



## **Operation, Maintenance, and Monitoring Plan**

**Folkertsma Refuse Site Walker, Kent County, Michigan**

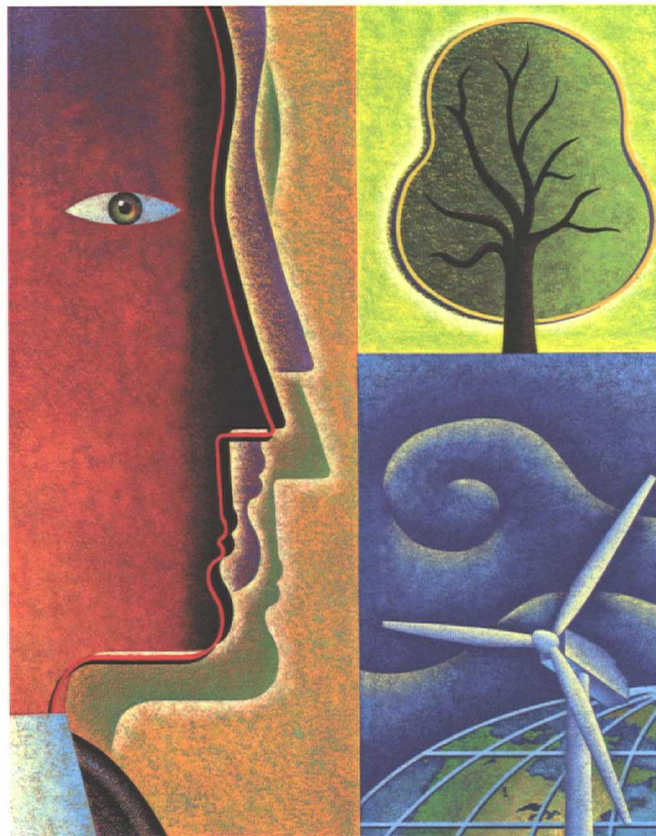
Original: September 1993 (by Warzyn, Inc.)

Revision 1: September 1999

Revision 2: June 2000

Revision 3: April 2001

Revision 4: April 2009



US EPA RECORDS CENTER REGION 5



489935



**CLOSED SITE MANAGEMENT GROUP - MIDEAST**

Autumn Hills RDF  
700 56th Avenue  
Zeeland, MI 49464-9328  
(616) 688-5777 ext. 17  
(616) 688-5781 Fax

July 1, 2009

Karen Cibulskis  
Project Manager  
USEPA Region 5 (SR6J)  
77 West Jackson  
Chicago, Illinois 60604

Subject: Folkertsma Refuse Site, Walker, Michigan  
Revised Operation, Maintenance, and Monitoring Plan

Dear Ms. Cibulskis;

Please find enclosed a copy of the revised Operation, Maintenance, and Monitoring Plan for the Folkertsma Refuse Site. The revisions to the document are based upon the recommendations contained within the USEPA Five-Year Review dated November 11, 2008 and the USEPA correspondence dated March 2, 2009.

A copy of this revised document will be sent to the Michigan Department of Environmental Quality as well.

If you have any questions or comments regarding the revised document, please do not hesitate to contact me at 616.688.5777 ext 17 or 616.822.3031.

Respectfully,

A handwritten signature in black ink that reads "Phillip M. Mazor". The signature is written in a cursive style.

Phillip M. Mazor  
Technical Committee Chairman  
Folkertsma Refuse Site Settling Defendants

Attachment

Cc: James C. Forney (w/o attachment)

***From everyday collection to environmental protection, Think Green® Think Waste Management.***



## **Operation, Maintenance, and Monitoring Plan**

*Folkertsma Refuse Site Walker, Kent County, Michigan*

**Original: September 1993 (by Warzyn, Inc.)**  
**Revision 1: September 1999**  
**Revision 2: June 2000**  
**Revision 3: April 2001**  
**Revision 4: April 2009**

*Prepared For*  
*Folkertsma Refuse Site Settling Defendants*

# Table of Contents

1.	Introduction .....	1-1
1.1	General .....	1-1
1.2	Background .....	1-2
2.	Operation .....	2-1
3.	Maintenance.....	3-1
3.1	Overview .....	3-1
3.2	Site Inspections .....	3-1
3.3	Restoration of the Landfill Cover .....	3-2
3.4	Vegetation Establishment and Cultivation.....	3-2
3.5	Biannual Mowing of the Landfill Vegetation .....	3-2
3.6	Sediment Removal in the Drainage Swales .....	3-2
3.7	Restoration of Damaged Sections of Drainage Swales.....	3-3
3.8	Restoration/Replacement of Fencing, Monitoring Wells, and Gas Probes.....	3-3
4.	Environmental Monitoring.....	4-1
4.1	Overview .....	4-1
4.2	Groundwater Monitoring Program .....	4-1
4.2.1	General Sampling .....	4-2
4.2.2	Water Level Measurements.....	4-2
4.2.3	Well Purging .....	4-3
4.2.4	Sample Collection.....	4-3
4.2.5	Sample Identification .....	4-4
4.2.6	Field Records.....	4-4
4.3	Surface Water Monitoring Program.....	4-5
4.4	Landfill Gas Monitoring Program.....	4-5
4.4.1	Monitoring Locations and Frequency .....	4-5
4.4.2	Monitoring Procedures.....	4-5
4.5	Data Analysis – Groundwater Monitoring .....	4-6
4.6	Data Analysis – Surface Water Monitoring .....	4-7
4.7	Data Analysis and Contingency Action – Landfill Gas Monitoring .....	4-7

5.	Alternative Operation, Maintenance, and Monitoring.....	5-1
6.	Safety During Maintenance.....	6-1
7.	Required Equipment.....	7-1
8.	Record Keeping.....	8-1
9.	Emergency Contacts.....	9-1

**List of Tables**

Table 3-1	Semiannual Site Inspection Record
Table 3-2	History of Site Inspection and Monitoring Activities
Table 4-1	<i>(Revised March 2009)</i> Environmental Monitoring Program Analytical Parameters, Methods, and Target Detection Limits for Groundwater and Surface Water
Table 4-2	Environmental Monitoring Program Sample Quantities, Containers, Preservatives, and Packaging Requirements
Table 4-3	Groundwater Stabilization Criteria
Table 4-4	Summary of the Sampling and Analysis Program
Table 4-5	<i>(Revised March 2009)</i> GSI Criteria for the Folkertsma Refuse Site

**List of Figures**

Figure 1	Site Locator Map
Figure 2	Environmental Monitoring Locations

**List of Appendices**

Appendix A	Amendments to the QAPP and SAP
Appendix B	Laboratory QA Manual and SOPs
Appendix C	Calculation of Generic GSI Criteria for Metals

# Section 1

## Introduction

---

### 1.1 General

This Operation, Maintenance, and Monitoring (OM&M) Plan has been revised in response to the USEPA Five-Year Review dated March 2, 2009 for the Folkertsma Refuse Site located in the city of Walker, Kent County, Michigan (Figure 1). This OM&M Plan is intended to serve as a guidance document to facilitate long-term implementation of the remedial action. Components of the Folkertsma remedial action requiring OM&M include the following:

- The landfill cover consisting of (from top to bottom) the following components:
  - Vegetation layer to aid in prevention of soil erosion
  - A minimum 0.5-foot thick topsoil layer that will sustain plant growth, and provide protection to the remainder of the cap
  - A minimum 1.0-foot thick rooting zone layer providing protection from frost penetration and preventing roots from the vegetative layer from damaging the low-permeability portion of the cap
  - A minimum 0.5-foot thick capillary break layer that will reduce the potential for head build-up on the barrier layer below by promoting drainage
  - A low-permeability, compacted clay barrier layer to minimize infiltration to the underlying waste. This layer is a minimum of 2.0 feet thick and has a maximum saturated hydraulic conductivity of  $1 \times 10^{-7}$  cm/s
- A 6-foot-high chain link fence with barbed wire surrounding the landfill
- Monitoring wells and gas probes associated with the Environmental Monitoring Program
- A groundwater drain within the waste to control groundwater migration
- Surface water drainage swales to handle off-site and on-site runoff

Information regarding the implementation of the Environmental Monitoring Program can be found in the Quality Assurance Project Plan for the Environmental Monitoring Program (Warzyn, September 1993), as amended by Appendix A of this OM&M Plan.

In addition to operation, maintenance, and monitoring guidelines, this document presents the following:

- Alternative operation and maintenance procedures
- Safety procedures during maintenance
- Required equipment for maintenance

- Record keeping requirements
- Emergency contacts

## **1.2 Background**

On July 29, 1999, Waste Management, Inc., one of the Settling Defendants, and the Settling Defendants' consultant, RMT, Inc. (RMT), met with the USEPA Region 5 Post-Construction Completion (PCC) Team to discuss the USEPA's 5-year review of the Folkertsma Refuse Site (letter dated February 17, 1999). In particular, the problems associated with the statistical analysis of the data for this site and potential modifications to the OM&M Plan that would resolve these problems were discussed. During that meeting, USEPA representatives described the PCC Program's goals, as outlined in the USEPA letter of March 30, 1999, to provide cost savings to PRPs, and ultimately NPL deletion, by streamlining the performance monitoring at sites with constructed remedies on the basis of collected data, while maintaining protection of human health and the environment.

At the July 29, 1999, meeting, RMT made an oral presentation of their technical assessment of the data collected to date and the limitations in attempting to apply the statistical methods required in the Scope of Work (SOW) to this data set. Also discussed at the meeting was how the completed repair work at the site and the proposed changes to the OM&M Plan would both address the USEPA's 5-year review comments and streamline future monitoring, while continuing to protect human health and the environment. At the end of the meeting, the PCC Team members requested a revised OM&M Plan, which would also include the technical justification for requesting the changes. At that time, it was agreed that the technical rationale for the changes to the OM&M Plan would be included as an appendix to the revised Plan. This information was submitted to the USEPA on September 30, 1999.

At the July 29, 1999, meeting, the PCC Team also discussed the administrative procedures that could be used to make changes to operation and maintenance programs at PCC sites. Additionally, it was noted that Paragraph 16a (Modification of the Scope of Work) of Section VII (Additional Work and Modification of the SOW) of the Administrative Order on Consent (AOC) for this site, allows either party to the AOC to propose changes to the SOW.

In a letter dated November 9, 1999, the USEPA and the MDEQ commented on the information submitted in September. The Settling Defendants responded to the agencies' comments in a letter dated January 3, 2000. The USEPA and the MDEQ met on January 27 to discuss the proposed changes and the Settling Defendants' responses to their comments, and, in a telephone conversation with RMT, the USEPA requested a separate response to each of the comments in their November 9, 1999, letter. RMT submitted the requested information in a letter dated March 3, 2000.

On April 25, 2000, the USEPA, the MDEQ, and RMT had further discussions about the OM&M Plan. This was followed by letter from the USEPA, dated May 1, 2000, which documented the MDEQ's concerns that were raised on April 25. The Settling Defendants provided responses to the agencies' May 1 comments in a June 30, 2000, letter, and incorporated all of the changes discussed since February 1999 into a second revision of the OM&M Plan, dated June 2000.

In a letter dated August 10, 2000, the USEPA advised the Settling Defendants that the agencies would not change the recommendations for groundwater monitoring in the Five-Year Review.

On November 1 and, December 5, 2000, the Settling Defendants requested a meeting with the agencies to discuss the proposed changes to the OM&M Plan. In response, the USEPA sent an e-mail message to RMT, Inc., (on February 8, 2001), asking for additional information. The Settling Defendants provided responses in a letter dated February 16, 2001.

In an April 25, 2001 letter, the USEPA approved many of the Settling Defendants' requested changes to the OM&M Plan. Through a series of telephone conversations and e-mail messages between the USEPA and RMT, on April 25, 27, and 30, 2001, the agencies and the Settling Defendants negotiated final modifications to the OM&M Plan.

On March 2, 2009, the USEPA completed the Third Five-Year Review and recommended changes to the OM&M Plan. Details of the OM&M Plan modifications are described in the cover letter accompanying this document. The recommended changes are incorporated in this latest revision to the OM&M Plan.



## Section 2 Operation

---

There are no operational tasks associated with the remedial action. The landfill cover systems (including surface water and groundwater management systems) are passive systems that do not require human involvement for normal operation.

Since there are no operational tasks required for the landfill remedial action, no operational problems are expected.

# Section 3

## Maintenance

---

### 3.1 Overview

Provide continued maintenance for the appropriate components of the remedial action as required, unless otherwise noted. Maintenance tasks include the following:

- Semiannual site inspections and identification of needed repairs
- Restoring damaged areas of the landfill cover, as needed
- Mowing of the landfill cover vegetation on a biannual basis
- Overseeding and fertilizing, as needed
- Removing sediment in the drainage swales, as needed
- Restoring damaged sections of drainage swales, as needed
- Restoring/replacing damaged fencing, monitoring wells, and gas probes, as needed

A detailed description of each activity is given below. A log of these activities will be maintained and a summary of the activities will be reported to the USEPA and the MDEQ with the annual monitoring reports.

### 3.2 Site Inspections

Conduct semiannual site inspections of the landfill. Inspect the final cover for suitable vegetation, and the presence of erosion rills or gullies, depressions, and subsidence. Inspect the drainage swales for excess sediment, clogging by debris, sparse vegetation, or damage, including erosion and surface ponding. Closely monitor areas identified during the last four semiannual inspections. In addition, inspect the drainage swales for flood damage after a major storm event (a 100-year event). Inspect site fencing, groundwater monitoring wells, and gas probes for damage. Closely monitor and make note of any changes in land or groundwater use at the site and adjacent properties.

Record the site inspection findings on the form shown in Table 3-1. Summarize the site inspection findings in the annual monitoring reports (see Section 8) by updating the record in Table 3-2.

Notify the USEPA and the MDEQ in advance of scheduled site inspections. If, during any site inspection, changes to land or groundwater use is observed, communications will be made to the both the USEPA and MDEQ. Contact information is listed in Section 9.

### **3.3 Restoration of the Landfill Cover**

Restore the landfill cover if any of the following conditions are identified during site inspections:

- Gullies or rills on the surface of the cover that are greater than 6 inches in depth
- Areas of subsidence that form depressions or otherwise impair the performance of the cap in promoting surface water runoff, preventing infiltration, or waste containment

To restore the cover, place the required materials to achieve the original profile and requirements of the landfill cover design. Re-establish vegetation over the repaired areas. Place silt fences downslope of the area under repair if significant erosion is expected to occur before vegetation is established.

### **3.4 Vegetation Establishment and Cultivation**

Fertilize, seed, and mulch as required promoting good vegetative growth. Fertilize with an appropriate mix for the condition of the grass (*e.g.*, 19-19-19). Reseed over areas where maintenance activities have disturbed the soil or areas greater than 8 feet in diameter where vegetation coverage is less than 75 percent. The seed mixture will be 50% Perennial Ryegrass, 35% Creeping Red Fescue, and 15% Kentucky Bluegrass, as specified in MDOT Section 8.21.09 (Table 8.21, roadside mix) unless the grass seed mixture appears to be ineffective. Modify the seed mixture, as appropriate.

### **3.5 Biannual Mowing of the Landfill Vegetation**

Mow the landfill surface at least once every 2 years to stimulate root growth and to allow the vegetation to reseed itself. Perform additional mowing if vegetation is matted down (due to excessive height) to the extent that it reduces the retardation of surface water runoff, if vegetation becomes overgrown and a nuisance, or if detrimental plant species with penetrating roots become established. Control noxious weeds with timely application of an approved herbicide and by mowing.

### **3.6 Sediment Removal in the Drainage Swales**

Sediment eroded from the landfill cover system may accumulate in the drainage swales. Remove sediment from the drainage swales, if reduced capacity is identified. Excavate the sediment when it is reasonably dry and disperse it uniformly over the soil cap area. Take care not to damage existing vegetation. Stockpile and vegetate the material, if necessary, to use for site maintenance at a later date. Restore the drainage swale if damaged by the sediment removal process.

### **3.7 Restoration of Damaged Sections of Drainage Swales**

Restore to original condition or better, damaged sections of drainage swales noted during semiannual site inspections or after major storm events.

### **3.8 Restoration/Replacement of Fencing, Monitoring Wells, and Gas Probes**

Restore or replace to original condition, or better, damaged sections of fencing, monitoring wells, or gas probes noted during site inspections. Maintain stamped aluminum identification tags on monitoring wells.

As necessary to replace a monitoring well, install the new well by blind drilling to the required depth using a 6-inch-diameter, hollow-stemmed auger. Install 2-inch-inner diameter, threaded PVC Schedule 40, well screen and riser pipe. Backfill the annular space with a well-graded sand to a height of 2 feet above the screen. Install a bentonite seal to ground surface. Set a 6-inch-diameter protective casing with locking cover over the well-riser stickup. Install guard posts around each well. A qualified geologist or geotechnical engineer will oversee the well installation activities and record details on the new well installation. Submit a record of this work to the USEPA and the MDEQ for file update.

In the event that a well is replaced, the replaced well will be abandoned in accordance with Rules 325.1663 and 325.1664 of the Michigan water well construction code (Part 127 of Act 368), which incorporates ASTM standard D5299-92. The wells will be abandoned by either overdrilling the casing and then filling the borehole with grout, or by cutting off the well casing 3 feet below grade and then filling the casing with bentonite. State well abandonment forms will be submitted for all wells abandoned.

For gas probe replacement, follow the same actions for installing a groundwater monitoring well, except that a borehole for a gas probe will be 4 inches in diameter, and the pipe will be a 1-inch-diameter, Schedule 40 PVC perforated pipe.

# Section 4

## Environmental Monitoring

---

### 4.1 Overview

Environmental monitoring for the landfill remedial action will be conducted once, 6-12 months prior to the next Five-Year Review in 2013, for the following media:

- Groundwater
- Surface water
- Landfill gas

The USEPA, in their November 2008 Third Five-Year Review, agreed to limit the environmental monitoring conducted at the landfill to only one complete round of monitoring, conducted six to twelve months prior to the next Five-Year Review expected in 2013.

This section presents an overview of the environmental monitoring actions required for these media and the associated data analysis. Further details pertaining to environmental monitoring are presented in the Quality Assurance Project Plan (QAPP) for the Environmental Monitoring Program (Warzyn, Inc., September 22, 1993; as modified by Appendix A of this document).

The QAPP presents the organization, objectives, functional activities, and specific quality assurance and quality control activities associated with the remedial action. Appendix A of the September 1993 QAPP contains the Sampling and Analysis Plan (SAP). That document describes specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory and field analysis. Appendix A of this OM&M Plan amends those portions of the QAPP and the SAP that are affected by the changes to the environmental monitoring program that have been agreed upon by the USEPA, the MDEQ, and the Settling Defendants.

### 4.2 Groundwater Monitoring Program

Collect and analyze a single groundwater sample from each of the eight on-site monitoring wells (MW-106, MW-107R, MW-108, MW-109, MW-201, MW-206, MW-207R, and MW-208) six to twelve months prior to the next Five-Year Review expected in 2013. The locations of the monitoring wells are shown on Figure 2. Analyze each sample for the organic and inorganic chemicals and methods listed in Table 4-1. Sample quantities, containers, preservatives, and packaging requirements are provided in Table 4-2. Sample custody and documentation procedures are described in the SAP (Appendix A)

#### **4.2.1 General Sampling**

The following tests, measurements, and procedures will be conducted at groundwater monitoring wells in the order specified below:

- Record the name of the sampler, the date, and the time sampling began.
- Note and record the weather conditions, including sky conditions, air temperature, wind speed, and wind direction. Note and record other conditions that could affect the sample results.
- Inspect and record the condition of the monitoring well. Note if repair is needed. Note if the well was locked.
- Measure and record the depth to water to the nearest 0.01 foot, using the procedure described in Subsection 4.2.2. Repeat the depth to water measurement. If the second measurement differs from the first, remeasure until a consistent water level is determined.
- Purge the well, and record the results as specified in Subsection 4.2.3. Note the type of equipment used to purge the well. Also note the presence or absence of sediment in the sample. Dispose purge water to the ground surface. Use low-flow sampling techniques (purge rate of 1 L/min or less) to collect groundwater samples.
- Sample the well as specified in Subsection 4.2.4 for parameters listed in Table 4-1.
- Replace the well cap, lock the well, and note the time sampling is completed.

#### **4.2.2 Water Level Measurements**

The following procedures for measuring the water levels in the monitoring wells will be followed:

- Use either a weighted steel tape with raised numerals, graduated in tenths and hundredths of a foot, or an electric water level indicator to measure the water levels. Check to make sure that the tape is long enough to plumb the bottom of the deepest well.
- Check the tape to ensure that it is not damaged.
- Decontaminate the tape immediately prior to use and between each well by flushing with nonphosphate soap and distilled water.
- Note the previously recorded depth to water. Use the previously recorded depth as an estimate of the expected water level.
- Lower the tape into the well; and align the "hold" mark with the top of the well casing, being careful not to overshoot the top of the casing with the "hold" mark. Read the measurement directly from the tape, and record it on a form similar to the Water Level Measurement Record.

- Repeat the water level measurement to verify the depth to water.
- Measure the water levels in all of the monitoring wells during each sampling event.

### **4.2.3 Well Purging**

The following procedures will be followed prior to sampling each well:

- Use a new, disposable pair of Latex gloves and disposable tubing at each monitoring well for purging and sample collection. Place a clean plastic dropcloth on the ground around the well to minimize contact between the sampling equipment and the ground.
- Determine the volume of water in the well using the following calculation: one well volume (gallons) = (0.163 gallons per foot) x (feet of water column). (Note: this calculation is applicable only for 2-inch-inside diameter wells.)
- Remove water from the well using low-flow techniques (purge rate of 1 L/min or less), and monitor field parameters until the purged water meets the stabilization criteria listed in Table 4-3.
- Record the field parameters, including purging rate, until the stabilization criteria are met.
- Decontaminate the downhole sampling pumps by pumping 5 gallons of nonphosphate soap solution through them, followed by 10 gallons of distilled water.

### **4.2.4 Sample Collection**

The following procedures will be followed for sample collection:

- Upon completion of the well purging step, collect the samples using low-flow sampling techniques (1 L/min or less). A summary of the numbers and types of samples to be collected is presented in Table 4-4. The preservatives will be premeasured by the laboratory and added to the appropriate sample bottles prior to shipment to the facility. Each sample bottle for a given monitoring well will have a label (affixed by the laboratory) that will contain the facility name, the monitoring well number, the date and time of sample collection, and the sampler's initials. The type of preservative (if present) will also be recorded on the label.
- Do not filter samples for metals analysis unless the well produces water with excessive turbidity. If excessive turbidity is observed, also collect a filtered sample.
- If necessary to collect a filtered sample, filter the sample in the field using a peristaltic pump, a new section of Tygon® tubing, and a disposable (one-use)

0.45-micron high-capacity capsule filter for each sample. The filtered sample will be discharged directly into the containers provided by the laboratory.

- Do not touch the inside of bottle necks or caps, to minimize potential cross-contamination.
- Record the number and type of samples taken and the time of sampling on the chain-of-custody record and in the field notebook. The container label and field notes will identify which sample aliquots were filtered.

Quality control samples (see Table 4-4) will be collected/prepared as follows:

- Collect a duplicate sample at a randomly selected location.
- Prepare an equipment blank sample in the field by pouring distilled water through the pump after it has been cleaned, and then into the sample bottle.
- Pack the samples in coolers with ice that is sealed in zip-lock bags, and ship to the Pace Analytical Laboratories in Madison, Wisconsin, via overnight delivery. Ship the samples within 24 hours from the time that they are collected. Seal the chain-of-custody forms in zip-lock bags and place in the cooler containing the samples listed on the form.

#### **4.2.5 Sample Identification**

The following procedures will be followed for identifying samples:

- Identify duplicate samples by using a "D" as the station number and a sequential suffix beginning with "1" (for example, D-01). Duplicate samples will be "blind" samples sent to the laboratory to check the quality of the laboratory procedures.
- Enter the well identification number of the duplicate samples in the field log book.
- Identify field equipment rinsate blanks by using an "F" as the station number and a sequential suffix (*e.g.*, F-01).
- Include the field sample identification numbers on the chain-of-custody forms.

#### **4.2.6 Field Records**

To provide a complete, accurate, unbiased, and understandable record of events so that situations and events can be later reconstructed without having to rely on memory, sampling activities will be documented in field sample logs as follows:

- Use an all-weather pen to record information.
- Cross out incorrect entries with a single line, and date and initial the cross outs.



- Insert the correct information near the original entry in a manner that clearly shows where the correction belongs.
- Include in the field sample logs the following information:
  - Sample date and time
  - Type and number of bottles/containers filled
  - Sample description (color, odor, and turbidity)
  - Sample handling procedures (preservation, filtration, shipping)
  - Water levels, specific conductance, and temperature
  - Problems encountered
  - Any other information or observations that, in the judgment of the field personnel, are relevant to meeting the objectives of the project

### **4.3 Surface Water Monitoring Program**

Six to twelve months prior to the next Five-Year Review expected in 2013, collect and analyze a surface water sample from the combined discharge from the unnamed creek and the excavated ditch (at SW-1), and a sample from the background location (SWBG-1, which is the influent to the 48-inch reinforced concrete pipe). The surface water sampling locations are shown on Figure 2. The monitoring frequency and analytical parameters are identical to the groundwater monitoring program and are presented in Table 4-1. Sample quantities, preservatives, containers, and holding times are presented in Table 4-2.

### **4.4 Landfill Gas Monitoring Program**

#### **4.4.1 Monitoring Locations and Frequency**

Monitor the two gas probes (GP1 and GP2) for methane concentration (measured as a percent of the LEL) and for gas pressure within the probe. The locations of the gas probes are shown on Figure 2. Perform this monitoring event six to twelve months prior to the next 5-year review expected in 2013.

#### **4.4.2 Monitoring Procedures**

Landfill gas monitoring will be performed using portable gas meters and pressure gauges. The following procedures will be implemented:

1. Attach the low pressure port of the pressure gauge to the monitoring port. If the gauge indicates a negative reading, switch the hose on the gauge to the high-pressure sampling port. Record the vacuum or pressure. (Note: It is important to

monitor soil gas pressure in the probes prior to obtaining combustible gas concentration data.)

2. With the meter at room temperature, calibrate the meter according to the manufacturer's directions before each monitoring event.
3. At each probe, attach the hose to the monitoring port; and, when the readout has stabilized, record the oxygen, carbon dioxide, and combustible gas concentration.
4. After each reading using the combustible gas meter, remove the hose and allow the methane and oxygen reading to return to zero.
5. If the pressure gauge cannot be exactly zeroed prior to opening the monitoring probe valve, note the initial reading. After the valve is opened, and the pressure needle stabilizes, note this value. The actual pressure may be calculated by subtracting the initial reading from the second reading.
6. On a field data sheet or in the field log book, record the following data for the gas probes:
  - Percent oxygen
  - Carbon dioxide
  - Combustible gas as methane (percent LEL or percent gas)
  - Pressure
7. In addition, for each day monitoring is performed, record the following general information:
  - Date
  - Weather conditions
  - Barometric pressure and trend
  - Temperature
  - Ground condition (saturated, frozen, etc.)
  - Name of monitoring personnel

#### **4.5 Data Analysis – Groundwater Monitoring**

The results of the sampling event will be compared with the Michigan Part 201 generic GSI criteria (MDEQ, January 23, 2006). This review will include the following steps:

- Compare downgradient concentrations with the Part 201 generic GSI criteria (Table 4-5). If downgradient concentrations are above these criteria, then use site background (from MW-201) as the generic GSI criteria.

- If site background is used as the generic GSI criteria, then determine the percentage of nondetect ("U") data in the background data set, and address per USEPA guidance (1993, Section 2), as follows.
  - Nondetects less than or equal to 15% - replace with PQL/2 or use Aitchison's adjustment
  - Nondetects between 16% and 50% - use Aitchison's adjustment
  - Nondetects between 51% and 90% - use nonparametric Tolerance Limits
  - Nondetects greater than 90% - use Poisson Tolerance Limits
  - Nondetects at 100% - no statistical test is valid

#### **4.6 Data Analysis – Surface Water Monitoring**

The results of the surface water sampling will be evaluated in the same way as the results of the groundwater sampling (Subsection 4.5). The surface water results for SW-1 will be compared with the Michigan Rule 57 (Rule R323.1057 of the Michigan Administrative Code) criteria. For this site, the applicable Rule 57 criteria are the final chronic values (FCVs) for aquatic organisms. The FCVs are also the generic GSI criteria for this site. Consequently, the surface water and groundwater results will be compared with the same criteria (Table 4-5). As appropriate, surface water background will be calculated using results for SWBG-1.

#### **4.7 Data Analysis and Contingency Action – Landfill Gas Monitoring**

Compare the methane levels in the gas probes to the Lower Explosive Limit (LEL) for methane, which is 5 percent in air by volume. In accordance with Rule R299.4433, MAC, the point of compliance for landfill gas is the LEL at the property boundary.

# Section 5

## Alternative Operation, Maintenance, and Monitoring

---

As recommended in the *Superfund Remedial Design and Remedial Action Guidance* (USEPA, June 1986), this section should address operational and maintenance alternatives in case of failure of the remedial action. No alternative operation and maintenance procedures are recommended for the landfill cover because, if it fails, it will be repaired. The maintenance activities proposed for the landfill cover are intended to maintain the site in a secure condition, which includes performing repairs as needed.

Because the site has a major surface water drainage channel along its western and southern side that is affected by action beyond the control of the site owner and operator, alternative operation, maintenance, and monitoring procedures may need to be developed pending changes to the amount of surface water entering the site, including the enlargement of drainage channels if necessary.

## **Section 6**

# **Safety during Maintenance**

---

Follow the health and safety procedures set forth in the Health and Safety Plan for Operation, Maintenance, and Monitoring when conducting operation, maintenance, or monitoring at the site.

## Section 7 Required Equipment

---

It is anticipated that Folkertsma Settling Defendants will have a contractor perform maintenance activities for the site rather than keep equipment at the site. Maintenance activity frequency will be low enough that dedicated mowing and earthmoving equipment for the site would not be economically prudent.

## Section 8

# Record Keeping

---

Prepare an annual report for submittal to the USEPA and the MDEQ. Include the following items in this report.

- List of monitoring activities, if any, and records of the site inspections (Table 3-2). If additional inspections were performed due to significant storm events, also describe the condition of the landfill cover and drainage swales for each storm-related inspection.
- Date and results of routine maintenance activities such as mowing and fertilizing.
- Note of any changes in land or groundwater use at the site or at adjacent properties.
- Date and description of activities for repairs and reseeded. The description shall include the materials used and a map indicating the location.
- Date, description, and completion diagram of any wells that have been repaired, abandoned, or replaced.
- Records of annual operating costs.
- Summaries of all contacts with representatives of the local community, public interest groups, or State government.
- Changes in key personnel.
- Certification stating institutional controls are effective and in place.

# Section 9

## Emergency Contacts

This section presents a list of contacts in the event of an emergency at the site.

	Address	Telephone	Contact Person
Ambulance	City operates an ALS system; Fire Dept. authorities will contact ambulance via radio. A separate call is not required.	(616) 458-1441	
Hospital E/R	Spectrum Downtown Hospital 100 Michigan Street Grand Rapids	(616) 774-1774	
Poison Control	Grand Rapids	1-800-632-2727	
Police	City of Walker	(616) 453-5441 or 911	
Fire	City of Walker	(616) 458-1441 or 911	
Regulatory Agencies	77 W. Jackson Blvd., SR-6J Chicago, IL 60604	(312) 886-7253	Karen Cibulskis, Remedial Project Manager, USEPA Region 5 Post-Construction Completion Group
	MDEQ-Environmental Response Division	(517) 373-8436	Daria Devantier, Superfund Section
	MDEQ Pollution Emergency Alerting System (PEAS)	(800) 292-4706	
Settling Defendants' Technical Committee Chairperson	700 56 <sup>th</sup> Avenue Zeeland, MI 49464	(616) 688-5777	Phillip Mazor, Waste Management, Inc. District Manager Closed Site Management Group







# Tables

---

**Table 3-1  
Semiannual Site Inspection Record**

Date: \_\_\_\_\_

Temperature: \_\_\_\_\_

Inspector: \_\_\_\_\_

Weather: \_\_\_\_\_

USEPA/MDEQ Notified of  
Scheduled Inspection Date: \_\_\_\_\_

Ground Conditions: \_\_\_\_\_

ITEM	Adequate	Requires Maintenanc e	Status	Comments
<b>Final Cover:</b>				
Vegetation <sup>(1)</sup>				
Erosion <sup>(2)</sup>				
Settlement <sup>(3)</sup>				
Drainage swales <sup>(4)</sup>				
Grass mowed or fertilized (record date and/or fertilizer mix)				
Gas probes <sup>(5)</sup>				
Groundwater monitoring wells <sup>(5)</sup>				
Fencing <sup>(6)</sup>				
Gates and locks <sup>(6)</sup>				
Land and Groundwater Use <sup>(7)</sup>				
<b>Other Observations:</b>				

**NOTES:**

- (1) Inspect for suitable growth and coverage, presence of detrimental plant species, and weeds.
- (2) Inspect for erosion rills or gullies greater than 6 inches in depth.
- (3) Inspect for depressions or damaging subsidence.
- (4) Inspect for siltation or erosion, clogging by debris, or other damage. Inspect for flood damage after major storm events (100-year event).
- (5) Inspect riser pipes, surface seals, and supports for structural integrity, the presence and condition of the locks and identification labels.
- (6) Inspect for breaks, deterioration, frost heave, and vandalism.
- (7) Inspect for changes to land and groundwater use on site and at adjacent properties.

**Table 3-2  
History of Site Inspection and Monitoring Activities**

<b>DATE</b>	<b>EVENT</b>	<b>OBSERVATIONS</b>
12/19/94	Quarterly sampling and site inspection	No significant observations were noted.
12/20/94	Quarterly sampling and site inspection	No significant observations were noted.
1/31/95	Sample gas wells and inspect site	No significant observations were noted.
2/15/95	Sample gas wells and inspect site	No significant observations were noted.
3/16/95	Quarterly sampling and site inspection	No significant observations were noted.
4/25/95	Sample gas wells and inspect site	No significant observations were noted.
5/95	Fertilized and overseeded vegetative cover	No significant observations were noted.
5/18/95	Sample gas wells and inspect site	No significant observations were noted.
6/15/95	Quarterly sampling and site inspection	No significant observations were noted.
9/95	Fertilized and mowed	No significant observations were noted.
9/26/95	Quarterly sampling and site inspection	No significant observations were noted.
12/19/95	Quarterly sampling and site inspection	No significant observations were noted.
3/27/96	Quarterly sampling and site inspection	No significant observations were noted.
6/20/96	Quarterly sampling and site inspection	No significant observations were noted.
9/25/96	Quarterly sampling and site inspection	No significant observations were noted.
12/30/96	Quarterly sampling and site inspection	No significant observations were noted.
3/31/97	Quarterly sampling and site inspection	No significant observations were noted.
4/97	Fertilized and overseeded vegetative cover	No significant observations were noted.
6/97	Mowed vegetative cover	No significant observations were noted.
6/30/97	Quarterly sampling and site inspection	No significant observations were noted.
9/29/97	Quarterly sampling and site inspection	No significant observations were noted.
12/22/97	Quarterly sampling and site inspection	No significant observations were noted.
3/23/98	Quarterly sampling and site inspection	No significant observations were noted.
5/98	Fertilized and overseeded vegetative cover	No significant observations were noted.
6/25/98	Quarterly sampling and site inspection	Darling Rendering Plant collapsed on top of the site fence. The damage to the fence caused the site to become unsecured, and the fence was repaired in June 1998.

**Table 3-2 (continued)  
History of Site Inspection and Monitoring Activities**

<b>DATE</b>	<b>EVENT</b>	<b>OBSERVATIONS</b>
9/23/98	Quarterly sampling and site inspection	Site fence had been repaired since last site visit. The site is now secure.
11/5/98	Five-Year Review Site Inspection	A small low area was noted in the center swale. Slight erosion was noted in the center swale.
12/28/98	Quarterly sampling and site inspection	No significant observations were noted.
5/99	Fertilizing, overseeding*, and mowing of vegetative cover	No significant observations were noted.
9/99	Quarterly sampling and site inspection	No visible evidence of erosion. The main gate lock and MW201 lock were replaced.
3/00	Fertilizing and site inspection	Small trees growing in the drainage swale outside the northern fence need to be cut down when the site is mowed.
5/00	Semiannual sampling and site inspection	No significant observations. All well locks were replaced.
8/00	LFG monitoring and site inspection	Grass had been mowed since 5/00 inspection
10/00	Quarterly groundwater and surface water sampling, LFG monitoring and site inspection	No significant observations were noted.
12/00	Quarterly groundwater and surface water sampling, LFG monitoring and site inspection	No significant observations were noted.
3/01	Quarterly groundwater and surface water sampling, LFG monitoring and site inspection	No significant observations were noted.
6/01	Quarterly groundwater and surface water sampling, LFG monitoring, and site inspection	No significant observations were noted.
9/01	Quarterly groundwater and surface water sampling, LFG monitoring, and site inspection	Fence was damaged in the northeastern corner.
4/02	Quarterly groundwater and surface water sampling, LFG monitoring, and site inspection	Fence was damaged in the northeastern corner. An old drum was present on-site.
5/02	Fertilizing of entire site	
7/02	Mowing of vegetative cover	
9/02	Quarterly groundwater and surface water sampling, LFG monitoring, and site inspection	Site was mowed in July. Fence has been repaired. Old drum has been removed.
5/03	Semiannual groundwater and surface water sampling, LFG monitoring, and site inspection; fertilizing of entire site	
6/03	Removal of trees in ditches, and chipping and scattering of chips on-site; removal of two empty drums from site	

**Table 3-2 (continued)  
History of Site Inspection and Monitoring Activities**

DATE	EVENT	OBSERVATIONS
10/03	Semiannual LFG monitoring and site inspection; replacement of protective casings at GP1 and GP2	GP1 and GP2 protective casings were broken and have been replaced.
2004	Quarterly fence inspections and minor repairs.	
4/04	Semiannual groundwater and surface water sampling, LFG monitoring, and site inspection; abandonment of GP-3	No significant observations were noted.
6/04	Fertilizing of entire site	-
10/04	Semiannual LFG monitoring and site inspection	No significant observations were noted.
2005	Quarterly fence inspections and minor repairs.	
4/05	Semiannual groundwater and surface water sampling, LFG monitoring	No significant observations were noted.
11/05	Semiannual LFG monitoring and site inspection	No significant observations were noted.
2006	Quarterly fence inspections and minor repairs.	
4/06	Semiannual groundwater and surface water sampling, LFG monitoring	No significant observations were noted.
9/06	Mowing of vegetative cover	-
10/06	Semiannual LFG monitoring and site inspection	No significant observations were noted.
2007	Quarterly fence inspections and minor repairs.	
4/07	Semiannual groundwater and surface water sampling, LFG monitoring	No significant observations were noted.
5/07	Fertilizing of entire site	-
10/07	Semiannual LFG monitoring and site inspection, mowing of vegetative cover	No significant observations were noted.
2008	Quarterly fence inspections and minor repairs.	
4/08	Semiannual groundwater and surface water sampling, LFG monitoring	No significant observations were noted.
5/08	Fertilizing of entire site	-
9/08	Semiannual LFG monitoring and site inspection, mowing of vegetative cover	Locks were replaced at MW-108, MW-109, and the south gate. Broken hinge was repaired on MW-108.

\* Overseeding involves adding seed to fertilizer and broadcasting over the site.

**Table 4-1 (Revised March 2009)  
Environmental Monitoring Program  
Analytical Parameters, Methods, and Target Detection Limits  
for Groundwater and Surface Water**

PARAMETER	FREQUENCY OF ANALYSIS (MONTHS)	METHOD REFERENCE	TARGET DETECTION LIMITS (µg/L)
Volatile Organic Compounds (VOCs)	✓	8260	various
Aluminum	✓	6010/6020	50
Arsenic	✓	6010/6020/7060	20
Barium	✓	6010/6020	100
Cadmium	✓	6010/6020	0.5
Chromium (total)	✓	6010/6020	5
Cobalt	✓	6010/6020	10
Copper	✓	6010/6020	5
Iron	✓	6010/6020	100
Lead	✓	6010/6020	3
Magnesium	✓	6010/6020	100
Manganese	✓	6010/6020	20
Mercury	✓	SOW ILM03.0 <sup>(3)</sup>	0.2
Nickel	✓	6010/6020	25
Potassium	✓	6010/6020	500
Selenium	✓	6010/6020	5
Silver	✓	6020/200.9	0.2
Sodium	✓	6010/6020	1,000
Thallium	✓	6010/6020	2
Zinc	✓	6010/6020	20

Notes:

- <sup>(1)</sup> Method References: USEPA, 1996. SW 846. Test methods for evaluating solid wastes, physical/chemical methods. The most recent method revisions will be used.
- <sup>(2)</sup> From the MDEQ ERD Operational Memorandum #6, Revision 5, November 1998.
- <sup>(3)</sup> USEPA, Statement of Work for Inorganic Analysis, August 1991 (from Table 7-1 of the September 1993 QAPP).



**Table 4-2 (Revised March 2009)  
Environmental Monitoring Program  
Sample Quantities, Containers, Preservatives, and Packaging Requirements**

<b>Groundwater</b>						
Volatile Organic Compounds (VOCs)	Three 40 ml, glass VOA vials	HCl to pH<2, iced to 4°C.	14 days from sampling	Fill to top, no air bubbles	Shipped daily by overnight carrier	Vermiculite or bubble wrap
Target metals <sup>(3)</sup>	One 1-liter, high-density polyethylene bottle	HNO <sub>3</sub> to pH<2, iced to 4°C.	180 days from sampling (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite or bubble wrap
<b>Surface Water</b>						
VOCs	Three 40 ml, glass VOA vials	HCl to pH<2, iced to 4°C.	14 days from sampling	Fill to top, no air bubbles	Shipped daily by overnight carrier	Vermiculite or bubble wrap
Target metals <sup>(3)</sup>	One 1-liter, high-density polyethylene bottle	HNO <sub>3</sub> to pH<2, iced to 4°C.	180 days from sampling (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite or bubble wrap

**Notes:**

- <sup>(1)</sup> The holding time begins at the time the sample is collected.
- <sup>(2)</sup> The packing material should completely cushion the sample bottles – bottom, sides, and top.
- <sup>(3)</sup> Target metals for groundwater and surface water monitoring are listed in Table 4-1.

**Table 4-3  
Groundwater Stabilization Criteria**

PARAMETER	CRITERIA
pH	Three successive readings within 0.2 pH unit
Conductivity	Three successive readings within 20 $\mu$ mhos/cm
Temperature	Three successive readings within 0.5°C
Turbidity	Three successive readings within 10 percent or <10 NTU
Dissolved oxygen	Three successive readings within 0.5 mg/L
Eh	Three successive readings within 30 mv

**NOTE:**

<sup>(1)</sup> Stabilization criteria may be modified during sampling based on field observations.

**Table 4-4 (Revised March 2009)  
Summary of the Sampling and Analysis Program**

Media	Parameters	Number of Wells	Frequency of Sampling				
			1/10	1	1/10	1	10
Groundwater	pH, Eh, specific conductance, temperature, turbidity, and dissolved oxygen	8 <sup>(3)</sup>	1/10	1	1/10	1	10
Surface water	VOCs, Target metals	2 <sup>(4)</sup>	1/10	1	1/10	1	4

**Notes:**

- <sup>(1)</sup> Field parameters will be measured at the time of sample collection.
- <sup>(2)</sup> Laboratory analyses will be performed by Pace Analytical Services. Specific analytes, laboratory methods, and target detection limits are listed in Table 4-1.
- <sup>(3)</sup> Samples will be collected from the following 8 wells: MW-106, MW-107R, MW-108, MW-109, MW-201, MW-206, MW-207R, and MW-208.
- <sup>(4)</sup> Samples will be collected at SW-1 and SWBG-1.

**Table 4-5 (Revised March 2009)  
GSI Criteria for the  
Folkertsma Refuse Site**

PARAMETER	GENERIC GSI CRITERIA (GSI) <sup>(1)</sup>
VOCs	various
Aluminum	NA
Arsenic	150
Barium	1,037 <sup>(2)</sup>
Cadmium	9 <sup>(2)</sup>
Chromium III	216 <sup>(2)</sup>
Cobalt	100
Copper	27 <sup>(2)</sup>
Iron	NA
Lead	107 <sup>(2)</sup>
Magnesium	NA
Manganese	1,079 <sup>(2)</sup>
Mercury	0.2 <sup>(3)</sup>
Nickel	239 <sup>(2)</sup>
Potassium	NL
Selenium	5.0
Silver	0.2
Sodium	NA
Thallium	4
Zinc	493 <sup>(2)</sup>

(1) Generic GSI criteria published by the MDEQ, January 23, 2006.

(2) Hardness-dependent generic GSI criterion calculated using 225 mg/L calcium carbonate for the Indian Mill Creek in Kent County. Calculations presented in Appendix C.

(3) Generic GSI criteria is less than the Target Detection Limit, therefore, the TDL will be used as the site-specific GSI criteria.

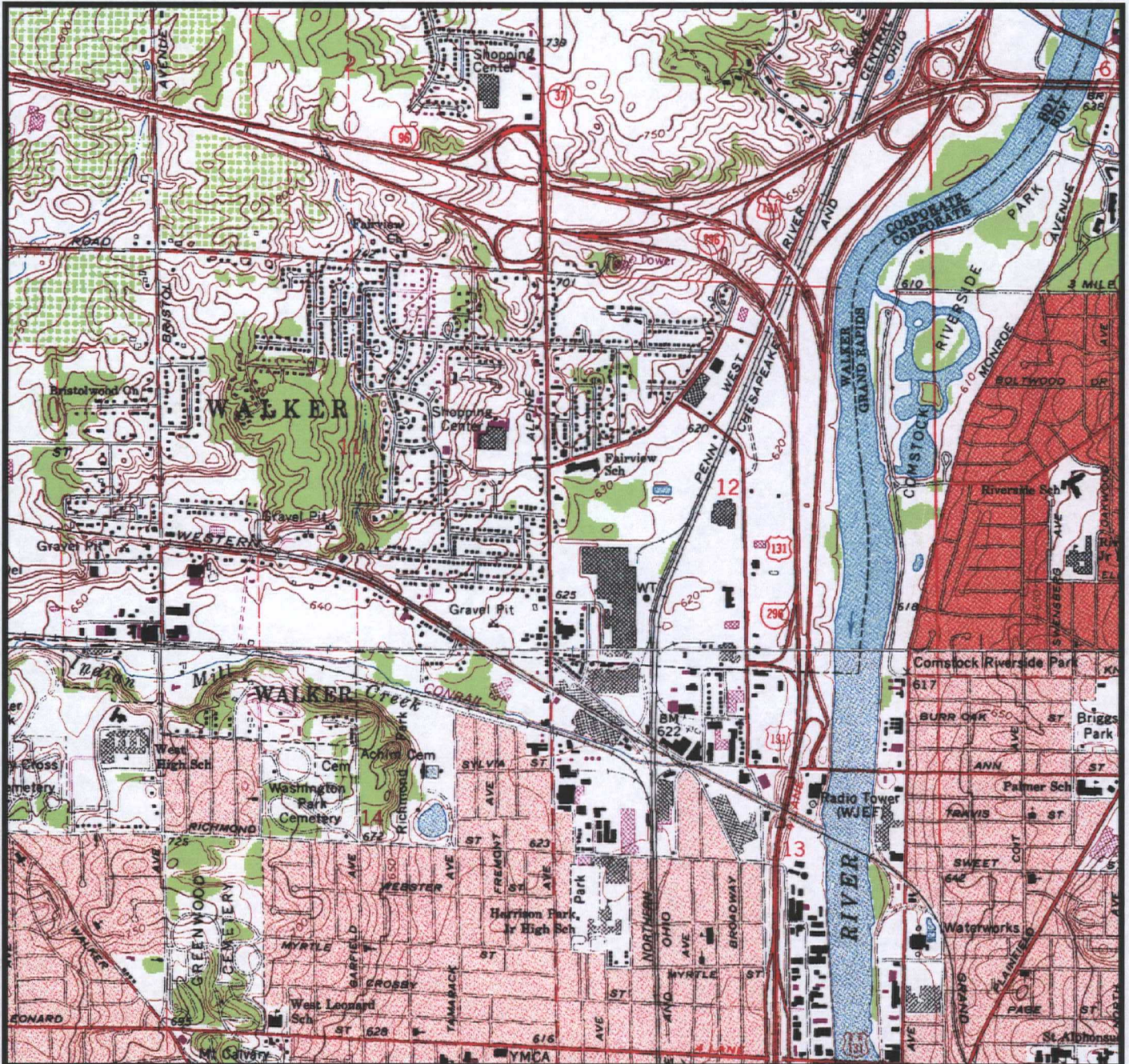
NA = not available.

NL = parameter not listed.



# Figures

---



Attached Xrefs:  
Cedar Springs SW Michigan; Grand Rapids West; Grand Rapids West; RMT Illustration;  
Site Layout (1)

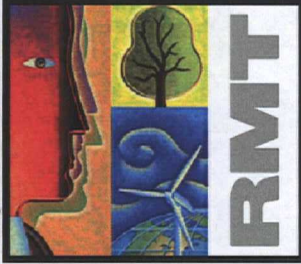
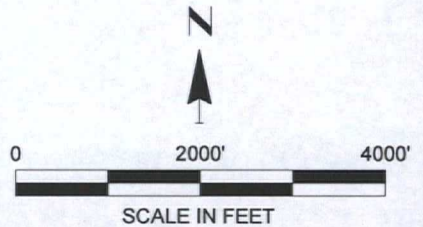
Attached Xrefs:  
Attached Images:  
Layout

Dwg Size: 0.17 MB  
Plot Date: April 16, 2009  
Plot Time: 10:45 AM

PLOT DATA:  
J:\05331\30\01.dwg  
Operator Name: SAM LUCIDO  
Drawing Plot Scale: 0.000500

**SOURCE:**

BASE MAP DEVELOPED FROM CEDAR SPRINGS SW AND GRAND RAPIDS WEST, 7.5 MINUTE U.S.G.S. TOPOGRAPHIC QUADRANGLE MAPS, DATED 1967, PHOTOREVISED 1972..



**FOLKERTSMA REFUSE SITE**  
**FOLKERTSMA, MICHIGAN**

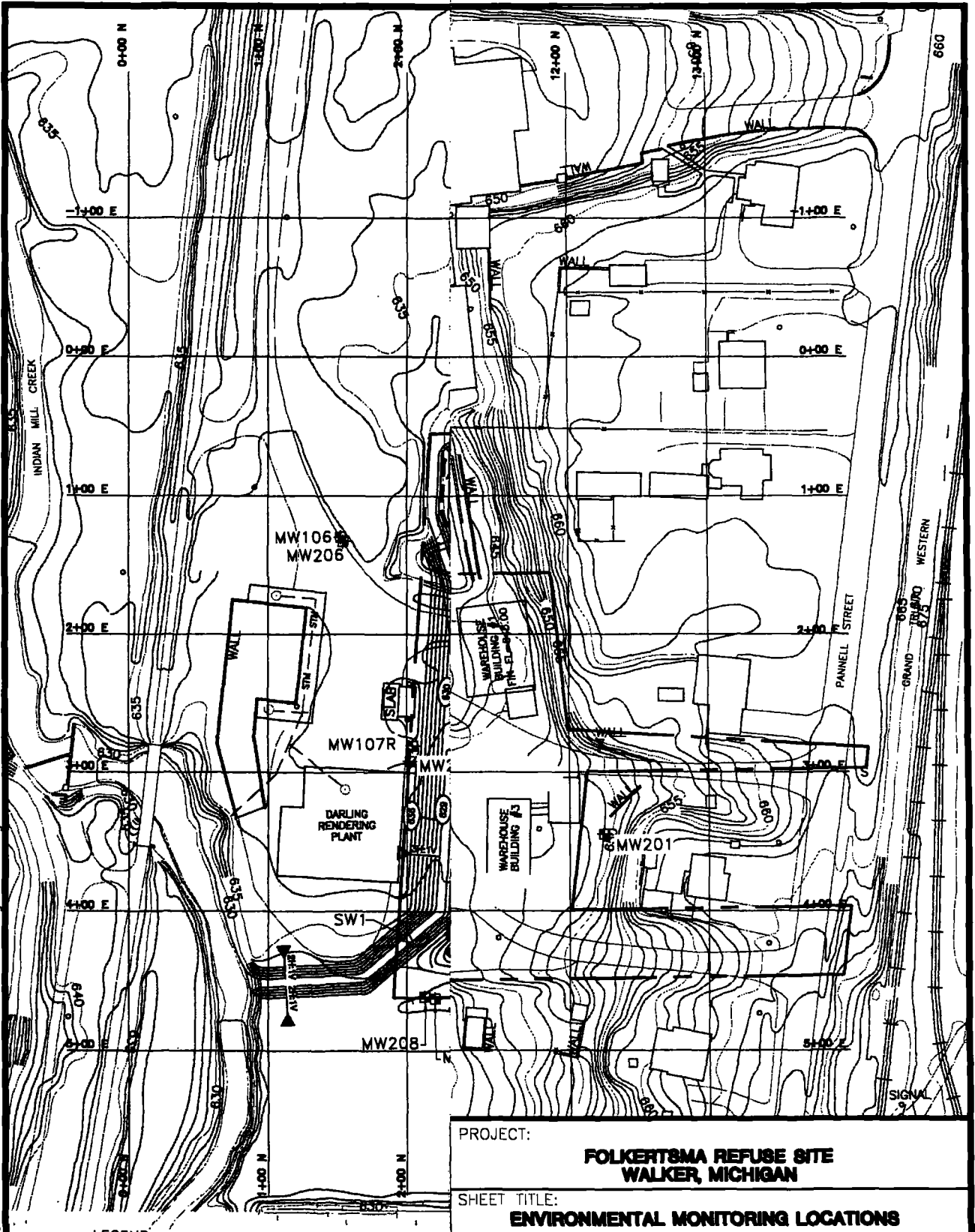
**SITE LOCATION MAP**

DRAWN BY:	SL
CHECKED BY:	JO
APPROVED BY:	JO
DRAWING SCALE:	SHOWN
PROJECT NUMBER:	J:\05331\30
FILE NUMBER:	5331.30.01.dwg
DATE:	April 2009

FIGURE 1

**PLOT DATA**  
 Drawing Name: J:\05331\30\5331.30.01.dwg  
 Operator Name: Lucido  
 Scale: 1"=100'  
 Dwg Size: 955874 Bytes

Plot Date: April 2009  
 Plot Time: 11:53:08 AM  
 Attached Xref's: No xref's Attached.  
 Attached Image's: No images attached



**LEGEND**

---	APPROXIMATE PROPERTY LINE
---	WATER LINE
---	GRAVEL ROAD
---	LIMITS OF FINAL COVER
---	DRAINAGE SWALE

MW109  
 GP1  
 SW1  
 SWBG-1

PROJECT:			
<b>FOLKERTSMA REFUSE SITE WALKER, MICHIGAN</b>			
SHEET TITLE:			
<b>ENVIRONMENTAL MONITORING LOCATIONS</b>			
DRAWN BY:	SL	SCALE:	PROJ. NO. 5331.30
CHECKED BY:	JO	1"=100'	FILE NO. 5331.30.01.DWG
APPROVED BY:	MA	DATE PRINTED:	<b>FIGURE 2</b>
DATE:	APRIL 2009		
<b>RMT</b>			
744 Heartland Trail Madison, WI 53717-1934 P.O. Box 8923 53708-8923 Phone: 608-831-4444 Fax: 608-831-3334			



11/11/11





# Appendix A

## Amendments to the QAPP and SAP

---

# Table of Contents

---

Introduction.....	A-iii
1. Project Description.....	A-1
1.1 Site Description.....	A-1
1.2 Past Data Collection Activity/Current Status.....	A-1
1.3 Project Objectives and Scope.....	A-1
1.3.1 Groundwater Monitoring.....	A-1
1.3.2 Surface Water Monitoring.....	A-2
1.3.3 Landfill Gas Monitoring.....	A-2
1.4 Sampling Network Design and Rationale.....	A-3
1.5 Parameters to be Tested and Frequency.....	A-3
1.6 Intended Data Usage and Data Quality Objectives.....	A-3
1.7 Project Schedule.....	A-4
2. Project Organization and Responsibilities.....	A-5
3. Quality Assurance Objectives for Measurement Data.....	A-6
4. Sampling Procedures.....	A-7
4.1 General Sampling.....	A-7
4.2 Water Level Measurements.....	A-8
4.3 Well Purging.....	A-8
4.4 Sample Collection.....	A-8
4.5 Sample Identification.....	A-9
4.6 Field Records.....	A-10
5. Sample Custody and Documentation.....	A-11
5.1 Field Sample Custody Procedures.....	A-11
5.2 Laboratory Custody Procedures.....	A-12

6.	Calibration Procedures and Frequency .....	A-13
7.	Analytical Procedures .....	A-14
8.	Internal Quality Control Check.....	A-15
9.	Data Reduction, Validation, and Reporting.....	A-16
9.1	Data Reduction .....	A-16
9.1.1	Field Data Reduction Activities.....	A-16
9.1.2	Laboratory Data Reduction Procedures.....	A-16
9.2	Data Reporting.....	A-17
9.2.1	Field Data Reporting.....	A-17
9.2.2	Laboratory Data Reporting .....	A-17
9.3	Data Validation .....	A-17
9.3.1	Procedures Used to Validate Field Data .....	A-17
9.3.2	Procedures Used to Validate Laboratory Data.....	A-17
10.	Performance and System Audits .....	A-19
11.	Preventive Maintenance.....	A-20
12.	Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness.....	A-21
13.	Corrective Action .....	A-22
14.	Quality Assurance Reports and Management.....	A-23
15.	References .....	A-24

**List of Tables**

Table 4-1	Environmental Monitoring Program Inorganic Parameters, Methods, and Target Detection Limits for Groundwater and Surface Water
Table 4-2	Environmental Monitoring Program Sample Quantities, Containers, Preservatives, and Packaging Requirements
Table 4-3	Groundwater Stabilization Criteria
Table 4-4	Summary of the Sampling and Analysis Program
Table 4-5	GSI Criteria for the Folkertsma Refuse Site

# Introduction

---

This amendment to the Quality Assurance Project Plan (QAPP) and its Sampling and Analysis Plan (SAP) describes only those procedures that have changed from the original QAPP and SAP for the site (Warzyn, 1993) and incorporates the unchanged portions of the original QAPP and SAP by reference.

The USEPA has completed its third 5-year review of the Folkertsma Refuse Site. An evaluation of the last 5 years of the post-construction monitoring data (2004-2008) indicates that certain modifications to the monitoring plan are appropriate. These modifications will focus future data acquisition on the site indicator parameters that provide useful information for ensuring the effectiveness of the site remedy. As a result of this evaluation, the monitoring program has been modified as follows:

- Analytical parameter list revised
- Sampling frequency reduced
- Laboratory changed
- Laboratory methods revised

# Section 1

## Project Description

---

### 1.1 Site Description

The Third Five-Year Review for the Folkertsma Refuse Site (USEPA, February 2009) describes the site history and changes to the site since the QAPP was approved. The significant changes to the site included the implementation of the remedy, which included installing a landfill cap and permeable underground drains, along with restoring site ditches. The Final Construction Completion Report, with a copy of the filed deed restriction, was submitted to the USEPA in February 1995 and was approved on March 30, 1995.

### 1.2 Past Data Collection Activity/Current Status

As described in the Third Five-Year Review (USEPA, 2009), the site has had continued quarterly groundwater monitoring since the first post-construction round of sampling in December 1994 through April 2001. Following April 2001, groundwater monitoring has been conducted on a semiannual basis from April 2001 through April 2003, and annually from April 2003 to the present. The next round of groundwater and surface water samples are scheduled to be collected 6-12 months prior to the next Five-Year Review, currently scheduled for 2013.

### 1.3 Project Objectives and Scope

#### 1.3.1 Groundwater Monitoring

The purpose of the groundwater monitoring program at the Folkertsma Refuse Site is to monitor the effectiveness of the Remedial Action and to evaluate the groundwater quality with respect to applicable standards.

The eight site monitoring wells (MW-106, MW-107R, MW-108, MW-109, MW-201, MW-206, MW-207R, and MW-208) will be sampled once, 6-12 months prior to the next Five-Year Review scheduled to be in 2013, for the list of analytes presented in Table 4-1 (table numbers are consistent with the numbers used in the main body of the OM&M Plan). This sampling event will confirm the landfill cover system and underground drains are continuing to function as intended.

The results of the sampling event will be compared with the Michigan Part 201 generic GSI criteria (MDEQ, January 23, 2006). This review will include the following steps:

- Compare downgradient concentrations with the Part 201 generic GSI criteria (Table 4-5). If downgradient concentrations are above these criteria, then use site background (from MW-201) as the generic GSI criteria.
- If site background is used as the generic GSI criteria, then determine the percentage of nondetect ("U") data in the background data set and address according to USEPA guidance (1993, Section 2) as follows:
  - Nondetects less than or equal to 15% - replace with PQL/2 or use Aitchison's adjustment
  - Nondetects between 16% and 50% - use Aitchison's adjustment
  - Nondetects between 51% and 90% - use nonparametric Tolerance Limits
  - Nondetects greater than 90% - use Poisson Tolerance Limits
  - Nondetects at 100% - no statistical test is valid

The results of this review will be reported to the USEPA with the annual report.

### **1.3.2 Surface Water Monitoring**

The purpose of the surface water monitoring is to assess the effect of the remedy on surface water quality. Surface water will be sampled on the same frequency as groundwater and analyzed for the same parameters (Table 4-1). The results of the surface water sampling will be evaluated in the same way as the results of the groundwater sampling (Subsection 1.3.1). The surface water results for SW-1 will be compared with the Michigan Rule 57 (Rule R323.1057 of the Michigan Administrative Code) criteria. For this site, the applicable Rule 57 criteria are the final chronic values (FCVs) for aquatic organisms. The FCVs are also the generic GSI criteria for this site. Consequently, the surface water and groundwater results will be compared with the same criteria (Table 4-5). As appropriate, surface water background will be calculated using results for SWBG-1.

### **1.3.3 Landfill Gas Monitoring**

The purpose for the landfill gas monitoring is to assess the potential for gas migration from the site. In accordance with Rule R299.5519, Michigan Administrative Code, the point of compliance for landfill gas is the property boundary. The two gas probes will be monitored for methane (percent lower explosive limit) and pressure. Testing will be performed once, 6-12 months prior to the next Five-Year Review scheduled to be in 2013.



If landfill gas is detected in a probe with measurable positive pressure, then the contingency response actions described in Subsection 4.7 of the OM&M Plan will be implemented.

## **1.4 Sampling Network Design and Rationale**

There are no changes to the sampling network design or rationale. All eight groundwater monitoring wells and two landfill gas probes will be included in the monitoring program. Downgradient surface water samples will continue to be collected at the combined surface water location (SW-1) and upstream surface water samples will continue to be collected from SWBG-1.

## **1.5 Parameters to be Tested and Frequency**

Sampling parameters for groundwater and surface water are presented in Table 4-1. A summary of sample volume, bottle, preservation, and packaging requirements is provided in Table 4-2. Sampling for groundwater and surface water will be conducted once, 6-12 months prior to the next Five-Year Review. Landfill gas will also be monitored once, 6-12 months prior to the next Five-Year Review.

## **1.6 Intended Data Usage and Data Quality Objectives**

Data will be collected to meet definitive data quality objectives (DQOs). Groundwater samples will be analyzed by a qualified laboratory to test for the presence of the analytical parameters included in Table 4-1. Sample analysis will be conducted in accordance with USEPA-approved methods for analytical and data validation procedures and Standard Operating Procedures (SOPs) for the laboratory in order to meet the target detection limits of the MDEQ's Part 201 Program, with the exception of mercury. The target detection limit for mercury will be the same as in the 1993 QAPP.

Analyses will include the QA/QC procedures and documentation and third-party data validation as described in the QAPP. These data will be used to evaluate the effect of the capped inactive landfill on the groundwater and surface water quality and landfill gas at the Folkertsma Refuse Site. The DQO process is a series of planning steps based on scientific methods that are designed to ensure that the type, quality, and quantity of environmental data used in decision-making are appropriate for the intended application.

DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO process that clarify the investigative objectives; define the type of data to collect; and determine conditions from which to collect the data. The DQOs are then used to develop a scientific and resource-effective data collection design.

The DQO process allows decision makers to define their data requirements and specify how different types of data will be used in the investigative process before data are collected.

**Screening Data:** These data are generated by less precise analytical methods with less rigorous sample preparation. Screening data provide analyte identification and quantification, although the quantification may be relatively imprecise. A portion of screening data may be confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Screening data without associated confirmation data are not considered to be data of known quality.

- Screening data will be used for field-measured parameters (pH, temperature, specific conductance, turbidity, and depth to groundwater and landfill gas).

**Definitive Data:** These data are generated using rigorous analytical methods, such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location as long as QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error or precision of the analytical method must be determined.

The following data will be collected to meet definitive data quality objectives:

- Groundwater and surface water samples to be analyzed for metals in accordance with EPA SW-846 protocol for analytical and data validation procedures.

These analyses will include QA/QC procedures and documentation and third-party data validation as described in this QAPP.

## 1.7 Project Schedule

Monitoring activities following these revisions in this QAPP will commence upon written approval by the USEPA.

## Section 2

# **Project Organization and Responsibilities**

Changes to the project organization are as follows:

- USEPA Region 5 Remedial Project Manager: Karen Cibulskis
- Consultant Project Manager: Jennifer Overvoorde
- Analytical Laboratory: Pace Analytical Services, Green Bay, Wisconsin
- Mr. Phillip Mazor of Waste Management, Inc., remains as the representative of the Folkertsma Site Settling Defendants' Technical Committee

# Section 3

## Quality Assurance Objectives for Measurement Data

---

The QA objectives for the measurement data have not changed. The level of QC sampling is summarized below:

	ORIGINAL SAMPLES	DUPPLICATE SAMPLES	TOTAL
Groundwater	8	1	10
Surface water	2	1	4
TOTAL	10	2	14

QC samples will be used to assess compliance with the data quality objectives of this project. Duplicate sample results are not independent data for the purposes of statistical evaluation and will not be used in statistical comparisons. They will be used to evaluate the validity of the original data point, and data analysis will include a discussion of any differences between original samples and their duplicates. Field blanks will not be collected if dedicated sampling equipment is used.

# Section 4

## Sampling Procedures

---

The sampling procedures described in the SAP will be followed with the following modifications:

- PID readings in the breathing zone are no longer required.
- Landfill gas testing will include pressure measurements inside the probe with a direct-reading instrument. Additionally, on each day during which gas monitoring is conducted, the ambient temperature and barometric pressure and trend will be recorded.

The specific procedures to be followed are described below.

### 4.1 General Sampling

The following tests, measurements, and procedures will be conducted at groundwater monitoring wells MW-106, MW-107R, MW-108, MW-109, MW-201, MW-206, MW-207R, and MW-208 in the order specified below:

- Record the name of the sampler, the date, and the time sampling began.
- Note and record the weather conditions, including sky conditions, air temperature, wind speed, and wind direction. Note and record other conditions that could affect the sample results.
- Inspect and record the condition of the monitoring well. Note if repair is needed. Note if the well was locked.
- Measure and record the depth to water to the nearest 0.01 foot, using the procedure described in Subsection 4.2. Repeat the depth to water measurement. If the second measurement differs from the first, remeasure until a consistent water level is determined.
- Purge the well, and record the results as specified in Subsection 4.3. Note the type of equipment used to purge the well. Also note the presence or absence of sediment in the sample. Dispose purge water to the ground surface. Use low-flow sampling techniques (purge rate of 1 L/min or less) to collect groundwater samples.
- Sample the well as specified in Subsection 4.4 for parameters listed in Table 4-1.
- Replace the well cap, lock the well, and note the time sampling is completed.

## 4.2 Water Level Measurements

The following procedures for measuring the water levels in the monitoring wells will be followed:

- Use either a weighted steel tape with raised numerals, graduated in tenths and hundredths of a foot, or an electric water level indicator to measure the water levels. Check to make sure that the tape is long enough to plumb the bottom of the deepest well.
- Check the tape to ensure that it is not damaged.
- Decontaminate the tape immediately prior to use and between each well by flushing with nonphosphate soap and distilled water.
- Note the previously recorded depth to water. Use the previously recorded depth as an estimate of the expected water level.
- Lower the tape into the well; and align the "hold" mark with the top of the well casing, being careful not to overshoot the top of the casing with the "hold" mark. Read the measurement directly from the tape, and record it on a form similar to the Water Level Measurement Record.
- Repeat the water level measurement to verify the depth to water.
- Measure the water levels in all of the monitoring wells during each sampling event.

## 4.3 Well Purging

The following procedures will be followed prior to sampling each well:

- Use a new, disposable pair of Latex gloves and disposable tubing at each monitoring well for purging and sample collection. Place a clean plastic dropcloth on the ground around the well to minimize contact between the sampling equipment and the ground.
- Determine the volume of water in the well using the following calculation: one well volume (gallons) = (0.163 gallons per foot) x (feet of water column). (Note: this calculation is applicable only for 2-inch-inside diameter wells.)
- Remove water from the well using low-flow techniques (purge rate of 1 L/min or less), and monitor field parameters until the purged water meets the stabilization criteria listed in Table 4-3.
- Record the field parameters, including purging rate, until the stabilization criteria are met.
- Decontaminate the downhole sampling pumps by pumping 5 gallons of nonphosphate soap solution through them, followed by 10 gallons of distilled water.

## 4.4 Sample Collection

The following procedures will be followed for sample collection:

- Upon completion of the well purging step, collect the samples using low-flow sampling techniques (1 L/min or less). A summary of the numbers and types of samples to be collected is presented in Table 4-4. The preservatives will be premeasured by the laboratory and added to the appropriate sample bottles prior to shipment to the facility. Each sample bottle for a given monitoring well will have a label (affixed by the laboratory) that will contain the facility name, the monitoring well number, the date and time of sample collection, and the sampler's initials. The type of preservative (if present) will also be recorded on the label.
- Do not filter samples for metals analysis unless the well produces water with excessive turbidity. If excessive turbidity is observed, also collect a filtered sample.
- If necessary to collect a filtered sample, filter the sample in the field using a peristaltic pump, a new section of Tygon® tubing, and a disposable (one-use) 0.45-micron high-capacity capsule filter for each sample. The filtered sample will be discharged directly into the containers provided by the laboratory.
- Do not touch the inside of bottle necks or caps, to minimize potential cross-contamination.
- Record the number and type of samples taken and the time of sampling on the chain-of-custody record and in the field notebook. The container label and field notes will identify which sample aliquots were filtered.

Quality control samples (see Table 4-4) will be collected/prepared as follows:

- Collect a duplicate sample at a randomly selected location.
- Prepare an equipment blank sample in the field by pouring distilled water through the pump after it has been cleaned, and then into the sample bottle.
- Pack the samples in coolers with ice that is sealed in zip-lock bags, and ship to Pace Analytical Services in Green Bay, Wisconsin, via overnight delivery. Ship the samples within 24 hours from the time that they are collected. Seal the chain-of-custody forms in zip-lock bags and place in the cooler containing the samples listed on the form.

## 4.5 Sample Identification

The following procedures will be followed for identifying samples:

- Identify duplicate samples by using a "D" as the station number and a sequential suffix beginning with "1" (for example, D-01). Duplicate samples will be "blind" samples sent to the laboratory to check the quality of the laboratory procedures.
- Enter the well identification number of the duplicate samples in the field log book.
- Identify field equipment rinsate blanks by using an "F" as the station number and a sequential suffix (e.g., F-01).

- Include the field sample identification numbers on the chain-of-custody forms.

## **4.6 Field Records**

To provide a complete, accurate, unbiased, and understandable record of events so that situations and events can be later reconstructed without having to rely on memory, sampling activities will be documented in field sample logs as follows:

- Use an all-weather pen to record information.
- Cross out incorrect entries with a single line, and date and initial the cross outs.
- Insert the correct information near the original entry in a manner that clearly shows where the correction belongs.
- Include in the field sample logs the following information:
  - Sample date and time
  - Type and number of bottles/containers filled
  - Sample description (color, odor, and turbidity)
  - Sample handling procedures (preservation, filtration, shipping)
  - Water levels, specific conductance, and temperature
  - Problems encountered
  - Any other information or observations that, in the judgment of the field personnel, are relevant to meeting the objectives of the project



# Section 5

## Sample Custody and Documentation

---

### 5.1 Field Sample Custody Procedures

Sample possession will be traceable from the time of collection to disposal through the use of chain-of-custody procedures. Chain-of-custody forms will accompany all sample shipments in order to document the transfer of the shipping containers and samples from the field to the laboratory. The procedures to be implemented are as follows:

- Prepare sample containers in the laboratory with pre-applied labels, and apply chain-of-custody seals to the shipping containers.
- Label and tag each sample in the field with indelible, waterproof ink.
- Complete the chain-of-custody forms indicating sample identification number; number of containers filled; sampling date; sampling time; sample collector's name; and sample preservation, if applicable. This information will also be noted in the field log books.
- Repack the shipping containers (coolers) with the samples, chain-of-custody forms (sealed in a zip-lock bag), and ice (sealed in zip-lock bags). Each group of samples to be shipped together will be listed on a chain-of-custody form, which will travel with that group of samples.
- Seal the containers, and ship them to the designated analytical laboratory via an overnight delivery service. Common carriers or intermediate individuals will be identified on the chain-of-custody form, and copies of all bills-of-lading will be retained.
- In the laboratory, check the shipping containers for broken seals or damaged sample containers. If no problems are noted, log the samples into the laboratory and complete the chain-of-custody form.
- Fill out the chain-of-custody form legibly in waterproof ink.
- Correct any errors by drawing a single line through the incorrect information and entering the correct information. All corrections will be initialed and dated by the person making the correction.
- Prepare a separate sample receipt whenever samples are split with a government agency. Mark the receipt to indicate with whom the samples were split. The person relinquishing samples to an agency will request the agency representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this will be noted on the receipt and in the field log book.

- The chain-of-custody is a triplicate form; keep the bottom copy (pink) with the field notes in the field. The top and second copy will accompany the samples to the laboratory. If a chain-of-custody form is damaged in shipment, the field copy (pink) will be available.
- Attach a copy of the chain-of-custody form(s) with the analytical report.

## **5.2 Laboratory Custody Procedures**

Laboratory custody procedures are detailed in the laboratory SOP (Appendix B).

# Section 6

## Calibration Procedures and Frequency

---

Instrument calibration procedures and frequency are described in the laboratory SOPs (Appendix B).

# Section 7

## Analytical Procedures

---

Groundwater and surface water samples collected for metals analyses will be analyzed by Pace Analytical Services, in Green Bay, Wisconsin, by the methods presented in Table 4-1 to meet the target detection limits of the MDEQ's Part 201 program with the exception of mercury. The target detection limit for mercury will be the same as in the 1993 QAPP. The Pace Analytical QA Manual and Laboratory SOPs are included as Appendix B to this OM&M Manual.

# Section 8

## Internal Quality Control Check

---

Internal quality control check procedures are described in the laboratory QA Manual.

# Section 9

## Data Reduction, Validation, and Reporting

---

### 9.1 Data Reduction

#### 9.1.1 Field Data Reduction Activities

Raw data from field measurements and sample collection activities will be recorded as specified in the QAPP and SAP. If the data are to be used in the project reports, they will be reduced or summarized, and the method of reduction will be documented in the report. With the exception of the temperature correction for specific conductance, no calculation will be involved in field data reduction. Only direct-reading instrumentation will be employed in the field.

#### 9.1.2 Laboratory Data Reduction Procedures

Pace Analytical Services will perform analytical data reduction under the direction of their QA Officer. The Laboratory QA Officer will be responsible for assessing data quality and advising of any data that were rated "preliminary" or "unacceptable" or of other notations that would caution the data user of possible unreliability. Data reduction, by the laboratory, will be conducted as follows:

- The raw data produced by the analyst will be turned over to the metals area supervisor.
- The supervisor will review the data for attainment of quality control criteria as outlined in the established USEPA methods and for overall reasonableness.
- Upon acceptance of the raw data by the supervisor, a computerized report will be generated.
- The Laboratory QA Officer verifies the accuracy and completeness of the final reports.
- The Laboratory QA Officer and the Laboratory Supervisor in conjunction with the RMT QA/QC Manager, will decide whether any sample reanalysis is required.
- Data reduction procedures will be those specified in the QA Manual and the laboratory SOPs in Appendix B.

## **9.2 Data Reporting**

### **9.2.1 Field Data Reporting**

Field data reporting will consist of field logs documenting site activities and the sample chain-of-custody forms.

### **9.2.2 Laboratory Data Reporting**

The analytical laboratory will prepare and retain full analytical and QC documentation. Such retained documentation need not be hard (paper) copy, but may be in other storage media (e.g., computer diskette or magnetic tape). As needed, the laboratory will supply a hard copy of the retained information.

The laboratory will provide the following information in each analytical data package submitted:

1. Chain-of-custody documentation
2. Sample results
3. Reporting limits
4. Method citations on LIMS summary report
5. Method blank results and batch associations
6. Laboratory control sample results
7. Matrix spike/matrix spike duplicate results from the analytical batch.

## **9.3 Data Validation**

### **9.3.1 Procedures Used to Validate Field Data**

Procedures to evaluate field data will primarily include checking for transcription errors and reviewing field notebooks, on the part of field crew members. This task is the responsibility of the On-site Coordinator. The data reviewer will review field notes and field chain-of-custody forms to determine that procedures specified in the QAPP and SAP have been followed.

### **9.3.2 Procedures Used to Validate Laboratory Data**

RMT's Applied Chemistry personnel will conduct third-party data validation. The RMT QA Manager or designated data reviewer will conduct a review of the data for compliance with the established QC criteria based on the spike, duplicate, and blank

results provided by the laboratory. Data validation will determine whether the procedures specified in this QAPP were implemented, the DQOs for confirmatory data specified in this QAPP were attained, the specified quantitation limits were achieved, and the sample holding times were met. An evaluation of data accuracy, precision, sensitivity, and completeness, based on method-specific criteria, will be performed according to the following guidance document:

- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review - USEPA, February 1994.

The procedures used to evaluate data include the following items:

- All technical holding times will be checked for inorganic analyses. Sample calculations will be checked.
- Field precision will be determined from blind field duplicate data.
- Completeness of the data package will be checked to determine that all samples and analyses required by the QAPP were processed, that the procedures specified in the QAPP were implemented, and that all deliverables specified in the QAPP are included.
- The Data Reviewer will identify any out-of-control data points and data omissions and will interact with the laboratory to correct data deficiencies.
- Decisions to repeat sample collection and analyses may be made by the RMT Project Manager based on the extent of the deficiencies and their importance in the overall context of the project.
- Data generated for the Folkertsma Refuse Site will be computerized in a format organized to facilitate data review and evaluation. The computerized data set will include the data flags provided by the analytical laboratories, as well as additional flags and comments of the third party data validator.
- The third party data reviewer will assess the usability of results against the DQOs.
- The data validation report will address the following items:
  - Overall quality and usability of the data
  - Evaluation of QC data, including precision, accuracy, and completeness of the data
  - Potential sample contamination due to blank contributions
  - Assessment of laboratory and field records
  - Actions regarding specific QC criteria exceedences



# Section 10

## Performance and System Audits

---

No change from the original QAPP and SAP except that the laboratory will be Pace Analytical Services of Green Bay Wisconsin and the consultant will be RMT, Inc., of Grand Rapids, Michigan.

# Section 11

## Preventive Maintenance

---

Preventive maintenance of the laboratory equipment is routine. Procedures are described in the QA Manual (Appendix B).

# Section 12

## Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

---

This section of the OM&M Plan is revised in part by the laboratory QA Manual and SOPs in Appendix B.

# Section 13

## Corrective Action

---

The consultant and the laboratory identified in the original QAPP have changed to RMT of Grand Rapids, Michigan and Pace Analytical Services of Green Bay, Wisconsin, respectively. The laboratory's corrective action procedures are summarized in the QA Manual.

# Section 14

## Quality Assurance Reports and Management

---

No change from the original QAPP and SAP.

# Section 15

## References

---

- MDEQ. 2000. Groundwater: residential and industrial-commercial Part 201 generic cleanup criteria and screening levels. June 7, 2000.
- USEPA. 1989. Statistical analysis of ground-water monitoring data at RCRA facilities, interim final guidance. EPA 530/SW 89-026. April 1989.
- USEPA. 1993. Statistical analysis of ground-water monitoring data at RCRA facilities, addendum to interim final guidance. EPA /530-R-93-003. January 1993.
- USEPA. 1993. Data quality objectives process for Superfund, interim final guidance. EPA 540-R-93-071. September 1993.
- USEPA. 1994. Contract laboratory program national functional guidelines for inorganic data review. USEPA, February 1994.
- USEPA. 1996. SW846-Test methods for evaluating solid waste, physical/chemical methods.
- USEPA. 1999. Five-year review for Folkertsma Refuse Site, Walker, Michigan. 12 pages. February 17, 1999.
- Warzyn. 1993. Quality assurance project plan for the environmental monitoring program, Folkertsma Refuse Site, Walker, Michigan. September 22, 1993.

**Table 4-1 (Revised March 2009)**  
**Environmental Monitoring Program**  
**Analytical Parameters, Methods, and Target Detection Limits**  
**for Groundwater and Surface Water**

Parameter	Method	Method Reference	Target Detection Limit
Volatile Organic Compounds (VOCs)	✓	8260	various
Aluminum	✓	6010/6020	50
Arsenic	✓	6010/6020/7060	20
Barium	✓	6010/6020	100
Cadmium	✓	6010/6020	0.5
Chromium (total)	✓	6010/6020	5
Cobalt	✓	6010/6020	10
Copper	✓	6010/6020	5
Iron	✓	6010/6020	100
Lead	✓	6010/6020	3
Magnesium	✓	6010/6020	100
Manganese	✓	6010/6020	20
Mercury	✓	SOW ILM03.0 <sup>(3)</sup>	0.2
Nickel	✓	6010/6020	25
Potassium	✓	6010/6020	500
Selenium	✓	6010/6020	5
Silver	✓	6020/200.9	0.2
Sodium	✓	6010/6020	1,000
Thallium	✓	6010/6020	2
Zinc	✓	6010/6020	20

Notes:

- (1) Method References: USEPA, 1996. SW 846. Test methods for evaluating solid wastes, physical/chemical methods. The most recent method revisions will be used.
- (2) From the MDEQ ERD Operational Memorandum #6, Revision 5, November 1998.
- (3) USEPA, Statement of Work for Inorganic Analysis, August 1991 (from Table 7-1 of the September 1993 QAPP).

*RMT, Inc.*

F:\PJT\00-05331\30\APPENDIXA\_533130\_001.DOC 6/15/09

*Folkertsma Refuse Site  
April 2001*



**Table 4-2 (Revised March 2009)**  
**Environmental Monitoring Program**  
**Sample Quantities, Containers, Preservatives, and Packaging Requirements**

<b>Groundwater</b>						
Volatile Organic Compounds (VOCs)	Three 40 ml, glass VOA vials	HCl to pH<2, iced to 4°C.	14 days from sampling	Fill to top, no air bubbles	Shipped daily by overnight carrier	Vermiculite or bubble wrap
Target metals <sup>(3)</sup>	One 1-liter, high-density polyethylene bottle	HNO <sub>3</sub> to pH<2, iced to 4°C.	180 days from sampling (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite or bubble wrap
<b>Surface Water</b>						
VOCs	Three 40 ml, glass VOA vials	HCl to pH<2, iced to 4°C.	14 days from sampling	Fill to top, no air bubbles	Shipped daily by overnight carrier	Vermiculite or bubble wrap
Target metals <sup>(3)</sup>	One 1-liter, high-density polyethylene bottle	HNO <sub>3</sub> to pH<2, iced to 4°C.	180 days from sampling (28 days for mercury)	Fill to shoulder of bottle	Shipped daily by overnight carrier	Vermiculite or bubble wrap

**Notes:**

- <sup>(1)</sup> The holding time begins at the time the sample is collected.
- <sup>(2)</sup> The packing material should completely cushion the sample bottles – bottom, sides, and top.
- <sup>(3)</sup> Target metals for groundwater and surface water monitoring are listed in Table 4-1.

**Table 4-3  
Groundwater Stabilization Criteria**

pH	Three successive readings within 0.2 pH unit
Conductivity	Three successive readings within 20 $\mu$ mhos/cm
Temperature	Three successive readings within 0.5°C
Turbidity	Three successive readings within 10 percent or <10 NTU
Dissolved oxygen	Three successive readings within 0.5 mg/L
Eh	Three successive readings within 30 mv

**NOTE:**

(1) Stabilization criteria may be modified during sampling based on field observations.

**Table 4-4 (Revised March 2009)  
Summary of the Sampling and Analysis Program**

Media	Parameters	Analytes	Frequency	Number of Wells				
				SW-1	SWBG-1	MW-106	MW-107R	MW-108
Groundwater	pH, Eh, specific conductance, temperature, turbidity, and dissolved oxygen	VOCs, Target metals	8 <sup>(3)</sup>	1/10	1	1/10	1	10
Surface water		VOCs, Target metals	2 <sup>(4)</sup>	1/10	1	1/10	1	4

**Notes:**

- <sup>(1)</sup> Field parameters will be measured at the time of sample collection.
- <sup>(2)</sup> Laboratory analyses will be performed by Pace Analytical Services. Specific analytes, laboratory methods, and target detection limits are listed in Table 4-1.
- <sup>(3)</sup> Samples will be collected from the following 8 wells: MW-106, MW-107R, MW-108, MW-109, MW-201, MW-206, MW-207R, and MW-208.
- <sup>(4)</sup> Samples will be collected at SW-1 and SWBG-1.

Table 4-5 (Revised March 2009)  
 GSI Criteria for the  
 Folkertsma Refuse Site

Parameter	Generic Criteria
VOCs	various
Aluminum	NA
Arsenic	150
Barium	1,037 <sup>(2)</sup>
Cadmium	9 <sup>(2)</sup>
Chromium III	216 <sup>(2)</sup>
Cobalt	100
Copper	27 <sup>(2)</sup>
Iron	NA
Lead	107 <sup>(2)</sup>
Magnesium	NA
Manganese	1,079 <sup>(2)</sup>
Mercury	0.2 <sup>(3)</sup>
Nickel	239 <sup>(2)</sup>
Potassium	NL
Selenium	5.0
Silver	0.2
Sodium	NA
Thallium	4
Zinc	493 <sup>(2)</sup>

Table 4-5 (Revised March 2009)

GSI Criteria for the  
Folkertsma Refuse Site



- (1) Generic GSI criteria published by the MDEQ, January 23, 2006.
- (2) Hardness-dependent generic GSI criterion calculated using 225 mg/L calcium carbonate for the Indian Mill Creek in Kent County. Calculations presented in Appendix C.
- (3) Generic GSI criteria is less than the Target Detection Limit, therefore, the TDL will be used as the site-specific GSI criteria.

NA = not available.

NL = parameter not listed.



# Appendix B

## Laboratory QA Manual and SOPs

---



# QUALITY ASSURANCE MANUAL

## Quality Assurance/Quality Control Policies and Procedures Revision 12.0

Pace Analytical Services – Green Bay, WI  
1241 E Bellevue Street, Suite 9  
Green Bay, WI 54302  
920-469-2436

### CORPORATE APPROVAL

Steve A. Vanderboom  
President, CEO  
1700 Elm Street SE, Suite 200  
Minneapolis, MN 55414  
(612) 607-1700

Date 1/30/09

Bruce Warden  
Director of Quality, Safety, and Training  
1700 Elm Street SE, Suite 200  
Minneapolis, MN 55414  
(612) 607-1700

Date 1/30/09

© 2002 - 2008 Pace Analytical Services, Inc. This Quality Assurance Manual may not be reproduced, in part or in full, without written consent of Pace Analytical Services, Inc. Whether distributed internally or as a "courtesy copy" to customers or regulatory agencies, this document is considered confidential and proprietary information.

Any printed documents in use within a Pace Analytical Services, Inc. laboratory have been reviewed and approved by the persons listed on the cover page. They can only be deemed official if proper signatures are present.

This is COPY# 14 distributed on 4/16/09 by KEG and is CONTROLLED or  UNCONTROLLED.





**PACE ANALYTICAL SERVICES – GREEN BAY, WI  
LOCAL APPROVAL**

This document has been approved as the Quality Assurance Manual, effective 01/29/09, as indicated by the following signatures:

*Nick Mellone*  
Laboratory General Manager  
920-469-2436

01/29/09  
Date

*Kevin E. Strand*  
Laboratory Quality Manager  
920-469-2436

1/29/09  
Date

*Alan A. Cohen*  
Laboratory Technical Director  
920-469-2436

1/29/09  
Date

*Jeffrey J. Bushman*  
Laboratory Technical Director  
920-469-2436

1/29/09  
Date

*Charlie*  
Laboratory Technical Director  
920-469-2436

1/29/09  
Date

**Additional Signatures**

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Title

\_\_\_\_\_  
Date

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Title

\_\_\_\_\_  
Date

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Title

\_\_\_\_\_  
Date

\_\_\_\_\_

## Table of Contents

---

<b>1.0 INTRODUCTION AND ORGANIZATIONAL STRUCTURE .....</b>	<b>5</b>
1.1 INTRODUCTION TO PASI .....	5
1.2 STATEMENT OF PURPOSE .....	5
1.3 QUALITY POLICY STATEMENT AND GOALS OF THE QUALITY SYSTEM.....	5
1.4 PACE ANALYTICAL SERVICES CORE VALUES.....	5
1.5 CODE OF ETHICS .....	6
1.6 STANDARDS OF CONDUCT .....	6
1.7 LABORATORY ORGANIZATION.....	7
1.8 LABORATORY JOB DESCRIPTIONS.....	8
1.9 TRAINING AND ORIENTATION .....	12
1.10 LABORATORY SAFETY.....	13
1.11 SECURITY AND CONFIDENTIALITY .....	13
<b>2.0 SAMPLE CUSTODY .....</b>	<b>14</b>
2.1 SAMPLING SUPPORT .....	14
2.2 PROJECT INITIATION.....	14
2.3 CHAIN-OF-CUSTODY.....	15
2.4 SAMPLE ACCEPTANCE POLICY.....	15
2.5 SAMPLE LOG-IN .....	17
2.6 SAMPLE STORAGE .....	17
2.7 SAMPLE PROTECTION .....	18
2.8 SUBCONTRACTING ANALYTICAL SERVICES.....	19
2.9 SAMPLE RETENTION AND DISPOSAL .....	20
<b>3.0 ANALYTICAL CAPABILITIES .....</b>	<b>21</b>
3.1 ANALYTICAL METHOD SOURCES.....	21
3.2 ANALYTICAL METHOD DOCUMENTATION.....	21
3.3 ANALYTICAL METHOD VALIDATION .....	21
3.4 DEMONSTRATION OF CAPABILITY (DOC).....	21
3.5 REGULATORY AND METHOD COMPLIANCE.....	22
<b>4.0 QUALITY CONTROL PROCEDURES.....</b>	<b>23</b>
4.1 DATA INTEGRITY SYSTEM .....	23
4.2 METHOD BLANK .....	23
4.3 LABORATORY CONTROL SAMPLE .....	24
4.4 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) .....	25
4.5 SURROGATES.....	25
4.6 SAMPLE DUPLICATE.....	26
4.7 INTERNAL STANDARDS .....	26
4.8 FIELD BLANKS.....	26
4.9 TRIP BLANKS.....	26
4.10 LIMIT OF DETECTION (LOD) .....	26
4.11 LIMIT OF QUANTITATION (LOQ) .....	27
4.12 ESTIMATE OF UNCERTAINTY.....	28
4.13 PROFICIENCY TESTING (PT) STUDIES .....	28
4.14 ROUNDING AND SIGNIFICANT FIGURES .....	28

---



<b>5.0</b>	<b>DOCUMENT MANAGEMENT AND CHANGE CONTROL.....</b>	<b>30</b>
5.1	DOCUMENT MANAGEMENT .....	30
5.2	DOCUMENT CHANGE CONTROL .....	31
<b>6.0</b>	<b>EQUIPMENT AND MEASUREMENT TRACEABILITY.....</b>	<b>32</b>
6.1	STANDARDS AND TRACEABILITY .....	32
6.2	GENERAL ANALYTICAL INSTRUMENT CALIBRATION PROCEDURES .....	32
6.3	SUPPORT EQUIPMENT CALIBRATION PROCEDURES.....	35
6.4	INSTRUMENT/ EQUIPMENT MAINTENANCE .....	36
<b>7.0</b>	<b>CONTROL OF DATA.....</b>	<b>38</b>
7.1	ANALYTICAL RESULTS PROCESSING.....	38
7.2	DATA VERIFICATION.....	38
7.3	DATA REPORTING.....	39
7.4	DATA SECURITY.....	40
7.5	DATA ARCHIVING .....	40
7.6	DATA DISPOSAL .....	40
<b>8.0</b>	<b>QUALITY SYSTEM AUDITS AND REVIEWS.....</b>	<b>41</b>
8.1	INTERNAL AUDITS .....	41
8.2	EXTERNAL AUDITS .....	42
8.3	QUARTERLY QUALITY REPORTS.....	43
8.4	ANNUAL MANAGERIAL REVIEW.....	43
<b>9.0</b>	<b>CORRECTIVE ACTION.....</b>	<b>45</b>
9.1	CORRECTIVE ACTION DOCUMENTATION.....	45
9.2	CORRECTIVE ACTION COMPLETION.....	46
<b>10.0</b>	<b>GLOSSARY .....</b>	<b>48</b>
<b>11.0</b>	<b>REFERENCES .....</b>	<b>54</b>
<b>12.0</b>	<b>REVISIONS.....</b>	<b>55</b>
	<b>ATTACHMENT I.....</b>	<b>60</b>
	<b>ATTACHMENT IIA.....</b>	<b>62</b>
	<b>ATTACHMENT IIB.....</b>	<b>63</b>
	<b>ATTACHMENT III.....</b>	<b>64</b>
	<b>ATTACHMENT IV.....</b>	<b>68</b>
	<b>ATTACHMENT V.....</b>	<b>69</b>
	<b>ATTACHMENT VI.....</b>	<b>73</b>
	<b>ATTACHMENT VII.....</b>	<b>75</b>
	<b>ATTACHMENT VIII.....</b>	<b>75</b>
	<b>ATTACHMENT VIII.....</b>	<b>76</b>

---

## 1.0 INTRODUCTION AND ORGANIZATIONAL STRUCTURE

**“Working together to protect our environment and improve our health”**

### *Pace Analytical Services Inc. - Mission Statement*

#### 1.1 Introduction to PASI

Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, industrial hygiene testing, explosives, high resolution mass spectroscopy (including dioxins, furans and coplanar PCB's), radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

#### 1.2 Statement of Purpose

To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

#### 1.3 Quality Policy Statement and Goals of the Quality System

The PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system. The overall objective of this quality system is to provide reliable data through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

All personnel within the PASI network are required to be familiar with all facets of the quality system and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel comply with all current applicable state, federal, and industry standards (such as the NELAC and ISO 17025 standards).

#### 1.4 Pace Analytical Services Core Values

- **INTEGRITY**
- **VALUE EMPLOYEES**
- **KNOW OUR CUSTOMERS**
- **HONOR COMMITMENTS**
- **FLEXIBLE RESPONSE TO DEMAND**
- **PURSUE OPPORTUNITIES**
- **CONTINUOUSLY IMPROVE**

## 1.5 Code of Ethics

PASI's fundamental ethical principles are as follows:

- Each PASI employee is responsible for the propriety and consequences of his or her actions.
- Each PASI employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where PASI does business or seeks to do business.
- Each PASI employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

Strict adherence by each PASI employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of PASI.

Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

## 1.6 Standards of Conduct

### 1.6.1 Data Integrity

The accuracy and integrity of the analytical results produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values and puts PASI and its employees at grave financial and legal risk. Therefore, employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations and databases. Employees are prohibited from making false entries or misrepresentations of data (e.g., dates, calculations, results or conclusions).

Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work; including commercial, financial, over-scheduling and working condition pressures.

### 1.6.2 Confidentiality

PASI employees must not (directly or indirectly) use or disclose confidential or proprietary information except when in connection with their duties at PASI. This is effective over the course of employment and for a period of two years thereafter.

Confidential or proprietary information, belonging to either PASI and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

### 1.6.3 Conflict of Interest

PASI employees must avoid situations that might involve a conflict of interest or appear questionable to others. The employee must be careful in two general areas:

- Participation in activities that conflict or appear to conflict with PASI responsibilities.

- Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced. This includes bribes, kickbacks or illegal payments.

Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other questionable activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company or participation in any outside business during the employee's work hours.

#### 1.6.4 Compliance

All employees are required to read, understand and comply with the various components of the standards listed in this document. As confirmation that they understand this responsibility, each employee is required to sign an acknowledgment form (either hardcopy or in electronic database) annually (or as revisions become finalized) that becomes part of the employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

#### 1.7 Laboratory Organization

The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety, and training activities. PASI's Director of Quality, Safety & Training is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

Each laboratory within the system operates with local management, but all share common systems and receive support from the Corporate Office.

A General Manager (GM) supervises each regional laboratory. Some operations may have an Assistant General Manager (AGM) in situations where the General Manager is responsible for multiple laboratory facilities and is not necessarily in the facility on a regular basis. Quality Managers (QM) at each lab report directly to their General Manager (or Assistant General Manager) but receive guidance and direction from the Director of Quality, Safety & Training.

The General Manager bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of the General Manager (and an Assistant General Manager), the Quality Manager serves as the next in command. He or she assumes the responsibilities of the GM until the GM is available to resume the duties of their position. In the absence of the GM and QM, management responsibility of the laboratory is passed to the Technical Director – provided such a position is identified – and then to the most senior department manager until the return of the GM or QM. The most senior department manager in charge may include the Client Services Manager or the Administrative Business Manager at the discretion of the General Manager.

A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory General Manager or Quality Manager has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

The Quality Manager has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set for in this Quality Assurance Manual, the Quality Manager has the authority to halt laboratory operations should he or she deem such an action necessary. The QM will immediately

communicate the halting of operations to the GM and keep him or her posted on the progress of corrective actions. In the event the GM and QM are not in agreement as to the need for the suspension, the Chief Operating Officer and Director of Quality, Safety and Training will be called in to mediate the situation.

Under the direction of the General Manager, the technical staff of the laboratory is generally organized into the following functional groups:

- Organic Sample Preparation
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Product Testing
- Equipment Maintenance
- Microbiology

Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – Green Bay is listed in Attachment IIA. In the event of a change in General Manager, Quality Manager or Technical Director(s), the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key personnel will also be noted by the additional signatures on the QAM Local Approval page. In any case, the QAM will remain in effect until the next scheduled revision.

## 1.8 Laboratory Job Descriptions

### 1.8.1 Senior General Manager

1. Oversees all functions of all the operations within their designated region.
2. Oversees the development of local General Managers within their designated region.
3. Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation.
4. Oversees the preparation of budgets and staffing plans for all operations within their designated region.
5. Ensures compliance with all applicable state, federal and industry standards.

### 1.8.2 General Manager

1. Oversees all functions of the operations.
2. Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation.
3. Prepares budgets and staffing plans.
4. Monitors the Quality Systems of the laboratory and advises the Quality Manager accordingly.
5. Ensures compliance with all applicable state, federal and industry standards.

### 1.8.3 Quality Manager

1. Oversees the laboratory Quality Systems while functioning independently from laboratory operations. Reports directly to the General Manager.
2. Monitors Quality Assurance policies and Quality Control procedures to ensure that the laboratory achieves established standards of quality.

3. Maintains records of quality control data and evaluates data quality.
4. Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives.
5. Reviews and maintains records of proficiency testing results.
6. Maintains the document control system
7. Assists in development and implementation of appropriate training programs.
8. Provides technical support to laboratory operations regarding methodology and project QA/QC requirements.
9. Maintains certifications from federal and state programs.
10. Ensures compliance with all applicable state, federal and industry standards.
11. Maintains the laboratory training records, including those in the Learning Management System (LMS).

#### **1.8.4 Technical Director**

1. Monitors the standards of performance in quality assurance and quality control data
2. Monitors the validity of analyses performed and data generated.
3. Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project
4. Serves as the general manager of the laboratory in the absence of the GM, AGM and QM.
5. Provides technical guidance in the review, development and validation of new methodologies.

#### **1.8.5 Administrative Business Manager**

1. Responsible for financial and administrative management for the entire facility.
2. Provides input relative to tactical and strategic planning activities.
3. Organizes financial information so that the facility is run as a fiscally responsible business.
4. Works with staff to confirm that appropriate processes are put in place to track revenues and expenses.
5. Provide ongoing financial information to the General Manager and the management team so they can better manage their business.
6. Utilizes historical information and trends to accurately forecast future financial positions.
7. Works with management to ensure that key measurements (mileposts) are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios.
8. Works with General Manager to develop accurate budget and track on an ongoing basis.
9. Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments.
10. Works with project management team and administrative support staff to ensure timely and accurate invoicing.

#### **1.8.6 Client Services Manager**

1. Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control.
2. Responsible for staffing and all personnel management related issues for Client Services.
3. Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure.
4. Performs or is capable of performing all duties listed for that of Project Manager.

#### **1.8.7 Project Manager**

1. Coordinates daily activities including taking orders, reporting data and analytical results.
2. Serves as the primary technical and administrative liaison between customers and PASI.



3. Communicates with operations staff to update and set project priorities.
4. Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.).
5. Works with customers, laboratory staff, and other appropriate PASI staff to develop project statements of work or resolve problems of data quality.
3. Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records.
4. Mediation of project schedules and scope of work through communication with internal resources and management.
5. Responsible for preparing routine and non-routine quotations, reports and technical papers.
6. Interfaces between customers and management personnel to achieve customer satisfaction.
7. Manages large-scale complex projects.
8. Supervises less experienced project managers and provide guidance on management of complex projects.
6. Arranges bottle orders and shipment of sample kits to customers.
7. Verifies login information relative to project requirements and field sample Chains-of-Custody.

#### **1.8.8 Project Coordinator**

1. Responsible for preparation of project specifications and provides technical/project support.
2. Coordinates project needs with other department sections and assists with proposal preparation.
3. Prepares routine proposals and invoicing.
4. Responsible for scanning, copying, assembling and binding final reports.
5. Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

#### **1.8.9 Department Manager/Supervisor**

1. Oversees the day-to-day production and quality activities of their assign department.
2. Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied.
3. Assesses data quality and takes corrective action when necessary.
4. Approves and releases technical and data management reports.
5. Ensures compliance with all applicable state, federal and industry standards.

#### **1.8.10 Group Leader/Supervisor**

1. Trains analysts in laboratory operations and analytical procedures.
1. Organizes and schedules analyses with consideration for sample holding times.
2. Implements data verification procedures by assigning data verification duties to appropriate personnel.
3. Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs.
4. Reports non-compliance situations to laboratory management including the Quality Manager.

#### **1.8.11 Laboratory Analyst**

1. Performs detailed preparation and analysis of samples according to published methods and laboratory procedures.
2. Processes and evaluates raw data obtained from preparation and analysis steps.
3. Generates final results from raw data, performing primary review against method criteria.

4. Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.
5. Reports data in LIMS, authorizing for release pending secondary approval.
6. Conducts routine and non-routine maintenance of equipment as required.
7. Performs or is capable of performing all duties associated with that of Laboratory Technician.

#### **1.8.12 Laboratory Technician**

1. Prepares standards and reagents according to published methods or in house procedures.
2. Performs preparation and analytical steps for basic laboratory methods.
3. Works under the direction of a Laboratory Analyst on complex methodologies.
4. Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies.
5. Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

#### **1.8.13 Sample Management Personnel**

1. Signs for incoming samples and verifies the data entered on the Chain-of-Custody forms.
2. Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting.
3. Stages samples according to EPA requirements.
4. Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

#### **1.8.14 Systems Administrator or Systems Manager**

1. Assists with the creation and maintenance of electronic data deliverables (EDDs).
2. Coordinates the installation and use of all hardware, software and operating systems.
3. Performs troubleshooting on all aforementioned systems.
4. Trains new and existing users on systems and system upgrades.
5. Maintains all system security passwords.
6. Maintains the electronic backups of all computer systems.

#### **1.8.15 Safety/Chemical Hygiene Officer**

1. Maintains the laboratory Chemical Hygiene Plan.
2. Plans and implements safety policies and procedures.
3. Maintains safety records.
4. Organizes and/or performs safety training.
5. Performs safety inspections and provides corrective/preventative actions.
6. Assists personnel with safety issues (e.g. personal protective equipment).

#### **1.8.16 Waste Coordinator**

1. Evaluates waste streams and helps to select appropriate waste transportation and disposal companies.
2. Maintains complete records of waste disposal including waste manifests and state reports.
3. Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.
4. Conducts a weekly inspection of the waste storage areas of the lab.

## 1.9 Training and Orientation

Each new employee receives a five part orientation: human resources, ethics and data integrity, safety, Quality Systems, and departmental.

The human resources orientation includes benefits, salary, and company policies. All records are stored with Human Resources.

The ethics and data integrity training covers the obligations of each employee to ensure the defensibility of laboratory data. Employees are provided with general policies related to ethics in the laboratory and specific examples of improper practices that are unacceptable in any PASI facility. The employee is trained to make the right decisions with regards to laboratory practices and where to go for answers in circumstances where they may be unclear as to the correct protocol.

The safety orientation includes an in-depth review of the PASI Chemical Hygiene Plan/Safety Plan, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200) and other pertinent regulations.

The Quality Systems orientation provides the new employee with information through an introduction to the Quality Assurance Manual and SOPs, acceptable record keeping practices, and the individual's responsibility to data quality. Quality Systems training is reinforced with the new employee as specific topics are covered during the departmental or analytical method training. Quality Systems training will address policies and practices that ensure the quality and defensibility of the analytical data. These topics include but are not limited to traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation and root cause analysis.

The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position.

Supervised training uses the following techniques:

- Hands-on training
- Training checklists
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.

Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability (see Section 3.4 for details on Demonstration of Capability requirements). Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the Learning Management System (LMS).

All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by lab management. Additional information can be found in SOP S-ALL-Q-020 *Orientation & Training Procedures* or its equivalent revision or replacement.

### 1.10 Laboratory Safety

It is the policy of PASI to make safety and health an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the corporate Safety Manual and Chemical Hygiene Plan.

### 1.11 Security and Confidentiality

Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Keyless door-lock combinations (and computer access codes/logins) are changed periodically. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facility must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the GM, QM or TD specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day should ensure that all outside access points to that area are secure.

Additional security is provided where necessary, e.g., specific secure areas for sample, data and customer report storage, as requested by customers or cases where national security is of concern. These areas are lockable within the facilities, or are in secure offsite storage. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out the associated internal Chain-of-Custody records.

Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for internal identification of samples and data by number only are implemented as required under contract specific Quality Assurance Project Plans (QAPPs).

All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so (i.e. federal or state subpoena).

## 2.0 SAMPLE CUSTODY

### 2.1 Sampling Support

Each individual PASI laboratory provides shipping containers, sample containers (including applicable chemical preservatives), custody documents, and field quality control samples (e.g., trip blanks) to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VIII. Note that all analyses listed are not necessarily performed at all PASI and there may be additional laboratory analyses performed that are not included in these tables. PASI – Green Bay may provide pick-up and delivery services to their customers when needed. PASI – Green Bay may provide pick-up and delivery services to customers when needed.

### 2.2 Field Services Division

Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of the other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Storm water and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

The Field Services Division operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services for a unit specific Quality Program. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

### 2.3 Project Initiation

Prior to accepting new work, the laboratory reviews performance capability. The laboratory establishes that sufficient resources (personnel, equipment capacity, analytical method capability, etc.) are available to complete the required work. The customer needs and data quality objectives are defined and appropriate environmental test methods are assured to meet customer's requirements by project managers or sales representative. Project Managers review laboratory certifications. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the General Managers and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between

customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-ALL-Q-006 *Review of Analytical Requests* or its equivalent revision or replacement.

#### 2.4 Chain-Of-Custody

A chain-of-custody (COC) (see Attachment VII) document provides the legal documentation of samples from time of collection to completion of analysis. Importance is stressed on completeness of COCs. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

Field personnel or client representatives complete a chain-of-custody form for all samples. Samples are received by the laboratory accompanied by these forms.

If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

The sampler is responsible for providing the following information on the chain-of-custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample type (matrix)
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks (if applicable)
- Custody Seal Number (if applicable)
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

The record is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain-of-custody in the "relinquished" and "received by" sections. All information except signatures is printed.

Additional information can be found in SOP S-ALL-C-001 *Sample Management* or its equivalent revision or replacement.

#### 2.5 Sample Acceptance Policy

In accordance with regulatory guidelines, PASI complies with the following sample acceptance policy for all samples received.

If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document

any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately qualified on the final report.

All samples must:

- Have unique customer identification that are clearly marked with durable waterproof labels on the sample containers and that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature
- Have the requested analyses clearly marked
- Have clear documentation of any special analysis requirements (data deliverables, etc.);
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer permission.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but  $\leq 6^{\circ}\text{C}$  (See Note 1), unless program requirements or customer contractual obligations mandate otherwise (see Note 2). The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the lab immediately after collection are considered acceptable if there is evidence that the chilling process has been started, for example by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data will be appropriately qualified on the final report.

**Note 1:** Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to  $0.1^{\circ}\text{C}$  will be read and recorded to  $\pm 0.1^{\circ}\text{C}$ . Measurements obtained from a thermometer graduated to  $0.5^{\circ}\text{C}$  will be read to  $\pm 0.5^{\circ}\text{C}$ . Measurements read at the specified precision are not to be rounded down to meet the  $\leq 6^{\circ}\text{C}$  limit (i.e.  $6.2^{\circ}\text{C}$  rounded and recorded as  $6^{\circ}\text{C}$ ).

**Note 2:** Some microbiology methods allow sample receipt temperatures of up to  $10^{\circ}\text{C}$ . Consult the specific method for microbiology samples received above  $6^{\circ}\text{C}$  prior to initiating corrective action for out of temperature preservation conditions.

Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers
- Sample condition: Intact, broken/leaking
- Sample holding time
- Sample pH when required
- Appropriate containers

Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

Additional information can be found in SOP S-ALL-C-001 *Sample Management* or its equivalent revision or replacement.

## 2.6 Sample Log-in

After sample inspection, all sample information on the chain-of-custody is entered into the Laboratory Information Management System (LIMS).

This permanent record documents receipt of all sample containers including:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of lab receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

All samples received are logged into the LIMS system within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

The Laboratory Information Management System (EPIC Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The 5 digit "X" number represents the project number followed by a 3 digit sample number. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represent the total number of containers of that particular type. Together the sample LIMS number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the client's field identification; it will be a permanent reference number for all future interactions.

Sample labels are printed from the LIMS system and affixed to each sample container.

Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or General Manager.

Additional information can be found in SOP S-ALL-C-001 *Sample Management* or its equivalent revision or replacement.

## 2.7 Sample Storage

### 2.7.1 Storage Conditions

Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross-contamination (e.g. volatile samples are stored separate from other samples). All sample fractions, extracts, leachates and



other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

#### 2.7.2 Temperature Monitoring

Samples are taken to the appropriate storage location (ambient, refrigerator, freezer) immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

The temperature of each refrigerated storage area is maintained at  $\leq 8^{\circ}\text{C}$  unless state or program requirements differ. The temperature of each freezer storage area is maintained at  $< -10^{\circ}\text{C}$  unless state or program requirements differ. The temperature of each storage area is monitored and recorded each workday. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after two hours to verify temperature exceedance. Corrective action is initiated if necessary.
- The Quality Manager and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified.
- Documentation is provided on analytical report.

#### 2.7.3 Hazardous Materials

Pure product or potentially heavily contaminated samples are tagged as "hazardous" or "lab pack" and are stored separately from other samples.

#### 2.7.4 Foreign/Quarantined Soils

Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are segregated. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure.

Additional information can be found in SOP S-ALL-C-001 *Sample Management* and SOP S-GB-S-001 *Regulated Soil* or its equivalent revision or replacement.

### 2.8 Sample Protection

PASI laboratory facilities are operated under controlled access to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted.

Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

Upon customer request, additional and more rigorous chain of custody protocols for samples and data can be implemented. For example, some projects may require complete documentation of sample custody within the secure laboratory.

Additional information can be found in SOP S-ALL-C-001 *Sample Management* or its equivalent revision or replacement.

## 2.9 Subcontracting Analytical Services

Every effort is made to perform chemical analyses for PASI customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory (inside or outside the PASI network) becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. Customers are notified in writing of the lab's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations.

Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

Additional information can be found in SOP S-ALL-Q-027 *Evaluation & Qualification of Vendors* or its equivalent revision or replacement. The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include :

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turnaround time requirements

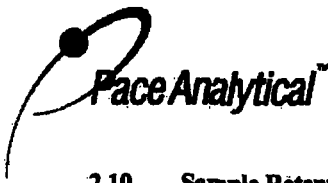
Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions (quick turn-around, required detection or reporting limits, unusual information known about the samples or analytical procedure).
- Signature in "Relinquished By"

All subcontracted sample data reports are sent to the PASI Project Manager.

Any Pace Analytical work sent to other labs within the PASI network is handled as subcontracted work (also known as inter-regional) and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-NELAC work is clearly identified. PASI will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

Additional information can be found in SOP S-ALL-Q-017 *Subcontracting Samples* or its equivalent revision or replacement.



## 2.10 Sample Retention and Disposal

Samples (and sample by-products) must be retained by the laboratory for a period of time necessary to protect the integrity of the sample or sample by-product (e.g. method holding time) and to protect the interests of the laboratory and the customer.

Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The sample retention time is a minimum of 45 days from receipt of the samples. Samples requiring storage beyond this time due to special requests or contractual obligations will not be stored under temperature controlled conditions unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires PASI to dispose of excess samples, PASI will arrange for proper disposal by an approved contractor.

Additional information can be found in SOP S-ALL-S-002 *Waste Handling* and S-ALL-C-001 *Sample Management* or their equivalent revisions or replacements.

## 3.0 ANALYTICAL CAPABILITIES

### 3.1 Analytical Method Sources

PASI laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, and State Agencies. Section 11 of this manual is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

### 3.2 Analytical Method Documentation

The primary form of documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

### 3.3 Analytical Method Validation

In some situations, PASI develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods (e.g. methods other than EPA, NIOSH, ASTM, AOAC, etc.) are required for specific projects or analytes of interest, or when the laboratory develops a method, or modifies a standard method, the laboratory validates the method prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method validation include determination of the limit of detection and limit of quantitation, evaluation of precision and bias, and evaluation of selectivity of each analyte of interest.

### 3.4 Demonstration of Capability (DOC)

Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel or test method (when a defined 'work cell' is in operation, the entire work cell must meet the criteria). The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard is calculated and compared to method criteria (if available) or established lab criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability and corresponding raw data for future reference and must document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

Alternative demonstration of capability procedures may be used for IDOC for methods that don't lend themselves to the "4 replicate" approach. For methods that only measure precision, the precision of four laboratory duplicate pairs will be assessed. The relative percent differences must be within the method acceptance limits. For procedures like TCLP or SPLP, the analyst will demonstrate making the buffered solution and performing the tumbling process. The trainer or supervisor will sign-off on demonstration of

capability of the tumbling process. Additional demonstration of capability options will be specified in Section 14 – Method Performance of the applicable method SOP.

For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples or any of the approaches utilized for IDOCs. For methods or procedures that do not lend themselves to the “4 replicate” approach, the demonstration of capability requirements will be specified in Section 14 – Method Performance of the applicable SOP.

Pace Analytical utilizes a peer review system for data review and approval. The data review staff are qualified to validate data conversion, transcription, and reporting in addition to assessing deviations from the standard operating procedures. The data review staff are familiar with the analytical method procedures with documentation maintained in their training files. The data reviewers also utilize a method specific checklist which contains the quality control acceptance criteria. Deviations from the standard operating procedure are documented on the checklist by the analyst. Further data review guidance is provided in SOP S-GB-Q-003 Data Reduction, Validation and Reporting in the Environmental Lab.

### **3.5 Regulatory and Method Compliance**

PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the Chain-of-Custody submitted with samples.

PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision-making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

It is PASI policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

## 4.0 QUALITY CONTROL PROCEDURES

### 4.1 Data Integrity System

The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to providing a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1. A data integrity training program: Standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics within this training include:
  - o Need for honesty in analytical reporting
  - o Process for reporting data integrity issues
  - o Specific examples of unethical behavior and improper practices
  - o Documentation of non-conforming data that is still useful to the data user
  - o Consequences and punishments for unethical behavior
  - o Examples of monitoring devices used by management to review data and systems
2. Signed data integrity documentation for all employees: This includes a quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual. The quiz along with the employee's electronic signature of agreement are maintained within the Learning Management System.
3. In-depth, periodic monitoring of data integrity: Including peer data review and validation, internal data audits, proficiency testing studies, etc.
4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be available for review for lab assessors and must be retained for a minimum of five years.

PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over-scheduling, and working condition pressures.

Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality, Safety and Training who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. The message line voice mail box is available at 612-607-6427.

### 4.2 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system. The method blank is processed through all preparation and analytical steps with its associated samples.

A method blank is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The method blank consists of a matrix similar to the associated samples that is known to be free of the analytes of interest. Laboratories will characterize a representative matrix as "clean" if the matrix contains contaminants at less than  $\frac{1}{2}$  the laboratory's reporting limit.

Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the

reporting limit and is greater than 1/10 of the amount of that analyte found in any associated sample for Inorganic Analysis. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater than 1/20 of the amount of that analyte found in any associated sample for Organic Analysis. Corrective actions include the re-preparation and re-analysis of all the samples (where possible) along with the full set of required quality control samples. Data qualifiers must be applied to any result reported that is associated with a contaminated method blank.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.3 Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

An LCS is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

The LCS contains all analytes specified by a specific method or by the customer or regulatory agency (which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix compounds when a full Appendix list of compounds is requested). In the absence of specified components, the lab will spike with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
  - For methods with 1-10 target compounds, the lab will spike with all compounds
  - For methods with 11-20 target compounds, the lab will spike with at least 10 compounds or 80%, whichever is greater
  - For methods with greater than 20 compounds, the lab will spike with at least 16 compounds.

The LCS is evaluated against the method default or laboratory-derived acceptance criteria. Method default control limits will be used until the laboratory has a minimum of 20 (preferably greater than 30) data points from which to derive internal criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier.

For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). NELAC has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but less than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria (this is a NELAC allowance). When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS (if possible) or reported with appropriate data qualifiers.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch (see LCS).

A Matrix Spike/Matrix Spike Duplicate (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per general matrix (i.e. soil, water, biota, etc.) per method.

The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Lab personnel spike customer samples that are specifically designated as MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible.

The MS and MSD contain all analytes specified by a specific method or by the customer or regulatory agency. In the absence of specified components, the lab will spike with the same number of compounds as previously discussed in the LCS section.

The MS and MSD are evaluated against the method or laboratory-derived criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site-specific information.

A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.5 Surrogates

Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

Surrogates are added to each customer sample (for organics), method blank, LCS and MS prior to extraction or analysis. The surrogates are evaluated against the method or laboratory-derived acceptance criteria. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-



analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.6 Sample Duplicate

A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

The sample and duplicate are evaluated against the method or laboratory-derived criteria for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.7 Internal Standards

Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

#### 4.8 Field Blanks

Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinseate blanks, or equipment blanks. The lab analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limit.

#### 4.9 Trip Blanks

Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the lab. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

#### 4.10 Limit of Detection (LOD)

PASI laboratories are required to use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. All sample-processing steps of the preparation and analytical methods are included in this determination. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or

when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

Where specifically stated in the published method, LODs (or MDLs) will be performed at the listed frequency.

The validity of the LOD must be verified by detection (a value greater than zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available (e.g. temperature).

The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the lab can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the lab must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The lab must verify the LOD on each instrument used for the reporting of sample data.
- The lab must be able to qualitatively identify all target analytes in the verification standard (distinguishable from noise).

Additional information can be found in SOP S-ALL-Q-004 *Method Detection Limit Studies* or its equivalent revision or replacement.

#### 4.11 Limit of Quantitation (LOQ)

A limit of quantitation (LOQ) for every analyte of concern must be determined. For PASI laboratories, this LOQ is referred to as the RL, or Reporting Limit. This RL is based on the lowest calibration standard concentration that is used in each initial calibration. Results below this level are not allowed to be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g. J flag).

There must be a sufficient buffer between the LOD and the limit of quantitation (LOQ). The LOQ must be higher than the LOD.

To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with target analytes at the concentration(s) equivalent to or less than the RL(s). This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits.

Additional information can be found in SOP S-ALL-Q-004 *Method Detection Limit Studies* or its equivalent revision or replacement.

#### 4.12 Estimate of Uncertainty

PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-GB-Q-010 *Estimation of Uncertainty* or its equivalent revision or replacement.

The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

#### 4.13 Proficiency Testing (PT) Studies

PASI laboratories participate in the NELAC-defined proficiency testing program. PT samples are obtained from approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

The lab initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the Quality Manager. A corrective action plan (including re-analysis of similar samples) is initiated and this report is sent to the appropriate state accreditation agencies for their review.

PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

Additional information can be found in SOP S-ALL-Q-010 *PE/PT Program* or its equivalent revision or replacement.

#### 4.14 Rounding and Significant Figures

In general, the PASI laboratories report data to no more than three significant digits. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program (Excel, etc.).

##### Rounding

PASI-Green Bay follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).



- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

#### **Significant Digits**

PASI-Green Bay follows the following convention for reporting to a specified number of significant figures. Unless specified by federal, state or local requirements or on specific request by a customer, the laboratory reports:

- Values  $> 10$  – Reported to 3 significant digits
- Values  $\leq 10$  – Reported to 2 significant digits



## 5.0 DOCUMENT MANAGEMENT AND CHANGE CONTROL

### 5.1 Document Management

Additional information can be found in SOP S-ALL-Q-002 Document Management or its equivalent revision or replacement.

Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures, Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system.

A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 Document Numbering.

As an alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the local or wide-area network (LAN or WAN). These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Electronic documents (i.e. pdf's) must be locked from printing. All hardcopy SOPs must be obtained from the Quality Department.

#### 5.1.1 Quality Assurance Manual (QAM)

The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the regional Quality Managers. The regional management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality, the General Manager, Quality Manager and Technical Director(s) sign the Quality Assurance Manual. Each regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis by all of the PASI Quality Managers and revised accordingly by the Director of Quality, Safety and Training.

#### 5.1.2 Standard Operating Procedures (SOPs)

SOPs fall into two categories: company-wide documents (starting with the prefix S-ALL-) and facility-specific documents (starting with the individual facility prefix).

The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the regional Quality Managers.



The regional management personnel sign the company-wide SOPs. The regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies.

Regional PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The regional facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The regional facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the regional Quality Manager according to the corporate document management policies.

SOPs are reviewed every two years at a minimum (a more frequent review may be required by state or federal agencies or customers). A review of the document does not necessarily constitute a re-issue of a new revision. Documentation of this review and any applicable revisions are made in the last section of each SOP. This provides a historical record of all revisions.

All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all PASI employees use the most current version of each SOP and provides the Quality Manager with a historical record of each SOP.

Additional information can be found in SOP S-ALL-Q-001 Preparation of SOPs or its equivalent revision or replacement.

### **5.1.3 Other Documentation**

Additional documents such as Forms and Spreadsheets are controlled through the document management system.

## **5.2 Document Change Control**

Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

All controlled copies of the previous document are replaced with controlled copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.





## 6.0 EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the (Green Bay) PASI facility.

### 6.1 Standards and Traceability

Each PASI facility retains all pertinent information for standards, reagents and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation and use.

Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique PASI identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

If a second source standard is required to verify an existing calibration or spiking standard, this standard is purchased from a different supplier. If no second source is available, a second standard from a different lot may be purchased from the same supplier if the lot can be demonstrated as prepared independently from other lots.

Additional information concerning standards and reagent traceability can be found in the SOP S-ALL-Q-025 Standard and Reagent Preparation and Traceability or its equivalent revision or replacement.

### 6.2 General Analytical Instrument Calibration Procedures

All types of support equipment and instrumentation are calibrated or checked before use to ensure proper functioning and verify that the laboratory's requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers (e.g. J flag) or explained in a narrative. The Minnesota Department Health requires that the reporting limit be verified upon initial calibration and monthly thereafter. The reporting limit verification must be within  $\pm 40\%$  of the true value of the reporting limit standard. The reporting limit may need to be adjusted accordingly to meet this criteria. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data

qualifiers (e.g. E flag) or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the Quality Manager. If the investigation indicates sample results have been impacted, the customer is notified within 30 days. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or replaced.

Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

#### **6.2.1 General Organic Calibration Procedures**

Calibration standards are prepared at a minimum of five concentrations for organic analyses. Results from all calibration standards must be included in constructing the calibration curve with the following exceptions:

- The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve.
- The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve.
- Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remain as established by method or standard operating procedure. The reporting limit or quantitation range, which is appropriate, must be adjusted accordingly.
- Results from a concentration level between the lowest and highest calibration levels can be excluded from the calibration curve for an acceptable cause with approval from the responsible department supervisor if the results for all analytes are excluded and the point is replaced by re-analysis. Re-analysis must occur within the same 12 hour time period for GC/MS methodologies and within 8 hours of the initial analysis for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed.

Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples.

Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. Calibration verification is performed at the beginning and end of each analytical batch (except if an internal standard is used only one verification at the beginning of the batch is needed), whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that contain a calibration verification requirement. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence is continued. If both CCVs fail, the analytical sequence is terminated. All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

When instruments are operating unattended, the autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. (The 12 hour clock begins with the injection of the second CCV.)
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples preceded by the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

### 6.2.2 General Inorganic Calibration Procedures

The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range is established,
- Zero point and single point calibration standards are analyzed with each batch

- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards. A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

A calibration blank is also analyzed with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs and CCBs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

### **6.3 Support Equipment Calibration Procedures**

All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications.

#### **6.3.1 Analytical Balances**

Each analytical balance is checked and calibrated annually by a qualified service technician. The calibration of each balance is checked each day of use with weights traceable to NIST. Calibration weights are ASTM Class 1 (or other class weights that have been calibrated against a NIST standard weight) and are re-certified annually against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

#### **6.3.2 Thermometers**

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified, at a minimum, yearly with equipment directly traceable to NIST.

Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

Laboratory thermometer inventory and calibration data are maintained in the Quality department.

### 6.3.3 pH/Electrometers

The meter is calibrated before use each day, using fresh buffer solutions. Additional information regarding Ph/Electrometers can be found in SOP S-ALL-GB-I-015 *Measurement of pH in Water, Soil, and Waste*.

### 6.3.4 Spectrophotometers

During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

### 6.3.5 Mechanical Volumetric Dispensing Devices

Mechanical volumetric dispensing devices including bottle top dispensers, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. Non-Class A glassware and disposable pipettes must be calibrated once per lot prior to first use. The accuracy of glass microliter syringes is verified and documented prior to use.

Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-ALL-Q-013 Support Equipment or its equivalent revision or replacement.

## 6.4 Instrument/ Equipment Maintenance

The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

The Laboratory Operations Manager and department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems and coordinate instrument repair and maintenance. The analysts have a primary responsibility to perform routine maintenance.

To minimize downtime and interruption of analytical work, preventative maintenance is routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation are, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

When maintenance is performed to repair an instrument problem, depending on the initial problem, demonstration of return to control may be satisfied by the successful analysis of a reagent blank or continuing calibration standard. The entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily.

## 7.0 CONTROL OF DATA

Analytical results processing, verification and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a well-defined, well-documented multi-tier review process prior to being reported to the customer. This section describes procedures used by PASI for translating raw analytical data into accurate, final sample reports and PASI data storage policies.

### 7.1 Analytical Results Processing

When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g. Run log or Instrument log) or copies of computer-generated printouts are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the lab chooses to minimize paper usage, these records can be kept as electronic records. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting discrepancies in logbooks and as footnotes or narratives, and uploading analytical results into the LIMS.

The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain-of-custody forms, and logbook copies.

Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

### 7.2 Data Verification

Data verification is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated and that any discrepancies are properly documented.

Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS.

The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data reviewer, the reviewer must:

1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note
2. Have a DOC on file for a similar method/technology (i.e. GC/MS) and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note
3. Supervise or manage a Department and have an SOP acknowledgement form on file for the method/procedure being reviewed; or,



4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

**Note:** Secondary reviewer status must be approved personally by the Quality Manager or General Manager in the event that this person has no prior experience on the specific method or general technology (i.e. GC/MS).

This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer also validates the data entered into the LIMS.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

### 7.3 Data Reporting

All data segments pertaining to a particular PASI project number are delivered to the Client Services Department (Project Manager) for assembly into the final report. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. A standard PASI final report consists of the following components:

1. A title which designates the report as "Final Report", "Laboratory Results", "Certificate of Results", etc.
2. Name and address of laboratory (or subcontracted laboratories, if used).
3. Phone number and name of laboratory contact where questions can be referred.
4. A unique number for the report (project number). The pages of the report shall be numbered and a total number of pages shall be indicated (usually in the cover letter).
5. Name and address of customer and name of project (if applicable).
6. Unique identification of samples analyzed (including customer sample numbers).
7. Identification of any sample that did not meet acceptable sampling requirements (from NELAC or other governing agency), such as improper sample containers, holding times missed, sample temperature, etc.
8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less.
9. Identification of the test methods used.
10. Identification of sampling procedures if sampling was conducted by the laboratory.
11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data.
12. Identification of whether calculations were performed on a dry or wet-weight basis.
13. Reporting limits used.
14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.
15. A signature and title of person accepting responsibility for the content of the report (can be an equivalent electronic identification) and date report was issued.
16. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory.
17. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory.
18. Identification of all test results provided by a subcontracted laboratory or other outside source.

19. Identification of results obtained outside of quantitation levels.

Any changes made to a final report shall be designated as "Revised" or equivalent wording. The laboratory must keep sufficient archived records of all lab reports and revisions. For higher levels of data deliverables, a copy of all applicable raw data is sent to the customer along with a final report of results. When possible, the PASI facility will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

The following positions are the only approved signatories for PASI final reports:

- Senior General Manager
- General Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

7.4 **Data Security**

All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and other information used to produce the technical report are maintained secured and retrievable by the PASI facility.

7.5 **Data Archiving**

All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis and personnel involved. NELAP-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the Quality Manager or a designated Data Archivist.

Records that are computer-generated have either a hard copy or electronic write-protected backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6 **Data Disposal**

Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements.

## 8.0 QUALITY SYSTEM AUDITS AND REVIEWS

### 8.1 Internal Audits

#### 8.1.1 Responsibilities

The Quality Manager is responsible for designing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be functionally independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The Quality Manager evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted by the Director of Quality, Safety and Training and/or designee. The corporate audits will focus on the execution of the Quality System as outlined in this manual but may also include other quality programs applicable to each laboratory.

#### 8.1.2 Scope and Frequency of Internal Audits

Internal systems audits are conducted yearly at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality-related system as applied throughout the laboratory.

Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual and all applicable addenda
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, standards, and associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.
- General procedures for data security, review, documentation, reporting and archiving.
- Data integrity issues such as proper manual integrations.

When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain-of-custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

A representative number of data audits are completed annually. The report format of any discrepancy is similar to that of other internal audits.

The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner until

such time as a follow up evaluation, full investigation, or other appropriate actions are completed and the issues clarified. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected, the impact on the data, the corrective actions taken by the lab and which final reports had to be re-issued. Customers are notified within 30 days when the investigation indicates analytical results are affected.

### 8.1.3 Internal Audit Reports and Corrective Action Plans

Additional information can be found in SOP S-ALL-Q-011 *Audits and Inspections* or its equivalent revision or replacement.

A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the Quality Manager writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within 3 business days, if investigations show that the laboratory results may have been affected.

Once completed, the internal audit report is issued jointly to the Laboratory General Manager and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The Quality Manager may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

The Quality Manager reviews the audit responses. If the response is accepted, the Quality Manager uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the Quality Manager determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

To complete the audit process, the Quality Manager performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been verified. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

## 8.2 External Audits

PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications, and by customers to maintain appropriate specific protocols.

Audit teams external to the company review the laboratory to assess the existence of systems and degree of technical expertise. The Quality Manager and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the Quality Manager. The Laboratory General Manager provides the necessary resources for staff to develop and implement the corrective action plans. The Quality Manager collates this information

and provides a written report to the audit team. The report contains the corrective action plan and expected completion dates for each element of the plan. The Quality Manager follows-up with the laboratory staff to ensure corrective actions are implemented.

### 8.3 Quarterly Quality Reports

The Quality Manager is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Results of internal systems or performance audits
- Corrective action activities
- Discussion of QA issues raised by customers
- Results of third party or external audits
- Status of laboratory certifications
- Proficiency Testing Study Results
- Results of internal laboratory review activities
- Summary of holding time violations
- Method detection limit study status
- Training activity summary
- SOP revision summary
- 3P Implementation summary (internal program)
- Other significant Quality System items

The Corporate Director of Quality, Safety & Technology utilizes the information from each laboratory to make decisions impacting the Quality Systems of the company as a whole. Each General Manager utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

Additional information can be found in SOP S-ALL-Q-014 *Quality System Review* or its equivalent revision or replacement.

### 8.4 Annual Managerial Review

A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

The managerial review must include the following topics of discussion:

- Policy and procedure suitability
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventative actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints

This managerial review must be documented for future reference by the Quality Manager and copies of the report are distributed to laboratory staff. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed timescale.



## 8.5 Customer Service Reviews

As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the lab management in order for them to evaluate and improve their management system, testing activities and customer service.

In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the lab's performance in relation to the work being performed for the customers.

## 9.0 CORRECTIVE ACTION

Additional information can be found in SOP S-ALL-Q-012 *Corrective Action/Preventive Action Process* or its equivalent revision or replacement.

During the process of sample handling, preparation and analysis, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of PASI provides systematic procedures for documentation, monitoring and completion of corrective actions. This can be done using PASI's LabTrack system that lists among other things, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

### 9.1 Corrective Action Documentation

The following items are examples of laboratory deviations or non-conformances that warrant some form of documented corrective action:

- Quality Control data outside of acceptance criteria
- Sample Acceptance Policy deviations
- Missed holding times
- Instrument failures (including calibration failure)
- Sample preparation or analysis errors
- Sample contamination
- Errors in customer reports
- Audit findings (internal and external)
- Proficiency Testing (PT) sample failures
- Customer complaints or inquiries

Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g. matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventative Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance problem, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance problem. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within Lab Track or on the Corrective/Preventative Action Report.

After all the documentation is completed, the routing of the Corrective/Preventative Action Report and /or Lab Track will continue from the person initiating the corrective action, to their immediate supervisor or the Project Manager and finally to the Quality Manager, who is responsible for final review and signoff of all formal corrective/preventative actions.

## **9.2 Corrective Action Completion**

### **9.2.1 Quality Control outside of acceptance criteria**

The analyst that is generating or validating Analytical data is responsible for checking the results against established acceptance criteria (quality control limits). The analyst must immediately address any deficiencies discovered. Method blank, LCS or matrix spike failures are evaluated against method, program, and customer requirements and appropriate footnotes are entered into the LIMS system. Some deficiencies may be caused by matrix interferences. Where possible, matrix interferences are confirmed by re-analysis.

Quality control deficiencies must be made known to the customer on the final report for their review of the data for usability. If appropriate, the supervisor is alerted to the QC failure and if necessary a formal corrective action can be initiated. This may involve the input of the Quality Manager or the General Manager.

The department supervisor and/or Operations Manager are responsible for evaluating the source of the deficiency and for returning the analytical system to control. This may involve instrument maintenance, analytical standard or reagent evaluation, or an internal audit of the analytical procedure.

See applicable analytical SOPs for further guidance on QC acceptance criteria.

### **9.2.2 Sample Acceptance Policy deviations**

Any deviation from the Sample Acceptance Policy listed in this Manual must be documented on the Chain-of-Custody or other applicable form by the sample receiving personnel or by the Project Manager. Analysts or supervisors that discover such deviations must contact the sample receiving personnel or appropriate Project Manager so they can initiate the proper documentation and customer contact. If a more formalized corrective action must be documented, the Quality Manager is made aware of the situation.

The customer is notified of these deviations as soon as possible so they can make decisions on whether to continue with the sample analysis or re-sample. Copies of this documentation are included in the project file.

### **9.2.3 Missed holding times**

In the event that a holding time requirement has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the Quality Manager must be made aware of these hold time exceedances.

The Project Manager must contact the customer for appropriate decisions to be made with the resolution documented and included in the customer project file. The Quality Manager includes a list of all missed holding times in their Quarterly Report to the corporate office.

### **9.2.4 Instrument Failures**

In the event of an instrument failure that either causes the necessity for re-analysis or questions the validity of generated results, a formal corrective action must be initiated. The analyst and supervisor evaluate any completed data for validity and usability. They are also responsible for returning the instrument to valid operating condition and for documenting that the system is in control (e.g. acceptable calibration verification).



#### **9.2.5 Sample Preparation or Analysis errors**

When there is an error in the preparation or analysis of samples, the analyst evaluates the impact on the usability of the analytical data with the assistance of the supervisor or manager. The affected samples will be re-processed or re-analyzed under acceptable conditions. In the event that no additional sample is available for re-analysis, the customer must be contacted for their decision on how to proceed. Documentation may take the form of footnotes or a formal corrective action form.

#### **9.2.6 Errors in customer reports**

When an error on the customer report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g. incorrect analysis reported, reporting units are incorrect, reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

#### **9.2.7 Audit findings**

The Quality Manager is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for reporting back to the auditing body, the root cause of the issue, and the corrective action taken to resolve the findings. The Quality Manager is also responsible for providing any back-up documentation used to prove that a corrective action has been completed.

#### **9.2.8 Proficiency Testing failures**

Any PT result returned to the Quality Manager as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The Quality Manager reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the Quality Manager and reported to the applicable regulatory authorities.

#### **9.2.9 Customer Complaints**

Project Managers are responsible for issuing corrective action forms for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor. After the corrective actions have been listed, the Project Manager reviews the corrective action to determine if the customer needs or concerns are being addressed.

### **9.3. Preventive Action Documentation**

Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems (technical, managerial, quality, etc.). These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems are discovered. These improvements can be made within a department or lab-wide.
- Annual managerial reviews- part of this NELAC-required review is to look at all processes and procedures used by the lab over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

When improvement opportunities are identified or if preventive action is required, the lab can develop, implement, and monitor preventive action plans.

## 10.0 GLOSSARY

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity and Performance. Best Practices are identified that can be used by all PASI labs.
Accuracy	The agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	A portion of a sample taken for analysis.
Analyte	The specific chemical species or parameter an analysis seeks to determine.
Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	A sample for submitted for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test analyst or laboratory proficiency in the execution of the measurement process.
Calibration	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard must bracket the range of planned or expected sample measurements.
Calibration Curve	The graphic representation of known values, such as concentrations for a series of calibration standards and their instrument response.
Chain-of-Custody (COC)	A record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, collector, time of collection, preservation, and requested analyses.
Confirmation	Verification of the identity of a component through the use of an alternate scientific approach from the original method. These may include, but are not limited to: <ul style="list-style-type: none"> <li>• second-column confirmation</li> <li>• alternate wavelength</li> <li>• derivatization derivative</li> <li>• mass spectral interpretation</li> <li>• additional cleanup procedures</li> </ul>
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.

Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is:  $\% \text{ Completeness} = (\text{Valid Data Points} / \text{Expected Data Points}) * 100$
Calibration Verification	The process of verifying a calibration by analysis of standards and comparing the results with the known amount.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of nonconforming data.
Corrective Action	The action taken to eliminate the causes of a nonconformity, defect, or other undesirable situation in order to prevent recurrence.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate acceptable accuracy.
Detection Limit (DL)	General term for the lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not a false positive value. See definitions for Method Detection Limit and Limit of Detection.
Document Control (Management)	Procedures to ensure that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled (managed) to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate or Replicate Analysis	The identically performed measurement on two or more sub-samples of the same sample within a short interval of time
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: <ul style="list-style-type: none"> <li>• Non Potable Water ( Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts)</li> <li>• Drinking Water - Delivered (treated or untreated) water designated as potable water</li> <li>• Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents</li> <li>• Sludge - Municipal sludges and industrial sludges.</li> <li>• Soil - Predominately inorganic matter ranging in classification from sands to clays.</li> <li>• Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes</li> </ul>
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.

Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Holding Time	The maximum time that samples may be held prior to preparation and/or analysis as defined by the method.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Internal Standards	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
Laboratory Control Sample (LCS)	A blank sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. Sometimes referred to as <b>Laboratory Fortified Blank, Spiked Blank or QC Check Sample.</b>
Limit of Detection (LOD)	An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent.
Limit of Quantitation (LOQ)	The minimum levels, concentrations or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each lab's learn centers are maintained by a local administrator.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.

Matrix	<p>The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions are used:</p> <ul style="list-style-type: none"> <li>• <b>Aqueous or Non-Potable Water:</b> any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.</li> <li>• <b>Drinking Water:</b> any aqueous sample that has been designated a potable or potentially potable water source.</li> <li>• <b>Saline/Estuarine:</b> any aqueous sample from an ocean or estuary, or other saltwater source.</li> <li>• <b>Non-aqueous liquid:</b> any organic liquid with &lt;15% settleable solids.</li> <li>• <b>Biological Tissue:</b> any sample of a biological origin such as fish tissue, shellfish or plant material. Such sample can be grouped according to origin.</li> <li>• <b>Solid:</b> includes soils, sediments, sludges, and other matrices with &gt;15% settleable solids.</li> <li>• <b>Chemical Waste:</b> a product or by-product or an industrial process that results in a matrix not previously defined</li> <li>• <b>Air and Emissions:</b> whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas vapor that are collected with a sorbent tube, impinger solution, filter, or other device.</li> </ul>
Matrix Spike (MS)	A sample prepared by adding a known quantity of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency. (sometimes referred to as Spiked Sample or Fortified Sample)
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte. (sometimes referred to as Spiked Sample Duplicate or Fortified Sample Duplicate)
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures; and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Limit of Detection (LOD); defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures required by a specific project.
Quality Assurance (QA)	An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC)	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.
Quality Control Sample	A sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.
Quality Assurance Manual	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g. tapes which have been transcribed verbatim, dated and verified accurate by signature), the exact copy or exact transcript may be submitted.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Reference Standard	A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels (concentrations) of a variable of interest.
Standard	A substance or material with properties known with sufficient accuracy to permit its use to evaluate the same property in a sample.

Standard Blank	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Operating Procedure (SOP)	A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Traceability	The property of a material or measurement result defining its relationship to recognized international or national standards through an unbroken chain of comparisons.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand ( i.e. the concentration of an analyte).

## 11.0 REFERENCES

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (September 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
- Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988
- National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent
- ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.





## 12.0 REVISIONS

The PASI Corporate Quality and Safety Manager files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual Revision 10.0	<p>Throughout the document, Pace was replaced with PASI or in some cases with Pace Analytical. Also, corrections were made to wording, grammar, spelling, and formatting.</p> <p><b>SECTION 1:</b></p> <ul style="list-style-type: none"> <li>• Updated the PASI mission statement</li> <li>• Deleted Financial Responsibility, Drug-Free Workplace, Non-Harassment, Proper and Professional Conduct, Protection of Property, and Communication sections.</li> <li>• Added Assistant General Manager/ Operations Manager, Technical Director, Administrative Business Manager, Project Manager, Project Coordinator, Field Analyst, Laboratory Technician &amp; Field Technician job descriptions</li> <li>• Added detailed Chain of Command to Laboratory Organization section</li> <li>• Updated the Training and Orientation section to reflect current practices</li> <li>• Deleted a portion of the Laboratory Safety section and added a reference to the Safety Manual and Chemical Hygiene Plan.</li> </ul> <p><b>SECTION 2:</b></p> <ul style="list-style-type: none"> <li>• Switched the order of Chain of Custody and Sample Acceptance Policy sections</li> <li>• Added details of project review documentation to Project Initiation section</li> <li>• Added steps to sample log in</li> </ul> <p><b>SECTION 3:</b></p> <ul style="list-style-type: none"> <li>• Deleted reference to local addenda for companywide SOPs</li> <li>• Rearranged sentences</li> <li>• Added "PASI will not be liable if the customer chooses not to follow PASI recommendations" to the Regulatory and Method Compliance section.</li> </ul> <p><b>SECTION 4:</b></p> <ul style="list-style-type: none"> <li>• Added details to the documentation of review or investigation of possible data integrity.</li> <li>• Corrected wording in Method Blank section</li> <li>• Deleted from LCS/LCSD section an out-of-control statement that said affected samples associated with a failing LCS must be re-analyzed</li> </ul> <p><b>SECTION 5:</b></p> <ul style="list-style-type: none"> <li>• Added "Electronic documents must be readily accessible to all facility employees" to Documents Management section</li> <li>• Updated the Standard Operating Procedure section to describe the new PASI corporate SOP Templates and distribution.</li> </ul> <p><b>SECTION 6:</b></p> <ul style="list-style-type: none"> <li>• Re-organized &amp; re-named sections</li> <li>• Updated the interpretation of the Calibration Verification policy</li> <li>• Added clarification to the definition of the Second Source Standard</li> <li>• Revised Single Point Calibration procedure to address NELAC requirement</li> <li>• Incorporated Spare Parts into Instrument/ Equipment Maintenance</li> </ul> <p><b>SECTION 7:</b></p>	20Jun2006

Document Number	Reason for Change	Date
	<ul style="list-style-type: none"> <li>• Updated Analytical Results Processing section to clarify data documentation policy.</li> <li>• Deleted "All data that are manually entered into the LIMS is reviewed at a rate of 100%" and deleted the use of checklists statement from Data Verification section</li> <li>• Integrated paragraphs for better flow</li> <li>• Deleted item # 15, "If required, a statement of the estimated uncertainty of the test results." from the Data Reporting section</li> <li>• Added Data Security section to describe PASI data security practices</li> <li>• Added fire, flood, and vermin protection requirement to Data Archiving section</li> <li>• Added statement to Data Archiving section describing that NELAP related records are available to accrediting authorities.</li> <li>• Added Data Disposal section</li> </ul> <p><b>SECTION 8:</b></p> <ul style="list-style-type: none"> <li>• Deleted first paragraph stating that Pace labs are subject to internal and external audits and reviews.</li> <li>• Added description of PASI internal audit program and investigations</li> <li>• Added requirement that corrective action be taken and customer notified within 3 days if audit findings show that test results may have been affected</li> <li>• Updated requirement for manager(s)/supervisor(s) to respond to audit findings with a plan to correct all deficiencies within 14 days. Statement included that allows Quality Manager to grant additional time for response.</li> <li>• Added to Annual Managerial Review section that "The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed timescale."</li> </ul> <p><b>SECTION 9:</b></p> <ul style="list-style-type: none"> <li>• Added documentation requirement for reporting discovery of deficiency or non-conformance, must be documented "on the Non-Conformance Corrective/ Preventative Action report and/or QA Trak."</li> <li>• Added "Preventative actions must be taken in order to prevent or minimize the occurrence of the situation."</li> <li>• Added a paragraph to describe the new PASI Root Cause Analysis procedure.</li> </ul> <p><b>SECTION 10:</b></p> <ul style="list-style-type: none"> <li>• Added the following definitions: Contract Required Detection Limit (CRDL), Contract Required Quantitation Limit (CRQL), Corrective and Preventative Action (CAPA), Non Potable Water (to Environmental Sample definition), Intermediate Standard Solution, Quality Control Sample, Stock Standard, Uncertainty Measurement, Working Standard Solution,</li> </ul> <p><b>SECTION 11:</b></p> <ul style="list-style-type: none"> <li>• Added ISO/IEC 17025:2005 reference</li> </ul> <p><b>Appendix:</b></p> <ul style="list-style-type: none"> <li>• Added Appendix I: Quality Control Calculations</li> </ul>	
<p>Quality Assurance Manual Revision 11.0</p>	<p>Overall conversion to template format. Removed all references to Addenda. Changes required based on conversion are not explicitly noted unless change represents a significant policy change.</p> <p><b>SECTION 1:</b></p> <ul style="list-style-type: none"> <li>• Add comment to address continuous improvement to quality system.</li> <li>• Changed statement of purpose in Section header to "Mission Statement".</li> <li>• Added requirements for appointment when Technical Director absent.</li> </ul>	<p>17Sep2007</p>

Document Number	Reason for Change	Date
	<ul style="list-style-type: none"> <li>• Added requirements for notification to AA's and updates to organizational charts when management changes.</li> <li>• Added Client Services Manager job description.</li> </ul> <p>SECTION 2:</p> <ul style="list-style-type: none"> <li>• Changed temperature requirements to "Not Frozen but <math>\leq 6^{\circ}\text{C}</math>".</li> <li>• Added flexible section concerning default sampling time in absence of customer-specified time.</li> <li>• Added flexible section to address sample and container identification by the LIMS.</li> <li>• Changed sample retention requirement to 45 days from receipt of samples. Added comment allowing for storage outside of temperature controlled conditions.</li> </ul> <p>SECTION 3:</p> <ul style="list-style-type: none"> <li>• Inserted allowance for use of older methods.</li> <li>• Changed references to work processing and training documents to allow for use of LMS and other types of training media.</li> <li>• Inserted allowance for alternative DOCs where spiking not possible.</li> </ul> <p>SECTION 4:</p> <ul style="list-style-type: none"> <li>• Inserted reference to Anonymous Message line.</li> <li>• Inserted reference to the use of default control limits.</li> <li>• Inserted allowance for release of data without corrective action for obvious matrix interferences.</li> <li>• Inserted reference to the treatment of internal standards.</li> <li>• Inserted allowance for use of MDL annual MDL verification in lieu of full 40 CFR Part 136 annual MDL studies.</li> <li>• Inserted general procedure for LOQ verification</li> </ul> <p>SECTION 5:</p> <ul style="list-style-type: none"> <li>• Added general process for approval and use of QAM template.</li> <li>• Removed specific reference of Work Process Manuals. Left flexible section to include all other controlled documentation.</li> </ul> <p>SECTION 6:</p> <ul style="list-style-type: none"> <li>• No changes noted.</li> </ul> <p>SECTION 7:</p> <ul style="list-style-type: none"> <li>• Added qualifications for secondary reviewers.</li> </ul> <p>SECTION 8:</p> <ul style="list-style-type: none"> <li>• Changed frequency listing for Corporate Audits.</li> </ul> <p>SECTION 9:</p> <ul style="list-style-type: none"> <li>• Changed references from QA Track to Lab Track – left flexible to accommodate information still in QA Track.</li> </ul> <p>SECTION 10:</p> <ul style="list-style-type: none"> <li>• No changes noted.</li> </ul> <p>SECTION 11:</p> <ul style="list-style-type: none"> <li>• No changes noted.</li> </ul> <p>ATTACHMENTS:</p> <ul style="list-style-type: none"> <li>• Standardized format for Attachments.</li> </ul>	
Quality Assurance Manual Revision 12.0	<p>General: replaced the word 'client' with 'customer', where applicable.</p> <p>SECTION 1:</p> <ul style="list-style-type: none"> <li>• Section 1.6.4: added language for clarity</li> </ul>	13Nov2008



Document Number	Reason for Change	Date
	<ul style="list-style-type: none"> <li>• Added new section 1.8.1; responsibilities of Senior General Managers.</li> <li>• Section 1.8.3: added reference to LMS.</li> <li>• Added new section 1.8.17: responsibilities of Waste Coordinators.</li> <li>• Section 1.9, last paragraph: changed 'annually' to 'periodically'. Next to last paragraph- added reference to LMS.</li> </ul> <p><b>SECTION 2:</b></p> <ul style="list-style-type: none"> <li>• Incorporated optional language into section 2.1 for laboratories with field services staff supervised by the laboratory</li> <li>• Added new section 2.2 entitled Field Services.</li> <li>• Section 2.3: added reference to the new Review of Analytical Requests SOP.</li> <li>• Changed optional text in 2.6 to explain how EpicPro assigns unique ID # to projects and samples including the unique container ID</li> <li>• Section 2.7.2: changed freezer temp requirement to match SOP.</li> </ul> <p><b>SECTION 3:</b></p> <ul style="list-style-type: none"> <li>• Section 3.4: Included optional language for performing IDOCs for tests not amenable to spiking using the "4 replicate" approach.</li> </ul> <p><b>SECTION 4:</b></p> <ul style="list-style-type: none"> <li>• Section 4.1: expanded language to allow electronic signature and storing of integrity training documentation within the LMS</li> <li>• Section 4.10: revised and added language regarding LOD studies, initial verification and annual verification, where applicable.</li> <li>• Section 4.11: changed PRL to RL.</li> <li>• Section 4.13: added editable line regarding PT study information. Changed wording to say approved PT providers are utilized</li> <li>• Section 4.14: added sentence regarding rounding rules listed applying only to LIMS.</li> </ul> <p><b>SECTION 5:</b></p> <ul style="list-style-type: none"> <li>• Section 5.1, last bullet point: changed language to reflect that SOPs must be locked from printing if controlled electronically.</li> </ul> <p><b>SECTION 6:</b></p> <ul style="list-style-type: none"> <li>• Section 6.3.1: adjusted language about classes of weights potentially used.</li> <li>• Section 6.3.3: removed customer-specific requirement to re-calibrate every four hours but added space for this to be added back in where applicable.</li> <li>• Added reference to Attachment III in the introductory paragraph to this section.</li> </ul> <p><b>SECTION 7:</b></p> <ul style="list-style-type: none"> <li>• Sections 7.1-7.3: added language for those labs that are minimizing or eliminating the need for paper copies.</li> <li>• Section 7.2: clarified language in numbered items so that it does not appear that all 4 criteria must be applicable at one time.</li> <li>• Section 7.3: added list of approved signatories for final reports.</li> </ul> <p><b>SECTION 8:</b></p> <ul style="list-style-type: none"> <li>• Section 8.1.2, last paragraph: revised language regarding data integrity issues and added a timeframe to notify customers of affected data.</li> <li>• Added section 8.5 "Customer Service Reviews"- ISO requirement</li> </ul> <p><b>SECTION 9:</b></p> <ul style="list-style-type: none"> <li>• Added new section 9.3 regarding Preventive Action.</li> </ul> <p><b>SECTION 10:</b></p> <ul style="list-style-type: none"> <li>• No revisions.</li> </ul>	

Document Number	Reason for Change	Date
	<p>SECTION 11:</p> <ul style="list-style-type: none"><li>• No revisions.</li></ul> <p>Attachments:</p> <ul style="list-style-type: none"><li>• Attachment IIb: updated corporate org chart</li><li>• Attachment VIII: revised to match the current Analytical Guides.</li></ul>	

ATTACHMENT I

Quality Control Calculations

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for theLCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:

TrueValue = Amount spiked (can also be the  $\overline{CF}$  or  $\overline{RF}$  of the ICAL Standards)

Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentration} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:

R1 = Result Sample 1

R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^N W_i * (X_i - \bar{X}) * (Y_i - \bar{Y})}{\sqrt{\left(\sum_{i=1}^N W_i * (X_i - \bar{X})^2\right) * \left(\sum_{i=1}^N W_i * (Y_i - \bar{Y})^2\right)}}$$

- With: N      Number of standard samples involved in the calibration  
 i          Index for standard samples  
 Wi        Weight factor of the standard sample no. i  
 Xi        X-value of the standard sample no. i  
 X(bar)   Average value of all x-values  
 Yi        Y-value of the standard sample no. i  
 Y(bar)   Average value of all y-values

ATTACHMENT I (CONTINUED)

Quality Control Calculations (continued)

STANDARD DEVIATION (S)

$$S = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{(n-1)}}$$

where:

- n = number of data points
- X<sub>i</sub> = individual data point
- $\bar{X}$  = average of all data points

AVERAGE ( $\bar{X}$ )

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n}$$

where:

- n = number of data points
- X<sub>i</sub> = individual data point

RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\bar{X}} * 100$$

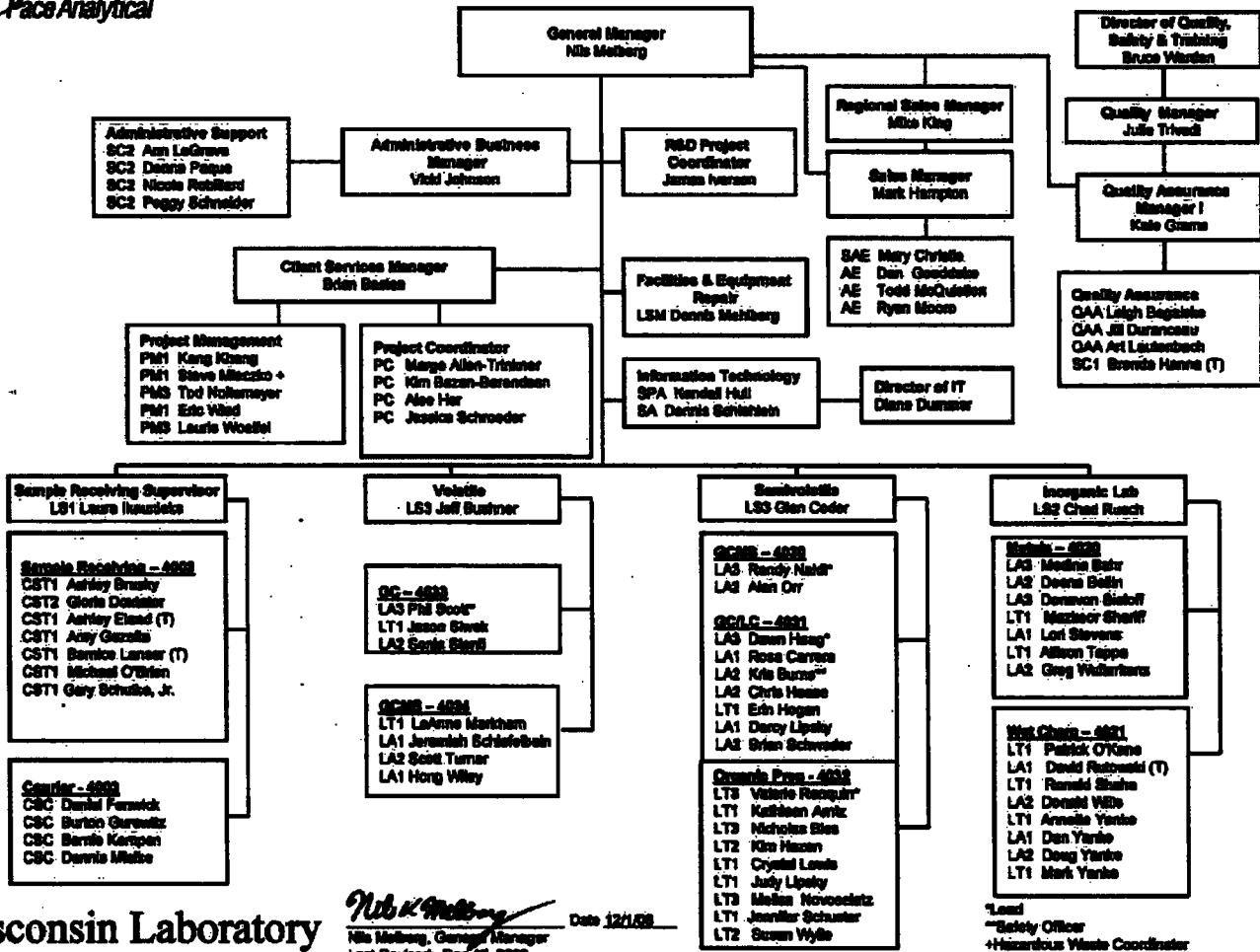
where:

- S = Standard Deviation of the data points
- $\bar{X}$  = average of all data points



ATTACHMENT IIA

PASI - GREEN BAY ORGANIZATION CHART



Wisconsin Laboratory

*Nils Melberg*  
 Nils Melberg, General Manager  
 Last Revised: Dec 1<sup>st</sup> 2008  
 Date 12/1/08

\*Lead  
 \*\*Safety Officer  
 +Hazardous Waste Coordinator

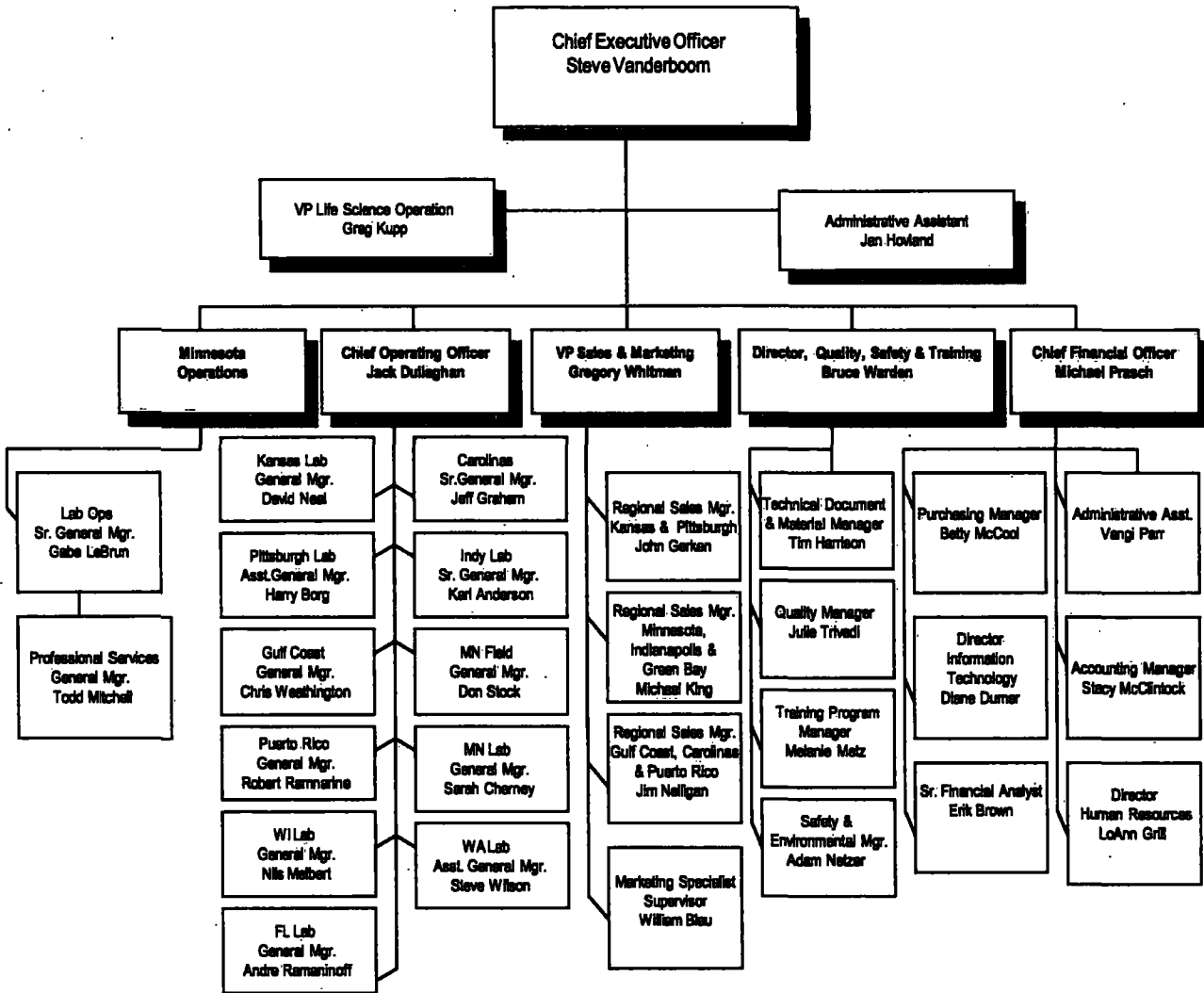




ATTACHMENT IIB

PASI – CORPORATE ORGANIZATIONAL CHART

CORPORATE/MANAGEMENT STRUCTURE



**ATTACHMENT III**

**PASI – GREEN BAY EQUIPMENT LIST**

<b>Analytical Instrument/Peripherals</b>	<b>Analysis</b>
Thermo Scientific ICAP 6500 ICP Spectrometer	Metals
ICPMS Thermo Scientific Xseries 2	Metals
PE FIMS (Flow Injection Hg System)	Mercury
PE FIMS (Flow Injector Mercury System)	Mercury
Hot Block Metals Digestion System	Metals Dig.
Hot Block Metals Digestion System	Metals Dig.
Hot Block Metals Digestion System	Metals Dig.
Hot Block Metals Digestion System	Metals Dig.
Hot Block Metals Digestion System	Metals Dig.
Denver Instrument XE-310 Top Loading Balance	
Mettler AJ 100	
American Scientific Balance	
A&D Balance	
Ohaus Analytical Balance	
Ohaus Analytical Balance	
Ohaus Analytical Balance	
Tekran-2500 Low-level Mercury Analyzer	1631E
Tekran 2600 Low Level Automated Mercury Analyzer	1631E
HP 5890 Series II GC	Pesticide
HP 6890 Series GC	Toxaphene
HP 6890 Series GC	PCB
HP 5890 Series GC	Pest/Tox (Screener)
HP 5890 Series II GC	Pesticide
HP 6890 Series GC	PCB
HP 6890 Series GC	PCB
HP 5890 Series II GC	PCB
HP 5890 Series II GC	
HP 5972 Mass Selective Detector	BNA
HP 6890 GC	
HP 5973 Mass Selective Detector	PAH
HP 6890 GC	
HP 5973 Mass Selective Detector	PAH
HP 6890 GC	
HP 5973 Mass Selective Detector	PAH
HP 5890 Series II GC	
HP 5972 Mass Selective Detector	BNA Screener

HP 5890E GC	
HP 5972A Mass Selective Detector	BNA/Phenols
HP 5890 Series II GC	DRO/TPH
HP 5890 Series II GC	DRO/TPH
HP 5890 Series II GC	DRO/TPH
HP 5890 Series II GC	DRO/TPH
HP 5890 GC	Alcohol
Lab-Line Automated Separatory Funnel Extractor	SVOA Ext.
Lab-Line Automated Separatory Funnel Extractor	SVOA Ext.
Lab-Line Automated Separatory Funnel Extractor	SVOA Ext.
Zymark TurboVap II Concentration Workstation	SVOA Ext.
Zymark TurboVap II Concentration Workstation	SVOA Ext.
Zymark TurboVap II Concentration Workstation	SVOA Ext.
Zymark TurboVap II Concentration Workstation	SVOA Ext.
Zymark TurboVap II Concentration Workstation	SVOA Ext.
Lab-Line 9334 Sonicator	SVOA Ext.
Fisher Isotemp Muffle Furnace	
Ohaus Analytical Balance	
Mettler Toledo Analytical Balance	
Mettler AE 200 Analytical Balance	
Sargent Welch TL 4000 DR Balance	
Mettler PM 480 Balance	
J2 Scientific Accuprep MPS GPC Cleanup System	SVOA Ext.
J2 Scientific Accuprep MPS GPC Cleanup System	SVOA Ext.
CEM Mars Xtraction Microwave System Model # 907501	SVOA Ext.
American Scientific Oven DK-42	
Scientific Products Oven DK-43	
Soxtherm Accelerated Soxhlet Extractor	SVOA Ext.
Soxtherm Accelerated Soxhlet Extractor	SVOA Ext.
Soxtherm Accelerated Soxhlet Extractor	SVOA Ext.
Soxtherm Accelerated Soxhlet Extractor	SVOA Ext.
Six place Soxhlet heater mantles and Glassware	SVOA Ext.
Sonifier Cell Disruptors with Horns	SVOA Ext.
Sonifier Cell Disruptors with Horns	SVOA Ext.
Sonifier Cell Disruptors with Horns	SVOA Ext.
HP 5890 Series II GC	
HP 5972 MSD	SW8260
Agilent 6850 GC	
Agilent 5975 MSD	SW8260
HP 5890 Series II GC	
HP 5972 MSD	SW8260
HP 6890 GC	
HP 5973 MSD	SW8260



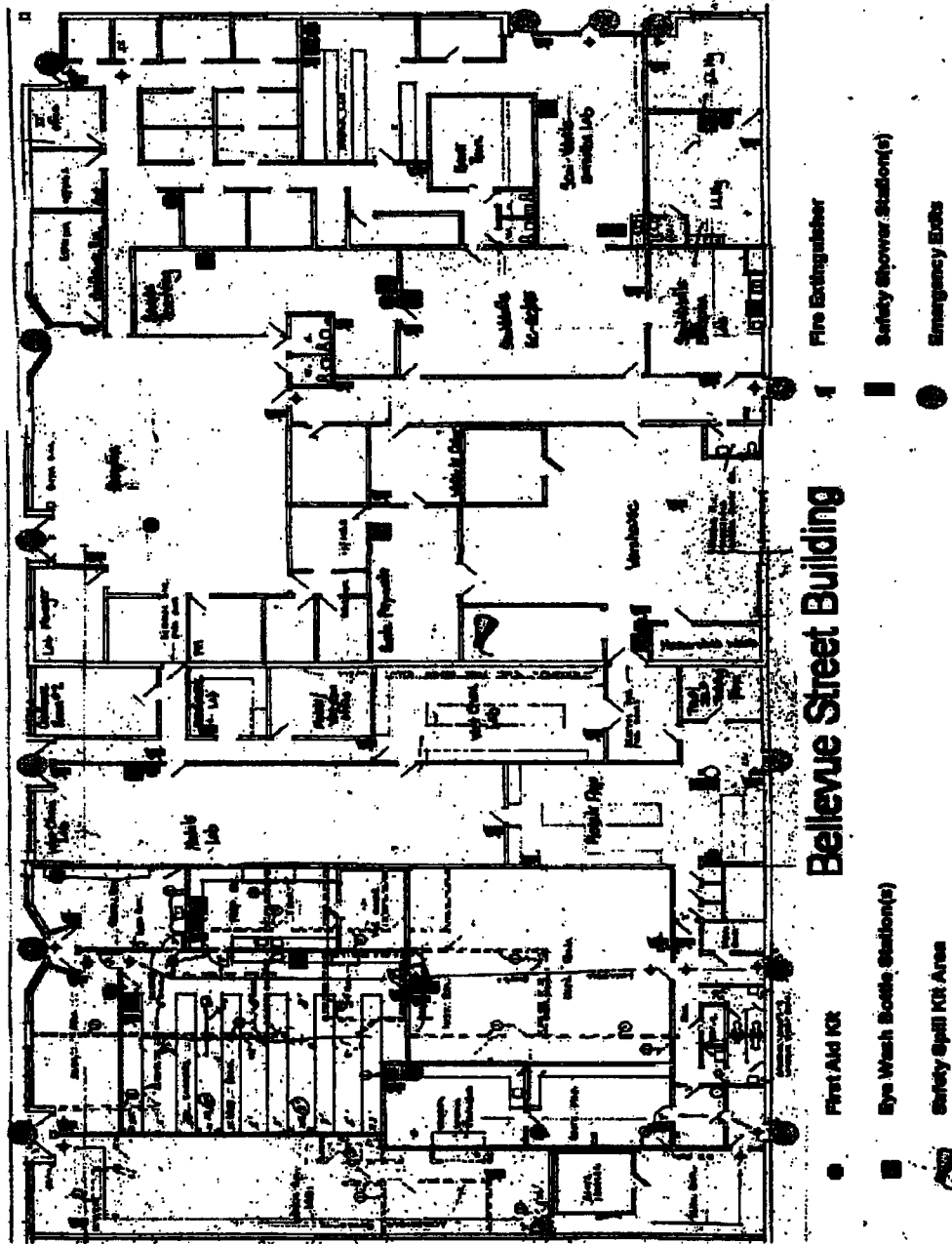
HP 5890 Series II GC	
HP 5972 MSD	SW8260
HP 6890 GC	
HP 5973 MSD	SW8260
Agilent Technologies 6850 Network GC System	
Agilent Technologies 5975B MSD	SW8260
HP 6890 GC	
HP 5973 MSD	SW8260
HP 6890 GC	
HP 5973 MSD	SW8260
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 Series II GC with PID/FID	BTEX/TPH/GRO
HP 5890 GC with FID	Methane, Ethane, Ethene
HP 5890 GC with FID	VOA Screen
HP 5890 GC with FID	VOA Screen
Mettler/Toledo Top Loading Balance	
Sartorius PT-210 Top Loading Balance	
Dohrmann DC-80 Total Organic Carbon Analyzer (TOC)	TOC
Horiba TOC Detector	TOC
Rosemount/Dohrmann TOC Boatsampler Lachat Quik Chem 8000	TOC
Lachat MICRO DIST Rapid Distillation System	CN, Alk, TKN, NH3, Phenolics, Total Phos, NO2-NO3
Lachat MICRO DIST Rapid Distillation System	Distillation
Lachat MICRO DIST Rapid Distillation System	Distillation
Lachat MICRO DIST Rapid Distillation System	Distillation
Dionex DX-120 Ion Chromatograph	Anions
Dionex DX-120 Ion Chromatograph	Anions
HACH DR 2000 Direct Reading Spectrophotometer	COD, Hex. Cr, Ferrous Fe, Color
Fisher Model FW 99-20-1385 Stereomaster Stereoscope	Bact



YSI 5000 Oxygen Meter	BOD
YSI 5010 5010 Oxygen Probe	BOD
HACH Turbidimeter	Turbidity
Accumet 30 Conductivity Meter	Conductivity
Blue M Bacteriological Incubator Model C-4008-Q	BOD
Millipore Incubator	BOD
Precision Scientific Low Temperature Incubator	BOD
National Steril-Quick Autoclave Model 704-9000-D	Autoclave
Precision Scientific – Flashpoint Instrument	Flashpoint
Fisher Scientific – Flashpoint Instrument	Flashpoint
Orion 720A – pH meter	pH
Coming 320 pH Meter	pH
Millipore YT31 ORA HW Rotary Agitator	TCLP/SPLP
TDS Oven	TDS
TSS Oven - Yamato DKN 600	TSS
Solids Oven	Solids
Total Solids Oven	Total Solids
Hot Block Digestion System	TKN, Total Phos
Hot Block Digestion System	TKN, Total Phos
Lindberg Muffle Furnace	
Mettler/Toledo Top Loading Balance	
Mettler/Toledo Top Loading Balance	
Satorius Top Loading Balance	
Mettler/Toledo Analytical Balance	
Ohaus Analytical Balance	
Ohaus Analytical Balance	
Mettler -Toledo PB602S Balance	
Tekmar/Dohrmann Apollo 9000 Total Organic Carbon Analyzer (TOC)	TOC
Dohrmann 183 S/SS Soil TOC Oven	TOC
Teledyne Model 14-9600-100 Total Organic Carbon Analyzer	TOC
Milton Roy Spectronic 21D Spectrophotometer	AVS/SEM

ATTACHMENT IV

PASI - GREEN BAY FLOOR PLAN





ATTACHMENT V

PASI – GREEN BAY SOP LIST

SOP Number	SOP Name
S-GB-ALL-C-001-REV.01	Sample Management
S-ALL-C-002-Rev.0	Bottle Order Database
S-GB-ALL-C-003-REV.01	Subcontracting Samples
S-GB-ALL-C-004-REV.02	Bottle Preparation
S-ALL-C-005-Rev.00	Operation of PacePort Customer Feedback Form
S-ALL-C-006-Rev.00	Review of Analytical Requests
S-GB-C-001-Rev.02	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035
S-GB-C-002-Rev.02	Extruding Sample from 25 g EnCores for Volatile and Semivolatile Analyses
S-GB-C-003-Rev.02	Tedlar Bag Preservation by Methanol
S-GB-C-005-Rev.02	Maintenance of Ice Chests and Shipping Containers
S-GB-C-007-Rev.02	Laboratory Tracking of Samples
S-GB-C-008-REV.0	Measurement of Percent Moisture in Soils and Solids
T-ALL-C-001-Rev.01	Project Management
GB-I-026-Rev.2	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy
S-GB-I-028-REV.2	Soil Fraction Preparation for Lead Analysis
GB-I-036-REV.2	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy
S-GB-I-039-REV.1	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry - Automated Tekran 2600
S-GB-I-053-Rev.0	Acid Volatile Sulfide / Simultaneously Extracted Metals
S-ALL-GB-M-002-Rev.1	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B
S-GB-M-001-REV.00	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry
S-GB-M-002-REV.00	Methyl Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry
S-GB-M-003-REV.00	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry Solids - Manual System
S-GB-M-004-REV.00	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (7470A/7471B)
S-GB-M-005-REV.00	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010C
S-GB-M-006-REV.00	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020A
S-GB-M-007-REV.00	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020
S-GB-M-008-REV.00	Cleaning Metals Glassware
S-GB-M-009-REV.00	Hardness by Calculation
S-GB-M-011-REV.00	The Determination of Mercury in Biological Samples by Cold Vapor Atomic Absorption Spectroscopy
S-ALL-GB-I-014-Rev.3	Measurement of Volatile Solids and Solids in Water
S-ALL-GB-I-015-Rev.00	Measurement of pH in Water, Soil, and Waste
S-ALL-GB-I-016-Rev.00	Measurement of Specific Conductance in Water
S-GB-I-001-Rev.4	Total Sulfide, Iodometric Titration
S-GB-I-002-Rev.2	Flash Point (Pensky-Martens Closed Cup Method For Ignitability)
S-GB-I-004-Rev.02	Acidity
S-GB-I-006-REV.01	Amenable Cyanide
S-GB-I-009-Rev.2	Ion Chromatography
S-GB-I-010-REV.1	Wet Chemistry Glassware Cleaning

S-GB-I-013-Rev.01	Free Liquids
S-GB-I-014-Rev.01	Alkalinity
S-GB-I-015-Rev.01	Oxidation - Reduction Potential (Eh) Measurement
S-GB-I-016-Rev.01	Specific Gravity
S-GB-I-017-Rev.01	Ferrous Iron
S-GB-I-018-Rev.01	Total Alkalinity Analyzed by Lachat 8000 Flow Injection
S-GB-I-019-Rev.02	Fecal Coliform Determination Using the Membrane Filter Technique
S-GB-I-020-Rev.02	Color Determination in Aqueous Samples
S-GB-I-023-Rev.02	Heterotrophic Plate Count
S-GB-I-024-REV.2	Colisure Presence/Absence Test for Detection and Identification of Coliform Bacteria and <i>Escherichia coli</i> in Drinking Waters
S-GB-I-025-Rev.2	TCLP - Toxicity Characteristic Leaching Procedure
S-GB-I-027-Rev.01	Dissolved Oxygen
S-GB-I-030-Rev.01	Turbidity (Nephelometric)
S-GB-I-031-REV.01	Determination of Total Organic Carbon Using the DC-80 Instrument
S-GB-I-037-REV.01	The Determination of Total Organic Carbon Using the Walkley-Black Procedure
S-GB-I-042-Rev.01	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument for MDEQ RRD Operational Memorandum No. 2
S-GB-I-043-Rev.01	Total Cyanide using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following SW846 9012A and EPA Method 335.4
S-GB-I-044-Rev.01	Biochemical Oxygen Demand
S-GB-I-045-Rev.01	Chromium, Hexavalent-Colorimetric
S-GB-I-046-Rev.02	Total Phenolics using Micro-Distillation and Lachat 8000 Flow Injection
S-GB-I-047-Rev.01	Total Kjeldahl Nitrogen using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 351.2
S-GB-I-048-Rev.01	Ammonia using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following EPA Method 350.1
S-GB-I-049-Rev.01	Total Phosphorus using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 365.4
S-GB-I-051-REV.01	Nitrate and Nitrite Analyzed by Lachat 8000 Flow Injection
S-GB-I-052-REV.01	Chemical Oxygen Demand, Colorimetric, Manual (Chemetric Vials)
S-GB-I-057-REV.00	SPLP - Synthetic Precipitation Leaching Procedure
S-GB-I-058-REV.00	ASTM Shake Extraction of Solid Waste with Water
S-GB-I-059-Rev.00	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument
S-GB-O-005-REV.02	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO
S-GB-O-006-Rev.02	Modified Method for Determination of Gasoline Range Organics
S-GB-O-008-REV.02	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors
S-GB-O-009-REV.02	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors
S-GB-O-010-Rev.02	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics
S-GB-O-017-REV.02	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography
S-ALL-GB-O-001-Rev.00	Determination of Semi-Volatile Organics by GC/MS (8270)
S-ALL-GB-O-003-Rev.00	Separatory Funnel Extraction
S-ALL-GB-O-005-REV.00	Ultrasonic Extraction
S-ALL-GB-O-008-REV.00	Determination of Semi-Volatile Organics by gc/ms (Selective Ion Monitoring)
S-GB-O-015-REV.02	Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics
S-GB-O-018-REV.1	Determination of DRO Sample Weight and Methylene Chloride Addition
S-GB-O-019-REV.02	WI Modified Method for Determination of Diesel Range Organics
S-GB-O-023-Rev.04	Total Petroleum Hydrocarbons
S-GB-O-025-Rev.01	Alcohols & Glycols by Direct Injection GC/FID
S-GB-O-026-REV.03	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography





S-GB-O-027-REV.2	Analysis of Organochlorine Pesticides by Gas Chromatography
S-GB-O-028-REV.02	Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes
S-GB-O-031-REV.1	Extraction of Biological Samples for Organochlorine Pesticides/PCBs
S-GB-O-032-REV.1	Gel Permeation Chromatography
GB-O-033-Rev.0	Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis
S-GB-O-034-REV.1	Sulfuric Acid Cleanup
S-GB-O-035-REV.1	Mercury Cleanup for the Removal of Sulfur from PCB Samples
S-GB-O-036-REV.1	Florisil Cleanup for PCBs
S-GB-O-037-REV.1	Florisil Cartridge Cleanup
S-GB-O-038-Rev.02	Silica Gel Cleanup for Organic Analysis
S-GB-O-039-REV.1	Copper Cleanup for the Removal of Sulfur from PCB Samples
S-GB-O-040-REV.02	Extraction of Wipes and Oil for PCB Analysis
S-GB-O-041-REV.02	Extraction of PCBs Using the Automated Soxhlet
S-GB-O-043-Rev.01	Extraction of Toxaphene Using the Automated Soxhlet
S-GB-O-044-REV.1	Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices
S-GB-O-045-REV.1	Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons and Base/Neutral/Acids in Solid Matrices
S-GB-O-047-REV.00	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography following 8082A
S-GB-ALL-O-002-REV.01	Determination of Volatile Organics by GC/MS
S-GB-O-001-REV.03	Sample Screening Volatile Organics Prior to Preparation
S-GB-O-012-REV.02	Cleaning of Syringes Used in the Analysis of Volatile Organics
ALL-IT-001-Rev.1	System Security and Integrity
S-ALL-IT-002-Rev.1	Server Back-UP
S-ALL-GB-IT-002-Rev.02	Server Back-UP - Green Bay Addendum
T-ALL-IT-001-rev.04	EPIC Pro 01: Basic System Functions
T-ALL-IT-002-rev.05	EPIC Pro 02: Client Setup
T-ALL-IT-004-rev.03	EPIC Pro: PMs / Sales II
T-ALL-IT-005-rev.04	EPIC Pro: Login
T-ALL-IT-006-rev.04	EPIC PRO: Lab Prep
T-ALL-IT-007-rev.03	EPIC Pro: Analyst / Lab Management
T-ALL-IT-008-rev.05	EPIC Pro: PM - Additional Knowledge
T-ALL-IT-009-rev.01	EPIC Pro: Detection, Reporting and Control Limits
T-ALL-IT-010-rev.02	EPIC Pro: Standard Traceability
S-ALL-Q-001-Rev.7	Preparation of Standard Operating Procedures
S-ALL-Q-002-Rev.02	Document Management
S-ALL-Q-003-REV.2	Document Numbering
S-ALL-Q-004-Rev.4	Method Detection Limit Studies
ALL-Q-005-Rev.2	Purchasing of Laboratory Supplies
S-ALL-GB-Q-005-REV.04	Purchasing of Laboratory Supplies - Green Bay Addendum
ALL-Q-006-Rev.1	Receipt and Storage of Laboratory Supplies
S-ALL-GB-Q-006-Rev.1	Receipt and Storage of Laboratory Supplies - Green Bay Addendum
S-ALL-Q-007-Rev.01	EPIC Pro: Acode Validation
S-ALL-Q-009-Rev.02	Laboratory Documentation
S-ALL-Q-010-REV.2	Proficiency Testing Program
ALL-Q-011-Rev.1	Audits and Inspections
ALL-Q-012-Rev.0	Corrective Action / Preventative Action Process
S-ALL-GB-Q-012-Rev.02	Corrective Action / Preventative Action Process - Green Bay Addendum
S-ALL-Q-013-REV.1	Support Equipment
ALL-Q-014-Rev.1	Quality system Review



All-Q-016-Rev.0	Manual Integration
S-ALL-Q-018-Rev.02	Monitoring Storage Units
S-ALL-Q-020-REV.1	Orientation and Training Procedures
S-ALL-Q-021-Rev.02	Sample Homogenization and Sub-Sampling
ALL-Q-022-Rev.1	3P Program Continuous Process Improvement
ALL-Q-025-Rev.1	Standard and Reagent Management and Traceability
S-ALL-GB-Q-025-REV.01	Standard and Reagent Management and Traceability - Green Bay Addendum
S-ALL-Q-027-REV.0	Evaluation and Qualification of Vendors
S-ALL-Q-028-Rev.0	Use and Operation of Lab Track System
S-ALL-Q-029-REV.0	MintMiner Data File Review
S-ALL-Q-030-Rev.01	Operation of Data Checker for Epic Pro
S-GB-Q-001-REV.01	Employee Master Signature Log
S-GB-Q-002-Rev.01	Training Record Files Maintained by the QAO
S-GB-Q-003-Rev.00	Data Reduction, Validation, and Reporting
S-GB-Q-004-Rev.01	Laboratory Notebooks and Logbooks
S-GB-Q-005-Rev.01	Precision and Accuracy Measurement and Evaluation
S-GB-Q-006-Rev.01	Data Archiving
S-GB-Q-007-Rev.01	Method of Syringe Technique
S-GB-Q-008-Rev.02	Preventative, routine, and non-routine maintenance
S-GB-Q-009-Rev.01	Common Laboratory Calculations and Statistical Evaluation of Data
S-GB-Q-010-Rev.01	Estimation of Measurement Uncertainty
S-ALL-S-001-REV.1	Hazard Assessment
ALL-S-002-Rev.0	Waste Handling
S-GB-ALL-S-002-REV.03	Waste Handling - Green Bay Addendum
S-GB-S-001-REV.2	Regulated Soil Handling (Green Bay Location)
S-GB-S-002-REV.01	Control of Hazardous Energy Program - Lockout/Tagout
GB-S-003-Rev.0	Electrical Generator Procedure
S-GB-S-004-Rev.01	Rescue Alert System Operation
S-GB-E-001-Rev.01	Use and Maintenance of the NANOpure Infinity Water Purification System
S-GB-E-002-Rev.01	Operation of Waste Disposal Equipment
S-GB-L-001-REV.1	Biological Tissue and Plant Preparation
S-GB-L-002-REV.1	Small Rodent Handling and Homogenization
S-GB-L-003-REV.1	The Determination of Lipids in Tissues, Fats, and Plants
S-GB-L-004-REV.1	Determination of Percent Solids in Tissue Samples
S-GB-L-005-REV.0	Reagent Water Quality
S-GB-L-006-REV.00	Procedure for Handling Aqueous Organic Extractable Samples Containing Sediment



ATTACHMENT VI

PASI – GREEN BAY CERTIFICATION LIST

Accrediting Authority	Program Category	Accrediting Agency	Certification #
Florida (NELAP)	Biological Tissue	Dept of Health, Bureau of Laboratories	E87948
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87951
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87948
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87951
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87948
Georgia	Hazardous Waste - Solid - NELAP stipulation	Environmental Protection Division	E87951
Georgia	Waste Water -NELAP stipulation	Environmental Protection Division	E87948
Georgia	Hazardous Waste - Solid - NELAP stipulation	Environmental Protection Division	E87951
Georgia	Waste Water - NELAP stipulation	Environmental Protection Division	E87948
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200051
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200050
Illinois (NELAP)	Waste Water	Illinois EPA	200050
Illinois (NELAP)	Waste Water	Illinois EPA	200051
Kentucky	UST	Environmental and Public Protection Cabinet	82
Kentucky	UST	Environmental and Public Protection Cabinet	83
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	04168
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	04168
Louisiana (NELAP)	Biological Tissue	Department of Environmental Quality	04168
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	04169
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	04169
Minnesota	Hazardous Waste	Dept of Health	055-999-334
Minnesota	Waste Water	Dept of Health	055-999-334
Minnesota	UST	Department of Health	055-999-334
New York (NELAP)	Solid - Hazardous Waste - NELAP	Dept of Health	11887
New York (NELAP)	Solid - Hazardous Waste - NELAP	Dept of Health	11888
New York (NELAP)	Waste Water - NELAP	Dept of Health	11888



New York (NELAP)	Waste Water - NELAP	Dept of Health	11887
North Carolina	Waste Water	Dept of Environment, Health & Natural Resources	503
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-150
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-200
North Dakota	Waste Water	Dept of Health Chemistry Division	R-150
North Dakota	Waste Water	Dept of Health Chemistry Division	R-200
South Carolina	Hazardous Waste	Dept of Hlth & Environmental Control	83006001
South Carolina	Waste Water	Dept of Hlth & Environmental Control	83006001
US Dept of Agriculture	Foreign Soil Permit	Dept of Argiculture	S-76505
Wisconsin	Drinking Water	Dept of Natural Resources	405132750
Wisconsin	Drinking Water	Dept of Agriculture, Trade & Consumer Protection	105-444
Wisconsin	Hazardous Waste	Dept of Natural Resources	405132750
Wisconsin	Waste Water	Dept of Natural Resources	405132750



ATTACHMENT VII

PASI - CHAIN OF CUSTODY

**CHAIN-OF-CUSTODY / Analytical Request Document**  
 The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.



<b>Section A</b> Required Client Information: Company: _____ Address: _____ Email To: _____ Phone: _____ Fax: _____ Requested Date/Time: _____		<b>Section B</b> Required Project Information: Request To: _____ Copy To: _____ Purchase Order No.: _____ Project Name: _____ Project Number: _____		<b>Section C</b> Invoice Information: Client: _____ Company Name: _____ Address: _____ Phone: _____ Fax: _____ Project Name: _____ Project Number: _____	
Sample ID (Must be unique) Sample ID MUST BE UNIQUE		Matrix Code (See field codes in left) Matrix Code Drinking Water Wastewater Product Soil/Sediment Air Other		Sample Type (e.g. Grab, Composite) Matrix Code (See field codes in left)	
Collected Composite Start Composite End		Date Time		Sample Temp at Collection	
Preservation/Preservatives Unpreserved H2SO4 HNO3 HCl NaOH Na2SO4 Methanol Other		# of Containers		Residual Chlorine (Y/N)	
Page: _____ of _____		Round Water <input type="checkbox"/> NPOES <input type="checkbox"/> UST <input type="checkbox"/> SWA		Other Water <input type="checkbox"/>	
Temperature in °C		Received on Ice (Y/N)		Custody Sealed (Y/N)	
Samples Intact (Y/N)		SAMPLE CONDITIONS		Face Project No./Lab I.D.	
PRINT NAME of SAMPLER SIGNATURE of SAMPLER		DATE Signed (MM/DD/YYYY)		SIGNATURE of SAMPLER	



**ATTACHMENT VIII  
METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE**

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
2, 3, 7, 8-TCDD	1613B	Soil	8oz Glass	None	90/40 Days
2, 3, 7, 8-TCDD	1613B	Water	1L Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	90/40 Days
2, 3, 7, 8-TCDD	8290	Water	1L Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	30/45 Days
Acidity	SM2310B	Water	Plastic/Glass	≤6°C	14 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤6°C	14 Days
Alpha Emitting Radium Isotopes	9315/903.0	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Anions by IC, including Br, Cl, F, NO <sub>2</sub> , NO <sub>3</sub> , SO <sub>4</sub>	300.0/300.1/ SM4110B	Water	Plastic/Glass	≤6°C	Br, Cl, F, SO <sub>4</sub> (28 Days) NO <sub>2</sub> , NO <sub>3</sub> (48 Hours)
Aromatic and Halogenated Volatiles	8021	Soil	5035 vial kit	See 5035 note*	14 days
Aromatic and Halogenated Volatiles	601/602/8021	Water	40mL vials	pH<2 HCl; ≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	24 Hours
Base/Neutrals and Acids	8270	Soil	8oz Glass	≤6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.1/525.2	Water	1L Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/30 Days
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag	None	48 Hours
Chloride	SM4500Cl/9250/ 9251/9252	Water	Plastic/Glass	None	28 Days
Chlorinated Herbicides	8151	Soil	8oz Glass Jar	≤6°C	7/40 Days
Chlorinated Herbicides	8151	Water	1L Amber Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/40 Days
Chlorinated Herbicides	515.1	Water	1L Amber Glass	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14/28 Days
Chorine, Residual	SM4500Cl	Water	Plastic/Glass	None	15 minutes
COD	SM5220C/ 410.3/410.4	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; ≤6°C	28 Days
Color	SM2120B,C,E	Water	Plastic/Glass	≤6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	6 Months
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN/9010/ 9012/335.4	Water		pH>12 NaOH; ≤6°C; ascorbic acid if Cl present	14 Days, 24 Hours if Sulfide present
Diesel Range Organics	8015	Soil	8oz Glass Jar	≤6°C	14/40 Days
Diesel Range Organics	8015	Water	1L Glass	≤6°C	7/40 Days
Dioxins & Furans	TO-9	Air	PUF	None	30/45 Days
EDB & DBCP	504.1/8011	Water	40mL vials	≤6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days
Explosives	8330/8332	Water	1L Glass	≤6°C	7/40 Days
Explosives	8330/8332	Soil	8oz Glass Jar	≤6°C	14/40 Days
Ferrous Iron	SN3500Fe-D	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010/1030	Water	Plastic/Glass	None	28 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Gas Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Soil	5035 vial kit	See 5035 note*	14 days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	48 Hrs

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH <sub>4</sub> Cl; <6°C	14/7 Days
Hardness, Total (CaCO <sub>3</sub> )	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	6 Months
Hexavalent Chromium	7196/218.6/ SM3500Cr	Water	Plastic/Glass	<6°C	24 Hours
Hydrogen Halide & Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Low Level Mercury	1631	Water	Glass	BrCl	90 days (if preserved and oxidized)
Mercury	7471	Soil	8oz Glass Jar	<6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	28 Days
Metals	7300/7303	Air	Filters	None	6 Months
Metals (and other ICP elements)	6010	Soil	8oz Glass Jar	None	6 months
Metals (and other ICP elements)	6010/6020/200.7/ 200.8	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	6 Months
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag	None	48 Hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Nitrogen, Kjeldahl	SM4500-Norg; 351.1/351.2	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/ 352.1	Water	Plastic/Glass	<6°C	48 Hours
Nitrogen, Nitrate & Nitrite	SM4500-NO3/ 353.2	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Nitrogen, Nitrite	SM4500-NO2/ 353.2	Water	Plastic/Glass	<6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/ 351.2	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag	None	48 Hours
Odor	SM2150B	Water	Glass	<6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/ 9070	Water	Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Organochlorine Pesticides & PCBs	TO-4	Air	PUF	None	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082/608	Water	1L Glass	<6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082	Soil	8oz Glass Jar	<6°C	14/40 Days
Organophosphorous Pesticides	8141	Soil	8oz Glass Jar	<6°C	14/40 Days
Organophosphorous Pesticides	8141	Water	1L Amber Glass	<6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/40 Days
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	6 Months
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag	None	48 Hours
pH	SM4500H+B/9040/ 9041/150.2	Water	Plastic/Glass	None	15 minutes
Phenol, Total	420.1/420.4/9065/ 9066	Water	Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; <6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> ; <6°C	28 Days
Polynuclear Aromatic Hydrocarbons	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons	8270 SIM	Soil	8oz Glass Jar	<6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons	8270 SIM	Water	1L Glass	<6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Radium-226 Radon Emanation Technique	903.1	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Radium-228	9320/904.0	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Silica, Dissolved	SM4500Si-D	Water	Plastic	<6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	<6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	<6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	<6°C	7 Days
Solids, Total Suspended	SM2540D	Water	Plastic/Glass	<6°C	7 Days
Solids, Total Volatile	SM2540E	Water	Plastic/Glass	<6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	<6°C	28 Days
Stationary Source Dioxins & Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	6 Months
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	6 Months
Sulfate	SM4500SO4/9036/9038/375.2/ASTMD516	Water	Plastic/Glass	<6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; <6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants	SM5540C	Water	Plastic/Glass	<6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/ 9060	Water	Glass	pH<2 H <sub>2</sub> SO <sub>4</sub> or HCl; <6°C	28 Days
Total Organic Halogen (TOX)	SM5320/9020/ 9021	Water	Glass; no headspace	<6°C	14 Days
Tritium	906.0	Water	Glass	pH<2 HNO <sub>3</sub>	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	<6°C	48 Hours
Uranium Radiochemical Method	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO <sub>3</sub>	180 days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Soil	5035 vial kit	See 5035 note*	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; <6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; <6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days (7 unpreserved)
Volatiles	524.1/524.2	Water	40mL vials	pH<2 HCl; <6°C; Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days
Alaska DRO	AK102	Soil	8oz Glass	<6°C	14/40 Days
Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; <6°C	14/40 Days
Alaska RRO	AK103	Soil	8oz Glass	<6°C	14/40 Days
Alaska GRO	AK101	Soil	5035 vial kit	See 5035 note*	14 Days
Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; <6°C	14 Days

**5035 Note:** 5035 vial kit typically contains 2 vials water, preserved by freezing or, 2 vials aqueous sodium bisulfate preserved at 4°C, and one vial methanol preserved at <6°C and one container of unpreserved sample stored at <6°C.







# Appendix C

## Calculation of Generic GSI Criteria for Metals

---

Calculation of Generic GSI Criteria for Metals at the Folkertama Refuse Site, Walker, MI

Chemical	FCVs Formula (ug/L)	FCV Conversion Factor (CF) for H=225 mg/L CaCO <sub>3</sub>	Dissolved-to-Total Metal Translator (T)	Formula to Calculate FCVs for H=225 mg/L (ug/L)	Calculated FCVs for H=225 mg/L (ug/L)	HNDV (ug/L)
Barium	$=EXP(1.0629*(LN(H))+1.1889)$	NA	NA	$=EXP(1.0629*(LN(225))+1.1889)$	1,037	180,000
Cadmium	$=(EXP(0.7852*(LN(H))-2.715))*CF*T$	$1.101672-(LN(225)*0.04184)-0.875$	2.1	$=(EXP(0.7852*(LN(225))-2.715))*0.875*2.1$	9	130
Chromium III	$=(EXP(0.819*(LN(H))+0.6848))*CF*T$	0.88	1.5	$=(EXP(0.819*(LN(225))+0.6848))*0.88*1.5$	216	9,400
Copper	$=(EXP(0.8545*(LN(H))-1.702))*CF*T$	0.98	1.5	$=(EXP(0.8545*(LN(225))-1.702))*0.98*1.5$	27	64,000
Lead	$=(EXP(1.273*(LN(H))-3.298))*CF*T$	$1.46203-(LN(225)*0.14571)-0.652$	4.5	$=(EXP(1.273*(LN(225))-3.298))*0.652*4.5$	107	190
Manganese	$=EXP(0.8784*(LN(H))+2.226)$	NA	NA	$=EXP(0.8784*(LN(225))+2.226)$	1,079	59,000
Nickel	$=(EXP(0.846*(LN(H))+0.0584))*CF*T$	0.997	2.31	$=(EXP(0.846*(LN(225))+0.0584))*0.997*2.31$	239	210,000
Zinc	$=(EXP(0.8473*(LN(H))+0.684))*CF*T$	0.966	2.1	$=(EXP(0.8473*(LN(225))+0.684))*0.966*2.1$	493	22,000
<b>Notes:</b>						
1. All formulae and values, except site-specific hardness and total suspended solids (TSS), are from Footnote (G) of the ERD Part 201 Integrated Table of Cleanup Criteria (June 7, 2000).						
2. Hardness for Indian Mill Creek in Kent County =225.0 mg/L CaCO <sub>3</sub> (per Jack Wuycheck, MDEQ).						
3. The dissolved-to-total metal translator for Nickel was calculated using 10 mg/L TSS for the Indian Mill Creek (as recommended by Brenda Sayles and Jack Wuycheck, MDEQ, June 26, 2000), as follows: $1+(0.49(ss)/0.4281)=2.31$ .						
4. For all chemicals, the surface water human non-drinking water value (HNDV) is greater than the calculated FCV. In accordance with Footnote (G) of the ERD Part 201 Integrated Table of Cleanup Criteria, the generic GSI criterion is the lesser of the calculated FCV and the HNDV.						