent Range Units: weight / weight
LD50: Oral rat LD50 = 4220 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: May cause irritation.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: White powder
Odor: Not determined
MAY CAUSE EYE, SKIN AND RESPIRATORY TRACT IRRITATION

HMIS:
Health: 1
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 1
Flammability: 0
Reactivity: 0
Symbol: Not applicable
Potential Health Effects:
Eye Contact: May cause irritiation
Skin Contact: May cause irritiation
Skin Absorption: None reported
Target Organs: None reported
Ingestion: Very large doses may cause: abdominal pain gastrointestinal disturbances alkalosis which causes abnormally high alkali reserve of the blood and other body fluids hypotension
Target Organs: None reported
Inhalation: May cause: respiratory tract irritation
Target Organs: None reported
Medical Conditions Aggravated: Pre-existing: Kidney conditions
Chronic Effects: None reported
Cancer / Reproductive Toxicity Information:
This product does NOT contain any OSHA listed carcinogens.

This product does NOT contain any IARC listed chemicals.

This product does NOT contain any NTP listed chemicals.

Additional Cancer / Reproductive Toxicity Information: None reported
Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.
Ingestion (First Aid): Give large quantities of water. Call physician immediately.
Inhalation: Remove to fresh air.

5. FIRE FIGHTING MEASURES

Flammable Properties: Does not burn, but may melt in a fire, releasing toxic fumes.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not applicable

Hazardous Combustion Products: Toxic fumes of: sodium monoxide nitrogen oxides. carbon monoxide, carbon dioxide.
Fire / Explosion Hazards: None reported
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Water. Carbon dioxide Dry chemical.
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.
Containment Technique: Stop spilled material from being released to the environment.
Clean-up Technique: Scoop up spilled material into a large beaker and dissolve with water. Flush the spilled material to the drain with a large excess of water. Decontaminate the area of the spill with a weak acid solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe dust. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Keep container tightly closed when not in use. Protect from: moisture oxidizers
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin Do not breathe: dust Wash thoroughly after handling. Keep away from: oxidizers
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: White powder
Physical State: Solid
Molecular Weight: Not applicable
Odor: Not determined
pH: Not determined
Vapor Pressure: Not applicable
Vapor Density (air = 1): Not applicable

Boiling Point: Not applicable
Melting Point: Not determined
Specific Gravity (water = 1): 2.10
Evaporation Rate (water = 1): Not applicable
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not applicable
Solubility:
Water: Slightly soluble
Acid: Slightly soluble
Other: Not determined
Metal Corrosivity:
Steel: Not determined
Aluminum: Not determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Excess moisture Heating to decomposition.
Reactivity / Incompatibility: Incompatible with: oxidizers
Hazardous Decomposition: Toxic fumes of: nitrogen oxides sodium oxides carbon monoxide carbon dioxide
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: None reported
LC50: None reported
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: Sodium Bicarbonate: Eye - rabbit - 100 mg/30 seconds - MILD; Skin - Human - 30 mg/3 days intermittent - MILD
Mutation Data: None reported
Reproductive Effects Data: None reported
Ingredient Toxicological Data: Sodium Bicarbonate: Oral rat LD_{50} = 4220 mg/kg; 1, 10-Phenanthroline: Oral rat LD_{50} = 132 mg/kg

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: --
No ecological data available for the ingredients of this product.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Dilute material with excess water making a weaker than 5% solution. Open cold water tap completely, slowly pour the material to the drain.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:

D.O.T. Proper Shipping Name: Not Currently Regulated

--

DOT Hazard Class: NA

DOT Subsidiary Risk: NA

DOT ID Number: NA

DOT Packing Group: NA

I.C.A.O.:

I.C.A.O. Proper Shipping Name: Not Currently Regulated

--

ICAO Hazard Class: NA

ICAO Subsidiary Risk: NA

ICAO ID Number: NA

ICAO Packing Group: NA

I.M.O.:

I.M.O. Proper Shipping Name: Not Currently Regulated

--

I.M.O. Hazard Class: NA

I.M.O. Subsidiary Risk: NA

I.M.O. ID Number: NA

I.M.O. Packing Group: NA

Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit Hazard Class: 9 UN Number 3316. ALSO NOTE: If the National Competent Authority declares this product an environmental hazard by Special Provision 909 (IMDG) and Special Provision A97 (IATA) the classification may be UN3077 or UN3082.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

C.P.S.C.: Not applicable

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): None

California Perchlorate Rule CCR Title 22 Chap 33:

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

TSCA CAS Number: Not applicable

16. OTHER INFORMATION

Intended Use: Iron determination
References: TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). In-house information. Technical Judgment. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Association, 1991.
Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2008

Procter & Gamble
Fabric and Home Care Division
Ivorydale Technical Center
5299 Spring Grove Avenue
Cincinnati, OH 45217-1087

MATERIAL SAFETY DATA SHEET

MSDS #: LDL 0001 Issue Date: 09/28/00
Supersedes: LDL9901 Issue Date: 02/17/99

SECTION I - CHEMICAL PRODUCT

Identity: **Liquid Hand Dishwashing Detergents**

Brands:

DAWN (All Variations)

IVORY (All Variations)

JOY (All Variations)

Hazard Rating:			
	Health:	2	4=EXTREME
	Flammability:	1	3=HIGH
	Reactivity:	0	2= MODERATE
			1=SLIGHT

Emergency Telephone Number: 24hr P&G Operator:

DAWN - 1-800-725-3296 (DAWN)

IVORY 1-800-253-2753 (IVORY)

JOY - 1-800-436-1569 (JOY)

or call Local Poison Control Center or your physician

SECTION II - COMPOSITION AND INGREDIENTS

Ingredients/Chemical Name: Cleaning and sudsing agents (anionic and nonionic surfactants), dispensing aid (ethyl alcohol), water, stabilizing agents, colorant and perfume.

Dawn and Joy Antibacterial Hand Soaps also contain the antibacterial active Triclosan.
Dawn Hand Care product contains a protease enzyme.

Hazardous Ingredients as defined by OSHA, 29 CFR 1910.1200.

Chemical Name	Common Name	CAS No.	Recommended Limits	Composition Range	LD50/LC50
Ethyl alcohol	Ethanol	64-17-5	ACGIH TLV: 1880 mg/m^3	5-10%	N/A
Subtilisin	Protease	9014-01-1	NIIOSH STEL 0.00006 mg/m^3	<0.01%	N/A

SECTION III - HAZARDS IDENTIFICATION

Health Hazards (Acute and Chronic):

Ingestions: Ingestion may cause transient gastrointestinal irritation.

Eye Contact: May cause mild, transient irritation.

Skin: Transient irritation with prolonged exposure to concentrated material.

Signs and Symptoms of Exposure:

Ingestion: May result in nausea, vomiting, and/or diarrhea.

Eye Contact: May cause stinging, tearing, itching, swelling, and/or redness.

Skin: Prolonged contact with concentrated material may be drying or transiently irritating to skin.

SECTION IV - FIRST AID INFORMATION

Emergency and First Aid Procedures:

Ingestion: Drink 1 or 2 glasses of water.

Eye Contact: Flush thoroughly with water for 15 minutes.

Skin: If prolonged contact occurs, rinse thoroughly with water. If spilled on clothing, change clothes. If symptoms persist or recur, seek medical attention.

Other: Consumer product package has a voluntary avoid accidents statement.

SECTION V - FIRE FIGHTING INFORMATION

Flash Point (Method Used): 105-130°F. Pensky-Martens (Closed cup) **Explosive Limits**: *LEL:* N/A *UEL*: N/A

Extinguishing Media: CO_2, water or dry chemical.

Special Fire Fighting Procedures: None. Although this product has a flash point below 200°F (closed cup), it is an aqueous solution containing ethyl alcohol which does not sustain combustion.

Unusual Fire Hazards: None

Stability *Unstable*: *Conditions to Avoid*: None known

Stable: X

Incompatibility (Materials to Avoid): None known

Hazardous Decomposition/By Products: None known

Hazardous Polymerization: *May Occur*: *Conditions to Avoid*: None known

Will Not Occur: X

SECTION VI - ACCIDENTAL RELEASE MEASURES

Personal Precautions: None

Environmental Precautions: DISPOSAL IS TO BE PERFORMED IN COMPLIANCE WITH ALL FEDERAL, STATE AND LOCAL REGULATIONS. **Ultra Dawn, Ultra Joy and Ultra Ivory undiluted waste products are considered RCRA Ignitable.**

Solutions of the Ultra Dawn, Ultra Ivory or Ultra Joy liquid hand dishwashing detergents, diluted in the course of use, may be allowed to be flushed down sewer. First check with your local water treatment plant. Recycling is recommended for undiluted scrap product. Do not landfill.

Steps To Be Taken in Case Material is Released or Spilled: Prevent spills from reaching a waterway. Sorbents may be used. Read "Waste Disposal Method" below for further information.

SECTION VII - HANDLING AND STORAGE

Precautions To Be Taken in Handling and Storing: No unusual precautions necessary.

Other Precautions: None known

SECTION VIII - EXPOSURE CONTROLS, PERSONAL PROTECTION

Respiratory Protection (Specify Type): None required with normal use.

Ventilation *Local Exhaust*: None required with normal consumer use. *Special*: None

Mechanical (General): Normal/general dilution ventilation is acceptable. *Other*: None

Eye Protection: None required with normal consumer use.

Industrial Setting: For splash protection, use chemical goggles. Eye Wash fountain is desirable.

Protective Gloves: None required with normal use.

Industrial Setting: Protective gloves (rubber, neoprene) should be used for prolonged direct contact.

Other Protective Equipment: None required with normal use.

SECTION IX - PHYSICAL AND CHEMICAL PROPERTIES

Boiling Point °F: Not known

Vapor Pressure (mm Hg): N/A

Vapor Density (Air=1): N/A

Odor Threshold: N/A

Coefficient of Water/Oil Distribution: N/A

Scooped Density: N/A

Appearance and Odor: Purple, Blue, Green, Yellow, Pink or Orange liquids. All products are perfumed.

Specific Gravity (H_2O=1): ca. 1

Percent Volatile by Volume (%): ~60-65%

Evaporation Rate (nBuOAc=1): Unknown

Freezing Point: ~ 30 F

pH (1% solution): ~ 8

Solubility in Water: Completely

Reserve Alkalinity: N/A

SECTION X - STABILITY AND REACTIVITY

Possible Hazardous Reactions/Conditions: None known

Conditions to Avoid: None

Materials to Avoid: None

Hazardous Decomposition Products: None known

Other Recommendations: None

SECTION XI - TOXICOLOGICAL INFORMATION

LD50 (rats oral): approx 12 mL/kg

ED50 approx 2.3 mL/kg

Liquid hand dishwashing detergents have a relatively low order of toxicity. They may be irritating, but they are not expected to be corrosive. They are expected to be emetic.

SECTION XII - ECOLOGICAL INFORMATION

All surfactants are readily biodegradable. These products are safe for septic tanks.

SECTION XIII - DISPOSAL CONSIDERATIONS

Waste Disposal Method: DISPOSAL IS TO BE PERFORMED IN COMPLIANCE WITH FEDERAL, STATE AND LOCAL REGULATIONS. RCRA hazardous under the classification "Ignitable." RCRA provides an exemption for household waste. Household product is safe for disposal down the drain during use or in the trash. *Industrial Setting*: Solutions of diluted detergent in the course of use, may be allowed to be flushed down sewer. First check with your local water treatment plant. Recycling is recommended for undiluted scrap product. **Hazardous waste incineration is necessary for the Ultra versions of these products if disposal is ultimately warranted**. Do not landfill.

SECTION XIV - TRANSPORT INFORMATION

Small household containers of Dawn, Joy and Ivory are not DOT regulated.

SECTION XV - ADDITIONAL REGULATORY INFORMATION

All components are listed on the US TSCA Inventory. No components are affected by Significant New Use Rules (SNURs) under TSCA §5.

No components of Dawn, Ivory or Joy are subject to California Proposition 65 labeling.

All ingredients are CEPA approved for import to Canada by Procter & Gamble only. This product has been classified with Hazard Criteria of the Canadian Control Products Regulation (CPR) and this MSDS contains all information required by the Canadian Products Regulation.

SECTION XVI - OTHER INFORMATION

*N/A. - Not Applicable *N/K. - Not Known

The submission of this MSDS may be required by law, but this is not an assertion that the substance is hazardous when used in accordance with proper safety practices and normal handling procedures. Data supplied is for use only in connection with occupational safety and health.

The information contained herein has been compiled from sources considered by Procter & Gamble to be dependable and is accurate to the best of the Company's knowledge. The information relates to the specific material designated herein, and does not relate to the use in combination with any other material or any other process. Procter & Gamble assumed no responsibility for injury to the recipient or third persons, for any damage to any property resulting from misuse of the controlled product.

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00222

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Nitric Acid
Catalog Number: 15249

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00222
Chemical Name: Nitric acid
CAS No.: 7697-37-2
Chemical Formula: HNO_3
Chemical Family: Inorganic Acid
Hazard: Causes severe burns. Oxidizer.
Date of MSDS Preparation:
Day: 19
Month: March
Year: 2007

2. COMPOSITION / INFORMATION ON INGREDIENTS

Nitric Acid
CAS No.: 7697-37-2
TSCA CAS Number: 7697-37-2
Percent Range: 65.0 - 75.0
Percent Range Units: weight / weight
LD50: Oral human LDLo = 430 mg/kg.
LC50: Inhalation rat LC_{50} = 625 ppm/4hours.
TLV: 2 ppm
PEL: 2 ppm
Hazard: Causes severe burns. Oxidizer.

Demineralized Water
CAS No.: 7732-18-5
TSCA CAS Number: 7732-18-5
Percent Range: 25.0 - 35.0
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: No effects anticipated.

3. HAZARDS IDENTIFICATION

Emergency Overview:

Appearance: Faint yellow to colorless

Odor: Suffocating

MAY BE FATAL IF SWALLOWED OR INHALED CAUSES SEVERE BURNS

STRONG OXIDIZER: CONTACT WITH OTHER MATERIAL MAY BE EXPLOSIVE

HMIS:

Health: 3

Flammability: 1

Reactivity: 1

Protective Equipment: X - See protective equipment, Section 8.

NFPA:

Health: 3

Flammability: 0

Reactivity: 0

Symbol: oxy

Potential Health Effects:

Eye Contact: Causes severe burns

Skin Contact: Causes severe burns

Skin Absorption: None reported

Target Organs: None reported

Ingestion: Causes: severe burns

Target Organs: None reported

Inhalation: Causes: severe burns May cause: bronchitis pneumonitis teeth erosion

Target Organs: None reported

Medical Conditions Aggravated: Pre-existing: Eye conditions Respiratory conditions Skin conditions

Chronic Effects: Chronic overexposure may cause erosion of the teeth

Cancer / Reproductive Toxicity Information:

O.S.H.A. Listed: No

IARC Listed: No

NTP Listed: No

Additional Cancer / Reproductive Toxicity Information: None reported

Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.

Skin Contact (First Aid): Wash skin with plenty of water for 15 minutes. Remove contaminated clothing. Call physician immediately.

Ingestion (First Aid): Do not induce vomiting. Give 1-2 glasses of water. Never give anything by mouth to an unconscious person. Call physician immediately.

Inhalation: Remove to fresh air. Give artificial respiration if necessary. Call physician.

5. FIRE FIGHTING MEASURES

Flammable Properties: Not Flammable, but reacts with most metals to form flammable hydrogen gas. Strong oxidizer. Contact with combustible materials may cause a fire or explosion.

Flash Point: Not applicable

Method: Not applicable

Flammability Limits:

Lower Explosion Limits: Not applicable

Upper Explosion Limits: Not applicable
Autoignition Temperature: Not applicable
Hazardous Combustion Products: Toxic fumes of: nitrogen oxides.
Fire / Explosion Hazards: Contact with metals gives off hydrogen gas which is flammable May react violently with: combustible materials
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Use media appropriate to surrounding fire conditions Water spray to cool containers
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear. Evacuate area and fight fire from a safe distance.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.
Containment Technique: Remove all combustible material from spill area. Absorb spilled liquid with non-reactive sorbent material. Stop spilled material from being released to the environment. Dike large spills to keep spilled material from entering sewage and drainage systems or bodies of water.
Clean-up Technique: Cover spilled material with an alkali, such as soda ash or sodium bicarbonate. Scoop up slurry into a large beaker. Dilute with a large excess of water. Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Flush reacted material to the drain with a large excess of water. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate local area (15 foot radius or as directed by your facility's emergency response plan) when: any quantity is spilled. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Product is regulated as RCRA hazardous waste. Product is regulated as a hazardous water pollutant.
304 EHS RQ (40 CFR 355): Nitric Acid 1000 lbs.
D.O.T. Emergency Response Guide Number: 157

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin clothing Do not breathe mist or vapors. Use with adequate ventilation. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Store in a cool, dry place. Keep away from: combustible materials heat metals organic material
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Have a safety shower nearby. Use a fume hood to avoid exposure to dust, mist or vapor. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: neoprene latex gloves
Inhalation Protection: laboratory fume hood
Precautionary Measures: Avoid contact with: eyes skin clothing Do not breathe: mist/vapor Keep away from: alkalies metals organic materials other combustible materials oxidizable materials Wash thoroughly after handling.
TLV: 2 ppm
PEL: 2 ppm

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Faint yellow to colorless
Physical State: Liquid
Molecular Weight: 63.006
Odor: Suffocating
pH: <0.5
Vapor Pressure: 62 mm Hg @ 20°C
Vapor Density (air = 1): 2 - 3
Boiling Point: 122°C (251.6°F)
Melting Point: -34°C (-29.2°F)
Specific Gravity (water = 1): 1.41
Evaporation Rate (water = 1): Not determined
Volatile Organic Compounds Content: None
Partition Coefficient (n-octanol / water): Not applicable
Solubility:
Water: Miscible
Acid: Miscible
Other: Not determined
Metal Corrosivity:
Steel: Corrosive
Aluminum: Corrosive

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Excess moisture
Reactivity / Incompatibility: May react violently in contact with: acids alkalies carbides hydrogen sulfide metals turpentine May explode in contact with: combustible materials
Hazardous Decomposition: Toxic fumes of: nitrogen oxides
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: Oral human LDLo = 430 mg/kg.
LC50: Inhalation rat LC_{50} = 625 ppm/4hours.
Dermal Toxicity Data: None reported.
Skin and Eye Irritation Data: None reported.
Mutation Data: None reported.
Reproductive Effects Data: Oral rat TDLo = 2345 mg/kg: biochemical and metabolic effects on newborn.
Ingredient Toxicological Data: Not applicable

12. ECOLOGICAL INFORMATION

Product Ecological Information: Shore crab LC50 = 180 mg/l/48H; Cockle LC50 = 330 - 1000 mg/l/48H; Starfish LC50 = 100-300 mg/l/48H

Ingredient Ecological Information: --
Not applicable

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: D002

Special Instructions (Disposal): Work in an approved fume hood. Working in a large container, cautiously add small portions of the material to cold water with agitation. Do not breathe the fumes. Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Open cold water tap completely, slowly pour the reacted material to the drain.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash. Rinsate from empty containers may contain sufficient product to require disposal as hazardous waste.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Nitric Acid (70%)
DOT Hazard Class: 8
DOT Subsidiary Risk: NA
DOT ID Number: UN2031
DOT Packing Group: II
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Nitric Acid (70%)
ICAO Hazard Class: 8
ICAO Subsidiary Risk: NA
ICAO ID Number: UN2031
ICAO Packing Group: II
I.M.O.:
I.M.O. Proper Shipping Name: Nitric Acid (70%)
I.M.O. Hazard Class: 8
I.M.O. Subsidiary Risk: NA
I.M.O. ID Number: UN2031
I.M.O. Packing Group: II
Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit Hazard Class: 9 UN Number 3316.

15. REGULATORY INFORMATION

U.S. Federal Regulations:
O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)
E.P.A.:
S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard Delayed (Chronic) Health Hazard Fire Hazard
S.A.R.A. Title III Section 313 (40 CFR 372): This product contains a chemical(s) subject to the reporting requirements of Section 313 of Title III of SARA.
Nitric Acid
302 (EHS) TPQ (40 CFR 355): Nitric acid: 1000 lbs.
304 CERCLA RQ (40 CFR 302.4): Nitric acid: 1000 lbs.
304 EHS RQ (40 CFR 355): Nitric Acid 1000 lbs.
Clean Water Act (40 CFR 116.4): Nitric acid - RQ 1000 lbs.
RCRA: Contains RCRA regulated substances. See Section 13, EPA Waste ID Number.

C.P.S.C.: The label for this product bears the signal word "POISON" because the concentration of Nitric Acid in the product is greater than/equal to 5%.

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): None

California Perchlorate Rule CCR Title 22 Chap 33:

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: TSCA Listed: Yes

TSCA CAS Number: 7697-37-2

16. OTHER INFORMATION

Intended Use: Laboratory reagent

References: Vendor Information. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. The Merck Index, 11th Ed. Rahway, New Jersey: Merck and Co., Inc., 1989. NIOSH Registry of Toxic Effects of Chemical Substances, 1985-86. Cincinnati: U.S. Department of Health and Human Services, April, 1987. IARC Monographs on the Evaluation of the Carcinogenic Risks to Humans. World Health Organization (Volumes 1-42) Supplement 7. France: 1987. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Association, 1991. CCINFO RTECS. Canadian Centre for Occupational Health and Safety. Hamilton, Ontario Canada: 30 June 1993. 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor).

Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2008

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00055

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: NitriVer ® 3 Nitrite Reagent
Catalog Number: 2107169

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00055
Chemical Name: Not applicable
CAS No.: Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Hazard: Causes eye burns.
Date of MSDS Preparation:
Day: 24
Month: March
Year: 2007

2. COMPOSITION / INFORMATION ON INGREDIENTS

Chromatropic Acid, Disodium salt
CAS No.: 129-96-4
TSCA CAS Number: 129-96-4
Percent Range: 1.0 - 5.0
Percent Range Units: weight / weight
LD50: Oral rat LD50 > 5000 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: May cause irritation.

Sodium Sulfanilate
CAS No.: 515-74-2
TSCA CAS Number: 515-74-2
Percent Range: 5.0 - 15.0
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: Toxic properties unknown. May cause irritation.

Potassium Pyrosulfate
CAS No.: 7790-62-7
TSCA CAS Number: 7790-62-7

Percent Range: 1.0 - 10.0
Percent Range Units: weight / weight
LD50: Oral rat LD50 = 2340 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: Causes eye burns.

Potassium Phosphate, Monobasic
CAS No.: 7778-77-0
TSCA CAS Number: 7778-77-0
Percent Range: 75.0 - 85.0
Percent Range Units: weight / weight
LD50: Oral rat LD50 = 7100 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: May cause irritation.

1,2-Cyclohexanediaminetetraacetic Acid Trisodium Salt
CAS No.: 36679-96-6
TSCA CAS Number: 36679-96-6
Percent Range: 1.0 - 5.0
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: Toxic properties unknown. May cause irritation.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: White powder
Odor: Not determined
CAUSES EYE BURNS MAY CAUSE SKIN AND RESPIRATORY TRACT IRRITATION

HMIS:
Health: 3
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 2
Flammability: 0
Reactivity: 0
Symbol: Not applicable
Potential Health Effects:
Eye Contact: Causes eye burns.
Skin Contact: May cause irritiation
Skin Absorption: None reported
Target Organs: None reported

Ingestion: May cause: irritation of the mouth and esophagus Very large doses may cause: gastrointestinal disturbances cardiac depression kidney damage

Target Organs: Heart Kidneys

Inhalation: May cause: irritation of nose and throat

Target Organs: None reported

Medical Conditions Aggravated: Pre-existing: Eye conditions Kidney conditions Central nervous system diseases

Chronic Effects: None reported

Cancer / Reproductive Toxicity Information:

This product does NOT contain any OSHA listed carcinogens.

This product does NOT contain any IARC listed chemicals.

This product does NOT contain any NTP listed chemicals.

Additional Cancer / Reproductive Toxicity Information: None reported

Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.

Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.

Ingestion (First Aid): Do not induce vomiting. Give 1-2 glasses of water. Call physician immediately. Never give anything by mouth to an unconscious person.

Inhalation: Remove to fresh air. Give artificial respiration if necessary. Call physician.

5. FIRE FIGHTING MEASURES

Flammable Properties: During a fire, this product decomposes to form toxic gases.

Flash Point: Not applicable

Method: Not applicable

Flammability Limits:

Lower Explosion Limits: Not applicable

Upper Explosion Limits: Not applicable

Autoignition Temperature: Not applicable

Hazardous Combustion Products: Toxic fumes of: phosphorus oxides carbon monoxide, carbon dioxide.

Fire / Explosion Hazards: None reported

Static Discharge: None reported.

Mechanical Impact: None reported

Extinguishing Media: Use media appropriate to surrounding fire conditions

Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:

Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.

Containment Technique: Stop spilled material from being released to the environment.

Clean-up Technique: Scoop up spilled material into a large beaker and dissolve with water. Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Flush the spilled material to the drain with a large excess of water.

Evacuation Procedure: Evacuate local area (15 foot radius or as directed by your facility's emergency response plan) when: any quantity is spilled.

Special Instructions (for accidental release): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe dust. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Protect from: light heat moisture
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves lab coat
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin Do not breathe: dust Wash thoroughly after handling. Protect from: light heat moisture
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: White powder
Physical State: Solid
Molecular Weight: Not applicable
Odor: Not determined
pH: of 5% solution = 3.2
Vapor Pressure: Not applicable
Vapor Density (air = 1): Not applicable
Boiling Point: Not applicable
Melting Point: 224°C (435°F)
Specific Gravity (water = 1): 3.12
Evaporation Rate (water = 1): Not applicable
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not applicable
Solubility:
Water: Soluble
Acid: Not determined
Other: Not determined
Metal Corrosivity:
Steel: 0.057 in/yr
Aluminum: 0.00 in/yr

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Excess moisture Extreme temperatures
Reactivity / Incompatibility: None reported
Hazardous Decomposition: Toxic fumes of: phosphorus oxides carbon dioxide carbon monoxide
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: None reported
LC50: None reported
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: None reported
Mutation Data: None reported
Reproductive Effects Data: None reported
Ingredient Toxicological Data: Chromatropic Acid: Oral rat LD50: >5000 mg/kg, Potassium Phosphate Monobasic: Oral rat LD50 = 7100 mg/kg, Potassium Pyrosulfate: Oral rat LD50 = 2340 mg/kg

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: --
No ecological data available for the ingredients of this product.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Dilute material with excess water making a weaker than 5% solution. Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Open cold water tap completely, slowly pour the reacted material to the drain.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
DOT Hazard Class: NA
DOT Subsidiary Risk: NA
DOT ID Number: NA
DOT Packing Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
ICAO Hazard Class: NA
ICAO Subsidiary Risk: NA
ICAO ID Number: NA
ICAO Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
I.M.O. Hazard Class: NA
I.M.O. Subsidiary Risk: NA

I.M.O. ID Number: NA
I.M.O. Packing Group: NA
Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit
Hazard Class: 9 UN Number 3316.

15. REGULATORY INFORMATION

U.S. Federal Regulations:
O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)
E.P.A.:
S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard
S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.
--
302 (EHS) TPQ (40 CFR 355): Not applicable
304 CERCLA RQ (40 CFR 302.4): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
Clean Water Act (40 CFR 116.4): Not applicable
RCRA: Contains no RCRA regulated substances.
C.P.S.C.: Not applicable
State Regulations:
California Prop. 65: No Prop. 65 listed chemicals are present in this product.
Identification of Prop. 65 Ingredient(s): None
California Perchlorate Rule CCR Title 22 Chap 33:
Trade Secret Registry: Not applicable
National Inventories:
U.S. Inventory Status: This product contains a chemical(s) exempt from the TSCA 8(b) Inventory due to a Low Volume Exemption held by Hach Company.
TSCA CAS Number: Not applicable
1,2-Cyclohexanediaminetetraacetic Acid Trisodium Salt. This chemical may only be used as a chelating reagent for chemical reactions.

16. OTHER INFORMATION

Intended Use: Determination of nitrite
References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Association, 1991. In-house information. Technical Judgment. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992.
Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2008

Material Safety Data Sheet

Nitrogen

Section 1. Chemical product and company identification

Product name : Nitrogen

Supplier : AIRGAS INC., on behalf of its subsidiaries
259 North Radnor-Chester Road
Suite 100
Radnor, PA 19087-5283
1-610-687-5253

Product use : Synthetic/Analytical chemistry. Liquid – cryogenic coolant.

Synonym : nitrogen (dot); nitrogen gas; Nitrogen NF, LIN, Cryogenic Liquid Nitrogen, Liquid Nitrogen

MSDS # : 001040

Date of Preparation/Revision : **2/13/2009.**

In case of emergency : 1-866-734-3438

Section 2. Hazards identification

Physical state : Gas. [NORMALLY A COLORLESS GAS: MAY BE A CLEAR COLORLESS LIQUID AT LOW TEMPERATURES. SOLD AS A COMPRESSED GAS OR LIQUID IN STEEL CYLINDERS.]

Emergency overview : WARNING!

GAS:
CONTENTS UNDER PRESURE.
Do not puncture or incinerate container.
Can cause rapid suffocation.
May cause severe frostbite.
LIQUID:
Extremely cold liquid and gas under pressure.
Can cause rapid suffocation.
May cause severe frostbite.

Do not puncture or incinerate container.

Contact with rapidly expanding gases or liquids can cause frostbite.

Routes of entry : Inhalation

Potential acute health effects

Eyes : Contact with rapidly expanding gas may cause burns or frostbite. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Skin : Contact with rapidly expanding gas may cause burns or frostbite. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Inhalation : Acts as a simple asphyxiant.

Ingestion : Ingestion is not a normal route of exposure for gases. Contact with cryogenic liquid can cause frostbite and cryogenic burns.

Potential chronic health effects : **CARCINOGENIC EFFECTS**: Not available.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.

Medical conditions aggravated by over-exposure : Acute or chronic respiratory conditions may be aggravated by overexposure to this gas.

See toxicological information (section 11)

Section 3. Composition, Information on Ingredients

Name	CAS number	% Volume	Exposure limits
Nitrogen	7727-37-9	100	Oxygen Depletion [Asphyxiant]

Section 4. First aid measures

No action shall be taken involving any personal risk or without suitable training.If it is suspected that fumes are still present, the rescuer should wear an appropriate mask or self-contained breathing apparatus.It may be dangerous to the person providing aid to give mouth-to-mouth resuscitation.

Eye contact : Check for and remove any contact lenses. Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical attention immediately.

Skin contact : In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Clean shoes thoroughly before reuse. Get medical attention immediately.

Frostbite : Try to warm up the frozen tissues and seek medical attention.

Inhalation : Move exposed person to fresh air. If not breathing, if breathing is irregular or if respiratory arrest occurs, provide artificial respiration or oxygen by trained personnel. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Ingestion : As this product is a gas, refer to the inhalation section.

Section 5. Fire-fighting measures

Flammability of the product : Non-flammable.

Products of combustion : Decomposition products may include the following materials:
nitrogen oxides

Fire-fighting media and instructions : Use an extinguishing agent suitable for the surrounding fire.

Apply water from a safe distance to cool container and protect surrounding area. If involved in fire, shut off flow immediately if it can be done without risk.

Contains gas under pressure. In a fire or if heated, a pressure increase will occur and the container may burst or explode.

Special protective equipment for fire-fighters : Fire-fighters should wear appropriate protective equipment and self-contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode.

Section 6. Accidental release measures

Personal precautions : Immediately contact emergency personnel. Keep unnecessary personnel away. Use suitable protective equipment (section 8). Shut off gas supply if this can be done safely. Isolate area until gas has dispersed.

Environmental precautions : Avoid dispersal of spilled material and runoff and contact with soil, waterways, drains and sewers.

Methods for cleaning up : Immediately contact emergency personnel. Stop leak if without risk. Note: see section 1 for emergency contact information and section 13 for waste disposal.

Section 7. Handling and storage

Handling : High pressure gas. Do not puncture or incinerate container. Use equipment rated for cylinder pressure. Close valve after each use and when empty. Protect cylinders from physical damage; do not drag, roll, slide, or drop. Use a suitable hand truck for cylinder movement.
Never allow any unprotected part of the body to touch uninsulated pipes or vessels that contain cryogenic liquids. Prevent entrapment of liquid in closed systems or piping without pressure relief devices. Some materials may become brittle at low temperatures and will easily fracture.

Storage : Cylinders should be stored upright, with valve protection cap in place, and firmly secured to prevent falling or being knocked over. Cylinder temperatures should not exceed 52 °C (125 °F).
For additional information concerning storage and handling refer to Compressed Gas Association pamphlets P-1 Safe Handling of Compressed Gases in Containers and P-12 Safe Handling of Cryogenic Liquids available from the Compressed Gas Association, Inc.

Section 8. Exposure controls/personal protection

Engineering controls : Use only with adequate ventilation. Use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure to airborne contaminants below any recommended or statutory limits.

Personal protection

Eyes : Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists or dusts.

When working with cryogenic liquids, wear a full face shield.

Skin : Personal protective equipment for the body should be selected based on the task being performed and the risks involved and should be approved by a specialist before handling this product.

Respiratory : Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.

The applicable standards are (US) 29 CFR 1910.134 and (Canada) Z94.4-93

Hands : Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary.

Insulated gloves suitable for low temperatures

Personal protection in case of a large spill : Self-contained breathing apparatus (SCBA) should be used to avoid inhalation of the product.

Product name

nitrogen	Oxygen Depletion [Asphyxiant]

Consult local authorities for acceptable exposure limits.

Section 9. Physical and chemical properties

Molecular weight : 28.02 g/mole

Molecular formula : N2

Boiling/condensation point : -195.8°C (-320.4°F)

Melting/freezing point : -210°C (-346°F)

Critical temperature : -146.9°C (-232.4°F)

Vapor density : 0.967 (Air = 1) Liquid Density@BP: 50.46 lb/ft3 (808.3 kg/m3)

Specific Volume (ft^3/lb) : 13.8889

Gas Density (lb/ft^3) : 0.072

Section 10. Stability and reactivity

Stability and reactivity : The product is stable.

Hazardous decomposition products : Under normal conditions of storage and use, hazardous decomposition products should not be produced.

Hazardous polymerization : Under normal conditions of storage and use, hazardous polymerization will not occur.

Section 11. Toxicological information

Toxicity data

Other toxic effects on humans : No specific information is available in our database regarding the other toxic effects of this material to humans.

Specific effects

Carcinogenic effects : No known significant effects or critical hazards.

Mutagenic effects : No known significant effects or critical hazards.

Reproduction toxicity : No known significant effects or critical hazards.

Section 12. Ecological information

Aquatic ecotoxicity

Not available.

Environmental fate : Not available.

Environmental hazards : No known significant effects or critical hazards.

Toxicity to the environment : Not available.

Section 13. Disposal considerations

Product removed from the cylinder must be disposed of in accordance with appropriate Federal, State, local regulation.Return cylinders with residual product to Airgas, Inc.Do not dispose of locally.

Section 14. Transport information

Regulatory information	UN number	Proper shipping name	Class	Packing group	Label	Additional information
DOT Classification	UN1066 UN1977	NITROGEN, COMPRESSED Nitrogen, refrigerated liquid	2.2	Not applicable (gas).		**Limited quantity** Yes. **Packaging instruction** **Passenger aircraft** Quantity limitation: 75 kg **Cargo aircraft** Quantity limitation: 150 kg
TDG Classification	UN1066 UN1977	NITROGEN, COMPRESSED Nitrogen, refrigerated liquid	2.2	Not applicable (gas).		**Explosive Limit and Limited Quantity Index** 0.125 **Passenger Carrying Road or Rail Index** 75

Nitrogen						
Mexico Classification	UN1066 UN1977	NITROGEN, COMPRESSED Nitrogen, refrigerated liquid	2.2	Not applicable (gas).		-

"Refer to CFR 49 (or authority having jurisdiction) to determine the information required for shipment of the product."

Section 15. Regulatory information

United States

U.S. Federal regulations : **United States inventory (TSCA 8b):** This material is listed or exempted.

SARA 302/304/311/312 extremely hazardous substances: No products were found.
SARA 302/304 emergency planning and notification: No products were found.
SARA 302/304/311/312 hazardous chemicals: nitrogen
SARA 311/312 MSDS distribution - chemical inventory - hazard identification: nitrogen: Sudden release of pressure

Clean Water Act (CWA) 307: No products were found.

Clean Water Act (CWA) 311: No products were found.

Clean Air Act (CAA) 112 accidental release prevention: No products were found.

Clean Air Act (CAA) 112 regulated flammable substances: No products were found.

Clean Air Act (CAA) 112 regulated toxic substances: No products were found.

State regulations : **Connecticut Carcinogen Reporting:** This material is not listed.
Connecticut Hazardous Material Survey: This material is not listed.
Florida substances: This material is not listed.
Illinois Chemical Safety Act: This material is not listed.
Illinois Toxic Substances Disclosure to Employee Act: This material is not listed.
Louisiana Reporting: This material is not listed.
Louisiana Spill: This material is not listed.
Massachusetts Spill: This material is not listed.
Massachusetts Substances: This material is listed.
Michigan Critical Material: This material is not listed.
Minnesota Hazardous Substances: This material is not listed.
New Jersey Hazardous Substances: This material is listed.
New Jersey Spill: This material is not listed.
New Jersey Toxic Catastrophe Prevention Act: This material is not listed.
New York Acutely Hazardous Substances: This material is not listed.
New York Toxic Chemical Release Reporting: This material is not listed.
Pennsylvania RTK Hazardous Substances: This material is listed.
Rhode Island Hazardous Substances: This material is not listed.

Canada

WHMIS (Canada) : Class A: Compressed gas.

CEPA Toxic substances: This material is not listed.
Canadian ARET: This material is not listed.
Canadian NPRI: This material is not listed.
Alberta Designated Substances: This material is not listed.
Ontario Designated Substances: This material is not listed.
Quebec Designated Substances: This material is not listed.

Section 16. Other information

United States

Nitrogen

Label requirements : GAS:
CONTENTS UNDER PRESURE.
Do not puncture or incinerate container.
Can cause rapid suffocation.
May cause severe frostbite.
LIQUID:
Extremely cold liquid and gas under pressure.
Can cause rapid suffocation.
May cause severe frostbite.

Canada

Label requirements : Class A: Compressed gas.

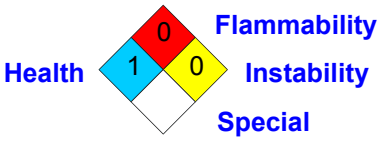
Hazardous Material Information System (U.S.A.) :

Health	1
Flammability	0
Physical hazards	0

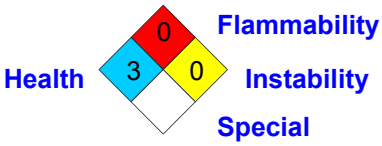
liquid:

Health	3
Fire hazard	0
Reactivity	0
Personal protection	

National Fire Protection Association (U.S.A.) :



liquid:



Notice to reader

To the best of our knowledge, the information contained herein is accurate. However, neither the above-named supplier, nor any of its subsidiaries, assumes any liability whatsoever for the accuracy or completeness of the information contained herein.
Final determination of suitability of any material is the sole responsibility of the user. All materials may present unknown hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards that exist.

Material Safety Data Sheet

June 1, 1999

YSI Incorporated
1725 Brannum Lane
Yellow Springs, OH 45387
USA

C-P# 05478-60

Information and Emergency Phone: (937) 767-7241

SECTION 1 - MATERIAL IDENTIFICATION

PRODUCT NAME: **YSI 3682 Zobell Solution** (ORP Cal. Solution) FORMULA: n/ap
Chemical Type: Inorganic chloride / cyanide
CAS No. n/app

SECTION 2 - HAZARDOUS / IMPORTANT INGREDIENTS

Chemical	CAS No.	PERCENT	PEL/TLV	CARCINOGEN (OSHA, NTP, IARC)
Potassium chloride	7447-40-7	72 - 78%	none	no
Potassium ferrocyanide, trihydrate	14459-95-1	10 - 15%	none	no
Potassium ferricyanide	13746-66-2	10 - 15%	none	no

SECTION 3 - CHEMICAL AND PHYSICAL PROPERTIES

Appearance: white powder
Odor: none
pH: neutral
Water Solubility: infinite
Evaporation Rate: n/av
Boiling Point: n/av
Melting Point: n/av
Specific Gravity: n/av
Vapor Pressure: n/ap
Vapor Density: n/ap

SECTION 4 - FIRE AND EXPLOSION HAZARDS

Flash Point: none Explosive Limits: none
Extinguishing Media: n/ap
Special Firefighting Procedures and Hazards: Material is not combustible. May emit toxic fumes when heated, such as NOx, HCN. HCl. Wear protection as described in Section 6.

SECTION 5 - REACTIVITY INFORMATION

Stable: X Unstable: Precautions: none known
Hazardous Polymerization: Occurs: Does Not Occur: X
Incompatibility: strong acids and oxcidizing agents.
Hazardous Decomposition Products: When heated, possibly NOx, HCN, HCl.

SECTION 6 - HEALTH HAZARDS / PROTECTIVE MEASURES / FIRST AID

Inhalation:
Possible irritation from dusts. (see CHRONIC below)
Use a NIOSH approved respirator for dusts. Get supplier recommendations. Provide adequate ventilation. Minimize dusty conditions.
Remove to fresh air and provide artificial respiration if needed.

Skin:
Possible irritation from dusts. (see CHRONIC below)
Wear dust-proof gloves and other body protection as needed. Minimize dusty conditions.
Wash exposed areas with soap and water for 15 minutes. Remove contaminated clothing, and wash before re-using.

Eyes:
Possible irritation from dust.
Wear dust barrier goggles. Eliminate dusty conditions.
Flush with water for 15 minutes.

Ingestion
No effects expected from normal use and minor amounts ingested. Large amounts, over 1 tablespoon, can cause digestive system upset s. (see CHRONIC below)
Reduce dusting. Avoid mouth breathing. Use facemask. Provide adequate ventilation.
Avoid swallowing. Spit out. Drink large amounts of water. Induce vomiting if person is conscious. Otherwise, and if effects persist, get medical attention.

CHRONIC EFFECTS: None reported for this material. "Cyanides" in general are often reported as toxic to humans. Therefore, it is recommended that exposure via skin, inhalation, and ingestion be limited.

IN ALL CASES: GET MEDICAL ATTENTION IF EFFECTS PERSIST.

Most likely routes of entry: skin, eyes, ingestion.

SECTION 7 - PRECAUTIONS FOR SAFE HANDLING AND USE

Spills and Leaks: Take up powder in any container and hold for disposal. Flush residual to sewer or ground. Provide personal protection as described in Section 6.

Storage and Handling: Keep containers closed. Discard any material that may be contaminated. Minimize dusting.

Waste Disposal: Is not listed as RCRA hazardous waste at this date. Cyanides are restricted in water disposed to streams and to sewers. Therefore, landfill disposal is indicated; check with local disposal companies.

Empty Containers: Rinse well. Dispose as appropriate for glass and plastic containers.

SECTION 8 - REGULATORY INFORMATION

DOT: Not regulated.
SARA Title III, S.313, Form R: Nothing reportable.

The information contained herein is based on data available at this time and is believed to be accurate. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof. Since information contained herein may be applied under conditions beyond our control, and with which we may be unfamiliar, no responsibility is assumed for the results of its use. The person receiving this information shall make his own determination of the suitability of the material for his particular use.

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00370

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Buffer Solution pH 10.01 ± 0.02
Catalog Number: 2283649

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00370
Chemical Name: Not applicable
CAS No.: Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Hazard: May cause irritation.
Date of MSDS Preparation:
Day: 27
Month: February
Year: 2008

2. COMPOSITION / INFORMATION ON INGREDIENTS

Demineralized Water
CAS No.: 7732-18-5
TSCA CAS Number: 7732-18-5
Percent Range: > 99.0
Percent Range Units: volume / volume
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: No effects anticipated.

Other components, each
CAS No.: Not applicable
TSCA CAS Number: Not applicable
Percent Range: < 1.0
Percent Range Units: volume / volume
LD50: Not applicable
LC50: Not applicable
TLV: Not established
PEL: Not established
Hazard: Any ingredient(s) of this product listed as "Other component(s)" is not considered a health hazard to the user of this product.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: Clear, blue
Odor: None

HMIS:
Health: 1
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 0
Flammability: 0
Reactivity: 0
Symbol: Not applicable
Potential Health Effects:
Eye Contact: May cause irritiation
Skin Contact: May cause irritiation
Skin Absorption: No effects anticipated
Target Organs: Not applicable
Ingestion: None reported
Target Organs: None reported
Inhalation: No effects anticipated
Target Organs: Not applicable
Medical Conditions Aggravated: None reported
Chronic Effects: None reported
Cancer / Reproductive Toxicity Information:
This product does NOT contain any OSHA listed carcinogens.

This product does NOT contain any IARC listed chemicals.

This product does NOT contain any NTP listed chemicals.

Additional Cancer / Reproductive Toxicity Information: None reported
Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with plenty of water. Call physician if irritation develops.
Ingestion (First Aid): Give large quantities of water. Call physician immediately.
Inhalation: None required.

5. FIRE FIGHTING MEASURES

Flammable Properties: Material will not burn.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not applicable

Hazardous Combustion Products: None
Fire / Explosion Hazards: None reported
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Use media appropriate to surrounding fire conditions
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.
Containment Technique: Stop spilled material from being released to the environment.
Clean-up Technique: Cover spilled material with a dry acid, such as citric or boric. Scoop up slurry into a large beaker. Adjust to a pH between 6 and 9 with an acid, such as sulfuric or citric. Flush reacted material to the drain with a large excess of water.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Protect from: heat Keep container tightly closed when not in use.
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves lab coat
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes Wash thoroughly after handling.
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Clear, blue
Physical State: Liquid
Molecular Weight: Not applicable
Odor: None
pH: 10.0
Vapor Pressure: Not determined
Vapor Density (air = 1): Not determined
Boiling Point: ~100°C (~212°F)
Melting Point: ~0°C (~32°F)
Specific Gravity (water = 1): 0.990

Evaporation Rate (water = 1): 0.76
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not determined
Solubility:
Water: Soluble
Acid: Soluble
Other: Not determined
Metal Corrosivity:
Steel: Not determined
Aluminum: Not determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Heat Evaporation
Reactivity / Incompatibility: None reported
Hazardous Decomposition: None reported
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: None reported
LC50: None reported
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: None reported
Mutation Data: None reported
Reproductive Effects Data: None reported
Ingredient Toxicological Data: None reported

12. ECOLOGICAL INFORMATION

Product Ecological Information: No information available for this product.

Ingredient Ecological Information: None reported

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Adjust to a pH between 6 and 9 with an acid, such as sulfuric or citric. Open cold water tap completely, slowly pour the reacted material to the drain.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--

DOT Hazard Class: NA
DOT Subsidiary Risk: NA
DOT ID Number: NA
DOT Packing Group: NA

I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
ICAO Hazard Class: NA
ICAO Subsidiary Risk: NA
ICAO ID Number: NA
ICAO Packing Group: NA

I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
I.M.O. Hazard Class: NA
I.M.O. Subsidiary Risk: NA
I.M.O. ID Number: NA
I.M.O. Packing Group: NA

Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit Hazard Class: 9 UN Number 3316.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable
304 CERCLA RQ (40 CFR 302.4): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
Clean Water Act (40 CFR 116.4): Not applicable
RCRA: Contains no RCRA regulated substances.

C.P.S.C.: Not applicable

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.
Identification of Prop. 65 Ingredient(s): None

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).
TSCA CAS Number: Not applicable

16. OTHER INFORMATION

Intended Use: Buffer

References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. Technical Judgment. In-house information.

Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2008

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00368

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Buffer Solution pH 4.01 ± 0.02
Catalog Number: 2283449

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00368
Chemical Name: Not applicable
CAS No.: Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Hazard: Practically non-toxic.
Date of MSDS Preparation:
Day: 09
Month: February
Year: 2008

2. COMPOSITION / INFORMATION ON INGREDIENTS

Demineralized Water
CAS No.: 7732-18-5
TSCA CAS Number: 7732-18-5
Percent Range: >95.0
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: No effects anticipated.

Other components, each
CAS No.: Not applicable
TSCA CAS Number: Not applicable
Percent Range: < 1.0
Percent Range Units: volume / volume
LD50: Not applicable
LC50: Not applicable
TLV: Not established
PEL: Not established
Hazard: Any ingredient(s) of this product listed as "Other component(s)" is not considered a health hazard to the user of this product.

Potassium Acid Phthalate
CAS No.: 877-24-7

TSCA CAS Number: 877-24-7
Percent Range: 1.0 - 5.0
Percent Range Units: weight / volume
LD50: Oral rat LDLo = 3200 mg/kg
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: May cause irritation.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: Clear, red liquid
Odor: None

HMIS:
Health: 0
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 0
Flammability: 0
Reactivity: 0
Symbol: Not applicable
Potential Health Effects:
Eye Contact: No effects are anticipated
Skin Contact: No effects are anticipated
Skin Absorption: No effects anticipated
Target Organs: Not applicable
Ingestion: No Effects Anticipated
Target Organs: Not applicable
Inhalation: No effects anticipated
Target Organs: Not applicable
Medical Conditions Aggravated: None reported
Chronic Effects: No effects anticipated
Cancer / Reproductive Toxicity Information:
This product does NOT contain any OSHA listed carcinogens.

This product does NOT contain any IARC listed chemicals.

This product does NOT contain any NTP listed chemicals.

Additional Cancer / Reproductive Toxicity Information: None reported
Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Flush eyes with water. Call physician if irritation develops.
Skin Contact (First Aid): Wash skin with soap and plenty of water.
Ingestion (First Aid): Give large quantities of water. Call physician immediately.

Inhalation: None required.

5. FIRE FIGHTING MEASURES

Flammable Properties: Material will not burn.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not applicable
Hazardous Combustion Products: Not applicable
Fire / Explosion Hazards: None reported
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Use media appropriate to surrounding fire conditions
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance.
Containment Technique: Stop spilled material from being released to the environment.
Clean-up Technique: Cover spilled material with an alkali, such as soda ash or sodium bicarbonate. Scoop up slurry into a large beaker. Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Flush reacted material to the drain with a large excess of water. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes Wash thoroughly after handling.
Storage: Keep container tightly closed when not in use.
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes Wash thoroughly after handling.
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Clear, red liquid
Physical State: Liquid
Molecular Weight: Not applicable
Odor: None
pH: 4.01
Vapor Pressure: Not determined
Vapor Density (air = 1): Not determined
Boiling Point: > 100°C (> 212°F)
Melting Point: < 0°C (< 32°F)
Specific Gravity (water = 1): 1.002
Evaporation Rate (water = 1): Not determined
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not determined
Solubility:
Water: Soluble
Acid: Soluble
Other: Not determined
Metal Corrosivity:
Steel: Not determined
Aluminum: Not determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Extreme temperatures
Reactivity / Incompatibility: None reported
Hazardous Decomposition: None reported
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: None reported
LC50: None reported
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: None reported
Mutation Data: None reported
Reproductive Effects Data: None reported
Ingredient Toxicological Data: Potassium Acid Phthalate: Oral rat LD_{Lo} = 3200 mg/kg

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: --
No ecological data available for the ingredients of this product.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Adjust to a pH between 6 and 9 with an alkali, such as soda ash or sodium bicarbonate. Open cold water tap completely, slowly pour the reacted material to the drain.

Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.

NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:

D.O.T. Proper Shipping Name: Not Currently Regulated

--

DOT Hazard Class: NA

DOT Subsidiary Risk: NA

DOT ID Number: NA

DOT Packing Group: NA

I.C.A.O.:

I.C.A.O. Proper Shipping Name: Not Currently Regulated

--

ICAO Hazard Class: NA

ICAO Subsidiary Risk: NA

ICAO ID Number: NA

ICAO Packing Group: NA

I.M.O.:

I.M.O. Proper Shipping Name: Not Currently Regulated

--

I.M.O. Hazard Class: NA

I.M.O. Subsidiary Risk: NA

I.M.O. ID Number: NA

I.M.O. Packing Group: NA

Additional Information: This product may be shipped as part of a chemical kit composed of various compatible dangerous goods for analytical or testing purposes. This kit would have the following classification: Proper Shipping Name: Chemical Kit Hazard Class: 9 UN Number 3316.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product does not meet the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): This product is not hazardous under 29 CFR.1910.1200 and therefore is not covered by Title III under SARA.

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

C.P.S.C.: Not applicable

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): --

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

TSCA CAS Number: Not applicable

16. OTHER INFORMATION

Intended Use: Buffer

References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. Technical Judgment. In-house information. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Association, 1991.

Revision Summary: Updates in Section(s) 14,

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2008

In-Situ Inc.
221 East Lincoln Avenue • Fort Collins, CO 80524 USA
1 800 446 7488 • 1 970 498 1500 (Tel)
1 970 498 1598 (Fax)
WWW.IN-SITU.COM

MATERIAL SAFETY DATA SHEET

I. Chemical Product and Company Identification

Product Name:	**Manufacturer:**	**Emergency Contact:**
Quick Cal Solution	In-Situ Inc. 221 East Lincoln Avenue Fort Collins, Colorado 80524 Telephone: 970 498 1500 Fax: 970 498 1598	INFOTRAC Emergency Response Hotline: 1-800-535-5053 (in the U.S. and Canada) 1-352-323-3500 www.infotrac.net
Hazardous Shipping Label:	**Dot** None	**IATA** None

II. Ingredients

Hazardous Components Specific Chemical Identity: Common Names	**CAS NO.**	**%**	**OSHA PEL**	**ACGIH TLV**	**LD_{50} (mg/Kg)**
Potassium Dihydrogen Phosphate (KH_2PO_4)	7778-77-0	< 1.0	None Listed	None Listed	None Listed
Potassium Chloride (KCl)	7447-40-7	< 1.0	None Listed	None Listed	3020 (ORL-RAT)
Sodium Hydroxide, NaOH	1310-73-2	< 1.0	None Listed	None Listed	1600 (ORL-RAT)
Potassium ferricyanide ($K_3Fe(CN)_6$)	13746-66-2	< 1.0	None Listed	None Listed	None Listed
Potassium ferricyanide trihydrate ($K_3Fe(CN)_6\ 3H_2O$)	14459-95-1	< 1.0	None Listed	None Listed	None Listed
Deionized Water, H_2O	7732-18-5	> 98	None Listed	None Listed	190,000 (IPR-MUS)

III. Physical Data

Boiling Point @ 750 mm Hg	100 °C	**Freezing Point**	0 °C
pH @ 25 °C	7.00	**Vapor Pressure @ 25 °C**	NA
Volatiles % By Wt.	NA	**Solubility in Water, % by Wt @ 25 °C**	Miscible
Vapor Density (Air = 1)	NA	**Evaporation Rate (Butyl, Acetate = 1)**	NA
Specific Gravity (Water = 1)	1.0	**Odor**	odorless
Appearance	Light green Liquid		

IV. Fire And Explosion Hazard Data

Flash Point (Test Method)	Not Flammable	**Autoignition Temperature**	NA
Flammable Limits in air, % by volume		**Lower** NA	**Upper** NA
Extinguishing Media	Water, CO_2, Dry Chemical , Foam Spray	**Special Fire-Fighting Procedures**	None, non-flammable
Unusual Fire & Explosion Hazards	None		

* NA – Not Applicable/not available

In-Situ Inc.

V. Reactivity Data

Stability	*Unstable*	*Stable* X
Conditions to Avoid	None	
Incompatibility (Materials to Avoid)	None	
Hazardous Decomposition Products	None	
Hazardous Polymerization	Cannot Occur	

VI. Health Hazard Data

Routes of Entry	***Inhalation*** Yes	***Skin*** Yes	***Ingestion*** Yes
Health Hazards	***Acute***	Irritation may occur in case of eye or skin contact. Irritation may also occur to mucous membranes from vapors.	
	Chronic	This substance is toxic to blood, lungs, mucous membranes.	
Carcinogenicity	***NTP*** Not Found	***IARC Monographs*** Not Found	***OSHA Regulated*** Not Found
Signs and Symptoms of Exposure	Irritation		
Medical Conditions Generally Aggravated by Exposure	Could Aggravate Diseases of Skin		
Emergency And First Aid Procedures	If skin contact occurs, wash off contact area with water. If ingested, give large amounts of water. Do not induce vomiting. Contact physician.		

VII. Precautions for Safe Handling and Use

Steps to be Taken In Case Material is Released or Spilled	Dilute with water and set aside for diposal
Waste Disposal Method	Consult Federal, State and Local laws for proper disposal.
Precautions To Be Taken In Handling and Storing	Suitable for any general handling storage. NFPA Rating: Scale (0-4); Health –2, Fire – 0, Reactivity – 0, Specific - None
Other Precautions	Do Not Ingest!

VI. Control Measures

Respiratory Protection (specific type)	None	
Ventilation	***Local Exhaust*** None	***Special*** None
	Mechanical (General) Use in Fume Hood	***Other*** None
Protective Gloves Yes	**Eye Protection** Safety Glasses	**Other Protective Clothing or Equipment** None
Work/Hygienic Practices	Emergency eyewash should be available. Wash hands after working with this product	

APPENDIX D

PORTABLE GENERATOR HAZARDS

Consumer Product Safety Commission Safety Alert

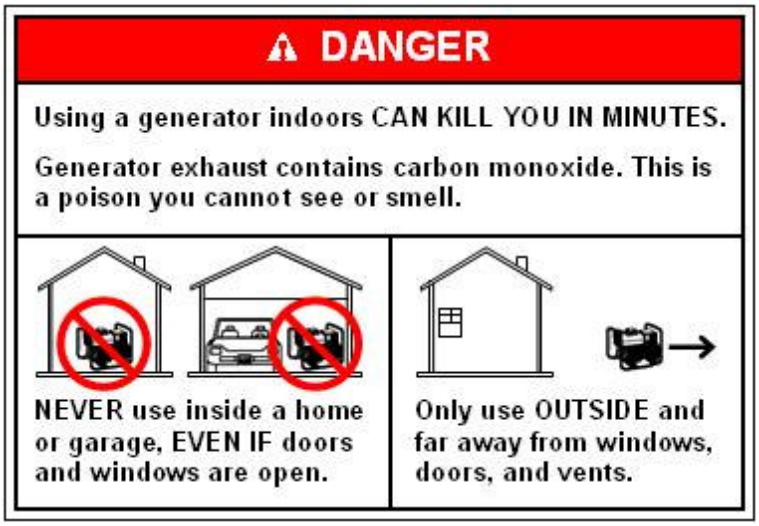
Portable Generator Hazards

Portable generators are useful when temporary or remote electric power is needed, but they also can be hazardous. The primary hazards to avoid when using a generator are carbon monoxide (CO) poisoning from the toxic engine exhaust, electric shock or electrocution, fire and burns.

Every year, people die in incidents related to portable generator use. Most of the incidents associated with portable generators reported to CPSC involve CO poisoning from generators used indoors or in partially-enclosed spaces.

Carbon Monoxide Hazards

When used in a confined space, generators can produce high levels of CO within minutes. When you use a portable generator, remember that you cannot see or smell CO. Even if you do not smell exhaust fumes, you may still be exposed to CO.



Danger labels are required on all portable generators manufactured or imported on or after May 14, 2007.

If you start to feel sick, dizzy, or weak while using a generator, get to fresh air **RIGHT AWAY. DO NOT DELAY**. The CO from generators can rapidly kill you.

Follow these safety tips to protect against CO poisoning.

- **NEVER** use a generator inside homes, garages, crawlspaces, sheds, or similar areas, even when using fans or opening doors and windows for ventilation. Deadly levels of carbon monoxide can quickly build up in these areas and can linger for hours, even after the generator has shut off.
- Follow the instructions that come with your generator. Locate the unit outdoors and far from doors, windows, and vents that could allow CO to come indoors.
- Install battery-operated CO alarms or plug-in CO alarms with battery back-up in your home, according to the manufacturer's instructions. CO alarms should be certified to the requirements of the latest safety standards (UL 2034, IAS 6-96, or CSA 6.19.01). Test batteries monthly.

To avoid CO poisoning when using generators:

- Never run generators indoors, including garages, basements, crawlspaces and sheds.
- Get to fresh air right away if you start to feel dizzy or weak.

Electrical Hazards

- Generators pose a risk of shock and electrocution, especially if they are operated in wet conditions. If you must use a generator when it is wet outside, protect the generator from moisture to help avoid the shock/electrocution hazard, but do so without operating the generator indoors or near openings to any building that can be occupied in order to help avoid the CO hazard. Operate the generator under an open, canopy-like structure on a dry surface where water cannot reach it or puddle or drain under it. Dry your hands, if wet, before touching the generator.

- Connect appliances to the generator using heavy-duty extension cords that are specifically designed for outdoor use. Make sure the wattage rating for each cord exceeds the total wattage of all appliances connected to it. Use extension cords that are long enough to allow the generator to be placed outdoors and far away from windows, doors and vents to the home or to other structures that could be occupied. Check that the entire length of each cord is free of cuts or tears and that the plug has all three prongs. Protect the cord from getting pinched or crushed if it passes through a window or doorway.

- **NEVER** try to power the house wiring by plugging the generator into a wall outlet, a practice known as “backfeeding.” This is extremely dangerous and presents an electrocution risk to utility workers and neighbors served by the same utility transformer. It also bypasses some of the built-in household circuit protection devices.

Fire Hazards

- **Never** store fuel for your generator in the home. Gasoline, propane, kerosene, and other flammable liquids should be stored outside of living areas in properly-labeled, non-glass safety containers. Do not store them near a fuel-burning appliance, such as a natural gas water heater in a garage.

- Before refueling the generator, turn it off and let it cool down. Gasoline spilled on hot engine parts could ignite.

5123/0407

Send the link for this page to a friend! Consumers can obtain this publication and additional publication information from the Publications section of CPSC's web site or by sending your publication request to info@cpsc.gov.

This document is in the public domain. It may be reproduced without change in part or whole by an individual or organization without permission. If it is reproduced, however, the Commission would appreciate knowing how it is used. Write the U.S. Consumer Product Safety Commission, Office of Information and Public Affairs, 4330 East West Highway, Bethesda, MD 20814 or send an e-mail via CPSC's On-Line Form.

The U.S. Consumer Product Safety Commission is charged with protecting the public from unreasonable risks of serious injury or death from thousands of types of consumer products under the agency's jurisdiction. The CPSC is committed to protecting consumers and families from products that pose a fire, electrical, chemical, or mechanical hazard. The CPSC's work to ensure the safety of consumer products - such as toys, cribs, power tools, cigarette lighters, and household chemicals - contributed significantly to the decline in the rate of deaths and injuries associated with consumer products over the past 30 years.

To report a dangerous product or a product-related injury, call CPSC's hotline at (800) 638-2772 or CPSC's teletypewriter at (800) 638-8270, or visit CPSC's web site at www.cpsc.gov/talk.html. To join a CPSC email subscription list, please go to https://www.cpsc.gov/cpsclist.aspx. Consumers can obtain this release and recall information at CPSC's Web site at www.cpsc.gov.

Consumer Safety (Home) | About CPSC | Library | Business

APPENDIX E

EMERGENCY CONTACTS

EMERGENCY SERVICES

Security (Police/Sheriff)	911
Fire/Rescue	911
Ambulance	911
Caribou Memorial Hospital Emergency Room	208-547-3341
Portneuf Medical Center Emergency Room (Pocatello)	208-239-1800
Star Valley Hospital Emergency Room (Afton, WY)	307-885-5821
Idaho Poison Control Center	800-860-0620

EMERGENCY CONTACT NUMBERS

Vance Drain (MWH Project Manager)	office: 801-617-3250 cell: (b) (6)
Cary Foulk (RI/FS Task Manager)	office: 970-871-4371 cell: (b) (6)
Emily Yeager (On Site Safety Officer)	cell: (b) (6)
Rachel Roskelley (P_4 Production Project Manager)	office: 208-547-1248 cell: (b) (6)
Emergency	911

Note: It may be necessary to dial a '9' to access an outside line at mine sites when calling.

HOSPITAL FACILITIES

The closest hospital and the specific route to the hospital will depend on where field activities are taking place. The following are the hospitals closest to the project sites:

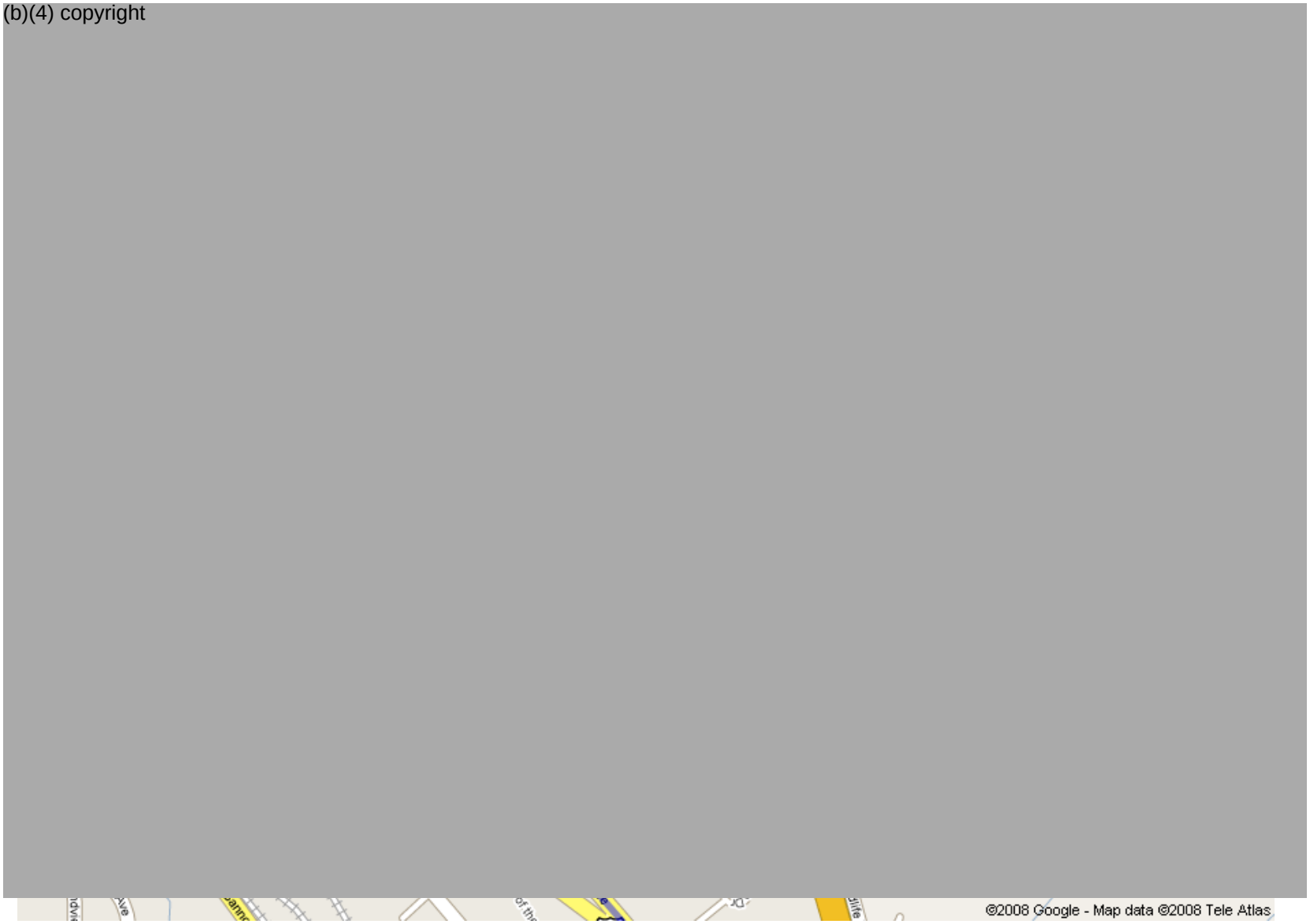
Caribou Memorial Hospital, 300 South 3rd West, Soda Springs, Idaho

(b)(4) copyright

Portneuf Medical Center (East Campus), 777 Hospital Way, Pocatello, Idaho, At Pine Ridge Mall

(b)(4) copyright

Portneuf Medical Center (West Campus), 651 Memorial Drive, Pocatello, Idaho



Star Valley Medical Center	307-886-5800 (Information)
110 Hospital Lane	307-886-5821 (Emergency Room)
Afton, Wyoming	911 (Search and Rescue/Sheriff's Dispatch)

In general, to get to the Caribou Memorial Hospital, proceed to Highway 30 in Soda Springs, which is also known as 2nd South. Proceed to 3rd West, and turn south to 3rd South. To get to the Portneuf Medical Center (East Campus), proceed to I-15 and take the Clark Street exit. The hospital is at the top of a hill and is visible from the freeway. To get to the Portneuf Medical Center (West Campus), proceed to I-15 through Pocatello and take the Clark Street exit. Drive West on Clark, turning South on 15th Street. The West Campus is on the right side. To get to the Star Valley Hospital, proceed to Highway 89 and turn onto Hospital Lane. When making calls from mine offices, it may be necessary to dial '9' to access an outside line.

APPENDIX F

ACKNOWLEDGEMENT FORM

PERSONAL ACKNOWLEDGMENT FORM

HEALTH AND SAFETY PROGRAM

PROGRAM OR SITE

As a component of the Health and Safety Plan designed to provide personnel safety during the investigation of the southeast Idaho phosphate resource area, you are required to read and understand the Health and Safety Plan. When you have fulfilled this requirement, please sign and date this personal acknowledgment form, and return the form to the On-Site Safety Officer.

Signature

Name (Printed)

Date

APPENDIX G

MONSANTO CONTRACTOR/GUEST ES&H SITE GUIDELINES

6.0 Contractor/Guest ES&H Guidelines

6.1.0 CONTRACTOR MANAGEMENT SYSTEMS

Contractors shall provide a full time Safety Representative in the following conditions:

a.) When the contract company has 10 or more employees on site.

b.) If they have less than 10 employees but the job has been defined as a high-hazard job through and ESH review, or if required by the Contractor Safety Specialist.

c.) The full-time Safety Representative shall:

- Have a minimum of three years experience in construction safety and have passed the OSHA 30 Hour Construction Course.
- Must have Accident/Incident Investigation Skills
- In some incidences, First Aid/CPR certification.

d.) The Safety Representative shall be responsible for all subcontractors, and to ensure they are following ESH requirements.

e.) The Safety Representative's primary responsibility will be to continuously monitor the contractor's personnel, the contractor's subcontractor personnel and equipment to ensure compliance with all applicable ESH requirements.

f.) If the Safety Representative does not demonstrate the ability to perform his/her work to the satisfaction of the Monsanto Representative, the Monsanto Representative may ask that this person be replaced by another Safety Representative meeting the criteria listed above.

g.) The Safety Representative must have the authority to correct any hazard or unsafe practice on the project, even if a work stoppage is required.

6.0 Contractor/Guest ES&H Site Guidelines

6.1.1 EMERGENCY RESPONSE

Purpose:

This plan provides for the immediate organization of all efforts to prevent injury to personnel, damage to property and the environment, and to ensure accountability of employees.

Scope:

The plant Emergency Plan shall be used for any abnormal incidents that may occur inside of the Soda Springs Site including, but not limited to, personnel injury, or serious property or environmental releases, which have the potential to impact the surrounding community.

General:

Plant emergencies can be reported to PROVOX operators by dialing 777 on any plant telephone or by using plant radios.

The Plant Emergency Plan identifies two levels of plant emergencies:

Level One- Emergencies controlled by the on-site emergency response team.

Level Two- Any plant emergency that requires more resources than are available in the plant and/or environmental incident that would impact the surrounding area.

When upgraded to a level two incident, the furnace or kiln PROVOX Operator will be instructed to activate the plant emergency sirens, initiating plant headcount procedures.

The emergency sirens are tested every Friday at 12:00 noon.

HEADCOUNT:

When the sirens are activated, Mark III will report to the Mark III Shop for headcount.

On weekends, holidays and night shifts, Mark III personnel assigned to manufacturing areas should report to the designated manufacturing headcount area.

Temporary contractors/visitors are to report directly to the guard station used for entry into the plant (East ext. 1310 or West ext. 1480) for headcount reporting.

NorthWest Cleaning personnel assigned to work in remote areas of the plant (Furnace Office, E&I Shop, etc.) should immediately phone East Security to report headcount and receive further instructions.

If the contractor/visitor cannot report to the assigned guard station, proceed to the closest designated area (listed below) and report by phone or radio to your assigned area (the guard station). The CMR assigned to the contractor/visitor is responsible to inform them of emergency procedures and if possible escort them to the assigned headcount.

Plant Headcount Areas

1. #8 & 9 Furnace Tapping Lunchroom
2. #7 & 8 Furnace Operating Level Lunchroom
3. Phos Dock Lunchroom
4. Main Shop Lunchroom
5. Machine Shop Lunchroom
6. Electric Shop Lunchroom
7. HEG Lunchroom
8. Department 10 Lunchroom
9. Mark III Shop Lunchroom
10. Security Office (East Entrance)
11. Security Office (West Entrance
12. Admin. Building Multipurpose Room #59
13. Storeroom Conference Room

Evacution:

In the event the evacuation becomes necessary, the emergency plan indicates two levels of evacuation:

Partial Evacuation-Instructions will be delivered via radio, telephone or runner.

Full Site Evacuation-The emergency sirens will be activated a second and third time. Indicating the need for all personnel to proceed to the assigned evacuation collection areas as listed below:

Plant Evacuation Collection Areas

South Area: (As indicated by the assigned CMR)

Primary - Administration Building Multi-Purpose Conference Room.
Secondary - HEG Shop

North Area: (as indicated by the assigned CMR)

Primary - HEG Shop
Secondary - Administration Building Multi-Purpose Conference Room.

If you are outside of, or cannot safely reach your designated collection area, proceed to the assigned secondary collection area and report by phone or radio.

The CMR assigned to the contractor/visitor, if possible, should escort them to the assigned evacuation collection area.

EMERGENCY EQUIPMENT:

1. The use of designated fire equipment for activities other than fire fighting is prohibited.

2. Know the location of, and how to use, emergency equipment in the area where you are working. Emergency equipment includes safety tubs, showers, eye wash stations, stretchers, positive air equipment and fire extinguisher.

3. Any contractor performing work on a fire protection system must first obtain an Impairment Permit from the Safety Department. Fire protection systems include fire water supply systems, sprinkler systems, FM-200 systems, etc.

4. Never block emergency equipment such as fire hydrants, hoses, extinguishers, emergency vehicles etc.

6.0 Contractor/Guest ES&H Site Guidelines

6.1.3 TRAINING

1. Contractors shall instruct each employee in the recognition and avoidance of unsafe conditions and regulations applicable to his/her work environment to control or eliminate any hazards or other exposure to illness or injury.

2. Contractors shall maintain records of all employees training. Training records shall contain, as a minimum:

   a.) Name and signature of the employee

   b.) Date of training

   c.) Subject and content of the training

   d.) Means used to verify that employee understood the training

   e.) Name of the instructor

3. When requested, the Certified Monsanto Representative shall be provided with documentation and certification of contractor training. In addition, the contract company must assure that all employees have the appropriate job skills, knowledge and proper technical certifications necessary to perform their work safely. Some certifications may include but are not limited to: Certified Welders, Crane Operators, Qualified Electrical Workers and Qualified Riggers. Monsanto reserves the right to audit Contractor Safety Training.

6.0 Contractor/Guest ES&H Guidelines

6.1.4 ENGLISH PROFICIENCY

1. All contractors must have the ability to read and speak English in order to perform their work safely.

6.0 Contractor/Guest ES&H Site Guidelines

6.2.0 WORKING IN AN OPERATING UNIT

Department Hazards

1. No one shall enter another department to perform work or use that department's equipment without first obtaining permission from that department's Operating Crew Supervisor.

2. No unauthorized person should ever start up or shut down a piece of equipment in any department (unless danger to another person's life exists).

3. Owning department will be responsible for clearing/isolating all process lines as well as initiating any and all required hazardous work permits.

4. Never reach under or between belts, pulleys, idlers, rollers, chains, gears, etc., unless the equipment is locked out.

5. Equipment shall not be operated without proper guards and safety chains in place.

6. Only qualified members of the Monsanto Electrical Department will open electrical boxes to remove fuses, energize resets or to perform other electrical functions. Electrical contractors must first get clearance from the Monsanto Electrical Department to perform any electrical work. The use of Ground Fault Circuit Interrupters is required when using tools.

7. Due to the high potential of injury resulting from the uncontrollable whipping of an air hose, which has unexpectedly uncoupled, whip checks will be required when using tools such as jackhammers and air drills.

8. Whip checks are also required when doing any high pressure water blasting. There are two types of whip checks available from the plant Storeroom.

9. All snap-on hose connections must be secured by wire pins.

10. No one shall use compressed air to clean his or her clothing or body. Compressed air shall not be directed toward anyone.

11. Compressed air shall not be used for cleaning purposes except when reduced to less than 30 P.S.I., or when a venting device is used along with all appropriate personal protective safety equipment. Note: Use industrial vacuums whenever possible.

12. Department Supervisor's approval must be obtained before using steam or air to unstop a pipe or line.

13. When working with Soda Ash or Lime; goggles and dust mask or respirators with Hepa filters are required.

14. Any cam-lock fitting used on Soda Springs Sites must be secured with a secondary safety device such as wire ties, pins, etc., or other means to prevent inadvertent disconnection.

6.0 Contractor/Guest ES&H Site Guidelines

6.2.1 OFFICE SAFETY

1. The following items, while not intended to be all-inclusive, are indications of some of the areas of concern for good office safety:

    a. Never carry pencils with points exposed in shirt pockets.

    b. Report spills, slipping and tripping hazards to the supervisor immediately. Clean up or correct them as soon as possible.

    c. Keep all machinery guards in place.

    d. Never leave paper cutter arm in an up, or open position.

2. The following items, while not intended to be all-inclusive, are indications of some of the areas of concern for good office housekeeping:

    a. Do not store used equipment in office.

    b. Do not stack large amounts of paper, and other items, on the tops of file cabinets, bookcases and tables.

    c. Clear desk and table tops at the end of each workday.

    d. Use coat racks or closets for coats and surplus clothing.

    e. Limit wall hangings to what is appropriate.

6.0 Contractor/Guest ES&H Site Guidelines

6.3.1 BASIC ENVIRONMENTAL SAFETY AND HEALTH RULES

Working safely is a condition of employment at this plant. Each employee (Monsanto & Contractor) must comply with the plant's safety rules and policies. Knowledge of these specific rules and policies is mandatory in order to perform work safely. In general, plant rules are provided to help insure everyone's safety. The following site-specific rules are provided to address specific safety concerns that are unique to our plant.

1. The best insurance against incidents and injuries:

    a) Know the job, the pertinent safety rules and area hazard.

    b) Keep focused, and your mind on the work at hand.

    c) Use common sense.

    d) If you can't do it safely, don't do it.

    e) If you don’t know, don’t be afraid to ask.

2. Only approved hard hats (ANSI 289.1-1969), which are equipped with goggles, shall be worn inside the plant.

3. Long hair below the nape of the neck must be pinned up or worn in a ponytail.

4. Radios, televisions, and cameras require permission from the Site Manager before being brought into the plant. The use of a cell phone camera for taking pictures requires permission from the Site Manager.

5. All individuals have a direct responsibility to immediately report unsafe acts or conditions to supervision. This includes reporting any near misses including, but not limited to: unexpected equipment condition, defeated

safety systems, or other process safety or personal safety items. If a potential injury is imminent, an attempt must be made to stop the unsafe act or condition, and report it through the use of the corrective action form.

6. It is the responsibility of the CMR to explain area hazards to the contractor before the contractor performs any work.

7. All "Hot" work will cease if the plant disaster alarm is sounded. (This includes cigarette smoking).

8. Eating or drinking is prohibited in production areas (designated eating areas are provided).

9. Contractors must observe safety signs while on Monsanto property.

10. Contractors performing work inside the main office, service building, laboratory or storeroom, will wear the appropriate safety protection (to be identified by the Safety Department).

11. Always use handrails when going up and down stairs. Remember the 3-point contact.

12. Objects or equipment must never be placed within 3 feet of electrical boxes or disconnects.

13. Equipment shall not be operated within 15 feet of power lines.

14. A full-face cutting hood or a face shield with goggles is required when using a cutting torch.

15. A face shield with goggles is required when doing any kind of grinding, cutting, sanding, water blasting, sawing, hammering or chipping that might have the potential for flying debris.

16. BUTANE lighters <u>are not</u> allowed in the plant.

17. No one under the age of eighteen may work on Monsanto property. This includes contractors and sub contractors.

18. Contractors should avoid strains when lifting by keeping their back as nearly upright as possible, using leg muscles instead of the back and stomach muscles. They should not attempt to lift more than 50 lbs per person without assistance.

PERSONAL CONDUCT:

Certain rules of personal conduct on the job are necessary for the successful operation of any organization. Some of the things that are prohibited while on Monsanto property are:

1. Smoking at Safety Meetings, in offices, or at any Monsanto function being conducted indoors.

2. Bringing intoxicating liquor or narcotics into the plant, or entering the plant under the influence of liquor or drugs, drinking or using narcotics on company property.

3. Bringing firearms or any type of explosive into the plant unless approved by the Site Manager.

4. Engaging in fights, horseplay, practical jokes or indecency.

5. Willfully damaging plant or personal property.

6. Using threatening or abusive language toward fellow employees or supervision.

7. Stealing.

8. Gambling on plant property.

9. Posting notices on company property without prior approval of Plant Management.

The above general rules cannot possibly cover all situations that might arise. As a rule of thumb however, it can be assumed that contractor/visitors are not to engage in any activity that interferes with another employee's or group of employees work, or engage in activity that interferes with the normal continuity of plant operations or the maintenance of order on its premises.

JEWELRY POLICY:

1. Rings, metal band watches, bracelets and necklaces will not be worn in the plant, quarry, storeroom, lab or mine.

2. Rings, metal band watches, bracelets and necklaces will not be worn when performing maintenance/electrical work on plant property.

3. Hoop: earrings, nose-rings and brow-rings will not be worn in the plant, mine quarry, storeroom or lab or when performing maintenance/electrical work on plant property.

4. Stud jewelry is allowed.

5. Medical alert necklaces and bracelets are allowed, and will be kept under clothing when performing maintenance.

CELL PHONE POLICY - such as talking, texting, and gaming is PROHIBITED:

1. When driving any motorized vehicle on Monsanto property.

2. When walking in “out of office” areas throughout plant and remote sites. (Take the call only when stopped in a safe area.)

3. When working in an area where a hazardous work permit exits; however, in this circumstance, a cell phone may be used for business and/or emergencies only.

4. Employees, contractors and visitors are asked to use good judgment when using their cell phones in manufacturing areas of the Soda Springs plant. Do not allow a cell phone to distract or otherwise cause inattention while performing a task. Keep your mind on your work until you can safely answer a call.

5. The best time to use cell phones for personal business is break time.

BARRICADE TAPE POLICY:

All excavations, holes in floors, areas where work is being performed overhead or temporary work areas where tripping or falling hazards exist will be covered over, roped off, or barricaded off. It is important that the proper barricade tape in conjunction with the proper tag be used when barricading an area. The Soda Springs plant uses two types of barricade tape: yellow & black barricade tape and orange barricade tape.

All barricade tape must be tagged with either a DANGER tag or a CAUTION tag. The DANGER tag or the CAUTION tag must include a description of the hazard, be signed by the person establishing the barricade, be placed on the barricade tape and be spaced frequently enough for easy identification.

1. Orange barricade tape is to be used to barricade an area, which has been determined to be hazardous for entry. Orange barricade tape is considered the same as a lockout. Once the orange barricade tape has been put up, NO ONE IS ALLOWED TO ENTER THAT AREA, not even

the person who applied the barricade tape. A DANGER tag must be attached to the barricade tape as described in #3 of this section. CAUTION tags are NOT to be used with orange barricade tape. Once the hazard has been identified and all safety precautions are taken to correct the hazard, the orange barricade tape can be taken down and replaced with yellow & black barricade tape (if necessary) so that work can proceed.

2. Yellow & black barricade tape is used to identify an area where a hazardous condition may exist. Either CAUTION or DANGER tags, as described in #3, may be used with yellow & black barricade tape.

3. CAUTION tags are used, entry into the barricaded area may occur, after reading the warning on the CAUTION tag. If DANGER tags are used, entry into the barricaded area can only occur after requesting permission from the person who established the barricade. If work proceeds beyond shift change, the CAUTION or DANGER tags must be replaced and signed by the oncoming shift.

4. All barricade tape is to be promptly taken down when the reason for the barricade no longer exists. Barricade tape is to be placed in trash containers when it is removed. Barricade tape is NOT to be left lying on the ground in the work area.

5. All open holes in the floor must be surrounded by barricade tape.

PEDESTRIAN TRAFFIC:

1. Pedestrians must use designated walkways and be aware of the beeping signal from backing equipment. They should also be aware of the limited visibility of heavy equipment operators.

2. Stay a safe distance from a moving piece of equipment until the attention of the operator has been gained.

3. Running up or down stairways or throughout the plant unnecessarily is not allowed.

4. Do not sit on handrails or lean on safety chains.

5. Never enter pot carrier area, nodule stockpile area, or the belt filter press cake/ore screen house area, without proper clearance.

RAILROAD SAFETY:

1. Cross railroad tracks only at regular crossings provided for that purpose. If railroad cars are blocking a road or passageway, do not climb over, between, or under them instead, walk around them.

2. You should allow at least eight feet of clearance between you and the car. This rule is in effect at all times whether or not a switch is being made. Anyone violating this rule will be subject to disciplinary action.

3. No one shall place material, equipment or do work within 8 feet of the center line of any railroad track without first contacting the Material Handling Department and/or Phos Dock personnel to have the tracks Blue Flagged and Locked Out.

BLUE FLAG

1. Whenever anyone is working around a railroad car at the phos dock, railroad shop, coke bunker(s), or anywhere on the plant tracks, a Blue Flag must be in place on the southernmost car on the affected track.

2. Keep derail closed at all times except when switching railroad cars.

3. Chock the wheels on lead car on a downgrade.

4. Any person(s) working in a railroad car area shall use a Blue Flag sign. Ownership and clearing of plant railroad tracks will be as follows:

   RR track ownership:
   Heavy Equipment Group – Track 721, commonly known as track #1.
   Phos dock – Track 722, commonly known as phos loading track #2.
   Reliability group (Railroad Shop) – Track 723, 724, 728.

Note: Work by the (authorized*)* **owning track group** requires a Blue Flag only.

Note*:* Work by the (affected) **non-owning employees** requires the use of some type of control method in addition to the blue flag. For example, a tag lock and tag, or lockbox must be used by both the owning track group and non-owning employees. This will ensure that all involved groups are recognized and that communication is maintained.

5. In the event that work is to be performed on a track by a group other than the owner, contact must first be made with the track owner and lockout tag affixed to the blue flag.

6. Persons needing to work across or within eight feet of the phos loading track shall contact the phos dock Crew Supervisor or backup, and fulfill the following requirements.

a. The phos dock will place a tripod Blue Flag sign at the south end of the phos loading track. The phos dock and persons performing the work on or near the tracks must lock out the car puller if there are any cars to the north of where the work is to be performed.

b. Skates or chocks will be used on the first car on either side of the work area.

c. Non-phos dock person(s) performing the work will affix a lockout tag bearing his/her name to the Blue Flag tripod sign. This is necessary to move rail cars before the work is completed. If a lockbox is being used then a tag must be affixed to the blue flag indicating that a lockbox is being used.

d. Work should be completed as promptly as possible to minimize any impact on loading and switching activities. When work is complete and the tracks clear, notification should be made immediately to the phos dock. The lockout tag must be removed from the tripod sign and the car puller if locked should be unlocked.

e. In the event that work extends beyond the end of the phos dock personnel's shift, a special written permit will be issued by the phos dock to authorize removal of the Blue Flag. This permit will contain a checklist for the person removing the Blue Flag who may not be familiar with the procedure to assure that the track is cleared.

f. No one may remove a Blue Flag from the phosphorus-loading track other than the phos dock or the holder of a written permit.

g. No one shall remove a Blue Flag bearing a lockout tag.

WORK-IN PROGRESS/ PERFORMANCE EVALUATIONS:

- Contractors must participate in work-in-progress audits when asked to by the Monsanto Representative.

WAPITI/ BEHAVIORAL BASED SAFETY PROCESS:

- Contractors will be asked to participate in the BBS Process.
- The BBS process identifies behaviors, which may lead to an injury or property damage.
- BBS is part of the safety culture and is an important part of changing an unsafe act.

HAZARDOUS ENTRY AND GENERAL ENTRY PROCEDURES:

A properly completed and approved hazardous work permit is required prior to performing hazardous jobs, entering vessels or entering designated confined spaces in the plant. The department Crew Supervisor may issue hazardous work permits or a responsible operator, provided the conditions of the hazardous work permit can be met. Otherwise, the job must stop until the permit can be properly completed and approved or a special written clearance is obtained.

1. Hazardous work permits are only valid during the shift in which it was created. If the work proceeds past the end of the shift, a new hazardous work permit must be completed.

2. Hazardous work permits must remain at the job site until completed and then should be returned to the operating crew supervisor.

3. Only Monsanto Employees are authorized to start/stop equipment or open/close valves. When these steps are needed, contact your assigned CMR.

NOTE: Nitrogen and inert gas are used to purge oxygen from most vessels in the plant. Inert gas sometimes contains hazardous amounts of Carbon Monoxide. Breathing pure nitrogen or inert gas can be fatal.

Permits:

There are many different permits in this plant that have been developed to assist in performing safe operations. It is the responsibility of each individual to know when and where a permit is required to perform a job. If in doubt about the need for or use of a permit, ask the CMR who represents you. Listed below are some of the permits that are used at this site. This list is not all-inclusive other permits maybe necessary and your assigned CMR will assist you in determining permits you need to complete your assigned work:

1. Hot Work Permit
2. Work Execution Permit
3. Confine Space Entry Permit
4. Line Entry Permit
5. Lifting Personnel with Crane Permit
6. Elevated Work Permit
7. Excavation Permit

6.0 Contractor/Guest ES&H Site Guidelines

6.3.5 INJURY AND ILLNESS RECORDKEEPING AND REPORTING

The Plant Dispensary is available for initial treatment of injuries, which occur on company property. The plant has a trained 24 Hour Emergency Response Team and EMT's for rescue work. Call 777 for emergency first aid support. Be sure to give your name, the location of the injured, and, if possible, the extent of the injury. Go or send someone to direct the emergency crew to the exact location of the injury.

1. Contractors/visitors are required to immediately report all injuries, no matter how small, to the CMR and Safety Department or on-shift EMT. A First Aid card (blue/Contractors) must be filled out immediately for any first aid or injury. Any transport to the hospital requires a blue first aid card to accompany the injured/ill employee. MD information is required on the back of the card. The first aid cards are located in the Administration Building Dispensary. The CAF (corrective action form) is attached and must also be submitted.

2. Contractors are required to submit a monthly Man-Hour & Illness/Injury Report to the Contractor Safety Specialist.

3. If you are personally exposed to blood/body fluids, immediately wash exposed area with soap/water, isolate the contaminated area, call EMT’s to perform decontamination, and report exposure to the Safety Department.

4. If blood/body fluids are discovered (without personal exposure), isolate the area from other personnel and call Plant EMT’s to perform decontamination procedures.

6.0 Contractor/Guest ES&H Site Guidelines

6.4.4 MONSANTO SECURITY POLICY

1. Contractors and their employees normally will be governed by the terms of their particular contract. In all cases, contractor's personnel are confined to the area in which they are working.

2. Contractors and their employees will use the entrance and exit gate, which has been designated by Monsanto.

3. Service Representatives and Service Company employees who are not under a formal contractual arrangement will be registered in and out. The plant guards will administer the above policy and enforce the necessary rules required to carry it out.

4. Contractor parking facilities will be separate from Monsanto Employee parking.

5. An approved Material Pass will be required for contract employees and visitors when leaving company property with bundles, packages, boxes, materials, equipment, etc. Plant guards may periodically inspect packages, even though the individual may have a Material Pass. Lunch boxes do not require a pass, but must be opened for inspection upon request. Vehicles leaving the plant are also subject to inspection at any time.

6. Each contractor's employees and/or visitors will be required to identify him or herself when entering or leaving company property. This shall include signing the log in the guard station.

7. Only the Plant Manager or their delegate can authorize the use of cameras, camera equipment or cell phone cameras. They will not be permitted within the plant without this authorization. Any photographs or video taken of the facilities will need the same authorization.

6.0 Contractor/Guest ES&H Site Guidelines

6.9.1 SUBSTANCE DETECTION

General Requirements:

In the general requirements for Low-Risk and High-Risk contractors, refer to 5.0 Contractor/Guest ES&H Guidelines, 5.9.1 Substance Detection.

This policy is in effect for all Monsanto Soda Springs locations including the Rock Springs, Wyoming Calciner facility, and all affiliated off-site mining operations and properties (hereinafter referred to as Soda Springs Facilities).

Contractors that do not have a substance abuse policy consistent with these guidelines will not be allowed in a Soda Springs facility without a Monsanto management exception.

All contractors have been classified into one of two categories, High-Risk or Low-Risk. This classification was determined as to the type of work that the contractor performs at Soda Springs Facilities.

Requirements for Both Low-Risk and High-Risk Contractors:

Low-Risk and High-Risk contractors must maintain a copy of an executed **Substance Abuse Policy Statement (Exhibit A)** for each contract employee who works at a Soda Springs Facility; and

Low-Risk and High-Risk contractors must present at the time of site orientation an executed **Substance Abuse Policy Statement (Exhibit A)** for each contract employee undergoing site orientation.

If the Certified Monsanto Representative (CMR) suspects any contract employee is impaired due to alcohol, drugs or other substances, that contract employee will be asked and required to immediately leave the Soda Springs Facility and not return until that contract employee has undergone a substance abuse test verifying that he or she is, in fact, substance free.

Additional Requirements for High-Risk Contractors:

A. Testing Program: All High-Risk contractors must implement one of the two following substance abuse testing programs, although please note that Monsanto prefers that its High-Risk contractors implement the first option:

Option One: Contractor must execute and provide to Monsanto the **Contractor Employer Substance Abuse Verification (Exhibit B)** and must implement a substance-abuse testing program that contains the following elements:

1. Prior to Working at Soda Springs Facilities – Contractor must have a program that documents that each contract employee working at a Soda Springs facility has passed a substance-abuse test prior to that individual starting work at any Soda Springs Facility.

2. Random Testing – Contractor must have a documented program providing evidence that at least 35 percent of its work force who have worked at Soda Springs Facilities has been randomly tested during the prior year. The testing of the 35 percent must occur evenly over an annual time frame. Testing all personnel on a single day does not qualify as random.

Option Two: Contractor must execute and provide to Monsanto the **Contractor Employer Substance Abuse Verification (Exhibit B)**. Within 15 days prior to any contract employee beginning work at the Soda Springs Facilities, and within six months of any previous test, Contractor must test that individual and maintain executed **Contractor Employee Substance Abuse Verification(s)** for that test(s). Contractor will provide to Monsanto a copy of the executed **Contractor Employee Substance Abuse Verification (Exhibit C)** at the time of that individual's site orientation. This testing requirement applies for as long as the contract employee continues to work at the Soda Springs Facilities. If there is a 45-day lapse in any assignment to the Soda Springs Facilities for any particular contract employee, contractor must test that individual within 15 days prior to him or her restarting work at the Soda Springs Facilities.

B. Test Requirements: The substance abuse testing conducted pursuant to the above guidelines must be for the following listed substances (or other such substances as indicated in writing by Monsanto) at levels set forth in the Federal Register of Health and Human Services Guidelines published in the Federal Register on April 11, 1988 [53 F.R. 11970], and any amends thereto.

Amphetamines
Cannabinoids (THC)
Cocaine Metabolite
Opiates
Phencyclidines

All tests shall be performed in a Substance Abuse and Mental Health Services Administration (SAMHSA) certified laboratory.

C. Positive Results: If a contract employee tests positive for substance impairment or contractor suspects a contract employee of substance impairment, contractors shall:

1. Immediately remove the contract employee from the Soda Springs Facilities.
2. Inform the CMR Leader and Purchasing Lead.

A contract employee who has tested positive for substance impairment shall not be allowed to return to the Soda Springs Facilities until the CMR Leader or Purchasing Lead has been provided verification and documentation that the contract employee has successfully completed a recognized substance abuse rehabilitation program.

EXHIBIT A

SUBSTANCE ABUSE POLICY STATMENT

Employee Name: _____

Contractor Name: _____("Company")

In connection with your employment, you may be assigned to work on the premises of Company's client, Monsanto Company ("Monsanto").

The use, possession, sale and distribution of alcohol or controlled, illegal or unauthorized substances, or the presence of an individual testing positive under Company's drug testing programs for such substances for non-medical reasons, are prohibited on any Monsanto work location, including project sites. Illegal drugs include, among others, marijuana, hashish, heroin, crack/cocaine and hallucinogens.

Entry to any Monsanto work location, including project sites, offices and vehicles, is conditional on Monsanto's right to search the entrant's personal effects and vehicle for prohibited drugs and paraphernalia, alcoholic beverages, or possession of unauthorized property or equipment. The Certified Monsanto Representative (CMR) has the right to remove any contract employee from any of the Soda Springs Facilities until a substance abuse test can verify that the contract employee is in fact substance free.

Violation of this policy or refusal to submit to a search or drug testing will be cause for immediate termination of Permission/Authorization to work on Monsanto's premises. I agree to abide by Monsanto's reasonable suspicion.

I HAVE READ AND UNDERSTAND THE ABOVE POLICY.

_____ _____

Employee Signature Date

EXHIBIT B

CONTRACTOR EMPLOYER SUBSTANCE ABUSE VERIFICATION

I hereby certify that our Company substance abuse program meets the standard set forth in the Monsanto Soda Springs site-specific document 6.9.1 as outlined in Option One or Two. I also agree to periodic audits of our substance abuse testing program to provide evidence that the program is in conformance.

Our selected option for the Soda Springs Site High Risk Contractor is:

Option One_____ Option Two _____

Name of Contract Company:_____

Printed Name:_____

Signature:_____

Title:_____

Date:_____

Please fax or return the above information:

Monsanto Company
Attn: Matt Kirby
(208) 547-1253 Office
(208) 547-3763 FAX
sodasprings.contractor-guest@Monsanto.com

EXHIBIT C

CONTRACTOR EMPLOYEE SUBSTANCE ABUSE VERIFICATION

Name of Employee:_____

Last four digits of Social Security Number:_____

Date Sample Taken:_____

Name of Testing Laboratory:_____

Test Utilized:_____

Negative Test Results* Yes_____ No_____*Do Not Send Actual Test Results

I HEREBY CERTIFY THE ABOVE TEST RESULTS TO BE CORRECT TO THE BEST OF MY KNOWLEDGE.

Name of Contract Company:_____

Printed Name:_____

Signature:_____

Title:_____

Date:_____

Please fax or return the above information:

Monsanto Company
Attn: Matt Kirby
(208) 547-1253 Office
(208) 547-3763 FAX
sodasprings.contractor-guest@Monsanto.com

APPENDIX H

PRE-JOB ANALYSIS WORKSHEET

Monsanto – Soda Springs Plant

Pre-Job Risk Analysis

Job #	
Date:	
Work Area:	

Work area inspected and the following hazards corrected:	

Work Team Signatures:				

Job Name/Title:	

Major Job Steps (Each major step is written below in this column)	**Potential Risks** (Run each job step thru the following list, identifying risks by entering the job step number from the prior column)	**Prevention Plan** (All risks identified from the previous column must have a prevention plan noted here, and detailed on the job order)
1.	Lockout/Tagout	
	Atmospheric Monitoring	
2.	Barriers/Guards	
	Housekeeping	
3.	Permitting	
	Head/face/neck protection	
4.	Eye Protection	
	Body Protection	
5.	Hand/Arm Protection	
	Legs/Feet Protection	
6.	Respiratory Protection	
	Fall Protection	
7.	Hearing Protection	
	Pinch Points	
8.	Lifting	
	Ascend/Descend	
9.	Line of Fire	
	Working Surface	
10.	Ergonomics	
	Work Pace	
11.	Tool Selection/Condition	
	Vehicle Inspection	
12.	Vehicle Operation	
	Assistance	
13.	Communication	
	Environmental Risks	
14.	Chemical Safety/MSDS	
	Electrical Clearance	

APPENDIX I

DEGERSTROM ORE HAULROAD TRAVEL REQUIREMENTS

ORE HAULROAD TRAVEL REQUIREMENTS

VISITORS

Haulroad Safety

There are two different types of haulroads used by Degerstrom Ventures. The system of roads used for the hauling of materials between the pit and dumps or pit and ore stockpile. This system is referred to as simply haulroads. The other type of haulroad is the Ore Haulroad which is a 19 mile paved haulroad used for hauling ore from the tipple to the ore stacker near the Monsanto Plant Site.

Both of these haulroads share some safety rules but also have some safety rules that are specific to that particular road. It is important to understand the differences between these two road systems and obey the traffic rules that assure personal safety and help maintain safe haulroad traffic.

Sections of the haulroads in the mine are shared through a co-operative agreement with Agrium. These present additional hazards and restrictions that are also addressed in this section.

Right hand traffic is observed on all roads to, from and at the mine; unless otherwise posted. Large pieces of mobile equipment have very limited visibility, large blind spots, and often have extremely long stopping distances: so be aware of this and give mobile equipment plenty of room when approaching them from any direction. It is important to be cautious, pay attention to what is going on around you at all times and particularly avoid equipment in blind spots. Always stay in areas where you can see the operators of mobile equipment – ***"See & Be Seen"*** when traveling on all roads in and around the mine. *If you cannot see the operator, the operator cannot see you.*

General Haulroad Safety Rules

1) Obey all posted speed limits.

2) Obey all posted warning, advisory, and traffic signs.

3) Always wear seatbelts.

4) Watch for wildlife crossing.

5) Always drive with headlights on.

6) Adjust your speed for weather and road conditions.

7) Loaded haul trucks have the right of way.

8) Always assume that unless you can see the operator they cannot see you.

9) Give plenty of clearance to all mobile equipment. Remember: large equipment has very limited visibility and large blind spots.

10) When following a haul truck stay far enough back that you can see the driver's side mirror, that way the operator can see you.

11) Never approach mobile equipment from the rear. Avoid this large blind spot and approach from the driver's side. Never park directly behind any mobile equipment.

12) Be alert for material that may fall off the beds of loaded trucks.

13) Never drive past, over or around any type of road barricade.

14) Pay attention to horns, alarms and signals on all mobile equipment.

Ore Haul Road

The Ore Haul Road runs from the tipple at the mine to the unloading facility at the plant. This paved road was designed for ore hauling, but the upper portion above the Blackfoot River Road/Ballard intersection serves as the access road to the mine. Traffic between the Ballard intersection and the plant is restricted to ore trucks, service equipment, and small vehicles equipped with the proper two-way radios. All other vehicles must make prior arrangements and/or be escorted.

The Ore Haulroad crosses the Blackfoot River Road at Ballard. Traffic at this intersection is controlled by traffic lights. These lights regulate traffic on the Blackfoot River Road to allow the ore trucks to proceed through the intersection safely, without stopping. This is very important to the operation of the ore trucks, especially when the trucks are loaded with 210 tons of ore. The traffic lights are triggered by trips set in the asphalt prior to the intersection and cycle through a preset time, which allows the ore trucks to pass through the intersection before the lights change. It is important to avoid driving over the trips and triggering the traffic light cycles, tripping them may effect the cycle time and interfere with an approaching ore truck. The trips are clearly marked on the asphalt and with signs. The ore trucks have the right of way, so avoid driving over the trips, stay clear of approaching traffic, and any ore trucks that may be following behind you.

Ore trucks may be parked on the ore haul road during lunch break and if they are experiencing mechanical problems. When you are approaching these parked trucks slow down and use caution, the operator or mechanics may step out from under or around these parked trucks.

Ore Haulroad Safety Rules

1) Obey all posted speed limits.

2) Obey all posted warning, advisory, and traffic signs.

3) Always wear seatbelts.

4) Always drive with your headlights on.

5) Watch for wildlife crossing.

6) Adjust your speed for the weather and road conditions.

7) Ore trucks have the right of way.

8) Stay as far to the right as possible when traveling the Ore Haulroad, especially when meeting ore trucks.

9) Use extreme caution when passing an ore haul truck. Never pass on corners or hills where visibility is limited.

10) Avoid driving over the traffic light trip at Ballard and Conda crossings.

11) Never park on the paved portion of the haulroad. Always park in a spot that is clear from traffic. In case of a mechanical problem, park as far to the right as possible and turn on emergency flashers.

12) Slow down and use caution when approaching and passing ore trucks that are stopped on the ore haulroad. Operators and mechanic may be walking around these stopped trucks.

Haulroads at the Mine

There are numerous roads throughout the mine itself. These roads are used for a variety of purposes such as, to accessing various areas of the mine, drilling & blasting, and surveying. The haulroads are designed for specifically for the hauling of mine ore and waste materials.

The traffic patterns on these roads change continually during each shift, as needed. The basic rule here is to stop before entering these roads, look at the flow of the traffic and then follow. This has been posted at main entrances to haulroads with signs that read – "STOP LOOK and FOLLOW." Remember it is very important to ***"See & Be Seen."***

During drilling, loading and blasting some roads may be barricaded with signs, traffic cones or other means to prevent access to the area. This is done to prevent access to a blasting area before, during and after a shot. Never enter these barricaded areas.

Mine Haulroad Safety Rules

1) Obey all posted speed limits.

2) Obey all posted warning, advisory, and traffic signs.

3) Always wear seatbelts.

4) Drive with your headlights on.

5) Adjust your speed for weather conditions.

6) Loaded haul trucks have the right of way.

7) Never enter areas that have been barricaded.

8) **Do Not Pass** any mobile equipment or vehicles unless you have received clearance (by radio/hand signals) from the operator of that piece of equipment.

9) Be alert for material that may fall from the bed of loaded haul trucks. Keep plenty of space between you and a haul truck when following up any grades.

10) Give plenty of clearance to all mobile equipment. These large pieces of equipment have very limited visibility and large blind spots.

11) When following a haul truck, stay far enough back that you can see the driver's side mirror, that way the operator can see you.

12) Never enter marked or barricaded blasting areas.

13) Never park directly behind any mobile equipment. Always park on the driver's side. If you cannot see the operator, the operator cannot see you.

14) Pay attention to horns, alarms, and signals from mobile equipment.

15) Never park on the haulroad. Always park in a spot that is clear from traffic. In case of a mechanical problem, park as far to the right as possible and turn on emergency flashers.

GENERAL REQUIREMENTS:

- All visitors planning to drive on any haul roads shall successfully complete a specific hazard awareness training session presented by Degerstrom Ventures.
- All visitors on any haul road shall obtain Degerstrom Ventures permission for access.
- All visitors must check in and out at the Degerstrom Ventures Office before and after each visit.
- Vehicles using haul roads shall not trip the traffic lights at the Conda or Ballard Crossings.
- Posted speed limits & traffic requirements shall be followed when using the haul roads.
- Seatbelts shall be worn at all times when using the haul roads.
- Vehicle headlights shall be used when traveling the haul roads.
- Ore haul trucks are considerably larger than regular semi trucks. It requires much longer distances to stop them and they are much less maneuverable.
- Never pass a haul truck traveling on the haul road.
- When approaching a haul truck stopped along the haul road – SLOW DOWN & SOUND HORN before passing.
- Be alert for wildlife and livestock that may be on, along or crossing the roadway.
- Leave all gates along the haulroads the way that you found them.

SPECIFIC REQUIREMENTS:

1. From the stacker dump station to the gravel pit, visitors can use the haul road if they have completed the general requirements.
2. From the gravel pit to the corrals by the Fish Pond, visitors must complete the general requirements and have an approved Monsanto or Degerstrom Ventures' escort at all times.
3. From the corrals by the Fish Pond to the Ballard light, visitors can use the haul road if they have completed the general requirements.
4. From the Ballard lights to the Degerstrom Ventures Office, visitors with business to complete at the mine can use the haul road by following the requirements of the road signs.
5. From the Degerstrom Ventures Office to the tipple and mine, visitors must have a Monsanto or Degerstrom Ventures' escort or complete the general requirements plus obtain Degerstrom Ventures permission.

Degerstrom Ventures haul trucks, equipment and other vehicles shall always have the right-of-way and all vehicles shall yield to them. Visitors shall travel at their own risk. Degerstrom Ventures shall not be held responsible for their safety.

USE OF THE HAUL ROAD IS A <u>PRIVLAGE</u> GRANTED BY DEGERSTROM VENTURES. ANY VIOLATIONS OF THIS POLICY WILL RESULT IN REMOVAL FROM THE PROPERTY

RELEASE FORM (Individual)

In consideration of the permission given to me by DEGERSTROM VENTURES and/or MONSANTO/P4 PRODUCTION, L.L.C. to enter upon land owned or leased by DEGERSTROM VENTURES for the sole and exclusive purpose of _____, I, the undersigned, do hereby:

(i) Voluntarily assume all risk, whether known or unknown, of accident, loss and damage to myself and others and property resulting or arising from or in any way connected with my presence on said land;

(ii) Release and discharge DEGERSTROM VENTURES and MONSANTO/P4 PRODUCTION, L.L.C., and its successors and assignees, and its and their respective directors, officers, employees, and agents, from any and all claims, obligations, liabilities, losses, damages, costs, or expenses of any kind, (including, without limitation, attorney's fees), whether known or unknown, sustained as a result of or arising from or in any way connected with my presence on said land; and

(iii) Agree to protect, indemnify, and save harmless DEGERSTROM VENTURES and MONSANTO.P4 PRODUCTION, L.L.C., their successors and assignees, and its and their respective directors, officers, employees, and agents, from any and all claims, demands, causes of action, losses, damages, obligations, liabilities, penalties, costs, and expenses of any kind (including, without limitation, amounts paid in settlement and attorney's fees and expenses), whether known or unknown, suffered by, imposed upon, incurred by, asserted against, or arising in any way against, DEGERSTROM VENTURES and MONSANTO/P4 PRODUCTION, L.L.C. or any of their successors or assignees, or the directors, officers, employees, or agents of any of them in connection with my presence on said land regardless of whether caused by the negligence of DEGERSTROM VENTURES and MONSANTO/P4 PRODUCTION,L.L.C. or their directors, officers, employees, or agents.

IN WITNESS WHEREOF, I hereunto set my hand this _____ day of _____, 200_.

_____

Address: _____

APPENDIX J

OSHA JOB SAFETY AND HEALTH PROTECTION POSTER

JOB SAFETY & HEALTH PROTECTION

The Occupational Safety and Health Act of 1970 provides job safety and health protection for workers by promoting safe and healthful working conditions throughout the Nation. Provisions of the Act include the following:

Employers

All employers must furnish to employees employment and a place of employment free from recognized hazards that are causing or are likely to cause death or serious harm to employees. Employers must comply with occupational safety and health standards issued under the Act.

Employees

Employees must comply with all occupational safety and health standards, rules, regulations and orders issued under the Act that apply to their own actions and conduct on the job.

The Occupational Safety and Health Administration (OSHA) of the U.S. Department of Labor has the primary responsibility for administering the Act. OSHA issues occupational safety and health standards, and its Compliance Safety and Health Officers conduct jobsite inspections to help ensure compliance with the Act.

Inspection

The Act requires that a representative of the employer and a representative authorized by the employees be given an opportunity to accompany the OSHA inspector for the purpose of aiding the inspection.

Where there is no authorized employee representative, the OSHA Compliance Officer must consult with a reasonable number of employees concerning safety and health conditions in the workplace.

Complaint

Employees or their representatives have the right to file a complaint with the nearest OSHA office requesting an inspection if they believe unsafe or unhealthful conditions exist in their workplace. OSHA will withhold, on request, names of employees complaining.

The Act provides that employees may not be discharged or discriminated against in any way for filing safety and health complaints or for otherwise exercising their rights under the Act.

Employees who believe they have been discriminated against may file a complaint with their nearest OSHA office within 30 days of the alleged discriminatory action.

Citation

If upon inspection OSHA believes an employer has violated the Act, a citation alleging such violations will be issued to the employer. Each citation will specify a time period within which the alleged violation must be corrected.

The OSHA citation must be prominently displayed at or near the place of alleged violation for three days, or until it is corrected, whichever is later, to warn employees of dangers that may exist there.

Proposed Penalty

The Act provides for mandatory civil penalties against employers of up to $7,000 for each serious violation and for optional penalties of up to $7,000 for each nonserious violation. Penalties of up to $7,000 per day may be proposed for failure to correct violations within the proposed time period and for each day the violation continues beyond the prescribed abatement date. Also, any employer who willfully or repeatedly violates the Act may be assessed penalties of up to $70,000 for each such violation. A minimum penalty of $5,000 may be imposed for each willful violation. A violation of posting requirements can bring a penalty of up to $7,000.

There are also provisions for criminal penalties. Any willful violation resulting in the death of any employee, upon conviction, is punishable by a fine of up to $250,000 (or $500,000 if the employer is a corporation), or by imprisonment for up to six months, or both. A second conviction of an employer doubles the possible term of imprisonment. Falsifying records, reports, or applications is punishable by a fine of $10,000 or up to six months in jail or both.

Voluntary Activity

While providing penalties for violations, the Act also encourages efforts by labor and management, before an OSHA inspection, to reduce workplace hazards voluntarily and to develop and improve safety and health programs in all workplaces and industries. OSHA's Voluntary Protection Programs recognize outstanding efforts of this nature.

OSHA has published Safety and Health Program Management Guidelines to assist employers in establishing or perfecting programs to prevent or control employee exposure to workplace hazards. There are many public and private organizations that can provide information and assistance in this effort, if requested. Also, your local OSHA office can provide considerable help and advice on solving safety and health problems or can refer you to other sources for help such as training.

Consultation

Free assistance in identifying and correcting hazards and in improving safety and health management is available to employers, without citation or penalty, through OSHA-supported programs in each State. These programs are usually administered by the State Labor or Health department or a State university.

Posting Instructions

Employers in States operating OSHA approved State Plans should obtain and post the State's equivalent poster.

Under provisions of Title 29, Code of Federal Regulations, Part 1903.2(a)(1) employers must post this notice (or facsimile) in a conspicuous place where notices to employees are customarily posted.

More Information

Additional information and copies of the Act, OSHA safety and health standards, and other applicable regulations may be obtained from your employer or from the nearest OSHA Regional Office in the following locations:

Atlanta, GA	(404) 562-2300
Boston, MA	(617) 565-9860
Chicago, IL	(312) 353-2220
Dallas, TX	(214) 767-4731
Denver, CO	(303) 844-1600
Kansas City, MO	(816) 426-5861
New York, NY	(212) 337-2378
Philadelphia, PA	(215) 596-1201
San Francisco, CA	(415) 975-4310
Seattle, WA	(206) 553-5930

Alexis M. Herman, Secretary of Labor

U.S. Department of Labor
Occupational Safety and Health Administration

Washington, DC
1997 (Reprinted)
OSHA 2203

This information will be made available to sensory impaired individuals upon request.
Voice phone: (202) 219-8615. TDD message referral phone: 1-800-326-2577

APPENDIX K

OCCUPATIONAL INCIDENT REPORT FORMS AND PROCEDURES

402 OCCUPATIONAL INJURY/ILLNESS REPORTING AND INVESTIGATION

I. PURPOSE

To set forth the essential components and responsibilities for implementation of the MWH accident investigation procedure. Accidents are investigated to identify unsafe conditions and acts which contribute to injury, illness, and/or property damage so that solutions for accident prevention may be developed.

II. ATTACHMENTS

Incident Reporting Process flowchart
MWH Occupational Incident Report Form (OIR)
Vehicle Accident Report Form (VAR)

III. DISCUSSION

Effective accident investigations lead to procedures which can reduce or eliminate occupational injuries and illnesses. It is important for MWH management and employees to understand and comply with the accident investigation procedures.

IV. DEFINITIONS

Accident/Incident: is an occurrence in a sequence of events that usually produces unintended injury or illness, or death, and/or property damage.

First Aid: is any one-time treatment and any follow up visit for the purpose of observation of minor scratches, cuts, burns, splinters, and so forth, which do not ordinarily require medical care, but may be administered by a physician.

Near-Miss Situations: are accidents without injury, illness or property damage, but have the potential for serious harm.

V. PROCEDURE

A. GENERAL

Every accident/incident, regardless of whether it results in injury, property damage, or a near miss, should be investigated to determine the actual cause and to take proper action to prevent recurrence. The attached flow chart presents a schematic representation of the incident reporting process.

It is the responsibility of the Business Unit Manager to assure that a thorough investigation into the cause of each employee injury occurs immediately after the event, and to initiate corrective action to prevent a similar recurrence.

The Business Unit Manager must ensure that accidents/incidents are reported to the company health and safety manager to comply with the Occupational Safety and Health Administration (OSHA) recordkeeping/reporting requirements for

occupational injuries and illnesses (see MWH Safety and Health Policies No. 403).

It should be noted that incidents resulting in a fatality or the hospitalization of 4 or more people must be reported to the local OSHA office within 8 hours of the incident. It is preferable for the company health and safety manager to place the report with OSHA, however, if the health and safety manager is not available, the most senior MWH person at the scene of the incident shall make the report. Contact the local telephone information service to obtain the OSHA phone number.

B. ACCIDENT/INCIDENT INVESTIGATIONS

Once an accident occurs (whether it involves personal injury or illness, property damage or is a near miss), whether the victim is a MWH employee, contractor, or visitor, the below listed procedures are to be carried out.

1. Attend to the victim's medical needs.

2. Secure the accident/incident area.

3. Complete the MWH Occupational Incident Report Form.

4. Assemble the personnel and tools needed to perform an accident investigation.

5. Call the Health and Safety Coordinator or Health and Safety Manager **immediately** after the scene is secure, to report the accident and to initiate workers compensation coverage, if needed.

6. Begin the accident investigation.

C. MWH OCCUPATIONAL INCIDENT REPORT FORM

The MWH Occupational Incident Report Form (OIR), Attachment A, shall be completed by the designated MWH employee representative after an employee injury occurs. Each section of the report should be addressed and completed or marked “na” for not applicable. The degree of detail in the report and any additional data should be commensurate with the degree of the incident. That is, the report should be simple and concise for a twisted ankle resulting from someone tripping out of a van. The report should be detailed with additional background for a near miss that resulted from a pressure grout packer being expelled from a well which could have caused a fatality.

If the incident was involving a vehicle, private, company owned/leased, or rented, or other, the attached Vehicle Accident Report Form (VAR) should be completed.

Note: Forms that provide essentially the same information may be used in lieu of the MWH form. Regardless of which form is used, the distribution stated on the MWH form must be used.

The OIR and VAR shall be promptly submitted to the Health & Safety Coordinator and to the Company Health & Safety Manager.

D. ACCIDENT INVESTIGATION REPORT FORMAT

The various sections of the OIR are intended to clearly identify the incident, describe its cause(s), and insure that information is gathered that can be used to prevent recurrence, either for this particular instance, or similar situations. While the OIIIR is basically self-explanatory, it is important that the persons completing and reviewing the form observe the following:

1. Do not speculate as to the facts, nor make unsubstantiated accusations. We all, however, are free to make reasonable assumptions based on a good faith understanding of the facts at hand. Remember that this form becomes an official, MWH legal document.

2. Handle any samples or physical evidence with care, insuring against even inadvertent tampering.

3. Keep all handwritten notes used in gathering the facts of the case in the office master file, along with the completed OIR. Raw data and notes may be helpful in a possible future evaluation.

E. RECORDKEEPING

Insure prompt distribution of complete OIR, i.e. within 24 hours, to:

1. Company Health & Safety Manager
2. The Local Health and Safety Coordinator
3. The Business Unit Manager or Program Director
4. The Direct Supervisor of the affected employee or project

VI. REFERENCES

A. Title 29, Code of Federal Regulations, Part 1904

B. Various States' Labor, Health and Safety, and Workers' Compensation Codes

MWH

BUILDING A BETTER WORLD

Incident Reporting Process
Work-Related Injury / Illness or Vehicle Accident

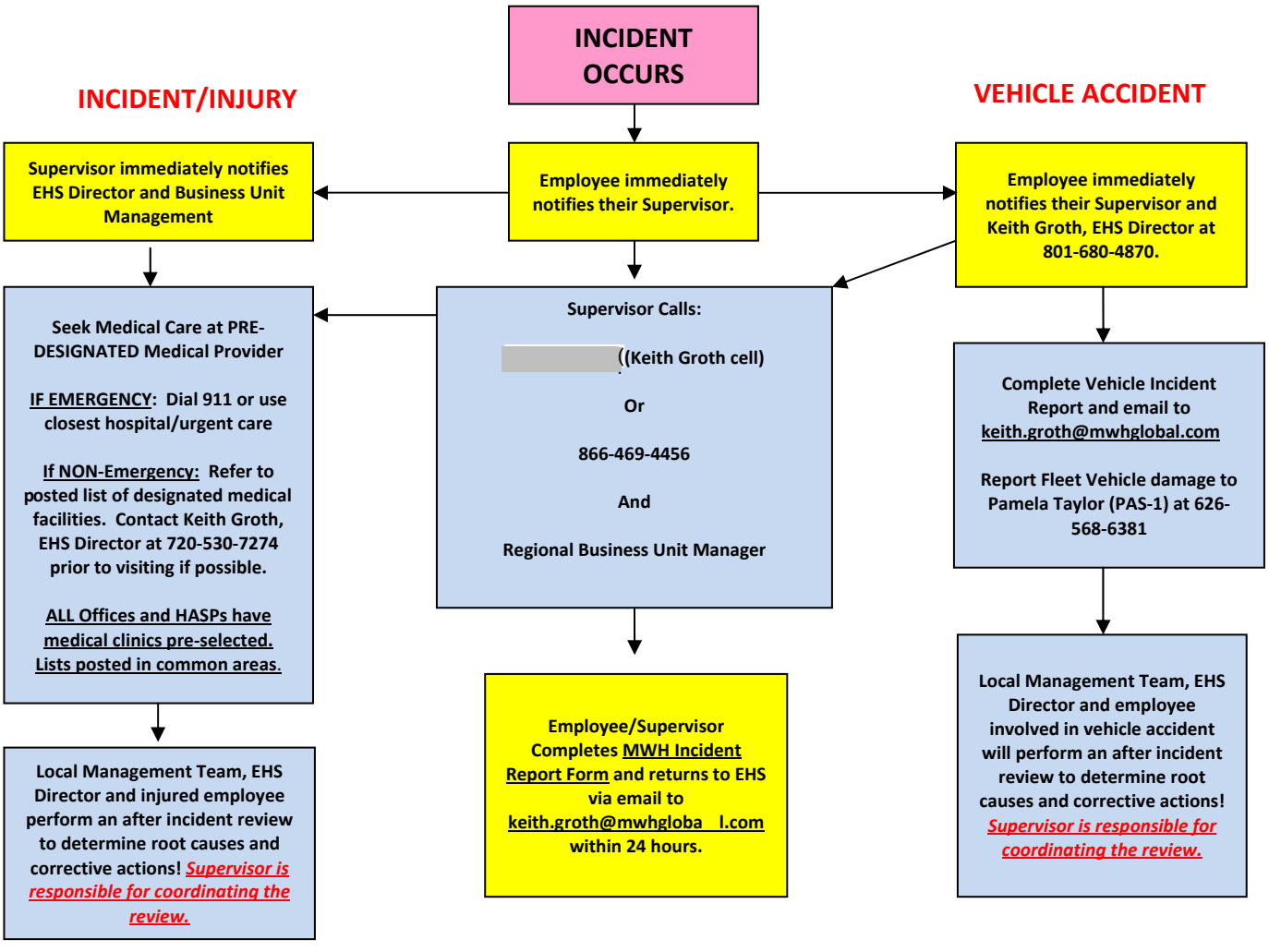
1. Employee(s) should seek necessary medical attention as appropriate:
   a. On-site first aid
   b. Local clinic or Emergency room – e.g., identified in safety plan
   c. Via 911 – transport by ambulance if there is any uncertainty about severity of injury

   If work related, inform medical personnel this is a work-related incident. ***If the medical provider asks for a claim number, advise them that*** **Travelers Insurance is your workers' compensation insurance carrier and your policy number is 5643B095.** Any concerns related to ensuring paperwork/billing is managed can be resolved by contacting Carol Brammell, Travelers Claim Account Executive at 602-861-8737 or 602-803-8597. cbrammel@travelers.com

2. MWH Employee(s) should immediately notify their supervisor. The MWHA employee or supervisor should then contact Keith Grogh, EHS Director at 801-680-4870 (cell) and their respective Business Unit Manager. Additionally, employees can utilize (866-469-4456) to report injuries or emergencies.

3. The MWH Occupational Incident and/or Vehicle Accident Report must be completed within 24 hours, preferably ASAP. Submit forms via email to Keith Groth at ***keith.groth@mwhglobal.com***

4. Ensure that any client or state-specific forms and notifications are complete.



MWH
BUILDING A BETTER WORLD

Occupational Incident Report Form

EMPLOYEE INFORMATION (Electronically, double click on the box, click "checked")

Employee Name

OFFICE ADDRESS	OFFICE PHONE	HOME ADDRESS	HOME PHONE

MWH EMPLOYEE (If not MWH employee, provide company name, address, phone) ☐ YES ☐ NO	BUSINESS UNIT

JOB TITLE	HIRE DATE	BIRTHDATE	SOCIAL SECURITY NO.	GENDER ☐ M ☐ F

SUBCONTRACTOR INVOLVED (If yes, provide company, address & phone)
☐ YES ☐ NO

INCIDENT INFORMATION

LOCATION (name of facility or location identification, addres, specific site)

DATE and TIME INCIDENT OCCURRED	EMPLOYEE'S WORK SCHEDULE ON DATE OF INCIDENT

TYPE OF INCIDENT:
☐ INJURY ☐ ILLNESS
☐ PROPERTY DAMAGE ☐ NEAR MISS
☐ OTHER INCIDENT

What was the employee doing just before the incident occurred? Describe the activity, as well as the tools, equipment, or material the employee was using. Be specific. Examples: *climbing a ladder while carrying roofing materials; daily computer key entry*

What happened? Tell us how the injury occurred. Examples: *when ladder slipped on wet floor, worker fell 4 feet; worker developed soreness in wrist over time.*

What was the injury or illness? Tell what part of the body was affected and how it was affected. Be more specific than "hurt", "pain" or "sore". Examples: *strained back, carpal tunnel syndrome*

What object or substance directly harmed the employee? Example: *concrete floor; radial arm saw*

INJURY / ILNESS TREATMENT INFORMATION

INJURY/ILLNESS TREATMENT:
☐ NOT APPLICABLE ☐ ON-SITE FIRST AID
☐ OFFERED & REFUSED
☐ OFF-SITE (If checked, list name of physician or other health care professional/facility, address & phone

Was employee treated in an emergency room? ☐ YES ☐ NO
Was employee hospitalized as an in-patient? ☐ YES ☐ NO
Was this a fatality? ☐ YES ☐ NO

WITNESS STATEMENTS ATTACHED: ☐ YES ☐ NO
Witness Names:

MWH
BUILDING A BETTER WORLD

Occupational Incident Report Form

ANALYSIS OF CAUSES AND CORRECTIVE ACTIONS

WHAT CONDITIONS OR ACTIONS CAUSED OR CONTRIBUTED TO THE INCIDENT?

CORRECTIVE ACTIONS TAKEN OR RECOMMENDED (Describe):

DISTRIBUTION
(Supervisor and Business Unit Manager Print & sign name; Original is forwarded to EHS SLC-1 Keith Groth or Brenda Zimmerman

What happened? Tell us how the injury occurred. Examples: *when ladders lipped on wet floor, worker fell 4 feet; worker developed soreness in wrist over time.*

1. EMPLOYEE or INDIVIDUAL REPORTED BY:	2. EMPLOYEE'S DIRECT SUPERVISOR:
3. BUSINESS UNIT MANAGER:	4. HEALTH AND SAFETY DIRECTOR: Keith Groth SLC-1 Telephone: 801-617-3302 Cell & Emergency: 801-680-4870 Facsimile: 801-617-4200 Email: keith.groth@mwhglobal.com OSHA Log Case Number:_____

Note: *Attach additional sheets as necessary to document incident.*

MWH
BUILDING A BETTER WORLD

VEHICLE ACCIDENT REPORT FORM

MWH DRIVER INFORMATION

Your Name: ☐ Leased (US Fleet Leasing Vehicle Number)

Office Address ☐ Rented ☐ Company Owned ☐ Other

City Year/Make/Model

State Zip Age

Work Phone Business Unit

Is Your Vehicle Drivable?

List Parts Damaged

MWH DRIVER'S INFORMATION

Driver's Name

Home Address

City

State **Zip**

Home Phone

OTHER VEHICLE OWNER'S INFORMATION

Owner's Name

Street

City

State Zip

License Plate Number

WITNESSES

Name

Home Address

City

State **Zip**

Home Phone

OTHER VEHICLE OWNER'S INSURANCE

Insurance Company

Address

City

State Zip

Policy Number Phone

INJURIES

Were you injured? ☐ Yes ☐ No

Any Passengers Injured? ☐ Yes ☐ No

Was the Other Party Injured? ☐ Yes ☐ No

Any Of Their Passengers Injured? ☐ Yes ☐ No

TYPE OF ACCIDENT

Collision With:

☐ Other Vehicle ☐ Animal

☐ Fixed Object ☐ Pedestrian

☐ Ran off Road ☐ Hit and Run

☐ Other (Non Hit and Run)

Manner of Collision:

☐ Backing ☐ Head-on

☐ Side Swipe ☐ Angle

☐ Read-End (We Hit)

MWH
BUILDING A BETTER WORLD

VEHICLE ACCIDENT REPORT FORM

STATEMENT OF FACTS

Accident Date _____ Purpose of Trip (circle one) Business Pleasure

Accident Location Street _____ Urban or Rural Area _____

City _____ State _____ Zip _____

Was a Police Report Made? ☐ At Scene ☐ At Station ☐ None Report Number _____

Road Conditions ☐ Dry ☐ Wet ☐ Mud ☐ Snow ☐ Ice ☐ Cinders ☐ Other

SPEED OF VEHICLES

Your Vehicle	Other Vehicle
Before Accident	Before Accident
At Impact	At Impact

CITATIONS

Was a Citation Issued? _____

To Whom? _____

Nature of Charge _____

DRIVER OF VEHICLE (Other Than Assigned Under Lease)

Driver's Name _____

Street _____

City _____ State _____ Zip _____

Relationship to Employee _____

MAIL OR FAX THIS REPORT TO

Keith Groth, EH&S Director – SLC-1
Phone: (801) 617-3302
Cell: [redacted]
Fax: (801) 617-4200
Email: SM-AccidentReporting@mwhglobal.com

Brenda Zimmerman
Phone: (303) 533-1967
Fax: (303) 410-4150

DESCRIBE WHAT HAPPENED (Be Specific. Attach Additional Sheets if Necessary)

_____

_____

_____

Signature _____ Date _____

ACCIDENT DIAGRAM

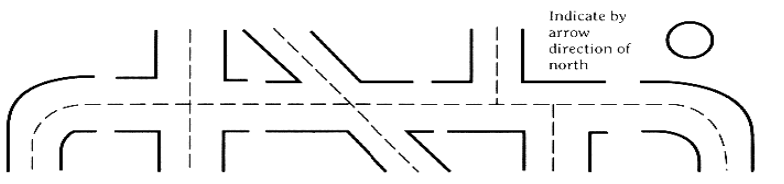


Diagram
Please draw what happened. Include signals and number the vehicles. #1 for your vehicle, #2 for the other vehicle, etc.

Directions
Use solid line to show path of vehicle before accident, dotted line after accident.