APPENDIX A5

TEST AMERICA

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TESTAMERICA ANALYTICAL TESTING CORPORATION QUALITY ASSURANCE / QUALITY CONTROL MANUAL

DAYTON DIVISION	INDIANAPOLIS DIVISION	PONTIAC DIVISION
3601 SOUTH DIXIE DRIVE	6964 HILLSDALE COURT	341 W. WALTON BLVD.
DAYTON, OH 45439 (937) 294-6856 FAX: (937) 294-7816	INDIANAPOLIS, IN 46250 (317) 842-4261 FAX: (317) 842-4286	PONTIAC, MI 48340 (248) 332-1940 FAX: (248) 332-5450

Effective Date: December 15, 2004

Prepared by:

TestAmerica Analytical Testing Corporation– Dayton, Indianapolis, and Pontiac Laboratories

and

TestAmerica Analytical Testing Corporation
122 Lyman St
Asheville, NC 28803
(828) 654-9242

_____ _____

Division Manager –Chris Weathington Date

_____ _____

Quality Assurance Officer – Jim Davis Date

This QA Manual is reviewed on an annual basis. This copy is not automatically scheduled to receive any updates unless a written request is sent directly to the laboratory's Quality Assurance Department.

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Section 3.0
Introduction / Policy Statement

The TestAmerica Analytical Testing Corporation –Quality Assurance Manual is a document prepared to define the overall policies, organizational objectives and functional responsibilities for achieving TestAmerica Analytical Testing Corporation's data quality goals. Each TestAmerica location maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

3.1 Policy Statement

The management of TestAmerica is committed to providing high quality and legally defensible data to its clients by adhering to approved methodologies and the QA/QC protocols described in this manual.

It is TestAmerica's policy to continually improve systems and provide support to quality improvement efforts. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire laboratory staff.

In this document we define the following terms as follows:

Quality Assurance -	the total integrated program for assuring reliability of monitoring and measurement data.
Quality Control -	the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

Elements of TestAmerica's QA program include:

- Standard Operating Procedures (SOPs) for instrumentation, field services, analytical services and applicable administrative systems.
- A quality control program that includes analysis of blanks, spikes, duplicates, calibration verification and other appropriate Quality Control samples to ensure that data quality objectives are met.
- Participation in performance testing (PT) programs such as Water Pollution, Water Supply, Solid Waste, and various client specified programs.
- Internal audit programs to monitor compliance with the Quality Assurance Manual, SOPs and to evaluate performance of analytical methods used in the laboratory.
- Maintenance of state and national laboratory accreditations.
- Implementation of a Corporate Quality Ethics Policy that defines ethical and legal responsibilities. A copy can be found in Appendix 1.

3.2 Fields of Testing Covered

The methods specifically covered by this manual include the most frequently requested water, industrial waste, and soil methodologies currently needed to provide analytical services in the United States and its territories. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by TestAmerica shall meet these criteria as

appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases the laboratory will abide by the more stringent criteria following review and acceptance of the requirements by the Division Manager, Department Supervisor, and the Quality Assurance Officer.

3.3 Management of the Manual

The manual has been prepared to include sections addressing the content requirements specified by the latest NELAC standards, ISO Guide 17025, and QAMS-005/80, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans USEPA Dec 29, 1980. The outline format is based on the Florida DEP publication outlining Quality Assurance Plan requirements, DEP Manual for Preparing Quality Assurance Plans – DEP-QA-001/90.

3.3.1 Review Process

The manual is reviewed annually by the Quality Assurance Officer and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica clients and regulators. From time to time the manual may need to be revised in order to meet new or changing regulations and operational changes. The Quality Assurance Officer will review the changes in the normal course of business and incorporate changes into revised sections of the document. The updates will be reviewed by the Quality Assurance Officer, Division Manager, Department Supervisors, relevant operational staff and the Corporate Director of Quality Assurance and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM template that is prepared and approved by the Senior Vice President of Operations and the Corporate Director of Quality Assurance. This template is reviewed annually by the Senior Vice President of Operations, Corporate Director of Quality Assurance and each laboratory. Necessary changes are coordinated by the Corporate Director of Quality Assurance and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QA Manual that require immediate attention may be addressed through the use of Corporate or division specific QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QA Manual and all other Standard Operating Procedures (see Section 3.3.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QA Manual between the cover page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy Memoranda are signed by the Senior Vice President of Operations and the Director of Quality Assurance. The QA/QC Policy memoranda are incorporated into the QA Manual during the periodic updates. An example format can be found in Figure 3-1.

3.3.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica Analytical Testing Corporation. If the document has been provided to external users or regulators it is for the exclusive purpose of reviewing TestAmerica's quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica Analytical Testing Corporation. The procedure for control of distribution is incorporated by reference to SOP CP01-02: Distribution and Control of Standard Operating Procedures and the QA Manual.

3.3.3 Order of Precedence

In the event of conflict or discrepancy between policies, the order of precedence is as follows:

1. TestAmerica Incorporated QA/QC Policy Memorandum - Corporate
2. TestAmerica Incorporated QA/QC Policy Memorandum – Division
3. Quality Assurance Manual
4. Corporate SOPs
5. Division SOPs
6. Other (memos, flow charts, etc.)

Figure 3-1

Example Format for a QA/QC Policy Memorandum

Corporate (or Local) QA/QC Policy Memorandum # _____

Effective Date: _____

Corporate: *(Only needed for Corporate Memorandum – Delete if Local)*

_____ _____
Senior Vice President of Operations Date
Director of Quality Assurance Date

Local:

_____ _____
Division Manager Approval DateQu

1. **Purpose**

2. **Procedure**

3. **Documentation**

4. **Attachments**

5. **References/Cross References**

Section 4.0
Organization and Responsibility

4.1 Organization

TestAmerica – Dayton, Indianapolis and Pontiac are part of a national network of laboratories known as TestAmerica Analytical Testing Corporation. This Quality Assurance Manual (QAM) is applicable to the Dayton, Indianapolis and Pontiac laboratories only. The corporate organization related to the laboratories can be found in Figure 4-1 and the laboratories' organizations can be found in Appendix 2. The locations of all TestAmerica laboratories/Sales Departments are as follows:

Asheville Corporate
122 Lyman St.
Asheville, NC 28801
Phone: (828) 258-3746

Cedar Falls Division
704 Enterprise Drive
Cedar Falls, IA 50613
Phone: (319) 277-2401

Pontiac Laboratory
339 W. Walton Blvd.
Pontiac, MI 48340
Phone: (248) 332-1940

Orlando Division
4310 E. Anderson Rd
Orlando, FL 32812
Phone: (407) 851-2560

Indianapolis Division
6964 Hillsdale Court
Indianapolis, IN 46250
Phone: (317) 842-4261

East Dundee Sales Department
1090 Rock Road Lane, Unit 11
East Dundee, IL 60118
Phone: (847) 783-4960

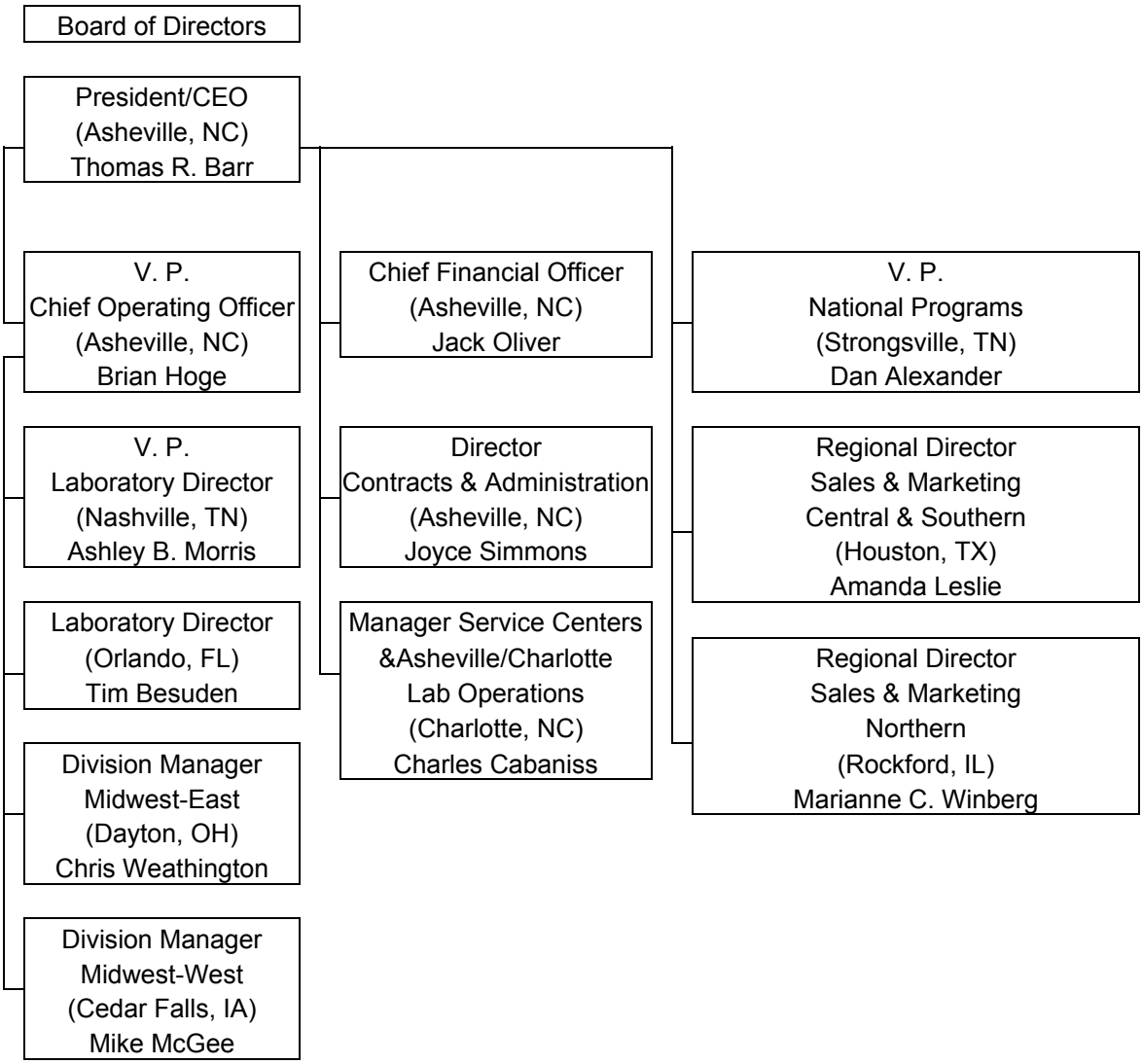
Dayton Division
3601 South Dixie Drive
Dayton, OH 45439
Phone: (937) 294-685

Watertown Division
602 Commerce Drive
Watertown, WI 53094
Phone: (920) 261-1660

Nashville Division
2960 Foster Creighton Dr.
Nashville, TN 37204
Phone: (615) 726-0177

Columbia Service Center
2417 Pine Tops Road
Columbia, SC 29210
(877) 240-3489

Figure 4-1 Corporate Organization Chart



4.2 Roles and Responsibilities

In order for the Quality Assurance program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to QA/QC. The descriptions following define each role in its relationship to the quality assurance program. Comprehensive job descriptions are maintained by laboratory management. Every employee has direct access to the Quality Assurance Manual (QAM) and training is provided in order to help each employee apply the QAM to his or her specific responsibilities.

4.2.1 Responsibility for the Quality Assurance Program

The responsibility for quality lies with every employee of TestAmerica. All employees are responsible for knowing the content of this manual and upholding the standards therein. Each person shall carry out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

4.2.2 President

The President reports directly to the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica Analytical Testing Corporation operations. The President establishes the overall quality standard for the company and provides the necessary leadership and resources to assure that the standard is met.

4.2.3 Vice Presidents of Laboratory Operations

The Vice Presidents of Laboratory Operations report directly to the President and are responsible for supporting the overall quality standard of the company by working with Division Managers and Laboratory Managers in providing the resources for implementing each Division or Laboratory's Quality Assurance program. The Vice Presidents review and approve the Corporate QAM template that is used by each laboratory to prepare a Division specific QAM. The Vice Presidents are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this Manual.

4.2.4 Director of Quality Assurance

The Director of Quality Assurance reports directly to the President. With the aid of the Vice Presidents, Division Managers, Laboratory Managers and Quality Assurance Officers, the Director of Quality Assurance has the responsibility for the establishment, general overview, and Corporate maintenance of the quality assurance program within TestAmerica Analytical Testing Corporation. Additional responsibilities of the Director of Quality Assurance include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QA Manual Template that is used by each laboratory to prepare their own Division specific QAM.
- Oversight of the QA/QC programs within each laboratory. This occurs through a final review of each Division or Laboratory specific QAM prior to finalization and by receiving a copy of each laboratory's QA monthly report.
- Participation, when requested, in the hiring of laboratory Quality Assurance staff.
- Maintenance of an archive for laboratory QA Manuals, Corporate Quality Policy memorandums and Corporate SOPs.
- Assisting laboratories with certification activities.

4.2.5 Division Manager

TestAmerica's Division Manager is responsible for the overall quality, financial, technical, human resource and service performance of the entire laboratory and reports to Corporate Operations. The Division Manager provides the resources necessary to implement and maintain an effective and comprehensive quality assurance program. In the absence of the Division Manager, the Department Supervisors and/or the Quality Assurance Officer fulfills the Division Manager's responsibilities. Specific responsibilities include, but are not limited to:

- Provides technical guidance to the analytical staff. The Division Manager may be the source point for technical help or designate an individual(s) to fill this role. A Division Manager appoints the technical directors for the appropriate fields of testing. The names of the technical director will be included in the national database.

  If a technical director is absent for a period of time exceeding 15 consecutive calendar days, the Division Manager must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function (this can be one of the other technical directors). If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.

- Ensures that personnel are free from any commercial, financial, and other undue pressures that might adversely affect the quality of their work.

- Ensure TestAmerica's human resource policies are adhered to and maintained.

- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be suspended by the Division Manager.

- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.

- Establishes and maintains a laboratory information system for tracking all samples in the laboratory and utilizes the system to ensure all sample holding times are met.

- Assumes the responsibilities of the QAO in the temporary absence of the QAO.

4.2.6 Quality Assurance Officer (QAO)

The QAO works with laboratory management to ensure that systems are maintained to produce data that is technically sound, legally defensible and of consistent quality in line with the laboratory's QAM and Standard Operating Procedures (SOPs).

The QAO reports directly to the Division Manager. The Corporate Director of Quality Assurance may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QAO serves as one of the Laboratories Technical Directors. Specific responsibilities include, but are not limited to:

- Serve as the focal point for QA/QC and be responsible for the oversight and/or review of Quality Control Data.
- Maintain and update the laboratory specific QAM. Ensure that the manual accurately reflects the procedures of the laboratory.
- Monitor and communicate regulatory changes that may affect the laboratory to management, marketing and the Corporate Director of Quality Assurance.
- Pursues and maintains appropriate laboratory certification and contract approvals. Arranges for the analysis of Performance Testing samples necessary to satisfy certification requirements.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or have the means of getting this information when needed).
- Arrange for or conduct internal audits.
- Notify laboratory management of deficiencies in the quality system and ensure corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs must be temporarily suspended or restricted following the procedures outlined in Section 13 (Corrective Action).

4.2.7 Operations Managers

The Operations Manager reports directly to the Division Manager and is responsible for the actual day-to-day supervision of laboratory procedures and reporting of results. Specific responsibilities include, but are not limited to:

- Monitoring standards of performance in quality control and quality assurance.
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Provide training direction to laboratory staff.
- Monitor production efforts of the lab. Look for ways to enhance production and improve quality through technical advances and improved LIMS utilization.
- Coordinate and review preparation of all test method Standard Operating Procedures.
- The Operations Manager can serve as a Technical Director and Department Supervisor.

4.2.8 Department Supervisors

Department Supervisors report directly to the Operations Manager and are responsible for ensuring that valid data are produced from those analyses within their departments. Department Supervisors serve as Technical directors. Responsibilities include:

- Supervise analysts to ensure adherence to applicable SOPs and the QA Manual.
- Ensure that all analysts in their group receive proper training and that training is documented following the procedures outlined in Section 8.
- Schedule sample analysis to ensure holding times and project due dates are met.
- Provide technical guidance to analysts in resolving analytical or instrument-related problems encountered during sample preparation and/or analysis. Technical guidance is also provided to Project Coordinators and the Quality Assurance Officer.

- Review or ensure review of all logbooks used by their department to document standard preparation, sample analysis, instrument maintenance and repair and other quality control activities outlined in this manual.

- Report all nonconformances, QC failures, and other problems with potential impact on data quality to the Quality Assurance Officer, Project Coordinator and to the Division Manager (when appropriate).

- Ensure preventive maintenance is performed on the instruments as outlined in the QAM or SOPs.

- Maintain adequate inventory of standards, reagents, and other materials required for the performance of routine analyses.

- Maintain current quality control data for updating lab control limits.

- Ensure MDLs (Method Detection Limit study) are determined for every instrument, matrix, and method.

- Assist the Division Manager with LIMS updates.

4.2.9 **Project Managers** – The Project Managers report to the Project Management Supervisor and serve as a liaison between the laboratory and its clients. The project manager's responsibilities include:

- Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory.

- Notify laboratory personnel of incoming projects and sample delivery schedules.

- Monitor the status of all projects in-house to ensure timely delivery of reports.

- Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.

- Coordinate client requests for sample containers and other services.

- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.

- Coordinate subcontract work.

- Prepare Data Packages

- Review laboratory data reports, quotes, and sample login sheets.

- Reviews and approves data reports prior to their release to the clients. Ensures client specific reporting and quality control requirements are met**.**

4.2.10 **Laboratory Analysts and Technicians** – Laboratory analysts and technicians are responsible for conducting analysis and performing all tasks assigned to them by the Department Supervisor and the Division Manager. The responsibilities of the analysts and technicians are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by SOPs, this QA Manual and project specific QAPPs.

- Document standard and sample preparation, instrument calibration and maintenance, data calculations and any observed nonconformance.

- Report all out-of-control situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to the Department Supervisor.

- Initiate corrective actions or seek technical assistance from Department Supervisors.

- Perform 100% review of the data generated prior to entering and submitting for secondary level review. Perform peer review of data as appropriate.

- Maintain control charts as appropriate to monitor analytical performance.

- Ensure sample analysis is completed within specified hold times or immediately inform the Department Supervisor if a hold time will not be met.

- Suggest method improvements to the Department Supervisor and the Quality Assurance Officer. These improvements, if approved, will be incorporated into the SOPs.

4.2.11 **Safety Officer** – The Safety Officer reports to the Division Manager and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer's responsibilities are outlined below:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.

- Administer dispersal of all Material Safety Data Sheet (MSDS) information.

- Perform regular chemical hygiene and housekeeping instruction.

- Give instruction on proper labeling and practice

- Serve as chairman of the laboratory safety committee.

- Ensure appropriately trained personnel and general protective equipment is available as needed.

- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.

- Supervise and schedule fire drills and emergency evacuation drills.

- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.

- When determined necessary, conduct exposure-monitoring assessments.

- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.

4.2.12 **Office Manager** – The Office Manager reports to the Division Manager. The responsibilities of the Office Manager are outlined below:

- Insure timely and correct shipment of sample containers to clients. Maintain accurate records of sample container shipments.

- Supervise purchasing, shipping, and receiving.

- Manages personnel records, actions.

- Supervise Reception and Invoicing.

4.2.13 **Project Coordinators** – The Project Coordinators report to the Project Management Supervisor and serve as the interface between the laboratory's technical departments and the laboratory's clients. The responsibilities of the Project Coordinator are outlined below:

- Assist clients in procuring the proper sampling supplies.

- Respond to client inquiries concerning sample status.

- Assist clients with resolution of problems concerning Chains-of-Custody.

- Prepare bottle Orders

- Assist the Project Managers with preparing client reports.

4.2.14 **Field Services Manager -** The Field Services Manager reports to the Division Manager. The responsibilities of the Field Services Manager are outlined below:

- Schedule sample collection events with clients and project management team.
- Maintain the necessary equipment and vehicles to conduct Field Sampling.
- Train new Field Services Technicians in proper sampling techniques.
- Supervise the completion of all field reports.

4.2.15 **Field Services Technician** – The Field Services Technician reports to the Field Services Manager. The responsibilities of the Field Services Technician are outlined below:

- Perform sample collection and sample pick-up
- Ensure sample containers media are prepared for sampling and assist in shipping sample containers to clients.
- Perform field tests and measurements and operate and maintain equipment used for those purposes.
- Prepare Field Reports

4.3 Education and Experience Requirements for Technical Personnel

The following are the education/experience requirements for laboratory technical personnel. Records of relevant qualifications, training, skills and experience of the technical personnel are maintained by the laboratory (see Section 8 – Analytical Procedures).

4.3.1 Division Manager– A bachelor's degree in science is required. If a bachelor's degree is in a field other than chemistry, the individual should have the number of credit hours in chemistry equivalent to a minor in chemistry.

4.3.2 Technical Directors – A bachelor's degree in the chemical, environmental, biological sciences or engineering, with at least 24 college semester credit hours in chemistry and at least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

4.3.3 Department Supervisors – A bachelor's degree in the chemical, environmental, biological sciences, physical sciences or engineering is required, with at least two years of experience in environmental laboratory analysis.

4.3.4 Quality Assurance Officer – A bachelor's degree in basic or applied science and at least 1 year of nonacademic analytical chemistry, or in lieu of a degree, 4 years of nonacademic analytical chemistry experience. Previous experience or

documented training in statistics or quality control procedures is required.

4.3.5 Analyst – An analyst must possess a high school diploma or equivalent. If the analyst operates ICP, ICPMS, GC or GC/MS equipment, the analyst must satisfactory complete a short course offered by an equipment manufacturer, professional organization, university, or other qualified training facility (formal in-house training is acceptable). A minimum experience requirement for the operation of GFAA, ICP, ICPMS, GC, and GCMS equipment is 1 year. The analyst must follow training and documentation as summarized in Section 8.

4.4 Physical Facilities

4.4.1 Environment

4.4.1.1 Laboratory accommodation, test areas, energy sources, lighting, heating and ventilation must be adequate to facilitate proper performance of tests.

4.4.1.2 The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement

4.4.1.3 The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate

4.4.1.4 In instances where monitoring or control of environmental conditions is specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

4.4.2 Work Areas

4.4.2.1 There shall be effective separation between neighboring areas when the activities therein are incompatible including culture handling or incubation areas and volatile organic chemicals handling areas.

4.4.2.2 Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

4.4.2.3 Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

4.4.2.4 Work areas must be available to ensure an unencumbered work area. Work areas include:

a) Access and entryways to the laboratory
b) Sample receipt areas
c) Sample storage areas
d) Chemical and waste storage areas
e) Data handling and storage areas

f) Analytical testing areas

4.4.3 Floor Plan

A floor plan can be found in Appendix 3.

4.4.4 Building Security

4.4.4.1 Each visitor to the laboratory must sign in and out in a visitor's logbook.

4.4.4.2 The laboratory maintains a contract with a building alarm services company. The keys and alarm code are distributed to key personnel who need access to the laboratory after normal business hours.

4.4.4.3 Signs are posted within the laboratory to limit access to non-laboratory personnel.

Section 5.0
QA Objectives for the Measurement of Data

The TestAmerica quality assurance objectives are described in terms of precision, accuracy, representativeness, comparability, completeness and uncertainty. Criteria for data quality indicators such as matrix spikes, laboratory control samples and duplicate sample precision as well as reporting limits are specified in the Control Limits Manual.

5.1 Precision

The laboratory objective for precision is to meet the precision demonstrated for these analytical methods on similar samples and to meet data for these analyses published by the US EPA. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike duplicate samples.

5.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for these analytical methods on similar samples and to meet the recovery data published by the US EPA. Accuracy is defined as the degree of bias in a measurement system. Accuracy is documented on the basis of recovery of matrix spikes. Accuracy may also be documented using laboratory control samples. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory can assist the customer with enacting proper sampling and handling methods in order to assure the integrity of the samples.

5.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated:

a) by other laboratories for similar samples, and
b) data generated by each individual TestAmerica division over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

5.8 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.6 Criteria for Quality Indicators

The Tables in this section summarize the precision and accuracy acceptability limits for analyses performed at the TestAmerica Division. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are provided. Where US EPA method limits are not provided, TestAmerica has developed limits from evaluation of data from similar matrices. Acceptability of QC will be determined as compared to these tables. Data may be accepted where QC falls outside these limits if probable cause can be attributed to the matrix and laboratory control samples (LCS) show that the method is in control. Deviations are documented in the final report to the client. In instances where an LCS limit is not available, a limit of 70 – 130% recovery is acceptable until in-house limits can be generated (Note: Ensure that an alternative default limit is not available in a published method). In some cases, lower default limits may be set with the quality assurance officer and technical director's approval. In the absence of in house or method defined limits, the following guidelines may be used to determine interim limits for matrix spike and matrix spike/matrix spike duplicates: MS: 60-140% MS/MSD: 20% RPD. Some compounds may need to be wider based on prior knowledge of the compound (e.g. Phenols in EPA 8270).

5.7 Control Limits:

The current standard reporting limits can be found in the Control Limits Manual. The limits listed in the Control Limits Manual are determined in three ways: statistically derived by the laboratory, from the listed method when available or derived from other means.

5.7.1 Statistically Derived Limits:

Statistically derived limits are generated by collecting a database of 20-30 LCS points for each analysis from multiple instruments (if applicable). The mean is calculated expressed as percent recovery and the standard deviation(s). The upper control limit (UCL) is equal to the mean of the database plus 3 standard deviations. The upper warning limit (UWL) is equal to the mean of the database plus 2 standard deviations. The lower control limit (LCL) is equal to the mean of the database minus 3 standard deviations. The lower warning limit (LWL) is equal to the mean of the database minus 2 standard deviations. Outliers may be excluded from the database for the following reasons: incorrect spike amount addition, no spike addition, instrument and/or preparation failure of which the cause can be determined and documented. The control limits and warning limits are generated yearly or whenever the process is changed. The data is plotted on a control chart. The purpose of control charting is to obtain real-time trend analysis of method performance. TestAmerica will routinely utilize statistically derived limits to evaluate method performance and determine when corrective action is appropriate.

5.7.2 Method Based Limits:

Method-based limits can also be used if there are not enough data points generated to determine the control limit range for the analysis. If the method based limits are used for an analysis, the limits will be noted in the Control Limits Binder.

5.7.3 Other Means of Determining Limits:

Arbitrarily determined limits will be used if there are not enough points to determine the statistical limits, if the statistically generated limits are considered to be too wide for the analysis, and there is not a method-based limits determination for an analysis. These limits will also be noted in the Control Limits Binder if they are used for an analysis.

The laboratory periodically updates the limits as stated in this manual. The limits are updated a minimum of once a year or more frequently if required/ The analysts are instructed to use the current limits posted in the laboratory or Control Limits Manual (dated and approved by the Department Supervisor and QA Officer). The Quality Assurance Officer maintains an archive of all limits used within the laboratory. The limits in the Control Limits Binder take precedence over those mentioned in individual SOPs or limits posted in the laboratory if there is a discrepancy.

5.8 Measurement Uncertainty

5.8.1 Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as

adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

5.8.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

5.8.3 The uncertainty associated with results generated at each TestAmerica Analytical Testing division can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS is used to assess the performance of the measurement system and is analyzed once for every 20 samples. The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

5.8.4 The range for each analyte is shown in a table of the Control Limits Manual generated for each laboratory; the tables are arranged by method and matrix (water or solid). Find the analyte for the method and matrix of interest and note the percent recovery range. To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the range percent value, and multiply the result by the decimal of the upper end of the range percent value. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

Section 6.0
Sampling Procedures

TestAmerica Analytical Testing Corporation provides sampling services. Many samples received by the laboratory are not sampled by laboratory field personnel. In these cases, the laboratory's responsibility in the sample collection process lies in supplying the sampler with the proper containers and preservatives.

6.1 Sampling Containers

TestAmerica offers pre-cleaned sampling containers for use by clients and laboratory field sampling personnel. These containers are obtained from reputable container manufacturers and are cleaned to EPA specifications.

Commercial pre-cleaned containers are used for all sampling.

6.1.1 Preservatives

Upon request, preservatives are provided to the client or laboratory field sampling personnel in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved by the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate –ACS grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS grade or equivalent
- Ascorbic Acid – ACS grade or equivalent

6.1.2 Preparing Bottle Orders

The date of receipt of the containers by TestAmerica Division is recorded on the pre-cleaned certificates received with the bottles (or on some type of documentation received with the bottles if certificates are not received). Upon request, the containers are then sent to clients for use in collecting samples or are used by field sampling personnel. The shipping date, type and number of containers are maintained by the lab. Shipping personnel will insure that bottle stock is rotated so that last in is first out. An example Bottle Order Form can be found in Figure 6-1. Completed forms are stored in Shipping and Receiving.

Figure 6-1: Example of Bottle Order Form

BOTTLE ORDER

		DATE: 12/14/2004	
ACCOUNT NAME:			
ACCOUNT ADDRESS:			
ATTN:		**PHONE:**	
BILLING:		**SHIPPING INSTRUCTIONS:**	
☐ Cash		☐ UPS	
☐ Account		☐ Pick-Up	
☐ Send Credit Application		☐ Route – Day/Date	
Project:			

TEST BOTTLES REQUIRED:

FORMS:
☐ Potable Chemical
☐ Total Coliform
___ Chain of Custody

SHIPPING CONTAINER:
☐ Box
☐ Cooler(s) w/packing
☐ Cooler(s) w/ blue ice

ROUTING:
_____ Initiated By
_____ Prepared By
_____ Date Sent

INSTRUCTIONS:
☐ Inorganic
☐ Bacteriological
☐ VOC's
☐ SOC's

THE FOLLOWING PARAMETERS ARE SUBCONTRACTED TO AN OUTSIDE LABORATORY:_____
_____

The laboratory also provides Encore sampling devices when requested.

If containers are provided direct to the client from the manufacturer or from other sources, TestAmerica Analytical Testing Corporation will not be responsible for any of the above records.

6.2 Field QC Samples

The common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the Quality Assurance Project Plan (QAPP). The recommended frequency is a minimum of one of each type per sampling day, per parameter class, or 5%, whichever is greater. TestAmerica analyzes trip blanks for VOC analysis with the sample containers when requested/provided. All blanks generated in the field will be analyzed in the analytical sequence along with the field samples.

6.2.1 Parameter Class

The parameter class is defined, for the purpose of determining the number of blank samples to be generated during the sampling episode, as a group of analytes for which the sample container type, container cleaning protocol, and preservation technique are identical. An example is total metals and hardness in water, which could be combined into a single sampling container.

6.2.2 Equipment Blank / Rinseate Blank

The equipment blank sometimes referred to as a rinseate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of analyte as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will be affected by the site and sample handling conditions evaluated by the other types of blanks.

6.2.3 Field Blank

The field blank is water that is as free of analyte as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination.

6.2.4 Trip Blank

The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers and are from the same batch as the empty containers being

taken to the field. They are filled with reagent grade water, sealed, and taken into the field with the empty containers that will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method.

6.2.5 Field Duplicates

Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

6.3 Field Sampling Procedures

The following procedures are followed when TestAmerica personnel perform field sampling. Sampling and ambient data collection will be performed in accordance with currently accepted guidelines outlined by EPA. The EPA Region IV Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual (May, 1996) is a reference and source of approved sampling procedures for field sampling. This reference is available for use by field personnel.

A list of TestAmerica's sampling capabilities is presented in Table 6-1. A number of different sampling protocols may need to be employed to satisfy specific project sampling requirements. The primary objective of any sampling program is to obtain a representative sample.

A thorough review of the sampling program and data collection objectives will be undertaken by the sampling project manager prior to establishing sampling stations and parameters to be analyzed. A sampling and analysis plan should be prepared to establish the best procedures to obtain representative data. This plan will be used to set each sampling point, determine the type of sample required, establish the analytical parameters, and determine the type of field equipment, sample containers and preservation techniques that will be necessary to complete the program.

The cleaning procedures outlined in this section are to be used by all laboratory and field personnel to clean sampling and other field equipment prior to sample collection. Whenever possible, a sufficient quantity of clean equipment will be transported to the sampling site to insure that the entire sampling event may be conducted without the need for cleaning (decontamination) in the field. Whenever field equipment cleaning is required, the procedures are recorded in the field records and logbooks.

Field equipment and instrumentation used by TestAmerica is listed in each section of sampling protocols (Tables 6.2 - 6.8). Routinely used field measurement instrumentation is listed in Section 9 (Calibration), Table 9-1. Miscellaneous equipment used in all types of sampling events is listed in Table 6-9.

Disposable gloves will be worn while sampling at all sampling sites involving organic or infectious wastes. New, unused gloves are worn for each separate sampling point (Note: Neoprene or rubber gloves may also be required for hazardous waste sampling).

The following cleaning and decontamination equipment and reagents may be present at the sampling site when field cleaning is anticipated: tap water; analyte-free water; Alconox or an equivalent laboratory-grade detergent; 10% nitric acid; cleaning brushes; wash tubs and waste receptacles. Tap water and deionized water are transported to the sampling site in pre-cleaned polyethylene containers. The 10% nitric acid is transported in an appropriately marked glass reagent bottle. All equipment that must be cleaned using solvents such as methylene chloride will be cleaned in the laboratory.

Analyte free water is defined as being free of interferences and analytes of interest (below laboratory detection limits). TestAmerica utilizes a commercial water treatment system, which employs anion/cation exchange resins, that produces water equivalent to ASTM type II water. Analyte-free water will be used for all blank preparations was well as final decontamination rinses. Analytical results from method blanks and/or sample equipment blanks are documented to demonstrate the reliability and purity of analyte-free water sources Analyte free water is stored and transported in either glass or HDPE containers, depending on its intended use.

Sampling container sets are assembled in the laboratory prior to transportation to the sampling site. If a preservative has been added to the container, it is noted on the container. A temporary field ID number may also be assigned to each container at this time. Fresh preservatives are obtained from stocks prior to the assemblage of each container set. Liquid sample fractions are preserved as specified in Table 6-10. Preservation methods and holding times for drinking water samples that differ from 40CFR Part 136; Table II are listed in Table 6-11. Soils, sediments, and sludge sample fractions are preserved as specified in Table 6-12.

Table 6-13 lists the sample preservation reagents and standards that the field personnel routinely use. Table 6-13 also includes the grade of reagents and method of storage and transportation. Reagents, standards, and solvents are maintained, stored, and transported segregated in regards to grade of reagents, potential for contamination, and safety.

Samples are collected from the least to most contaminated area whenever possible. The following is the preferred order of sample collection: VOC; TOX; TOC; extractable organics (including TRPH and oil and grease); total metals; dissolved metals; microbiological; phenolics; cyanide; inorganics; turbidity, and radionuclides.

Duplicates are collected by sampling from successively collected volumes (i.e., samples from the next bailer of sample water).

Split samples are taken from consecutive sample volumes from the same sampling device (i.e., samples from the same bailer) or by mixing in a large intermediate vessel. Note: for large volume samples that may require more than one bailer full, the first half volume of the first bailer full is poured into the first container, and the second half is poured into the second container; then the first half volume of the second bailer full is poured into the second container, and the second half is poured into the first container. This procedure is continued until both containers are full.

In general, sample collection equipment and containers are rinsed with sample water before the actual sample is taken. Exceptions include oil and grease; TRPH; bacteriological; volatile organics, and sample containers containing a pre-measured preservative.

The pH will be checked for acid or base preserved samples with narrow range pH paper for all samples per each parameter on the first sampling event, and on one sample per each parameter for all successive events at the same site. An aliquot of the sample is poured into a disposable container or extra container “dummy sample”, used for the pH check, and then discarded. Any additional chemicals used to augment preservation in the field will be from the same source as the chemical used to preserve the sample. Use of additional preservative chemicals is documented on the field data sheets. If an additional pH adjustment is made to a sample, then the same amount of that chemical is also added to the appropriate equipment blank.

Careful attention is given to the collection of VOCs. Aqueous and solid samples should never be mixed or composited in the field unless called for in an approved Project Plan. Soil samples are collected using a variety of possible procedures depending on project requirements. Soils are packed into wide mouth glass containers with a glass, stainless steel, or Teflon implement suitable for this purpose. Headspace is minimized in soil sample containers. Please see Method 5035 for use of Encore samplers on volatile samples. Liquid samples are collected using a bottom draining bailer or by pouring the sample slowly down the inside edge of the sample vial to minimize aeration. The VOC vial is filled to the point of creating a convex meniscus. The cap of the vial is secured with the Teflon side of the septum contacting the sample. To check for headspace, the vial is inverted and lightly tapped. If air bubbles are present, more sample is added to the vial and again checked for air bubbles. Additional sample can only be added a maximum of three times. If unsuccessful, the sample, vial and septum are discarded. A new vial is used and the collection procedure begins again. Samples for organic analysis may be collected using a peristaltic pump. Figure 6-2 is a diagram of a system used for the collection of extractable organics with a peristaltic pump. Oil and grease and TRPH samples are collected in a well-mixed area of the sampling site and never from the surface of the water. Oil and grease and TRPH samples should not be composited (if collected for Method 1664, follow additional procedures outlined in method).

If required by the sampling and analysis plan, samples collected for the analysis of trace metals can be field filtered through a 0.45-micron filter. Figure 6-3 shows a diagram of the field filtration system used for the collection of trace metals. These samples are acidified following filtration. Figure 6-4 shows an alternative system using a positive pressure Teflon bailer arrangement. Unfiltered samples for the analysis of trace metals may also be collected in conjunction with filtered samples. Samples requiring field filtration for analysis other than trace metals may also be collected using these field filtration systems (i.e., 0.45 micron filter for Ortho-phosphorous).

Microbiological samples are never composited. Precautions are taken to maintain the sterility of the sample containers (i.e., not touching the top of the sample container). The samples are analyzed by membrane filter within six hours of collection. If necessary, commercial carriers are used to transport the samples to the laboratory for analysis before the expiration of the sample holding time. This limit is applied to fresh waters,

seawaters, and shellfish-bed waters. The exception is drinking water supply Total Coliform samples collected from water treatment systems. Current regulations permit these samples to be help up to 30 hours. If sampling and transit conditions for environmental microbiological samples require more than six hours, and the use of field laboratories is impossible, the delayed incubation procedure for total and fecal coliforms and fecal streptococci should be considered. These problems can be addressed in site-specific sampling and analysis plans.

Following the collection of the samples, the samples are placed in insulated coolers containing wet ice if the travel time is greater than 2 hours or if weather conditions may cause the samples to degrade and, if necessary, materials to avoid breakage. VOC samples from different locations may be placed into the same cooler to reduce the number of trip blanks required, provided that the samples are segregated (multiple vials from the same sampling site are placed into the same container, usually Ziplock bags or cans containing vermiculite). The samples are then transported to the laboratory by the sampling team or shipped by commercial carrier. Field generated wastes, with the exception of purge waters from groundwater monitoring wells, are segregated and containerized by the field personnel and returned to the laboratory for proper disposal. These wastes include: standards used for calibrations; solvents; acids and other chemicals that are used as a part of the cleaning/decontamination process, and other miscellaneous wastes that the sampling crew may generate. In the event that a monitoring well is known or suspected to be contaminated, then the purge water from the well is containerized and returned to the laboratory for proper disposal

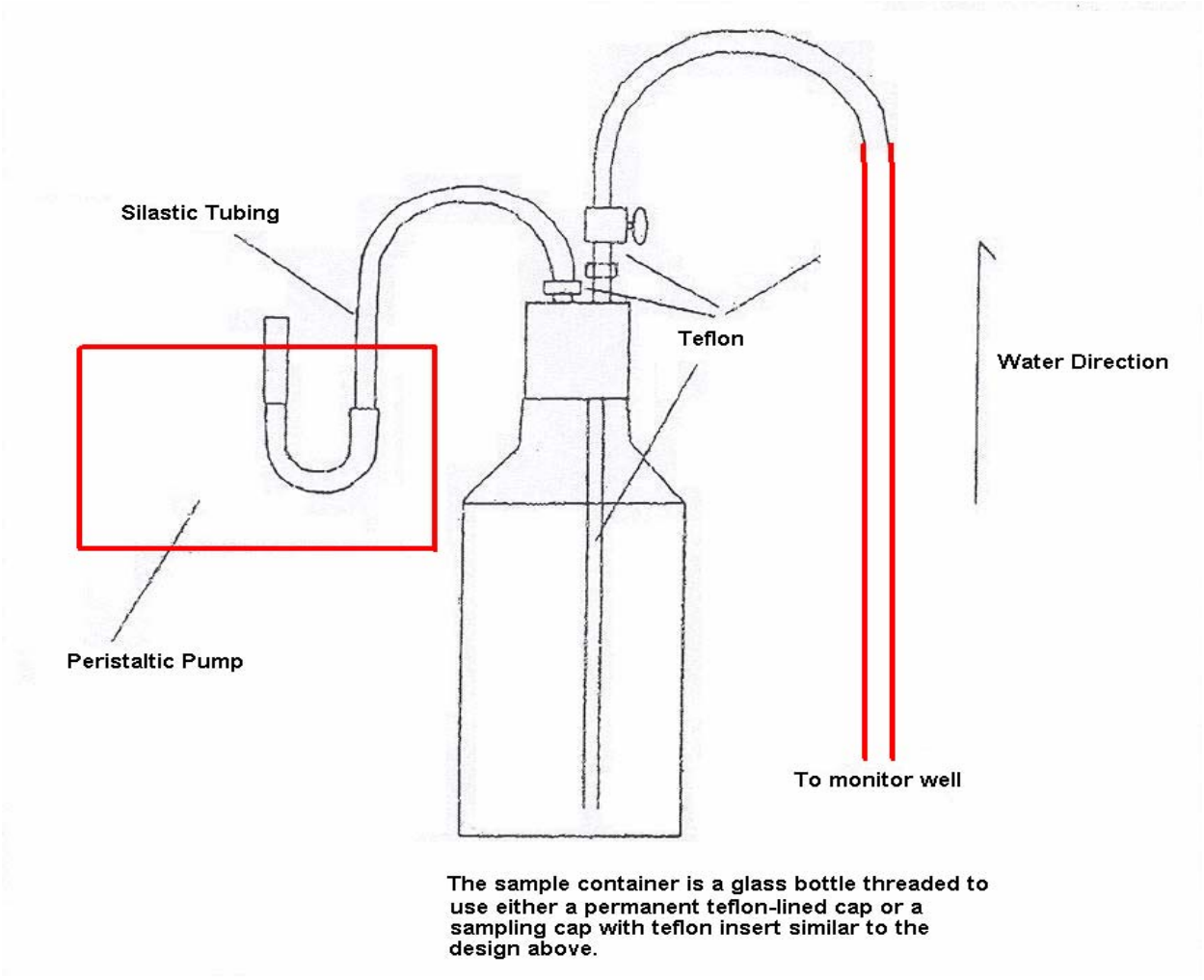
The field personnel are responsible for maintaining records for each sampling event. This is accomplished by using detailed field data sheets. These are field sheets for the sampling of ground water and surface water, soils, and sediments.

TABLE 6-1
SAMPLING CAPABILITIES

PARAMETER GROUP	SAMPLING SOURCE
Volatile and Extractable Organic Compounds	Groundwater, surface water, drinking water, wastewater, effluents, stormwater runoff, soils, sediments, chemical/hazardous wastes, domestic waste water sludges, hazardous waste sludges
Metals	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, tissues, chemical/hazardous wastes, domestic wastewater sludges, hazardous waste sludges
Inorganic Anions (Inorganic anions and other non-metallic tests)[1]	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, chemical/hazardous wastes, domestic wastewater sludges, hazardous waste sludges
Organics (BOD, COD, O&G, TOC, TRPH, Phenolics and Surfactants)	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, chemical/hazardous wastes, domestic wastewater sludges, hazardous waste sludges
Physical Properties	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, chemical/hazardous wastes, domestic wastewater sludges, hazardous waste sludges
Microbiology	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, domestic wastewater sludges
Cyanide	Groundwater, surface water, drinking water, wastewater effluents, stormwater runoff, soils, sediments, chemical/hazardous wastes, domestic wastewater sludges, hazardous waste sludges
Bioassay	Surface water, wastewater effluents, stormwater runoff

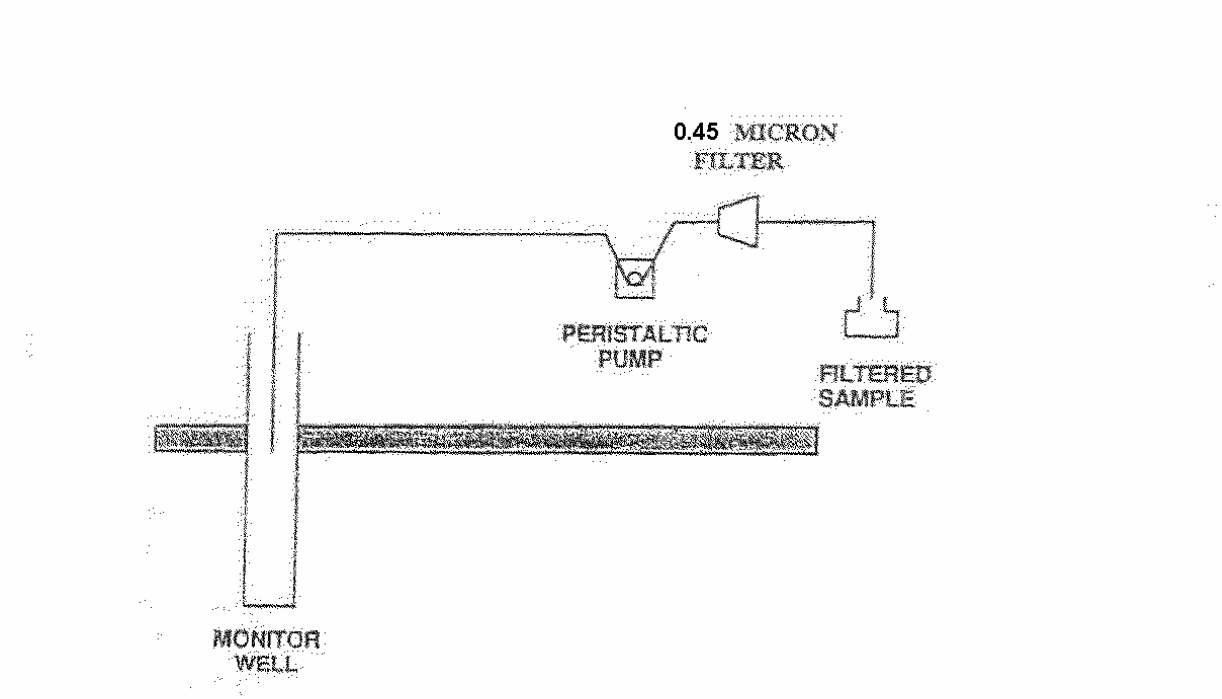
Key
1 Includes Bromide, Bromine, Chloride, Chlorine, Iodide, Nutrients, Sulfate, Silica, Sulfite, Acidity, Alkalinity, Dissolved Oxygen and Dissolved Silica.

Figure 6-2
DIAGRAM OF SYSTEM FOR COLLECTION EXTRACTABLE ORGANICS THROUGH A PERISTALTIC PUMP



The sample container is a glass bottle threaded to use either a permanent teflon-lined cap or a sampling cap with teflon insert similar to the design above.

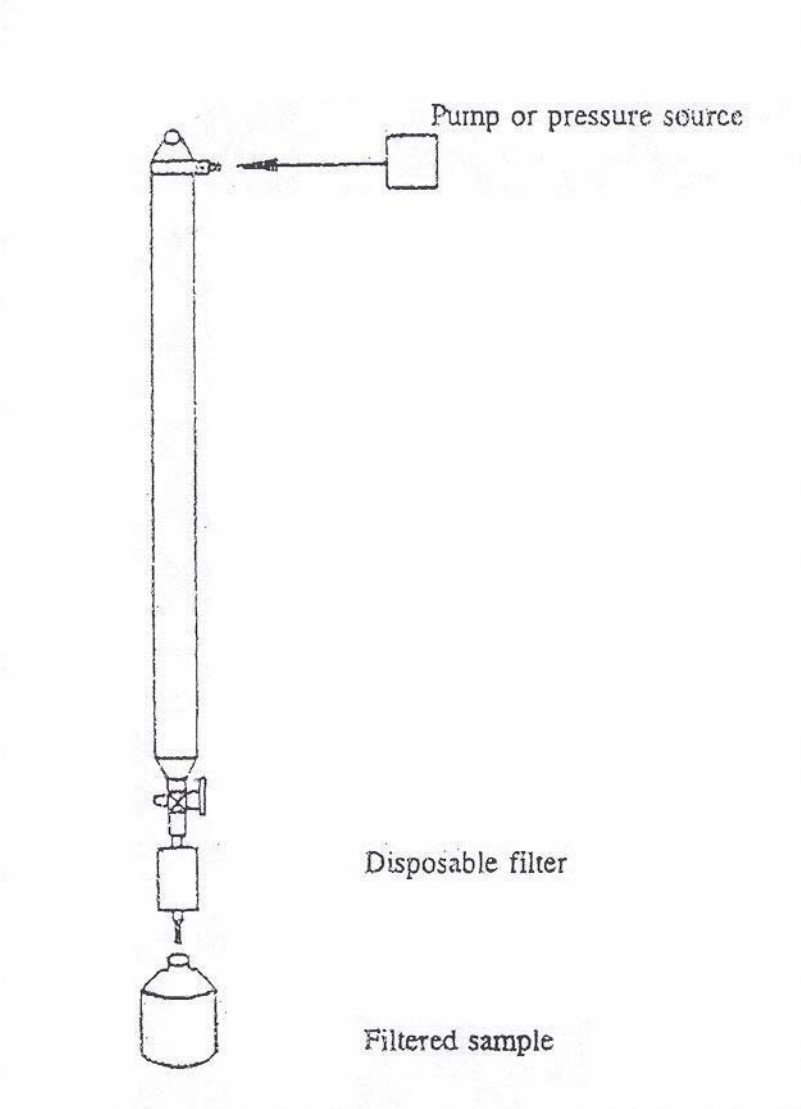
Figure 6-3
DIAGRAM OF RECOMMENDED FIELD FILTRATION SYSTEM FOR TRACE METALS



FIELD FILTRATION SCHEMATIC

1 Use one piece, molded, in-line high capacity disposable 1.0-micron filter.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the positive pressure side of the peristaltic pump.
4 If well is deeper than 25 feet; a submersible pump may be necessary to bring the sample o the surface. Sample should be collected in a suitable container and filters immediately, using the above system.
5. At least one filtered equipment blank using deionized water must be collected and analyzed.

Figure 6-4
DIAGRAM OF ALTERNATIVE FIELD FILTRATION SYSTEM FOR TRACE METALS USING A POSITIVE PRESSURE BAILER ARRANGEMENT



FIELD FILTRATION SCHEMATIC

1. Use one piece, molded, in-line high capacity disposable filter.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the exit side on the bailer stopcock.
4. At least one filtered equipment blank using deionized water must be collected and analyzed.

6.3.1 Cleaning Procedures

6.3.1.1 Laboratory Cleaning Procedures for Field Equipment

a) Cleaning Procedures for Teflon or Glass Field Sampling Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analysis[1]

1. Equipment will be washed thoroughly with Liquinox laboratory detergent or equivalent and hot tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment with at least a 10 percent nitric acid solution.[2]
4. Rinse equipment thoroughly with tap water.
5. Rinse equipment thoroughly with analyte-free water.
6. Rinse equipment twice and allow to air dry for at least 24 hours.
7. Rinse the Teflon or glass sampling equipment thoroughly with tap water in the field as soon as possible after use.

b) Cleaning Procedures for Stainless Steel or Metal Sampling Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analysis[3]

1. Wash equipment thoroughly with Liquinox laboratory detergent or equivalent and hot tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment thoroughly with analyte-free water.
4. Rinse equipment twice with isopropanol and allow to air dry for at least 24 hours.
5. Wrap equipment completely with aluminum foil to prevent contamination during storage and/or transportation to the sampling site.
6. Rinse the stainless steel or metal sampling equipment thoroughly with tap water in the field as soon as possible after use.

6.3.1.2 Cleaning Procedures for Automatic Sampling Equipment

a) ISCO Automatic Sampler

1. The exterior and accessible interior (excluding the waterproof timing mechanism) portions of automatic samplers will be washed with Liquinox laboratory detergent or equivalent and rinsed with hot tap water.
2. The face of the timing case mechanism will be cleaned with a clean damp cloth.
3. All tubing (sample intake and pump tubing) will be cleaned using Alconox Solution, Tap Water, 10 % Nitric, and DI water. Pump tubing will be discarded if it won't come clean or begins to show signs of wear (1 to 2 months depending upon usage).
4. New precleaned, silastic pump tubing will be installed.

5. When utilizing the samplers for collecting samples for metals and/or organic compound analyses, the metal distributor tubes should not be used; only glass or silastic pump tubing should be used for this purpose.

b) ISCO Metal Tube Automatic Sampler Rotary Funnel and Distributor and Automatic Sampler

1. Use only for non-organic sample collection using individual sequential bottles.
2. Clean with hot tap water, Liquinox laboratory detergent or equivalent and a brush.
3. Rinse thoroughly with analyte-free water.
4. Replace in sampler.

c) All Automatic Sampler Headers

1. Disassemble header and using a bottlebrush, wash with hot tap water and Liquinox laboratory detergent or equivalent.
2. Rinse thoroughly with analyte-free water.
3. Reassemble header, let dry thoroughly.

d) Reusable Glass Composite Sample Containers[4]

1. Wash containers thoroughly with hot tap water and Liquinox laboratory detergent or equivalent, using a bottle brush to remove particulate matter and surface film.
2. Rinse containers thoroughly with tap water.
3. Rinse containers with at least 10 percent nitric acid.
4. Rinse containers thoroughly with tap water.
5. Rinse containers thoroughly with analyte-free water.
6. Rinse twice and allow to air dry for at least 24 hours.
7. After using, rinse with tap water in the field, seal with aluminum foil to keep the interior of the container wet, and return to the laboratory for proper decontamination.

e) Plastic Reusable Composite Sample Containers[5]

1. Proceed with the cleaning procedures for the reusable glass composite sample container but omit the solvent rinse.

f) ISCO Glass Sequential Sample Bottles Automatic Sampler Base for Sequential Mode

1. Wash with Liquinox laboratory detergent or equivalent and hot tap water.
2. Rinse thoroughly with tap water.
3. Rinse with at least a 10 percent nitric acid solution.
4. Rinse thoroughly with analyte-free water.
5. Replace bottles in covered, automatic sampler base for storage.

g) ISCO Glass Sequential Sample Bottles (Automatic Sampler Base for

Sequential Mode) to be used for Collecting Samples for the Analysis of Organic Compounds

1. Proceed as outlined above.
2. Rinse twice and allow to air dry for at least 24 hours.
3. Replace in covered, automatic sampler base for storage and mark the base "Cleaned for organic analyses".

h) Bottle Siphons Used to Transfer Sample from Composite Container

1. Use a new siphon for each sampling location.
2. Use 3/8-inch Teflon tubing for samples collected for organic compound analyses. The tubing should be rinsed with solvent and dried in an oven overnight before use. The ends of the siphon should be capped with Teflon film for storage. The siphon should be flushed with sample thoroughly before use.
3. Use 3/8-inch PVC tubing utilized for samples, other than those collected for organic compound analysis. The tubing should be thoroughly flushed with sample before use.

6.3.1.3 Cleaning Procedures for Sample Tubing

a) Silastic Rubber Pump Tubing Used in Automatic Samplers and Other Peristaltic Pumps

1. Flush tubing with tap water and Liquinox laboratory detergent or equivalent.
2. Rinse tubing thoroughly with tap water.
3. Rinse tubing with analyte-free water.
4. Install tubing in automatic sampler or peristaltic pump.
5. Cap both ends of tubing.

New pre-cleaned tubing must be used for each automatic sampler set-up. The silastic rubber pump tubing need not be replaced in peristaltic pumps where the sample does not contact the tubing or where the pump is being used for purging purposes (i.e., not being used to collect samples) [11].

b) Teflon Sample Tubing

Use only new Teflon tubing pre-cleaned as follows for collection of samples for organic compound analyses:

1. Teflon tubing should be precut in convenient lengths before cleaning.
2. Rinse outside of tubing.
3. ~~3.~~ Flush interior of tubing.
4. Wrap tubing and cap ends with aluminum foil to prevent contamination during storage.

c) Stainless Steel Tubing

1. Wash tubing thoroughly with Liquinox laboratory detergent or equivalent and hot tap water using a long, narrow, bottlebrush to remove any particulate matter or surface film.
2. Rinse tubing thoroughly with tap water.
3. Rinse tubing thoroughly with analyte-free water.
4. Rinse tubing twice and allow to air dry for at least 24 hours.
5. Rinse the stainless steel or metal sampling tubing thoroughly with tap water in the field as soon as possible after use.

d) Glass Tubing

Use new glass tubing, pre-cleaned as follows:

1. Rinse tubing twice and allow to air dry for at least 24 hours.
2. Discard tubing after use.

6.3.1.4 Miscellaneous Equipment Cleaning Procedures

a) Submersible Pumps, Bladder Pumps and Hoses Used to Purge Ground Water Wells, Well sounders and Tapes Used to Measure Ground Water Levels[6]

1. Wash with Liquinox laboratory detergent or equivalent and hot tap water.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment thoroughly with analyte-free water.
4. Well sounders and tapes should be placed in a polyethylene bag or wrapped with polyethylene film to prevent contamination during storage or transportation to the sampling site.

b) Pressure Field Filtration Apparatus[6,7]

1. Equipment will be washed thoroughly with Liquinox laboratory detergent or equivalent, and hot tap water using a brush to remove any particulate matter or surface film.
2. The equipment will be rinsed thoroughly with tap water.
3. Rinse equipment with at least a 10 percent nitric acid solution.
4. Rinse equipment thoroughly with tap water.
5. Rinse equipment thoroughly with analyte-free water.
6. Rinse equipment twice and allow to air dry for at least 24 hours.
7. Assemble the apparatus and cap both the pressure inlet and sample discharge lines.
8. Rinse the Teflon or glass sampling equipment thoroughly with tap water in the field as soon as possible after use.

c) Augers and Soil Boring Equipment

1. Wash thoroughly with Liquinox laboratory detergent or equivalent, and hot water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment with at least a 10 percent nitric acid solution.
4. Rinse equipment thoroughly with tap water.
5. Rinse equipment thoroughly with analyte-free water.
6. Rinse equipment twice and allow to air dry for at least 24 hours.
7. Rinse equipment with tap water in the field as soon as possible after use.

d) Miscellaneous Sampling and Flow Measuring Equipment

Miscellaneous flow measuring and sampling equipment shall be washed with Liquinox laboratory detergent or equivalent, rinsed with hot tap water, followed by a thorough deionized water rinse, and dried before being stored. This procedure is not used for any equipment utilized for the collection of samples for trace organic compounds or metals analyses.

e) ISCO Flow Meters, Field Analytical Equipment, and Other Field Instrumentation

The exterior of sealed, watertight equipment such as ISCO flow meters should be washed with Liquinox laboratory detergent or equivalent, and rinsed with hot tap water before storage .The interior of such equipment may be wiped with a damp cloth if necessary.

Other field instrumentation should be wiped with a clean, damp cloth: pH meter probes, conductivity probes, DO meter probes, etc. should be rinsed with deionized water before storage.

The desiccant in flow meters and other equipment should be checked and replaced if necessary each time the equipment is cleaned.

f) Ice Chests and Shipping Containers

All ice chests and reusable containers will be washed with Liquinox laboratory detergent or equivalent, (interior and exterior) and rinsed with tap water and air-dried before storage. In the event that an ice chest becomes severely contaminated, with concentrated waste or other toxic material, it shall be cleaned as thoroughly as possible, rendered unusable, and disposed of properly.

6.3.1.5 Cleaning Procedures for Field Vehicles

All vehicles utilized by field personnel should be cleaned when necessary. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. When vehicles are used in conjunction with hazardous waste site inspection, or on studies where pesticides, herbicides, organic compounds or other toxic materials are known or suspected

to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of the sampling event.

All vehicles are equipped with trash containers to facilitate vehicle cleaning. All field personnel are responsible for keeping the vehicles clean by removing all trash and other debris before it accumulates. All contaminated trash and equipment must be kept separate from ordinary trash and must be disposed of properly on-site or upon return to the laboratory

a) Equipment Used for Routine Sample Collection Activities

For routine operations involving conventional parameter analyses, water quality sampling equipment such as Van Dorns, Kemmerers, grab samplers, buckets, dredges, etc., may be cleaned with sample or deionized water between sampling locations. A brush may be used to remove deposits of material or sediment, if necessary. If deionized water is used, water samplers should be flushed with sample at the next sampling location before the next sample is collected. It should be emphasized that these procedures cannot be used to clean equipment for the collection of samples for organic compounds; trace metals analyses, oil and grease, TRPH or bacteriologicals.

Flow measuring equipment such as weirs, velocity meters and other stream gauging equipment may be cleaned with tap water after use between measuring locations.

6.3.1.6 Emergency Disposable Sample Container Cleaning

Large polyethylene Whirl-paks may be used to collect samples of water or soils which do not require organics. New one-pint or one-quart mason jars may be used to collect samples of waste and soil for organics. These containers would also be acceptable on an emergency basis for the collection of water samples. The jars cannot be used for the collection of water samples for purgeable organics analysis. In some cases, jars may not be acceptable for soils where method 5035 is required.

The rubber-sealing ring should not be in contact with the jar and aluminum foil should be used, if possible, between the jar and the sealing ring. If possible, the jar and aluminum foil should be rinsed with pesticide grade isopropanol[18] and allowed to air dry before use. Empty bottles and lids or Whirl-paks from the sample lot should be submitted to the laboratory as blanks for quality control purposes.

6.3.1.7 Keys to Restrictions Noted Above

1. When this sample equipment is used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone or hexane to remove the materials before proceeding with Step 1. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with Step 1.

If the field equipment cannot be cleaned utilizing these procedures, it should be discarded.

2. Small and awkward equipment such as vacuum bottle inserts and small bailer parts may be soaked in the nitric acid solution instead of being acid-rinsed. Fresh nitric acid solution should be prepared for each cleaning session.
3. When this sampling equipment is used to collect samples that contain oil and grease or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone or hexane to remove the materials before proceeding with Step 1. In extreme cases, when equipment is painted, badly rusted or coated with materials that are difficult to remove, it may be necessary to steam clean, wire brush, or sandblast equipment before proceeding with Step 1. Any stainless steel sampling equipment that cannot be cleaned using these procedures should be discarded.
4. When these containers are used to collect samples that contain oil and grease or other hard to remove materials, it may be necessary to rinse the container several times with pesticide-grade acetone before proceeding with Step 1. If these materials cannot be removed with acetone, the container should be discarded. Glass reusable composite containers used to collect samples from pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds shall be disposed of properly (preferably at the facility) at the conclusion of sampling activities and will not be returned for cleaning. Also, glass composite containers used to collect in-process wastewater samples at industrial facilities should be discarded after sampling. Any bottles that have visible film, scale or discoloration remaining after this cleaning procedure will also be discarded.
5. Plastic reusable sample containers used to collect samples from facilities that produce toxic or noxious compounds or collect in-process waste steam samples at industrial facilities will be disposed of properly (preferably at the facility) at the conclusion of the sampling activities and will not be returned for cleaning. Any plastic composite sample containers that have a visible film, scale or other discoloration remaining after this cleaning procedure will be discarded.
6. The same procedure applies whether this equipment is cleaned in the laboratory or in the field.
7. Assemble and apply pressure to the filtration apparatus after each rinse step (water and acid) to drive rinse material through the porous glass filter holder in the bottom of the apparatus
8. Pesticide-grade petroleum ether, hexane or methanol may also be used. The specific solvent used should be specified in the field equipment decontamination logbook.

6.3.1.8 In-Field Equipment Cleaning Procedures

a) <u>Cleaning Procedures for Teflon or Glass Field Sampling Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analysis</u>

1. Equipment will be washed thoroughly with Liquinox laboratory detergent or an equivalent and tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment with at least a 10 percent nitric acid solution.
4. Rinse equipment thoroughly with de-ionized water.
5. Allow to air-dry for as long as possible.
6. Wrap equipment completely with (in bags) to prevent contamination during transportation to the next sampling site.

b) Cleaning Procedures for Stainless Steel or Metal Sampling Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analysis

1. Wash equipment thoroughly with Liquinox laboratory detergent or equivalent and tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment thoroughly with deionized water.
4. Rinse equipment twice with isopropanol and allow to air dry for as long as possible.
5. Wrap equipment completely with aluminum foil to prevent contamination during transportation to the next sampling site.

c) Cleaning Procedures for Teflon Sampling Equipment Used for the Collection of Samples for Nutrient Analysis

1. Wash equipment thoroughly with Liquinox laboratory detergent or equivalent and tap water using a brush to remove any particulate matter or surface film.
2. Rinse equipment thoroughly with tap water.
3. Rinse equipment with at least a 10 percent hydrochloric or nitric acid solution.
4. Rinse equipment thoroughly with de-ionized water.

d) Cleaning Procedures Sampling Equipment Used for the Collection of Inorganic Ions (SO4, SO3, SO2, NO3, NO2, PO4)

1. Rinse equipment with analyte-free water.
2. Rinse equipment with sample water.

e) Cleaning Procedures for Equipment Used for Routine Sample Collection Activities

For routing operations involving conventional parameter analyses, water quality sampling equipment (i.e., Van Dorn, Kemmerer, grab sampler, buckets, dredges, etc.) may be cleaned with sample or deionized water between sampling locations. If necessary, a brush may be used to remove deposits of material or sediment. If deionized water is used, water samplers should be flushed with sample at the next sampling location before the sample is collected. These procedures cannot be used to clean equipment for the collection of samples for organic compounds, trace metals analyses, oil and grease, TRPH or microbiologicals.

Flow measuring equipment such as weirs, velocity meters, and other stream gauging equipment should be cleaned with tap water after use between measuring locations.

6.3.2 Sampling Protocols

6.3.2.1 Wastewater Influent and Effluent

a) Sampling Equipment

A descriptive list of equipment used for the sampling of wastewater influents and effluents is presented in Table 6-3.

b) Sample Handling and Compositing

Sampling of wastewater influents and effluents should reflect the characteristics of the waste over normal operating cycles. Grab samples may be suitable for batch processes or for uniform effluent streams. For most cases a composite sample will be necessary. Composite samples can be obtained manually or with an automatic sampling device in either a time or flow proportioned mode. The quantity of each collected sub-sample is recorded on the field data sheets. Sub-samples collected for the analysis of trace organics (excluding VOCs) or metals will be mixed with glass, stainless steel or Teflon equipment. Compositing is accomplished by mixing the sub-samples together thoroughly prior to dividing the composited sample into containers used for analysis (VOCs are not to be composited except when specified in a FDEP approved QAPP.)

A. Grab Samples

Grab sampling is conducted when:

1. The water or wastewater stream is not continuous (i.e., batch-discharges or intermittent flow);
2. The characteristics of the water or waste stream are known to be constant or nearly so;
3. The sample is to be analyzed for parameters whose characteristics are likely to change significantly with time (i.e., dissolved gasses, bacteria, etc.);
4. The sample is to be collected for analysis of a parameter such as oil and grease where compositing process could significantly affect the actual concentration; and
5. Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.

Analysis for which samples of water shall always be collected on a grab basis or for which measurements shall be made in-situ include:

pH Phenol

Temperature	Oil and Grease
Dissolved Oxygen	Bacterial
Sulfide	Volatile Organic Compounds
Residual Chlorine	Specific Conductance
Cyanide	Other Dissolved Gases

B. Composite Samples

1. Timed Composite - A sample containing a minimum of eight equal (minimum of 100 mL), discrete samples collected at equal time intervals over the compositing period (18-24 hours). Timed composites may be collected where water or wastewater flows vary widely and are not dampened by wastewater treatment units.
2. Flow Proportional Composite - A sample containing a minimum of eight (minimum of 100 mL) discrete samples collected proportional to the flow rate over the compositing period (18-24 hours). Flow proportional samples may be collected where water or wastewater flows vary widely and are not dampened by wastewater treatment units.
3. Timed and Flow Proportional Composite Samples - The following guidance is given concerning the collection of composite samples:
   a. Composite samples are collected when average waste concentrations are of interest and are always associated with average flow data (where appropriate).
   b. Composite sampling is used when the water or wastewater stream is continuous, when it is necessary to calculate mass/unit time loadings, or when analytical capabilities are limited.
   c. A timed composite will be collected continuously or with a constant sample volume and a constant time interval between samples.
   d. A flow proportional composite will be collected continuously (proportional to the stream flow), with constant sample volume and the time between samples proportional to the stream flow or with a constant time interval between samples and the sample volume proportional to flow at the time of sampling.

c) Preliminary Site Preparation

Where applicable, wastewater influent samples should be collected at the location specified in the NPDES permit. If the source does not have such a permit, the field supervisor will select a sampling point where the most representative sample may be collected.

The following protocols are employed for sampling site selection for wastewater influents. A point of highly turbulent flow, near the center of the flow channel, is the most preferred. If this location is inaccessible the sample will be collected from either the upflow siphon following a comminutor, the upflow distribution box following pumping from a main plant wet well, the aerated grit chamber, the flume throat or the pump wet well when the pump is operating.

The sampling site for wastewater effluents will be as stated in the NPDES permit. If no site is specified in the permit the point of sampling will be at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters. When samples are collected from receiving waters it will be necessary to collect composite samples unless homogeneous mixing can be demonstrated. Samples should not be collected from the surface or from the bottom of any wastewater stream or receiving waters.

Sampling Procedures

A. Manual Sampling by Grab

1. The decontaminated grab sampler is lowered into the wastewater stream and rinsed three times with sample water (pre-rinse is not done for the sampling of oil and grease TRPH, VOCs or bacteriologicals).
2. The grab sampler is again lowered into the wastewater stream and allowed to fill up.
3. The full grab sampler is removed from the wastewater stream.
4. Precleaned sample containers are then filled with the sample.
5. The samples are assigned field ID numbers that are recorded on the field data sheet with the time and location of the sampling.
6. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
7. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed. The amount of preservative added is noted on the field data sheet along with the field ID of the sample needing the pH adjustment.
8. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
9. The pH, temperature, and conductivity of the samples are measured in the field parameter measurement cup and recorded on the field data sheet.

B. Grab Sampling at a Specific Depth

1. The total depth of the water column at the sampling point is measured using a sounding device and measuring tape. The total depth is recorded on the field data sheet.
2. The dissolved oxygen temperature pH and conductivity/salinity of the water at the depth of sampling is determined and recorded on the field data sheet.
3. A previously decontaminated sampler is lowered into the water column to the specified depth for that sample.
4. The messenger for the sampler is dropped.
5. The sampler is removed from the water and precleaned sample containers are filled.
6. The samples are assigned field ID numbers, which are recorded on the field data sheet with the time and location of the sampling.

7. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
8. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
9. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed. The amount of preservative added is noted on the field data sheet along with the field ID of the sample needing the pH adjustment.
10. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
11. The sampler is rinsed with deionized water prior to re-use at another depth or location. The sampler is also rinsed with sample prior to subsequent sampling. Exceptions include samples collected for bacteriologicals, oil and grease, TRPH or VOCs.

C. Automatic Sampling in the Flow Proportioned Mode

1. The decontaminated automated sampler is programmed and checked out in the laboratory to assure proper operation prior to transportation to the sampling site.
2. An equipment blank is collected prior to transportation to the sampling site.
3. The automated sampler is positioned on a flat surface at the sampling site and the sample collection tubing is positioned either in the direction of the flow or perpendicular to the flow in the wastewater stream.
4. The automated sampler and flow meter are then activated and the time of activation is recorded on the field data sheet.
5. The sample container(s) are retrieved from the sampling site. The date and time of retrieval is recorded on the field data sheet.
6. The sample(s) are removed from the automated sampler and poured into pre-preserved bottles prior to being transported to the laboratory in a cooler containing wet ice.
7. All bottles are appropriately preserved in the field in the correct bottles for analysis.

D. Automatic Sampling/Time Mode

1. The decontaminated automated sampler is programmed and checked out in the laboratory to assure proper operation.
2. At the request of the client, an equipment blank is collected in the field prior to sampling.
3. The automated sampler is positioned on a flat surface at the sampling site and the sample collection tubing is positioned in the wastewater stream.
4. The automated sampler is activated and the time of activation is recorded on the field date sheet.
5. The sample container(s) are retrieved from the sample site. The date and time of retrieval is recorded on the field data sheet.
6. The sample(s) are removed from the automated sampler and poured into pre-preserved bottles prior to being transported to the laboratory in a cooler containing wet ice.

7. All bottles are appropriately preserved in the field in the correct bottles for analysis.

TABLE 6-2
FIELD EQUIPMENT USED FOR THE SAMPLING OF WASTEWATER INFLUENTS AND EFFLUENTS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS
Automatic Sampler (ISCO Models 2700, 3710, 3700)	New Dow Corning medical grade Silastic tubing or equal in the pump and either Teflon or Tygon, or equal, in the sample train and reusable glass or plastic sample containers	Sampling, Compositing	Demand, Nutrients, Inorganic Anions, Cyanide, Metals	None
Bomb Sampler	Acrylic plastic with Silicon Seals	Grab Samples at a specific depth	Demands, Nutrients, Inorganic Anions, Cyanide, Metals, Microbiologicals	None
Kemmerer	SS with Teflon coated seals	Grab samples at a specific depth	All parameter groups	a
Extension Pole Grab Sampler	Glass beaker attached to a SS pole or string	Grab samples	All parameter groups	a
Misc. Equipment See Table 6-10	---	---	---	---

SS – Stainless Steel

Key to Restrictions and Precautions
a. Intermediate vessels are not recommended for the collection of Oil and Grease or TRPH samples.

6.3.2.2 Surface Water Sampling

a) Sampling Equipment

A descriptive list of equipment used for the collection of surface water samples is presented in Table 6-4.

b) Sample Handling and Compositioning

Surface water samples may be obtained from a number of distinctly different water systems. Each system will dictate the conditions necessary to obtain a representative sample. These will include such factors as water classification, point source discharges, non-point source discharges, small streams, rivers, estuaries, lakes, ponds and impoundments. The characteristics of the body of water to be sampled will dictate the sampling techniques. Care is taken not to disturb sediments in the immediate area of sample collection. When sampling of a site necessitates wading into the body of water, samples will be taken upstream from the sampler's body. If a fuel-powered boat is utilized, samples will be taken from the bow of the boat, and/or upwind and upstream from the motor to avoid contamination from fumes or effluent from the boat motor.

c) Preliminary Site Preparation

A. Ditches and Small Streams

Samples can be obtained by wading if water flow is sufficient to clear the area of disturbed materials. Road culverts or bridge stations are suitable locations to obtain free flowing well-mixed samples. The use of dippers (plastic, glass, Teflon), Kemmerer samplers or Van Dorn samplers will be used depending on the stream characteristics and objectives of the sampling program. These systems are usually well mixed and the water quality is uniform across the streambed. Sampling in flow restricted areas will be avoided whenever possible.

B. Rivers, Streams and Tributaries

Large flowing bodies of water may exhibit significant differences in water quality along a cross sectional transect Careful consideration of the location of sampling stations and water depths to be sampled should be done prior to starting the sampling program. As a general rule, mid-stream/mid-depth samples will be taken.

Tributary and estuaries systems may present situations when water quality is not uniform due to temperature, salinity or density gradients. These situations should be investigated in the field prior to sampling to determine the specific conditions that exist prior to or during sampling. Conductivity, salinity and temperature variations can be utilized to define existing water quality characteristics. Samples may be obtained from road or bridge stations. The use of a boat may be required to access the sampling points.

Lakes, Ponds, Estuaries and Impoundments

The sampling of lakes, ponds, estuaries and impoundments may involve multi-depth sampling and data collection. The objectives of the sampling program will be used to establish the sampling required Surface, mid-depth and bottom samples may be required to provide a true picture of the water quality and to identify lake stratification. A composite sample combining water from several levels may be used to provide a uniform sample of the entire water column.

d) Sampling Procedures

A. Manual Sampling by Grab

1. The decontaminated grab sampler is lowered into the water and rinsed once with sample water (pre-rinse is not done for the sampling of oil and grease, TRPH, VOCs or bacteriologicals).
2. The grab sampler is again lowered into the water and allowed to fill up.
3. The full grab sampler is removed from the water.
4. Pre-cleaned sample containers are filled with sample.
5. The samples are assigned field ID numbers, which are recorded on the field data sheet with the time and location of the sampling.
6. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
7. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
8. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed. The amount of preservative added is noted on the field data sheet along with the field ID of the sample needing the pH adjustment.
9. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
10. The pH, temperature and conductivity of the samples are measured in the field parameter measurement cup. These measurements are then recorded on the field data sheet.

B. Grab Sampling at a Specific Depth

1. The total depth of the water column at the sampling point is measured using a sounding device and measuring tape and the total depth is recorded on the field data sheet.
2. The dissolved oxygen, temperature, pH, and conductivity/salinity of the water at the depth of sampling is determined and recorded on the field data sheet.
3. A decontaminated sampler is lowered into the water column to the specified depth for that sample.
4. The messenger for the sampler is dropped.
5. The sampler is removed from the water and pre-cleaned sample containers are filled.

6. The samples are assigned field ID numbers, which are recorded on the field data sheet with the time and location of the sampling.
7. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
8. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
9. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed The amount of preservative added is noted on the field data sheet along with the field ID of the sample needing the pH adjustment.
10. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
11. The sampler is then rinsed deionized water rinses prior to re-use at another depth or location The sampler will also be sample rinsed prior to subsequent sampling unless the samples collected are to be analyzed for bacteriologicals, oil and grease, TRPH or VOCs.

e) Special Sampling Procedures

Samples requiring analysis for dissolved metals will be filtered in the field at the time of collection.

TABLE 6-3
FIELD EQUIPMENT USED FOR THE SAMPLING OF SURFACE WATER

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS
Bomb Sampler	Acrylic plastic with Silicon Seals SS with Teflon coated seals	Grab Samples at a specific depth	Demands, Nutrients, Inorganic Anions, Cyanide, Metals, Microbiologicals	None
Extension Pole Grab Sampler	Glass beaker attached to a SS pole	Grab samples	All parameter groups	a
Misc. Equipment See Table 6-9	---	---	---	---

SS – Stainless Steel

Key to Restrictions and Precautions
a. Intermediate vessels are not recommended for the collection of Oil and Grease or TRPH samples.

6.2.3.2 GROUND WATER/MONITORING WELL SAMPLING

a) Sampling Equipment

A descriptive list of equipment used for the collection of ground water from monitoring wells is presented in Table 6-4.

b) Sample Handing and Compositing

Monitoring wells exhibit a wide range of well characteristics, well depths, well diameters, recovery times, and materials of construction. The monitoring well casings should be enclosed in a protective sleeve and be capped and locked when not being sampled. Several techniques are available to successfully sample a well.

Water levels are measured in the field with an electronic level detection meter or a lanyard. Lanyards will not be allowed to come in contact with the ground during sampling. Nylon or braided rope lanyards will be disposed of after each use. Monofilament, stainless steel or Teflon coated lanyards will be decontaminated prior to reuse. The water level will be measured from the same point for each sampling event Measurements should be accurate to within $1/10^{th}$ of a foot, except when more stringent measurements are called for in the project plan.

The basic goals for the sampling of monitoring wells is to remove stagnant casing water and allow a representative portion of groundwater to re-charge the well prior to sampling. This is accomplished by purging five well volumes for stainless steal (three well Volumes for PVC), one dry purge or three well volumes with subsequent stabilization of pH, conductivity and temperature prior to sampling. Stabilization is determined as two consecutive reading values within five percent of each other. The following is the calculation used to determine the well volume:

$$V = (r2)(.163)(h)$$

Where: V = Volume in gallons
d = Diameter of well in inches
h = Depth of water in feet

Purging of the well begins just below the top of the water level. The purging equipment is lowered into the well to follow the water level as it falls. The maximum time between purging and sampling is six hours (or for slow recovery wells, ten hours). If the well has not sufficiently recovered within this time period, the sampling event will be canceled. Sampling should begin immediately after the well has been purged.

Before sampling can begin, it may be necessary to place protective covering, such as plastic sheeting on the ground around the wellhead. Wells that do not require analysis for organics are sampled with a PVC or Teflon bailer, centrifugal pump or submersible pump. Samples may be collected through

the pump depending on the analytical requirements of the monitoring program. For wells deeper than 25 feet, a submersible pump may be used to bring the sample to the surface, and also filter the sample.

Monitoring well requiring analysis for organic contaminants must be sampled with care to avoid contamination of the samples. Fuel powered sampling equipment will be placed away from the downwind of any site activities. These activities include purging, sampling and decontamination.

Monitoring well samples which require analysis for organics will be sampled with laboratory decontaminated Teflon bailers, Teflon or stainless steel bladder pumps, a battery operated peristaltic pump, stainless steel, or submersible pumps.

Temporary well points well be used when permanent wells cannot be installed or in emergency situations only (i.e., DOT right-of-way, private property, etc.).

c) Preliminary Site Preparation

It is extremely important to sample the unconfined or surficial aquifer down gradient of potential pollution sources or spills to determine if the ground water has been affected. If both shallow and deep groundwater levels are involved in the zone of interest, a screening study will reveal whether or not the deeper ground water level needs to be sampled and if a more detailed study is required. To adequately assess subsurface conditions, a minimum of three wells are required, one in the up gradient portion of the area of interest, one in the middle portion, and one in the down gradient portion. Site conditions and the scope of the sampling project will determine the total number of wells required. The sampling and analysis plan will determine the sampling protocols necessary to obtain a representative sample from the monitoring well.

d) Purging and Sampling Procedures

A. Submersible Pump, Bladder Pump and Peristaltic or Centrifugal Lift Pump

1. Determine the water volume in the well.
2. Measure the distance from the bottom of the well to the static water level.
3. Measure the inside diameter of the well or casing.
4. Calculate the well volume and record the results on the field data sheet.
5. The pump or pump hose assembly is lowered into the top of the standing water column (not deep in the column).
6. Pump until five well volumes for stainless steel (three well volumes for PVC) are purged dry or three well volumes are purged with subsequent stabilization of pH, conductivity and temperature.
7. Record the volumes of water purged, the pH, conductivity, and temperature on the field data sheet.
8. Remove the pump from the well after purging.

9. All wetted portions of the pump and hose are cleaned as previously outlined in field cleaning procedures.
10. An equipment blank is collected to determine the adequacy of cleaning prior to the collection of any sample.
11. Pre-cleaned containers are then filled with the samples.
12. The samples are assigned field ID numbers that are recorded on the field data sheet with the time and location of the sampling.
13. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation to the laboratory.
14. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
15. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed. The amount of preservative added is noted on the field sheet along with the field ID of the sample needing the pH adjustment.
16. The acid preserved samples are then placed in ice chests containing wet ice for transportation to the laboratory.

B. Bailer

1. Determine the water volume in the well.
2. Measure the distance from the bottom of the well to the static water level.
3. Measure the inside diameter of the well or casing.
4. Calculate the well volume and record the results on the field data sheet.
5. Remove water from the well until five well volumes for stainless steel (three well volumes for PVC) are purged, the well is purged dry or until three well volumes are purged with subsequent stabilization of pH, conductivity, and temperature.
6. Samples are then collected using another pre-cleaned bailer or with the bailer used for purging after it is cleaned as outlined in field cleaning procedures.
7. Proceed with steps 10 through 16 from the previous sampling procedure.

TABLE 6-4
FIELD EQUIPMENT USED FOR THE SAMPLING OF GROUNDWATER MONITORING WELLS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Pumps: General Note: Pumps may not be used in sampling for volatile organic components except when pumps are permanently installed as part of a drinking water system, or if positive displacement stainless steel and Teflon bladder pumps are used. If installed as a part of a drinking water system, the material construction of the pump and holding tank must be noted in the field documentation.				
Submersible[2] (Turbine, helical, rotor, gear driven)	Housing[3] SS, Teflon Tubing[3] SS, Teflon	Purging Sampling	All parameter groups All parameter groups (excluding VOCs)	a,b; in-line check valve required a,b; in-line check valve required
	Housing[3] SS, Teflon Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients, Metals, Radiochemistry	a,b: in-line check valve required a,b; in-line check valve required; polishing required[4] None
	Housing[3] Steel Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients, Metals, Radiochemistry	a,b: in-line check valve required a,b; in-line check valve required; polishing required[4] None

TABLE 6-4 (cont.)
FIELD EQUIPMENT USED FOR THE SAMPLING OF GROUNDWATER MONITORING WELLS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Bladder Pump (no gas contact)	Housing[3] SS, Teflon Tubing[3] SS, Teflon	Purging Sampling	All parameter groups All parameter groups	a,b a,b; bladder must be Teflon if sampling for organics
	Housing[3] SS, Teflon Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients, Metals, Radiochemistry	a,b: polishing required[4]; Not recommended None, Housing and tubing non-metallic if not SS
	Housing[3] Steel Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients	a,b: in-line check valve required a,b; in-line check valve required; polishing required[4] None
Centrifugal Pump	Housing NA Tubing SS, Teflon	Purging Sampling	All parameter groups All parameter groups	b; Foot valve required b: Foot valve required; Polishing required

TABLE 6-4 (cont.)
FIELD EQUIPMENT USED FOR THE SAMPLING OF GROUNDWATER MONITORING WELLS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Peristaltic Pump	Housing N/A Tubing SS, Teflon	Purging Sampling	All Parameter Groups Demand, Nutrients Metals, Radiochemistry Extractable Organics	b; Foot valve for continuous pumping required None b; Medical grade silicone tubing in pump head b; Configured as in Figure 6-2
	Housing[3] N/A Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients Metals, Radiochemistry	b; polishing required None b; Medical grade silicone tubing in pump head
	Housing[3] Steel Tubing[3] Polyethylene	Purging Sampling	All parameter groups Demands, Nutrients	a,b: in-line check valve required a,b; in-line check valve required; polishing required[4] None

TABLE 6-4 (cont.)
FIELD EQUIPMENT USED FOR THE SAMPLING OF GROUNDWATER MONITORING WELLS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Bailers	SS, Teflon	Purging Sampling	All Parameter Groups All Parameter Groups	None; Not recommended None
Misc. Equipment See Table 6-9	---	---	---	---

N/A: Not applicable
SS: Stainless Steel
HDPE: High Density Polyethylene
PVC: Polyvinyl Chloride
VOC: Volatile Organic Compound

Footnotes:
1. Key to Restrictions and Precautions listed below.
2. Submersible pumps may be used for purging or sampling only if no other pumping device is available, since lines (power cords, gas pressure tubing) may not be (practically) constructed of inert materials.
3. This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.
4. "Polishing": When purging for organics, the entire length of tubing or portion that comes in contact with the formation water should be constructed of Teflon or stainless steel If other materials (i.e., PVC, HDPE, or polypropylene) are used, the following protocols must be followed: 1) Contact with formation waters is minimized by slowly withdrawing the pump from the water column during the last phase of purging, thus removing from the well any water which may have contacted the exterior of the pump and/or tubing; 2) a single well volume must be removed with the sampling device before sampling begins Tygon must never be used for purging when organics are of interest Note: The use of non-inert (i.e., PVC, HDPE, etc.) is not recommended.

Key To Restrictions and Precautions:
a) If used as a non-dedicated system, pump must be completely dissembled, if practical, and cleaned between wells.
b) Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between uses.

6.3.2.4 Potable Water Sampling

a) Sampling Equipment

No specific sampling equipment is used Grab samples are collected directly into appropriate containers.

b) Sample handling and Compositing Drinking Water Supply Systems will be sampled with care to avoid contamination and to assure that a representative sample is collected. This is important not only from a technical and public health perspective, but also from a public relations standpoint. Poor sampling techniques may result in incorrect results. If incorrect results are disclosed to the public, it may be impossible to change public opinion when correct results are reported. Grab samples are generally collected and analyzed. Compositing may be (in some cases) done in the laboratory.

c) Preliminary Site Preparation

Potable water samples are taken to assess water quality within a given segment of the distribution network. Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest, and should not be separated from the segment of interest by a storage tank. The sampling tap must be protected from exterior contamination associated with being too close to the sink bottom or to the ground. Aerator, strainer, and hose attachments on the tap must be removed before sampling to reduce the risk of bacteriological contamination. Only taps where the water flow is steady will be sampled.

Before a sample may be collected, the line must be purged to remove all standing water. Purging should commence at maximum velocity for a minimum of fifteen minutes (for unknown well volumes), or for the volume of the holding tank (if known). For intermittently running pumps, purging should commence at maximum velocity for a minimum of fifteen minutes (for unknown well volumes) and with stabilization of pH, conductivity, and temperature (two consecutive measurements with five percent). Flow rate from the faucet should be reduced to approximately 500mL per minute for sample collection.

When sampling potable water from the water treatment plant, samples will be collected from the raw water supply and also following chlorination.

d) Sampling Procedures

A. Routine Sample Collection

1. The exact location of the tap being sampled, along with the date and name of the sampler(s) is recorded on the field data sheet.
2. Remove any attachments from the tap.
3. The cold water is turned on and allowed to flow in a steady stream for a minimum of 15 minutes.

4. Samples are collected. (If for Total Coliform the tap must be flamed for thirty seconds or treated with a bleach solution and reflushed.)
5. The samples are assigned field ID numbers that are then recorded on the field data sheet.
6. The samples are placed in an ice chest containing wet ice for transportation back to the laboratory.

B. Collection for Lead and Copper Analysis (For Lead and Copper Rule)

1. The water is allowed to sit in the pipes for a minimum of eight hours.
2. Collect 1L of first draw water from spigot inside building.
3. The samples are assigned field ID numbers that are then recorded on the field data sheet.
4. The samples are then transported to the laboratory.
5. Samples are preserved with 1:1 Nitric Acid to pH < 2.

6.3.2.5 Sediment, Soil, Residual, and Solid Waste Sampling

a) Sampling Equipment

A descriptive list of equipment used for the collection of sediments, soils and residuals is presented in Table 6-6.

b) Sample Handling and Compositing Sediments samples from surface water systems may be collected for different purposes. Sediment samples for chemical analysis can be collected by scooping surface material into a wide mouth container. Dredge or core samples may also be obtained. Core samples are used to sample a vertical portion of sediment. Coring devices can be used with glass or Teflon liners for sampling of sediment material for organic analysis. Core samplers can be push tube or weight driven, depending on the nature of the substrate to be sampled. All sediment grab samples are well mixed (except VOCs) before containerization. Samples collected for the analysis of VOCs will not be composited, except when specified by the FDEP.

Soil, sludge, and solid waste samples can be taken with either hand or power driven equipment. Hand equipment is used for shallow sampling. This includes the use of scoops, shovels, hand augers, and push tubes. Power equipment such as hollow augers, Shelby tubes, and split spoons may be required for deeper samples. Sampling performed with open scoops should be confined to quiescent waters (impoundments, lakes, etc.). When sampling sediments from moving waters (rivers, streams, etc.); closed containers such as Ponar dredges should be used to minimize the loss of fine particles.

Disturbed portions of samples collected by augers or split spoon samplers will be discarded and not taken as part of the sample. Samples for chemical analysis will not be taken from auger flights or cuttings from hollow stem auger flights. Samples used for geological, lithological, or vapor meter determinations will not be used for trace contaminant analysis.

c) Preliminary Site Preparation

A. Sediment Samples From Surface Water Systems

In order to obtain a representative sediment sample from a surface water system, a grid of the area to be sampled is first constructed. The sample sites are then selected at random from the grid.

B. Soil Samples

Areas selected for soil sampling will be strategically located in order to collect a representative fraction of the soils with the minimum number of samples and effort. A surface inspection of the subject area will be made to located pertinent features such as wet areas, permanent structures, fill areas, and erosional and depositional areas. An evaluation will then be made to access the relationship between these features and potential sources of pollution. After this initial evaluation is complete, several surface or near-surface samples will be taken and analyzed for the contaminants in question. This will serve as a screening procedure. Sampling of this type will be conducted in depositional areas on the periphery of the study area, primarily at the downstream or down gradient portion of the area of interest. An upgradient location will also be selected for obtaining background and/or control samples.

A more in-depth investigation will be conducted after the above screening procedure is complete. The number of samples and the number of test pits and/or borings and the specific depth that samples are collected can then be determined and the project sampling will begin.

C. Municipal Sludge Samples

Municipal sludge may be collected from several areas. Municipal sludge being drawn to a drying bed will be collected from the pipe flowing to the sludge bed or at the discharge point into the bed. To account for variations in the sludge concentrations and consistency, a minimum of three aliquots of the sludge will be collected during the draw period.

Municipal sludge from an anaerobic digester will be collected from sample ports in the recirculation lines or from “thief holes” in the floating top of the digester.

Municipal sludge from an aerobic digester will be collected while the contents of the digester are being mixed from the 1/3 - 2/3 depth zone.

Municipal sludge can also be collected from a quiescent holding basin. In this case, it is impossible to collect one sample that will be representative of the total contents of the basin since the material is generally stratified. It will therefore be necessary to collect a minimum of three samples that will be

composited prior to chemical analysis. These samples will be collected from a platform or boat using sediment-sampling devices.

Municipal sludge from drying beds will be collected using a stainless steel spoon, shovel, or similar device. Samples will be collected from more than one location in the drying bed to insure a representative sample. The sample should be collected from the entire depth of the sludge on the bed.

D. Solid Waste Samples

Sampling locations should be selected which will yield a sample that is representative of the solid waste being investigated. While a representative sample from a small solid waste area can often be obtained by collecting a single sample, the collection of a representative sample(s) from large waste piles is much more difficult. For the sample(s) to be representative, a statistical approach will be used in the selection of both the number of samples and the location where they are to be collected. This statistical approach involves the mapping out of the solid waste pile in a three-dimensional grid. Sample locations and depths will then be randomly selected from this grid.

d) Sampling Procedures

A. Surface Soil Sampling for Non-Volatile Samples

1. Carefully remove leaves, grass and surface debris with a pre-cleaned stainless steel spoon or shovel.
2. Collect samples with a pre-cleaned stainless steel scoop or spoon.
3. Place sample in a pre-cleaned stainless steel or glass pan for mixing.
4. Fill pre-cleaned sample containers completely with well-mixed sample with no headspace and tightly cap.
5. Assign the sample a field ID number and record with sample location and time on field data sheet.
6. Place the sample in an ice chest containing wet ice for transportation to the laboratory.

B. Shallow Subsurface Sampling for Non-Volatile Samples

1. Carefully remove leaves, grass and surface debris with a pre-cleaned stainless steel spoon or shovel.
2. Dig a hole or trench with a stainless steel shove.
3. Remove the loose soil from the hole or trench.
4. Collect the sample at the desired depth with a pre-cleaned stainless steel spoon, a stainless steel hand auger, or a Shelby tube.
5. Place the sample in a pre-cleaned stainless steel or glass pan for mixing.
6. Fill pre-cleaned sample containers with well-mixed sample, allowing for no headspace and tightly cap.
7. Assign the sample a field ID number and record along with the sample location and time on the field data sheet.

8. Place the sample in an ice chest containing wet ice for transportation back to the laboratory.

C. Deeper Subsurface Soil Sampling for Non-Volatile Samples

1. Carefully remove leaves, grass and surface debris with a pre-cleaned stainless steel spoon or shovel.
2. Bore a sampling hole to the desired depth with a pre-cleaned stainless steel auger.
3. Collect the sample with another clean auger bucket or Shelby tube.
4. Place the sample in a pre-cleaned stainless steel or glass pan for mixing.
5. Fill pre-cleaned sample containers with well-mixed sample, allowing for no headspace.
6. The samples are assigned field ID numbers that are then recorded on the field data sheet.
7. The samples are placed in an ice chest containing wet ice for transportation back to the laboratory.

D. Surface, Shallow and Deeper Subsurface Soil Sampling for Volatile Samples

1. Follow site preparation and sample retrieval steps in Sections A, B, and C above.
2. Using an EnCore™sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil has been exposed to the atmosphere: generally within a few minutes at most.
3. Collect at least two replicate EnCore™ samples from the same soil stratum or the same section of the solid waste being sampled, and with close proximity to the location from which the original sample was collected.
4. Collect an additional aliquot of the sample in a pre-cleaned 4 oz glass jar with a Teflon lined lid or 40 ml VOC vial. Fill sample container allowing for no headspace.
5. The samples are assigned field ID numbers that are then recorded on the field data sheet.
6. The samples are placed in an ice chest containing wet ice for transportation back to the laboratory.

TABLE 6-5
FIELD EQUIPMENT USED FOR THE SAMPLING OF SEDIMENTS, SOILS RESIDUALS AND SOLID WASTES

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Core Barrel (or liner)	SS, Teflon, Glass, Teflon coated, or Aluminum Polyethylene liner	Sampling	All parameter groups Demands, Nutrients Metals, Radiochemistry	a, b, c None b
Trowel, scoop, spoon or spatula	SS, Teflon, or Teflon coated Polyethylene	Sampling and Compositing	All parameter groups Demands, Nutrients, Metals, Radio-chemistry	VOC samples may not be taken from composite samples None
Mixing Tray (Pan)	SS, Teflon, Glass, Teflon coated, or Aluminum Polyethylene, Porcelain enamel coated pans	Compositing or homogenizing	All parameter groups (except VOCs) Demands, Nutrients Metals, Radio-chemistry	c None c
Shovel, Hand Auger, Bucket Auger	SS	Sampling	All parameter groups	none
Split Spoon	SS or carbon steel w/Teflon insert	Sampling	All parameter groups	a,b

TABLE 6-5 (cont.)
FIELD EQUIPMENT USED FOR THE SAMPLING OF SEDIMENTS, SOILS RESIDUALS AND SOLID WASTES

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Shelby tube	SS	Sampling	All parameter groups	c
	Carbon steel		All parameter groups	c,d; Samples for VOC and Metals must be taken from the interior of the core sample
EnCore™ sampler, T-handle	Inert Materials	Sampling	VOC	None
	SS	Sampling	VOC	None

SS: Stainless Steel
VOC: Volatile Organic Compound

Footnotes
1. Key to Restrictions and Precautions listed below

Key to Restrictions and Precautions

a. If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part.
b. Liners must be constructed of stainless steel or a suitable non-metallic material. If a carbon-steel liner is used with the core barrel, the samples for metals shall be taken from the interior part of the core sample.
c. Aluminum foil, trays, or liners may be used only if aluminum is not an analyte of interest

6.3.2.6 Biological Sampling

a) Sampling Equipment

A descriptive list of equipment used for the collection of biological specimens is presented in Table 6-7.

b) Sample Handling and Compositing

Biological sampling is a means of evaluating water quality. Samples collected should be representative of the populations present. Water quality affects the abundance, species composition, stability, productivity, and physiological condition of indigenous populations of aquatic organisms. The distribution of the diverse macroinvertebrates within water systems is extremely heterogeneous. Therefore, replicate samples must be taken at several locations within the system.

Benthic macroinvertebrates may be collected for enumeration using an Ekman, Ponar, or petite Ponar grab. These samples are field washed through a 30-mesh sieve and then transferred to wide mouth containers partially filled with water. Macroinvertebrates may also be collected with Hester-Dendy artificial substrate samplers. These samplers are placed in the water column for a predetermined time to allow for the colonization of macroinvertebrate communities. The optimum length of time that the samplers are left in the field is six weeks. Before the samplers are removed from the water, they are enclosed in oversized, double wrapped plastic bags. They are then transferred back to the laboratory for enumeration.

c) Preliminary Site Preparation

A. Benthic Macroinvertebrates

Many taxa of benthic macroinvertebrates are not distributed uniformly over the bottom of a lake, pond, estuary, river, or stream. The different habitats (mud, sand, gravel, or organic matter) support different densities and species of organisms. Even on a homogeneous bottom, benthic macroinvertebrates tend to aggregate. Therefore, the selection of sites to be sampled is of utmost importance to insure a truly representative sampling of the population. To permit comparison of the benthic macroinvertebrates present, the substrate of the sample stations should be ecologically similar. Sampling locations for benthic macroinvertebrates should also be located in an area not influenced by atypical conditions. A reference station(s) upstream or at a point remote from all discharges of concern should be sampled to serve as a comparison of the benthic macroinvertebrates in areas affected, as well as those not affected by the discharge of concern. In rivers and streams, the upstream sample location will serve as the reference station. Samples will also be collected at various sites downstream from the last discharge of concern to determine the linear extent of damage to the benthic macroinvertebrate community.

B. Macroinvertebrates in the Water Column

For maximum abundance and diversity of macroinvertebrates, the artificial substrate samplers are positioned in the euphotic zone (0.3 M) of the water column. At stations that are less than 1.2 M deep, the samplers will be positioned midway in the water column at low flow. The samplers will not be allowed to touch or rest on the bottom. A reference station will be sampled upstream or at a point remove from all discharges of concern. Sampling stations will also be located downstream from the discharge point of concern. The number of stations selected for sampling will be dependent on the specifications of the project.

d) Sampling Procedures

A. Benthic Sampling

1. A sounding device used to measure the total depth at the sample location
2. The total depth is recorded on the field sheet.
3. The grab is lowered into the water column slowly until it rests on the bottom.
4. The messenger for the grab is dropped.
5. The grab is removed from the water and placed in a plastic tub.
6. The grab is opened and the contents are removed.
7. Before the grab is removed from the tub, it is rinsed to remove any remaining sample.
8. The contents of the tub are transferred to a 30-mesh sieve bucket.
9. The sample is then sieved by raising and lowering the sieve bucket in the water.
10. The sieved sample is transferred to a wide mouth container along with a sufficient amount of sample water.
11. The sample is assigned a field ID number, which is then recorded on the field sheet along with all other pertinent information.
12. The sample is placed in an ice chest containing wet ice and transported back to the laboratory.

B. Water Column Sampling

1. Three samplers are placed at each sample location.
2. The date, time, and location of the samplers are recorded on the field data sheet, along with all other pertinent data.
3. The samplers are left in place for approximately 6 weeks.
4. Oversized plastic bags are used to remove the samplers from the water column.
5. The plastic bag, which contains the sampler and is full of sample water is removed from the water column and sealed.
6. The plastic bag and sampler are placed in another plastic bag.
7. The outer bag is labeled with a field ID, which is recorded on the field data sheet.
8. The samplers are placed in an ice chest containing wet ice for transportation to the laboratory.

TABLE 6-6
FIELD EQUIPMENT USED FOR THE SAMPLING OF BIOLOGICAL SPECIMENS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Ekman Dredge	Brass	Sampling	Macroinvertebrates	None
Petite Ponar Grab	Steel, Lead	Sampling	Macroinvertebrates	None
Hester-Dendy	Particle Board	Sampling	Macroinvertebrates	None
Misc. Equipment See Table 6-9	---	---	---	---

6.3.2.7 Microbiological Sampling

a) Sampling Equipment

A descriptive list of equipment used for the collection of microbiological specimens is presented in Table 6-8.

b) Sample Handling

All microbiological sampling is done under sterile conditions. Samples are collected in pre-sterilized, commercially available containers. These containers are sealed at the manufacturer and are not opened until sampling. Microbiological samples are never composited.

c) Preliminary Site Preparation

The minimum number of microbiological samples that adequately represent the sampling site are collected. The location and number of samples to be collected is predetermined before sampling begins and will meet the requirement necessary to determine water quality. Microbiological samples can be collected from potable water, surface water, groundwater, wastewater treatment plants, and hazardous waste sites. Refer to these sections for more detailed information concerning preliminary site preparation.

d) Sampling Procedures

Samples are collected by hand or with a sampling device. The sample bottle contains a dechlorinating agent, usually Sodium thiosulfate. In no case is a composite sample collected or bacteriological examination.

A. Surface Sampling By Hand

1. Open bottle or Whirl-Pak.
2. Lower the sample container into the water.
3. Move the sample container horizontally away from the sampler's body.
4. Tip the container upwards and remove from the water, allow 1-2 inches of air
5. space.
6. Seal the container and record the field ID number on the field data sheet.

B. Sampling at a Specific Depth

Special devices are used for collection microbiological samples at a specific depth, such as a ZoBell J-Z sampler or a Nisken Sampler A Kemmerer or Van Dorn type sampler should not be used for the collection of microbiological samples.

TABLE 6-7
FIELD EQUIPMENT USED FOR THE SAMPLING OF MICROBIOLOGICAL SPECIMENS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
ZoBell J-Z	Acrylic Plastic, Silicon Seals	Grab samples at a specific depth	Microbiologicals	None
Niskin	Stainless Steel with Teflon coated seals	Grab samples at a specific depth	Microbiologicals	None
Surface Grab	Taken by hand	Grab samples	Microbiologicals	None
Misc. equipment See Table 6-9	---	---	---	---

6.3.2.8 Liquid Hazardous Wastes and Drums and Tank Trucks

a) Sampling Equipment

A descriptive list of equipment used for the collection of liquid hazardous wastes and drum samples is presented in Table 6-8. Sampling equipment to collect hazardous samples should be disposable or easily decontaminated. The equipment should be easy to operate, because personnel may be wearing cumbersome safety clothing and respiratory equipment.

b) Sample Handling and Compositing

When collecting hazardous samples, it is advisable to collect only grab samples. Compositing samples poses a safety risk if it involves samples of unknown hazardous content. The chemical changes that may occur during compositing also support collection of grab samples. Compatibility testing must be performed prior to sample compositing for hazardous materials.

Hazardous wastes are usually complex mixtures of semi-solids, liquids, or solids. The liquid and semi-solid mixtures vary greatly in viscosity, corrosivity, volatility, explosiveness, and flammability. The wastes are containerized in drums, barrels, sacks, bins, tanker trucks, vacuum trucks, ponds, and other containers. No single type of sampler can collect representative samples of all types of waste, therefore, a number are commonly employed.

Sampling at known or hazardous waste sites can involve sampling operations that are inherently dangerous to the personnel involved. Therefore, the procedures outlined by OSHA will be observed during all sampling operations at known or suspected hazardous waste sites. Sampling operations at hazardous waste sites include the collection of samples from open and closed containers, waste piles, pits, lagoons, leachate steams, spillage of materials, and contaminated soil, as well as sampling of ground water and soil.

All samples collected on known or potential hazardous waste sites shall be considered to be “hot” or “concentrated” samples, unless field personnel have valid reasons to believe otherwise. All “hot” or “concentrated” samples shall be clearly labeled as such when they are submitted for laboratory analysis. These samples will be handled as hazardous materials in accordance with OSHA guidelines.

Sampling of closed containers (drums, barrels, and tanks) should only be conducted when absolutely necessary. When container sampling is necessary, the first priority should be the collection of samples from open containers. Open containers generally present less hazard to the samplers than closed containers (i.e., volatile components have already evaporated, and extreme acute toxicity would probably be evident from dead animal life or vegetation around the site). Closed containers must be considered as extremely hazardous from either toxicity, explosion, or fire standpoints. Chronic toxicity may be a danger in both open and closed containers.

Because of the dangers involved with container sampling, the sampling of drums, barrels, or the containers containing either unknown material or known hazardous material shall be considered a hazardous duty.

A remotely controlled device may be used when deemed necessary. Such a device involved the use of a remotely operated pneumatic wrench, along with a brass pressure-fitted bung socket.

A problem, which often arises in container sampling, is stratification and/or phase separation of the container contents. When this condition occurs or is suspected, care must be taken to insure that the sample collected is representative of the container contents. If only one layer or phase is sampled, this should be noted and taken into account when interpreting analytical results.

Where possible, samples should be composited with depth (i.e., collected throughout the entire depth of the container, or at several different depths) to provide a representative sample. When a drum or cylindrical container is standing vertically, depth compositing provides a good quantitative estimate of the container content In other cases where such containers are tipped, horizontal, or deformed, etc., depth compositing will provide a representative sample at least on a qualitative basis.

Note: A quantitatively representative sample could be collected, but it would require sophisticated sampling methodology involving multi-layer sampling and volume measurements. This is not recommended unless initial screening indicates that it is absolutely necessary.

The Coliwasa is a device employed to sample free-flowing liquids and slurries contained in drums, shallow tanks, pits, and similar containers. It is especially useful for sampling wastes that consist of several immiscible liquid phases. The coliwasa consists of a glass, plastic, or metal tube equipped with an end closure that can be opened and closed while the tube is submerged in the material to be sampled.

Samples from drums or barrels can be collected using a four-foot length of glass tube. In most instances, glass tubes with one-half inch or less inside diameter work best. An optional method involves the use of a piercer valve, which is inserted into the drum or barrel using a remotely operated hydraulic jack. However, this method should be used only as the last resort. Several valves may be required at different depths on the drum or barrel if stratification has occurred.

Other sampling procedures that include the use of automatic samplers, pumps, siphons, multiple valves and ports, etc., may be used depending on the specific container involved. These procedures should not be used unless it can be established that their use will not constitute a fire or explosion hazard. This determination will be made only after field reconnaissance, collection of appropriate field data (explosion meter, photoionizer, etc.), and consideration of available file information on the site.

Tank trucks and storage tanks containing liquid wastes are a special case. Samples may be collected from access ports on to of these tanks or trucks using the techniques outlined above. Tank trucks are often compartmentalized, and the field personnel should insure that all compartments of the tank truck are sampled. Sampling from discharge valves usually found on tank trucks is not recommended due to the potential stratification of the tank contents. However, if the investigator has to sample from a tank truck discharge valve, the valving arrangement of the particular tank truck being sampled must be clearly understood to insure that the contents of all compartments are sampled. The same precautions apply to sampling from storage tank valves. In either case, the field personnel must realize that samples obtained from valves (particularly those at or near the bottom of the truck and storage tanks) may not yield representative samples.

c) Preliminary Site Preparation

Guidance given in previous sections will be used when selecting sampling locations for environmental assessments of these sites.

When selecting a sampling location at a landfill or hazardous waste site, the following should be determined: surface water flow (drainage pattern), discharge and recharge areas, direction of groundwater flow, topography, leachate flow, location of streams in relation to the subject area, vegetation, wells in the area, seeps, springs, wet areas, soil conditions, and general geology. All of these items are important in evaluating a site for sampling both solids and liquids, and have been discussed at length in the sections on water and soil sampling.

When a site is being screened for sampling, the most productive method of determining sampling locations is to walk the boundary of the site (after it has been located and defined), focusing attention on the areas where surface runoff leaves the site. The site will be checked for leachate flow or surface spills. Springs, seeps, ponds, and wet areas will be examined to see if they display signs of leachate. Obvious signs of leachate in the water are discoloration and/or odor. The soil will be inspected to see if it is discolored. Areas of excessive dead vegetation and/or dead animals are good indicators of a potential hazard. Nearby water bodies and wells downgradient from the site will be located for sampling to check for off-site migration of contaminants. Test holes can be bored with hand or power equipment to define the general direction of groundwater movement and subsurface stratigraphic conditions.

d) Sampling Procedures

A. Manual Sampling By Grab

1. The decontaminated grab sampler is lowered into liquid to be sampled and rinsed once with sample water, except for the sampling of bacteriologicals, oil and grease, TRPH and VOCs

2. The grab sampler is again lowered into the liquid to be sampled.
3. The full grab sampler is removed.
4. Precleaned sample containers are filled with the sample.
5. The samples are assigned field ID numbers that are recorded on the field data sheet with the time and location of the sampling.
6. The unpreserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
7. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
8. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed The amount of preservative added is noted on the field sheet, along with the field ID of the sample needing the pH adjustment.
9. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
10. The pH, temperature, and conductivity of the samples are measured in the field parameter measurement cup. These measurements are then recorded on the field data sheet.

B. Grab Sampling at a Specific Depth

1. The total depth of the water column at the sampling point is measured using a sounding device and measuring tape and the total depth is recorded on the field data sheet.
2. The dissolved oxygen, temperature, pH, and conductivity/salinity of the water at the depth of sampling is determined and recorded on the field data sheet A decontaminated sampler is lowered into the water column to the specified depth for that sample.
3. A decontaminated sampler is lowered into the water column to the specified depth for that sample.
4. The messenger for the sampler is dropped.
5. The sampler is removed from the water and pre-cleaned sample containers are filled.
6. The samples are assigned field ID number which are recorded on the field data sheet with the time and location of the sampling.
7. The un-preserved and bacteriological samples are placed in ice chests containing wet ice for transportation back to the laboratory.
8. The pH-preserved samples are checked with narrow range pH paper to assure a proper pH.
9. If the pH of the preserved samples is incorrect, additional preservative will be added until the proper pH is observed The amount of preservative added is noted on the field sheet along with the field ID of the sample needing the pH adjustment.
10. The preserved samples are then placed in ice chests containing wet ice for transportation back to the laboratory.
11. The sampler is then rinsed with deionized water prior to re-use at another depth or location The sampler will also be sample rinsed prior to subsequent sampling unless the samples collected are to be analyzed for bacteriologicals, oil and grease, TRPH or VOCs.

C. Bailer

1. Determine the water volume in the well (for well sampling).
2. Measure the distance from the bottom of the well to the static water level.
3. Measure the inside diameter of the well or casing.
4. Calculate the well volume and record the results on the field data sheet.
5. Purge the well of five well volumes, one dry purge, one dry purse, or three well volumes with subsequent stabilization of pH, conductivity, and temperature.
6. Pre-cleaned sample containers are then filled.
7. Proceed with steps 6 through 11 from the previous sampling procedures.

D. Capillary Tube Sampling of Multiphase Liquids and Slurries

1. The tube is inserted into the liquid to be sampled.
2. The open end is sealed either with the thumb or a rubber stopper to hold the sampling the tube while removing the tube from the liquid.
3. Pre-cleaned containers are filled with sample.
4. The samples are assigned a field ID number that is recorded on the field data sheets.
5. The samples are packaged properly (see special sampling procedures) for transportation.
6. The capillary tube is disposed of properly (for drum sampling, the tube will be left in the drum that was sampled.)

E. Colawasa Composite Waste Sampler

1. Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod handle until it sits against the sampler's locking block.
2. Slowly lower the sampler into the liquid to be sampled so that the liquid level inside and outside of the tube are about the same. If the level inside the sampler tube is lower than the level outside of the sampler, then the sampling rate will be too fast and a non-representative sample will be collected.
3. When the sampler stopper hits the bottom of the waste container, the tube is pushed downward against the stopper to close the sampler.
4. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block.
5. Slowly withdraw the sampler from the liquid being sampled.
6. Carefully discharge the sample into pre-cleaned sample containers. Open the sampler by slowly pulling the lower end of the T-handle away from the locking block, while the lower end of the sampler is positioned in the sample container. Carefully discharge the sample into the pre-cleaned sample container.
7. The sample is then assigned a field ID number that is recorded on the field data sheet.
8. The samples are packaged properly (see special sampling procedures) for transportation.

9. The sampler is wrapped and sealed in either aluminum foil or plastic bags for transportation back to the laboratory for proper cleaning.

e) Special Sampling Procedures

Any observations (odor, appearance, container labeling, etc.) made by the field personnel should alert the laboratory to potential dangers. The field supervisor will provide the laboratory personnel with information on possible constituents in the samples (high concentration, etc.). The information will be documented on the sample tag and explained verbally to the sample custodian.

Samples not determined to be environmental samples or samples known or expected to contain hazardous material must be considered hazardous substance samples and transported according to the following requirements. If the material in the sample is know or can be identified, then it should be packaged, marked, labeled, and shipped according to the specific instructions for the material (if listed) found in the DOT Hazardous Materials Table, 49 CFR 172.101. For those samples of hazardous substances where the contents are unknown, the selection of the appropriate transportation category will be based on the DOT Hazardous Material Classification, a prioritized system of transportation categories. Liquid and solid samples will be classified no lower than “flammable” for purposes of packaging safety.

TABLE 6-8
FIELD EQUIPMENT USED FOR THE SAMPLING OF LIQUID HAZARDOUS WASTES, DRUMS AND TANK TRUCKS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Colawasa	Glass, or SS	Sampling multi-phase liquids and slurries	All parameter groups	none
Capillary Tube	Glass	Sampling multi-phase liquids and slurries	All parameter groups	none
Bomb Sampler	Acrylic Plastic with Silicon Seals	Grab samples at a specific depth	Demands, Nutrients, Inorganic Anions, Cyanide, Metals	none
Kemmerer	SS with Teflon coated	Grab samples at a specific depth	All parameter groups	a
Extension Pole Grab Sampler	Glass beaker attached to a SS pole	Grab samples	All parameter groups	a
Bailers	SS, Teflon	Sampling	All parameter groups	none
Misc. Equipment See Table 6-9	---	---	---	---

SS = Stainless Steel

Key to Restrictions and Precautions

a. Intermediate vessels are not recommended for the collection of Oil and Grease or TRPH samples.

TABLE 6-9
MISCELLANEOUS FIELD EQUIPMENT USED FOR ALL SAMPLING EVENTS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Decontamination Equipment and Supplies (if equipment is cleaned in the field)				
Basins, buckets or bowls used to hold wash water and various rinse waters	SS, HDPE, Glass	In-field equipment decontamination	All parameters	none
Brushes or other implements used to clean equipment	Non-inert handle w/nylon bristles	see above	All parameters	none
Detergent: Liquinox	N/A	see above	All Parameters	none
Reagent Grade Acids: 10% $HN0_3$ 10% HCl	 Transported in HDPE bottle Transported in HDPE bottle	 see above see above	 All parameters All parameters	 a,b b
Solvents: Isopropanol (pesticide grade)	Transported in glass bottle	See above	All parameters	none
Protective wrapping: Aluminum foil Zip-Lock bags	 Aluminum Plastic	 Wrapping and packaging of decontaminated equipment	 All parameters	none
Analyte-free water containers	Glass HDPE	Transportation of organic-free water Transportation of DI water	All parameters All parameters	c c

TABLE 6-9 (cont.)
MISCELLANEOUS FIELD EQUIPMENT USED FOR ALL SAMPLING EVENTS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Supplies and reagents for field measurements (i.e., pH buffers, conductivity standards – See Table 6-13)				
Preservation Supplies				
Reagent grade acids: 1:1 HCl 1:1 H_2SO_4 1:1 HNO_3	 Transported in HDPE bottle Transported in HDPE bottle Transported in HDPE bottle	 Preservation Preservation Preservation	 VOCs, O & G Nutrients, O & G Metals	 none none none
Reagent grade base: NaOH	Transported in HDPE bottle	Preservation	Cyanide, Sulfide	none
Reagent grade chemicals: Zinc acetate	Transported in HDPE bottle	Preservation	Sulfide	none
Dispensing equipment: Disposable Pipettes	Glass	Dispensing Preservatives	All parameters	One use only
Narrow range pH paper	N/A	Checking preservation	All parameters	Intermediate container used
Filtering equipment: Syringe 1.0 or .45 µm filters	 Polyethylene Commercially prepared	 Field filtering Field filtering	 Metals, OP Metals, OP	 none
Sample Transportation Supplies				
Ice chests	Polyethylene	Transportation of samples	All parameters	none
Ice	N/A	Cooling samples	All parameters	none
Sealing tape	Polyethylene	Sealing ice chests for shipment	All parameters	none

TABLE 6-9 (cont.)
MISCELLANEOUS FIELD EQUIPMENT USED FOR ALL SAMPLING EVENTS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Sample Transportation Supplies (cont.)				
Shipping labels and forms	Paper	Shipping samples	All parameters	none
Sample labels	Paper	Labeling samples	All parameters	none
Bubble wrap	Polyethylene	Packaging	All parameters	none
Protective Clothing				
Gloves	Powder-free latex	Personal protection, sample contamination prevention	All parameters	d
Coveralls	Cotton, Tyvek	Personal protection	All parameters	none
Respirators	Materials differ	Personal protection	All parameters	none
Documentation Supplies				
Logbooks, Field sheets	Paper	Documentation	All parameters	none
Pens, Markers	N/A	Documentation	All parameters	none
Chain of Custody	Paper	Documentation	All parameters	none
Camera	N/A	Documentation	All parameters	none
Calculator	N/A	Calculations	All parameters	none
Reference Materials				
CQAP	Paper	Reference	All parameters	none
Site Maps	Paper	Reference	All parameters	none
Other Equipment				
Water level indicator tape	SS tip with polyethylene-coated cable	Water level determination	All parameters	Must be field-cleaned between each use

TABLE 6-9 (cont.)
MISCELLANEOUS FIELD EQUIPMENT USED FOR ALL SAMPLING EVENTS

EQUIPMENT	CONSTRUCTION	USE	PERMISSIBLE PARAMETERS	RESTRICTIONS PRECAUTIONS[1]
Other Equipment (cont.)				
Lanyards for Bailers	Monofilament Nylon	Bailer lanyard	All parameters	Disposable, one use only
Paper towels	Paper	Absorbing	All parameters	none
Protective sheeting	Polyethylene	Placement around well heads	All parameters	none
Drums	Metal, HDPE	Contain and dispose purge waters and wastes	All parameters	Must be properly labeled
Field Test Instruments – See Table 9.1				

Key to Restrictions and Precautions

a. Nitric acid should not be used on steel sampling equipment.
b. If sampling for nutrients, a 10-15% reagent grade HCl rinse should be used (except stainless steel equipment). If BOTH metals and nutrients are to be sampled, the HCl rinse must replace the HNO_3 rinse must be followed by HCl rinse.
c. Analyte-free water should not be left in these containers for extended periods, especially HDPE. These containers should be filled for a single sampling event and then emptied at the end of the sampling day.
d. New, disposable gloves, un-powdered latex gloves are changed and discarded after every sampling point. Other type of gloves may be used as long as the construction materials do not contaminate the sample or if safety protocols require greater protection.
e. 0.45 µm filter used for Ortho-phosphorous and FDEP surface water dissolved metals. 1.0 µm high capacity cartridge filter used in line with pressurized bailer for FDEP groundwater dissolved metals samples.

6.4 Definition of Holding Time

The date and time of sampling documented on the field chain-of-custody (COC) form establishes the day and time zero. When the maximum allowable holding time is expressed in days, the holding time is based on day measured. Holding times expressed in 72 hours or less are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis.

6.4.1 Semi-Volatile

Holding times for sample preparation for semi-volatile organics are measured from the date and time of sampling until the solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding times for analysis are measured from the date and time of initiation of extraction to the time of injection into the gas chromatograph.

6.4.2 Volatiles

Holding times for volatile organics are measured from the date and time of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must record the start-of-purging time in the instrument log. Extractions, e.g. for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

6.4.3 Inorganic

For inorganics and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date and time.

6.5 Recommended Containers, Preservation, Holding Times

The preservation and holding time criteria specified in Tables 6-10, 6-11 and 6-12 are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. If criteria are not specified in a source document, internal TestAmerica – Dayton Division guidelines have been set and are marked with an "*". In the tables, items marked with an "*" will not result in the report data being qualified if the requirement is not met. "Analyze immediately" is an EPA designation reserved for tests which, for compliance monitoring projects, should be performed by field instrumentation or a laboratory "generally within 15 minutes" of sampling (Federal Register, Vol. 48, No. 209, p 11). TestAmerica will qualify data for these parameters if analysis cannot be performed within 15 minutes of sampling. "ASAP" is an EPA designation for tests for

which rapid analysis is advised, but for which neither EPA nor TestAmerica – Dayton Division have a basis for a holding time.

The table for water (Table 6-11, 6-12) follows the criteria of Table 2 in 40CFR Part 136 when applicable. The table of analysis of soils, solids and wastes (Table 6-13) evaluated under the Resource Conservation and Recovery Act (RCRA) follow criteria in SW-846 and the TCLP method. In regard to determining hazardous waste characteristics, aqueous wastes are considered “solid waste” and so both aqueous and non-aqueous wastes are covered in that portion of this table.

TABLE 6-10
SAMPLE PRESERVATION, HOLDING TIMES, CONTAINER/LID TYPES AND REQUIRED SAMPLE VOLUMES[1] FOR WATER/WASTEWATER

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
Acidity	P,G/P	Cool, 4ºC	14 days	100 ml
Alkalinity, Total	P,G/P	Cool, 4ºC	14 days	100 ml
Ammonia Nitrogen	P,G/P	Cool, 4ºC H_2SO_4 to pH<2	28 days	400 ml
Bicarbonates	P,G/P	Cool, 4ºC	14 days	100 ml
BOD 5 Day	P,G/P	Cool, 4ºC	48 hours	1000 ml
Bromide	P,G/P	None required	28 days	100 ml
CBOD 5 Day	P,G/P	Cool, 4ºC	48 hours	1000 ml
Carbon Dioxide	P,G/P	Cool, 4ºC	14 days	100 ml
Free Carbon Dioxide	P,G/P	Cool, 4ºC	14 days	100 ml
Total Carbon Dioxide	P,G/P	Cool, 4ºC	14 days	100 ml
TOC	P,G/P	Cool, 4ºC H_2SO_4 or HC1 pH<2	28 days	25 ml
COD	P,G/P	Cool, 4ºC H_2SO_4 to pH<2	28 days	50 ml
Chloride	P,G/P	None required	28 days	50 ml
Residual Chlorine	P,G/P	None Required	Analyze Immediately	200 ml
Chlorophyll a,b,c	P,G/P[5]	14 days in dark	30 days[5]	1000 ml
Color	P,G/P	Cool, 4ºC	48 hours	50 ml
Corrosivity	P,G/P	Cool, 4ºC	7 days[6]	100 ml
Specific Conductance	P,G/P	Cool, 4ºC	28 days	100 ml

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
Hexavalent Chromium	P,G/P	Cool, 4ºC	24 hours	200 ml
Cyanide, Total and Amenable to Chlorination	P,G/P	Cool, 4ºC NaOH pH> 12 0.6g Ascorbic acid[7]	14 days[8]	500 ml
Dissolved Oxygen (Probe)	G/G	None required	Analyze Immediately	300 ml
Dissolved Oxygen (Mod. Winkler)	G/G	Fix on site and store in dark	8 hours	300 ml
Foaming Agents	P,G/P	Cool, 4ºC	48 hours	800 ml
Fluoride	P/P	None required	28 days	300 ml
Total Fluoride	P/P	None required	28 days	300 ml
Total Hardness	P,G/P	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months	100 ml
Ignitability	G/P	None Required	N/A	100 ml
Dissolved Mercury	P,G/P	Filter on site HNO_3 to pH<2	28 days	100 ml
Total Mercury	P,G/P	HNO_3 to pH<2	28 days	100 ml
Metals Dissolved (except Hg and Cr^{+6})	P,G/P	Filter on site HNO_3 to pH<2	6 months	200 ml
Total Metals	P,G/P	HNO_3 to pH, <2	6 months	200 ml
Microbiologicals	P,G/P	Cool, 4°C Na_2S2O_3	6 hours	100 ml
Nitrate-Nitrite	P,G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	100 ml
Nitrate-Nitrogen	P,G/P	Cool, 4°C	48 hours	100 ml
Nitrite-Nitrogen	P,G/P	Cool, 4°C	48 hours	50 ml

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
Organic Nitrogen	P,G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	500 ml
TKN	P, G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	500 ml
Odor	G/P	Cool, 4°C	24 hours	200 ml
Oil and Grease	G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	1000 ml
pH	P, G/P	None required	Analyze Immediately	25 ml
Phenols, 4AAP	G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	500 ml
Ortho-Phosphate	P,G/P	Filter Immediately Cool, 4°C	48 hours	50 ml
Total Phosphorous	P,G/P	Cool, 4°C H_2SO_4 to pH <2	28 days	50 ml
Total Solids	P,G/P	Cool, 4°C	7 days	100 ml
Dissolved Solids	P,G/P	Cool, 4°C	7 days	100 ml
Suspended Solids	P,G/P	Cool, 4°C	7 days	1000 ml
Volatile Solids	P,G/P	Cool, 4°C	7 days	100 ml
Salinity	G/Wax Seal	Analyze Immediately or use was seal	30 days[9]	100 ml
Dissolved Silica	P/P	Cool 4°C	28 days	50 ml
Sulfate	P,G/P	Cool4°C	28 days	50 ml
Sulfite	P,G/P	None required	Analyze Immediately	50 ml

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
Sulfide	P,G/P	Cool, 4°C add 2 ml zinc acetate plus NaOH to pH >9	7 days	500 ml
Temperature	P,G/P	None required	Analyze Immediately	1000 ml
Turbidity	P,G/P	Cool, 4°C	48 hours	100 ml
Purgeable Halocarbons	G/Teflon-lined septum	Cool, 4°C 0.088% $Na_2S_2O_3$[7]	14 days	40 mL
Purgeable Aromatic Hydrocarbons	G/Teflon-lined septum	Cool, 4°C 0.088% $Na_2S_2O_3$[7] HCl to pH2	14 days	40 mL
Acrolein and Acrylonitrile	T/Teflon-lined septum	Cool, 4°C 0.088% $Na_2S_2O_3$[7] Adjust pH to 4-511	14 days	40 ml
Benzidines[12]	G/Teflon-lined cap	Cool, 4°C 0.098% $Na_2S_2O_3$[7]	7 days until extraction [13]	1000 ml
Phthalate Esters[12]	G/Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction	1000 ml
Nitrosamines [12,14]	G/Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[7]	7 days until extraction; 40 days after extraction	1000 ml

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
PCBs[12] acrylonitrile	G/Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction	1000 ml
Nitro-aromatics and Isophorone[12]	G/Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[7]; Store in the dark	7 days until extraction; 40 days after extraction	1000 ml
Polynuclear Aromatic Hydrocarbons	G/Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[7]; store in the dark	7 days until extraction; 40 days after extraction	1000 ml
Haloethers[12]	G/Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[7]	7 days until extraction; 40 days after extraction	1000 ml
Chlorinated Hydrocarbons[12]	G/Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction	1000 ml
TCDD[12]	G/Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[7]	7 days until extraction; 40 days after extraction	1000 ml

PARAMETER (**Table 6-10**: Sample Preservation, Holding Times, Container/Lid Types and Required Sample Volumes for Water/Wastewater)	CONTAINER/ LID[2]	PRESERVATION[3,4]	HOLDING TIME	REQUIRED SAMPLE VOLUME
Pesticides[12]	G/Teflon-lined cap	Cool, 4°C pH 5-9[15]	7 days until extraction; 40 days after extraction	1000 ml
TPH – Gasoline Range	G/Teflon-lined septum	Cool, 4°C HCl to pH2	14 days	40 mL
TPH – Diesel Range	G/Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction	1000 ml
Herbicides	G/Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction	1000 ml
Haloacetic Acids	G/Teflon-lined cap	Cool, 4°C, NH_4Cl	14 days	60 mL

Key to Table 6-11

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, pages 72-82 (1976) Method D0-3370.
2. Polyethylene (P) or Glass (G) For Metals, polyethylene with a polypropylene cap (no liner) is preferred.
3. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
4. When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance. For the preservation requirements of Table 6-8, the Office of Hazardous Materials,

Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid, (HCl) in water, solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Chlorophyll A (modified): sample filtration in lab within 24 hours, add magnesium carbonate to filter while last of sample passes through, analyze or freeze filter for later analysis (within 21 days).
6. Temperature and pH must be measured on-site at the time of sample collection. Seven days is the maximum holding time for laboratory analysis of total alkalinity, calcium ion and total solids.
7. VOCs: If residual chlorine is present, Sodium thiosulfate is added to the sample vial first. The vial is then filled to at least half volume with sample, acid is added, and finally the vial is filled as per procedure (may be filled to top before adding acid). Note: It is no longer recommended to mix the two preservatives (and sample) together in an intermediate vessel.
8. Cyanide: If residual chlorine is present, 0.6 g ascorbic acid should be added. If sulfide is present, samples must be pretreated in the field or must be shipped un-preserved at 4°C to laboratory for analysis within 24 hours. Sulfide must be tested using lead acetate paper, and can be removed by adding cadmium nitrate powder until a negative spot test is obtained. The sample should then be filtered and NaOH added to pH > 12.
9. The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.
10. Sample receiving no pH adjustment must be analyzed within seven days of sampling.
11. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.
12. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, adjusting the pH to 6-9. Samples preserved in this manner may be held for seven days before extraction and for 40 days after extraction. Exceptions for this optional preservation and holding times procedures are noted in footnote 7 (re: the requirement for thiosulfate reduction of residual chlorine); and footnotes 13, and 16 (re: the analysis of benzidine)
13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
14. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_20_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_20_3$.
16. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 $\pm$ 0.2 to prevent rearrangement to benzidine.

TABLE 6-11
PRESERVATION METHODS AND HOLDING TIMES FOR DRINKING WATER SAMPLES THAT DIFFER FROM 40 CFR PART 136, TABLE II

Parameter	DRINKING WATER		40 CFR PART 136 TABLE II	
	Preservation[1]	Holding Time[2]	Preservation[1]	Holding Time
Microbiologicals[3]	Cool, 4°C	30 hour	Cool, 4°C $Na_2S_2O_3$[4]	6 hours
Radiologicals:				
Group A[5]	HCl, HNO_3 pH<2	6 months	HNO_3 pH<2	6 months
Group B[6]	None	6 months	N/A	N/A
Nitrate:				
Chlorinated[7]	Cool, 4°C	28 days	Cool, 4°C	48 hours
Non-chlorinated[7]	H_2SO_4 pH<2	14 days[8]	Cool, 4°C	48 hours

Key to Table 6-11

1. Preservation, when required, must be done upon sample collection.
2. States values are the maximum regulatory holding times.
3. Parameters included are fecal coliform, total coliform, and Fecal Streptococci.
4. Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test.
5. Group A parameters are: Gross Alpha, Gross Beta, Strontium 90, Radium 226, Radium 228, Uranium, and Photon Emitters.
6. Group B parameters are: Cesium -134, Iodine-131, and Tritium.
7. Chlorinated means that the source water has a detectable amount of residual chlorine, as indicated by a field test. Non-chlorinated means that the source water contains no detectable amount of residual chlorine (i.e., is below the method detection limit).
8. Ion chromatographic methods using conductivity as the detector cannot be used to analyze for nitrate in such samples.

TABLE 6-12
SAMPLE PRESERVATION, HOLDING TIMES, CONTAINER/LID TYPES, AND REQUIRED SAMPLE VOLUMES FOR SOIL, SEDIMENT, AND RESIDUAL SAMPLES[1]

PARAMETER	CONTAINER/ LID	PRESERVATION	MAXIMUM HOLDING TIME	REQUIRED SAMPLE VOLUME
Total Metals (except Cr^{+6} and Hg)	Glass or plastic, 8 oz wide-mouth	None	6 months	200 g
Cyanide	Glass or plastic, 8 oz wide-mouth	Cool, 4°C +/- 2 °C	14 days	60 g
Nitrate, Nitrite, Phosphorus, Misc. Wet Chemistry Parameters	Glass or plastic, 8 oz wide-mouth	Cool, 4°C +/- 2 °C	28 days	100 g
Hexavalent Chromium (Cr^{+6})	Glass or plastic, 8 oz wide-mouth	Cool, 4°C +/- 2 °C	One month to extraction, 4 days after extraction	100 g
Mercury (Hg)	Glass or plastic, 8 oz wide-mouth	Cool, 4°C +/- 2 °C	28 days	200 g
Volatile Organics (See Note 2)	EnCore™ sampler and Glass, 40 ml vial Teflon septa or 4 oz wide-mouth, with Teflon-lined cap	Cool, 4°C	14 days	3 EnCore™ samplers and one 40 ml vial or 4 oz wide mouth jar]
Semivolatile Organics (See Note 3)	Glass, 8 oz wide-mouth, with Teflon-lined cap	Cool, 4°C	14 days until ex-traction, 40 days after extraction	60 g

Key to Table 6-13

1 Adapted from Tables 3-1 and 4-1 in Test methods for Evaluating Solid Waste, EPA SW-846

2 **Method 5035 Low-Level Volatile analysis of Soils**

Two options per sample:

Option 1: Two preweighed VOA vials with 5 ml of Sodium Bisulfate solution and one stirring bar. Sampler should add 5.0 +/- 0.5 gram of soil to each VOA vial. Sodium Bisulfate solution is equal to 1 gram sodium bisulfate per 5 ml organic free water. One disposable 10 ml plastic syringe with ends cut off may be used to obtain sample: fill syringe to the 2-3 ml mark with soil---this will be about 5 grams. **Also**, a soil sample submitted in a 2-ounce glass jar is needed for medium prep and for determination of % moisture (dry weight).

Option 2: Two 5-gram EnCore Samplers submitted to laboratory within 48 hours of collection. The laboratory **must** preserve these samples with the Sodium Bisulfate solution within 48 hrs of collection. **Also,** a soil sample submitted in a 2-ounce glass jar is needed for medium prep and for determination of % moisture (dry weight).
Submit to laboratory at 4 degrees C.

3 Includes Chlorinated Herbicides, Organochlorine Pesticides, PCBs, TPH

TABLE 6-13
REAGENT AND STANDARD STORAGE FOR FIELD USE

CHEMICAL/GRADE	METHOD OF STORAGE	METHOD OF TRANSPORTATION TO FIELD
Reagent Grade Hydrochloric Acid	Conc. acid is diluted to the required normality and stored in properly labeled glass containers.	Acid-proof carrying caddy
Reagent Grade Nitric Acid	See above	See above
Reagent Grade Sulfuric Acid	See above	See above
Reagent Grade Sodium Hydroxide	Conc. base is diluted to required normality and stores in properly labeled polyethylene containers.	Separate carrying caddy for bases only
Reagent Grade Zinc Acetate	Stored in properly labeled glass containers.	General use carrying caddy
Reagent Grade Residual Chlorine Test Chemicals	Stored in properly labeled glass and/or polyethylene containers.	See above
Reagent Grade Conductivity Standards	See above	See above
Reagent Grade pH Standards	Stored in properly labeled polyethylene containers	pH meter carrying case
Pesticide Grade Isopropanol	Stored in original container	Original container
Pesticide Grade Methylene Chloride	See above	See above
Tap, Deionized and Analyte-free water	Stored in properly labeled polyethylene containers	General use carrying caddy
Liquinox	Stored in original container	General use carrying caddy

Section 7.0
Sample Custody and Handling

The sample management procedures used at each TestAmerica division is designed to ensure that sample integrity is maintained and documented.

7.1 Procedure to Assess Capability to Perform Work

It is the primary responsibility of [Project Management] and Operations Management to manage workload in the laboratory. Availability of capacity in the laboratory is contingent on both labor and instrumentation. Because these variables can change, TestAmerica has developed mechanisms to pro-actively manage capacity. These mechanisms involve constant communication and utilization of our Laboratory Information Management System (LIMS). Outlined below are TestAmerica procedures for managing workload:

All analytical work is checked into LIMS and is given a sample and job number. A personal responsibility list (PRL) has been developed in each analytical department (Wet Chemistry, Metals, Organics). The PRL list includes the following information: number of samples checked into the laboratory by test, due date of the samples and hold date of the sample. It is the responsibility of the analysts to review the PRL daily and bring any problems with scheduling forward to the Department Supervisor.

The Project Management Team print a status report that provides detailed information on all jobs that are logged into the laboratory. This information includes the job due date and incomplete analyses on a per job basis. A scheduling meeting is held to review the detailed job list and department PRLs. Scheduling and instrument issues on a department-by-department basis are discussed and resolved. The Project Management Team also presents any large quotation information so that new work capacity can be discussed. Departments with limited capacity or scheduling issues are noted and this information is delivered to Project Management so that clients can be notified.

All samples that are quoted for Rush Turn-Around-Time are written on a Sample Notification Form (See Figure 7.1). All rush work must be pre-approved prior to acceptance by the laboratory. Rush work is defined as any work received with a due date of less than 5 working days.

Operations/Supervisors are responsible for notifying Project Management of any issues that may cause backlog problems. Project Management is responsible for informing clients of issues leading to delayed results and informing new clients of the laboratory's available capacity.

7.2 Field Sample Custody

The collection personnel must first consider the analyses to be performed so that proper sample containers and shipping containers are assembled and the proper preservatives are added. All records required for documentation of field collection, including the Chain Of

Custody (COC) (Figure 7-2), pertinent data on sample labels (Figure 7-3) and field data worksheets must be completed by the field personnel.

The purpose of the COC is to supply a detailed record of the sample description, collection information, and any transfer of custody from sample collection through sample receipt into the laboratory. The sample collector is responsible for the care and custody of the samples until properly dispatched to the receiving laboratory or turned over to the sample custodian or designee. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. Samples should be delivered to the laboratory as soon as possible.

NOTE: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier must be kept with the chain-of-custody document.

7.3 Sample Receipt Protocols

Sample acceptance, receipt, tracking, and storage procedures are fully detailed in TestAmerica sample management standard operating procedures. These procedures are summarized in the following sections:

7.3.1 When samples arrive at TestAmerica, the login personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any problems or deviations are recorded on the chain of custody or on the Sample Receipt Record (Figure 7-4). If no COC is provided a Sample Receipt Record must be used.

Inspection of samples include a check for:

7.3.1.1 Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.

7.3.1.2 Complete sample labels to include unique identification in indelible ink.

7.3.1.3 Use of appropriate sample containers.

7.3.1.4 Adherence to holding times as specified in the test method and/or summarized in Section 6.

7.3.1.5 Adequate sample volume for required analyses.

7.3.1.6 Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace.

7.3.1.7 Checking and recording the temperature of the samples that require thermal preservation. Samples shall be deemed within temperature limits if arrival

temperature is 4° C ± 2° C, or the method specific range. Samples that are hand delivered immediately after collection may not be at the required temperatures; however if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. Both the receiving temperature and "Received on Ice" shall be recorded. This will be documented on the COC or Sample Receipt Record. If the sample temperature is outside of range, it will be documented on the COC or Sample/Cooler Receipt Record.

7.3.1.8 Checking for sample preservation as specified in the test method. The pH of the sample is recorded prior to preparation/analysis in the logbooks. In the case of volatiles it is recorded after analysis. Chlorine is checked on: Extractable Organics, BOD, CBOD, TOX, Cyanide, Fluoride, Ammonia, TKN, and Nitrate is recorded prior to preparation/analysis in the logbooks.

7.3.2 Samples received after normal working hours or on weekends are to be left in their coolers and placed in the walk-in cooler. The person receiving the samples must sign, date and record the time and temperature the cooler was received on the COC (Figure 7.2).

7.3.3 Any deviations from the checks described in section 7.3.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance criteria are not met, the laboratory shall either:

1. Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

2. Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

7.4 Sample Log-in and Laboratory Tracking

All samples that are received by each TestAmerica division are logged into the LIMS to allow the laboratory to track and evaluate sample progress. The LIMS assigns a job identification number to the project and a sample identification number. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. The LIMS will generate a sample label that is attached to each bottle of the sample. If there are multiple bottles for the same parameter, LIMS will assign a unique number for each bottle (i.e. 123456-01, 123456-02, 123456-03, etc.).

The login person verifies that the client information on the bottle matches the COC. The sample(s) are then logged in according to the instructions provided by the client on the COC.

7.4.1 The login person logs in each sample contained in the job recording the following information:

7.4.1.1 Client/Project Name, Address, Phone, Fax, Report to information, invoice to information

7.4.1.2 Date Received, Date Taken, Time Received, and Time Taken

7.4.1.3 Job Description, Sample Description, Job location

7.4.1.4 Matrix of the Sample, Special Sample remarks

7.4.1.5 Reporting requirements (QC, Report Format, Invoicing Format)

7.4.1.6 Turn-around-time requirements

7.4.1.7 Parameters, methods, reporting limits

7.4.2 After the job is completely logged in, the login person prints the login information. The LIMS prints a logsheet, order confirmation sheet and bottle labels. Bottle labels are then applied to the sample bottles. The login person is responsible for reviewing the logsheet,

7.4.3 The initialed logsheet, order confirmation, COC, and all corresponding sample information is then stapled together for review.

7.4.4 The login person at the end of each business day shall print the detailed standard daily log which chronologically records the following login information:

7.4.4.1 Date of laboratory receipt of samples

7.4.4.2 Unique laboratory identification code

7.4.4.3 Field identification code as supplied by the sample submitter

7.4.4.4 Requested analyses

7.4.4.5 Comments unique to login

7.4.5 All projects that are logged in undergo the following review:

7.4.5.1 Parameter subcontract review

7.4.5.2 Appropriate methods and analysis selected

7.4.5.3 Short holdtime parameters handled correctly

7.4.5.4 Appropriate reporting limits

7.4.5.5 Appropriate Turn-around-time

7.4.5.6 Adequate sample volumes collected

7.4.5.7 COC discrepancies resolved with client

7.4.5.8 Clerical and typographical errors

7.4.5.10 Order confirmation reviewed for correct pricing

7.4.5.11 Special QC requirements

7.4.5.12 Special sample handling requirements

7.4.6 If all login information is correct the project manager or project coordinator initials and dates the job logsheet and places in the logsheet file.

If there are problems with the login of the job, the project manager is responsible for resolving the problem. Once the problem is resolved, the project manager initials the job logsheet, order confirmation, and sample comment sheet and places in the logsheet file cabinet.

7.5 Storage Conditions

The primary considerations for sample storage are temperature, holding times, contamination, and security.

Section 6 summarizes the temperature and holding time protocols for various analyses. Samples, sample fractions, extracts, leachates or other sample preparation products that require thermal preservation shall be kept at $\pm$ 2° C unless otherwise specified in the test method requirements. Those samples that have a specified storage temperature of 4°C may be stored at 2°-6° C. Samples from North Carolina or New Jersey have specification of 1° – 4.4° C.

All samples distributed into the lab are stored separately from standards and reagents used for analyses to prevent any contamination. Samples are also stored away from food and other potentially contaminating sources. Samples may not be stored in the refrigerator compartment of a unit that has standards stored in the freezer compartment.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to TestAmerica – division personnel only. Visitors to the laboratory are prohibited from entering the laboratory areas unless accompanied by an employee of TestAmerica Analytical Testing Corporation. Samples are returned to the appropriate refrigerator after sufficient sample has been obtained to complete the analysis.

7.6 Sample Disposal

Several possibilities for sample disposal exist: the sample may be consumed completely

during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be stored after analysis (samples are normally maintained no longer than 30 days from analysis completion unless otherwise requested).

The TestAmerica standard operating procedure for sample disposal describes the details of all disposal procedures.

7.7 LIMS Security

The LIMS uses a password security system. All personnel have a login name that restricts the access to system files based upon the level of security they are granted. All personnel are given a unique password that must be entered each time the system is accessed.

7.7.1 The following are the levels of security granted lab personnel:

7.7.1.1 Level A: Access to all files, including LIMS configuration files and client data. Data modification and database modification is allowed.

7.7.1.2 Level B: Access to data entry and sample inquiry.

7.7.1.3 Level C: Review only. No entry of data or modification of data is allowed.

7.7.2 The Division Manager, Project Management, Quality Assurance Officer, Operations Manager, and Department Supervisors are assigned level A. Analysts and clerical personnel are assigned level B. Clients are assigned security level C where they can only view their specific data.

7.7.3 This password records each computer operation with the person initiating that operation, e.g., data entry or data modification. An explanation must accompany the data change with initials before the system will implement the data change.

7.8 Computer Maintenance

The LIMS database is maintained by the TestAmerica Information Technology Group. Technical support for the database system is provided by Decision System Plus, Inc. SCO and hardware support is provided by IBM. The entire LIMS is backed up nightly (11:00pm M-F) and archived with limited access.

7.9 Sub-contracting

A sub-contract laboratory is defined by TestAmerica as a laboratory external to the TestAmerica Analytical Testing Corporation network. However, there are some situations where a network lab must be defined as a sub-contract laboratory and requires client or agency approval prior to use on a project. These requirements are discussed at the start of the project. In these cases, work will only be sub-contracted to an approved laboratory (internal or external) and the client will be notified as requested.

A sub-contract laboratory will be used only after approval is obtained from the client and the quality of the laboratory is determined to be acceptable to the Quality Assurance Officer using the criteria outlined. The Project Management Team is responsible for identifying and initiating pre-qualification of the subcontract laboratory and managing the sub-contractor throughout project implementation.

7.9.1 The Project Management Team must complete the Sub-Contracting Approval form (Figure 7-6) and have the following on file with TestAmerica Analytical Testing Corporation prior to initiation of any work (the following information is already on file with TestAmerica Analytical Testing Corporation for internal laboratories – a Sub-Contracting Approval form is not needed):

7.9.1.1 Copy of Quality Assurance Manual. Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.

7.9.1.2 SOP for method. Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. The SOP can be examined if an on-site audit is performed.

7.9.1.3 The most recent 2 sets of WP/WS reports and any associated corrective action. A score of 80% or higher is required for relevant methods.

7.9.1.4 Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included.

7.9.1.5 Example final report.

7.9.1.6 Technical staff summary – position, education, and years of experience. (This may be part of QA Manual or Statement of Qualifications)

7.9.1.7 Copy of recent on-site audit with corrective actions. If this is not available due to internal lab policy, consider need for TestAmerica Incorporated on site audit.

7.9.1.8 Insurance Certificate

7.9.2 Sub-contractors in use prior to the effective date of this Section shall have six months to come into compliance with these procedures.

7.9.3 These procedures do not apply to laboratories where the clients have previously qualified the sub-contractor laboratory. The client will assume responsibility for the quality of the data generated from use of the sub-contractor that TestAmerica Incorporated has not qualified.

7.9.4 The Division Manager may waive this process temporarily to meet emergency program needs. In the event this provision is utilized, the Corporate Director of Quality Assurance will be informed and the Quality Assurance Officer will be

required to verify adequacy of WP/WS scores and certifications. The Quality Assurance Officer or a member of the Project Management Team will immediately request full documentation and qualify the sub-contractor under the provisions above within 30 calendar days.

Figure 7.1: Sample Notification Form

TestAmerica INCORPORATED

Dayton Division

Sample Notification Form

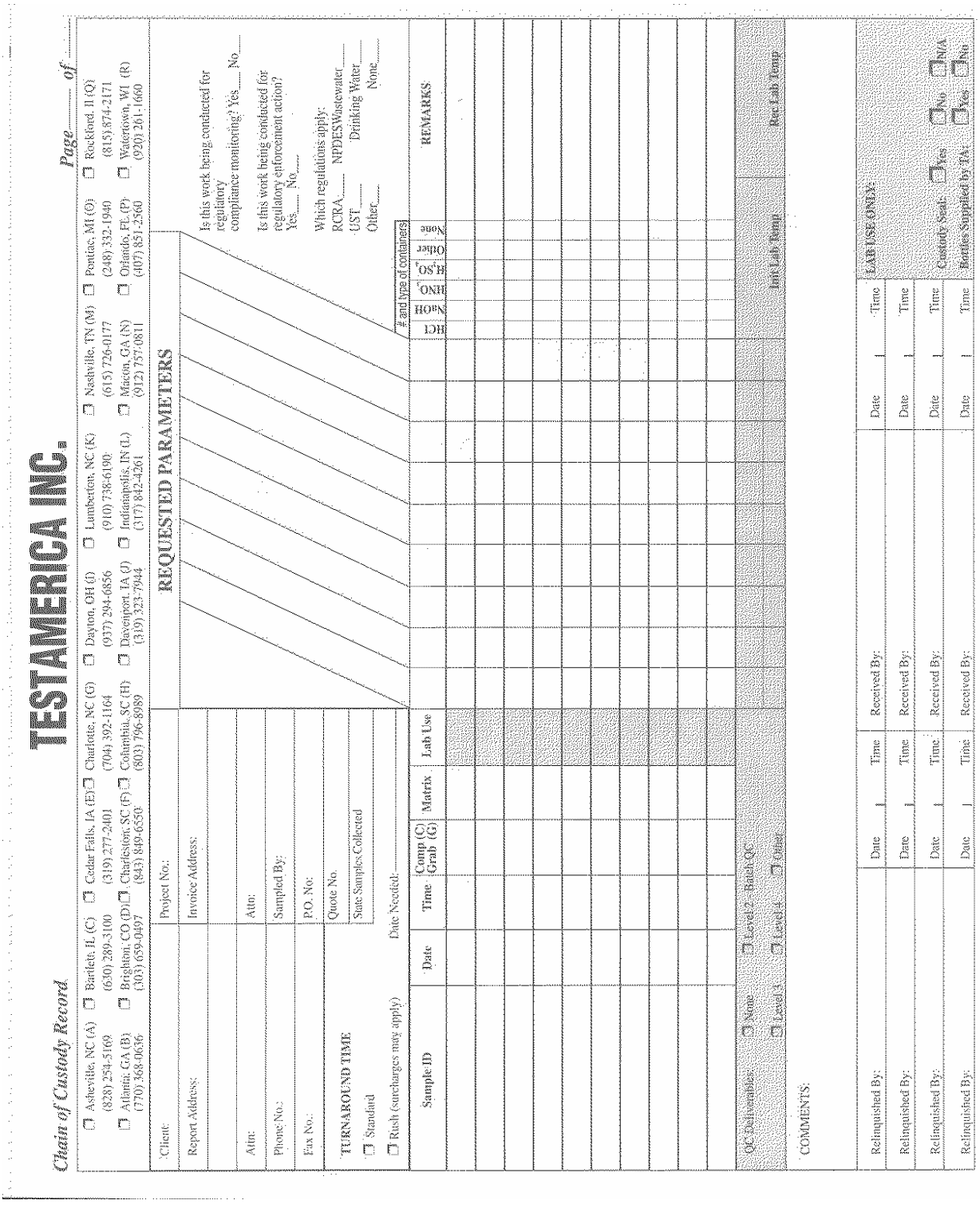
KER ☐ DLJ ☐ SKS ☐
JCS ☐ JAS ☐

Client: _____ **Project Manager:**

Matrix	Number of Samples	Test Parameters	Date/Time Expected
Waste Water { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Bacteria { } Hex Chrome { } BOD { } MBAS { } OTHER _____	
Surface Water { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Bacteria { } Hex Chrome { } BOD { } MBAS { } OTHER _____	
Ground Water { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Bacteria { } Hex Chrome { } BOD { } MBAS { } OTHER _____	
Potable Water { } Yes { } No		Metals { } Wet Chem { } Bacteria { } GC/MS VOA { } OTHER _____	
Soil { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Hex Chrome { } OTHER _____	
Waste Solid (Miscellaneous, i.e. Sludge) { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Hex Chrome { } OTHER _____	
Oil { } Yes { } No		DRO { } Herbicide { } TCLP { } GRO { } BTEX { } BNA { } GC/MS VOA { } Pesticide { } PNA { } Metals { } Wet Chem { } Hex Chrome { } OTHER _____	
Date Report Due:		Turn Around Time:	
Additional information:			

Distribution: Sample Control, Department Supervisors, Project Manager, Operations Manager, Customer Service Representative

Figure 7-2: Example of Chain of Custody Form.



TESTAMERICA INC.

Chain of Custody Record

Page ____ of ____

☐ Asheville, NC (A) (828) 254-5169
☐ Atlanta, GA (B) (770) 368-0636
☐ Barlett, IL (C) (630) 289-3100
☐ Brighton, CO (D)
☐ Cedar Falls, IA (E) (319) 277-2401
☐ Charleston, SC (F) (843) 849-6550
☐ Charlotte, NC (G) (704) 392-1164
☐ Columbia, SC (H) (803) 796-8989
☐ Dayton, OH (J) (937) 294-6856
☐ Davenport, IA (J) (319) 323-7944
☐ Lumberton, NC (K) (910) 738-6190
☐ Indianapolis, IN (L) (317) 842-4261
☐ Nashville, TN (M) (615) 726-0177
☐ Macon, GA (N) (912) 757-0811
☐ Pontiac, MI (O) (248) 332-1940
☐ Orlando, FL (P) (407) 851-2560
☐ Rockford, IL (Q) (815) 874-2171
☐ Watertown, WI (R) (920) 261-1660

Client:
Project No.:
Report Address:
Invoice Address:
Attn:
Attn:
Phone No.:
Sampled By:
Fax No.:
P.O. No:
TURNAROUND TIME
Quote No.
☐ Standard
State Samples Collected
☐ Rush (surcharges may apply)
Date Needed:

REQUESTED PARAMETERS

Is this work being conducted for regulatory compliance monitoring? Yes____ No____
Is this work being conducted for regulatory enforcement action? Yes____ No____
Which regulations apply:
RCRA____ NPDES/Wastewater____
UST____ Drinking Water____
Other____ None____

Sample ID	Date	Time	Comp (C) Grab (G)	Matrix	Lab Use	# and type of containers: HCl	HNO3	ONE	2SO4	Other	None	REMARKS

QC Deliverables: ☐ None ☐ Level 2 - Batch QC
☐ Level 3 ☐ Level 4 ☐ Other

Inf Lab Temp | Rec Lab Temp

COMMENTS:

Relinquished By:	Date	Time	Received By:	Date	Time	LAB USE ONLY:		
Relinquished By:	Date	Time	Received By:	Date	Time			
Relinquished By:	Date	Time	Received By:	Date	Time	Custody Seal: ☐ Yes ☐ No ☐ N/A		
Relinquished By:	Date	Time	Received By:	Date	Time	Bottles Supplied by TA: ☐ Yes ☐ No		

Figure 7-3: Example Sample Labels.

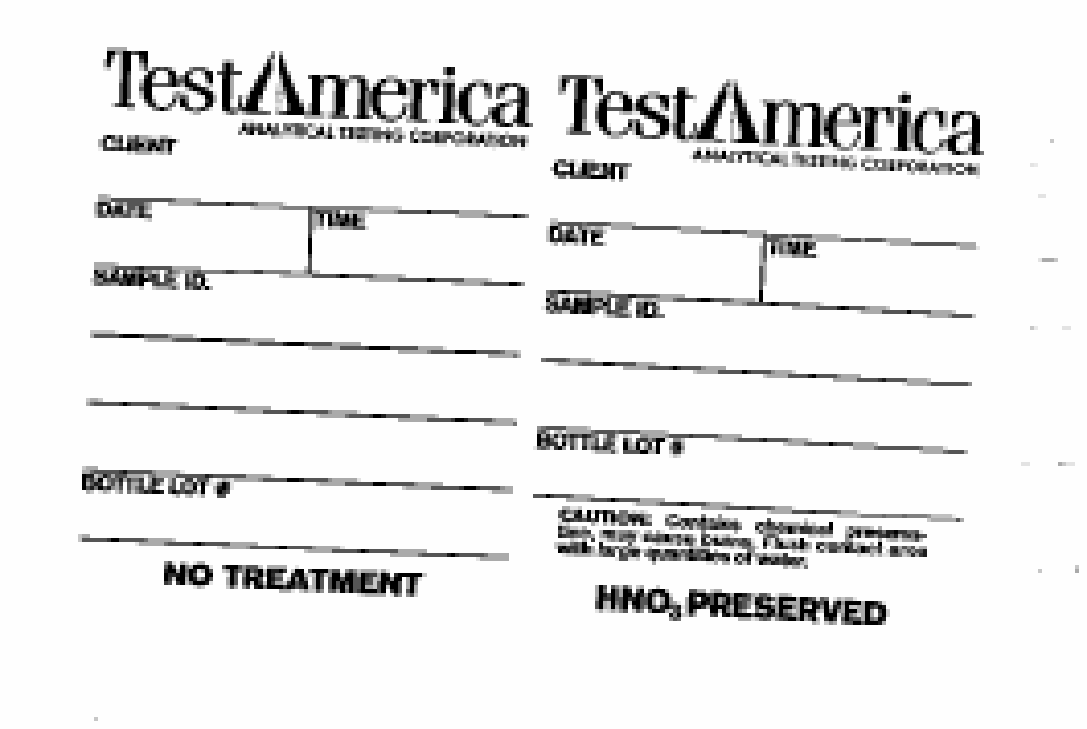
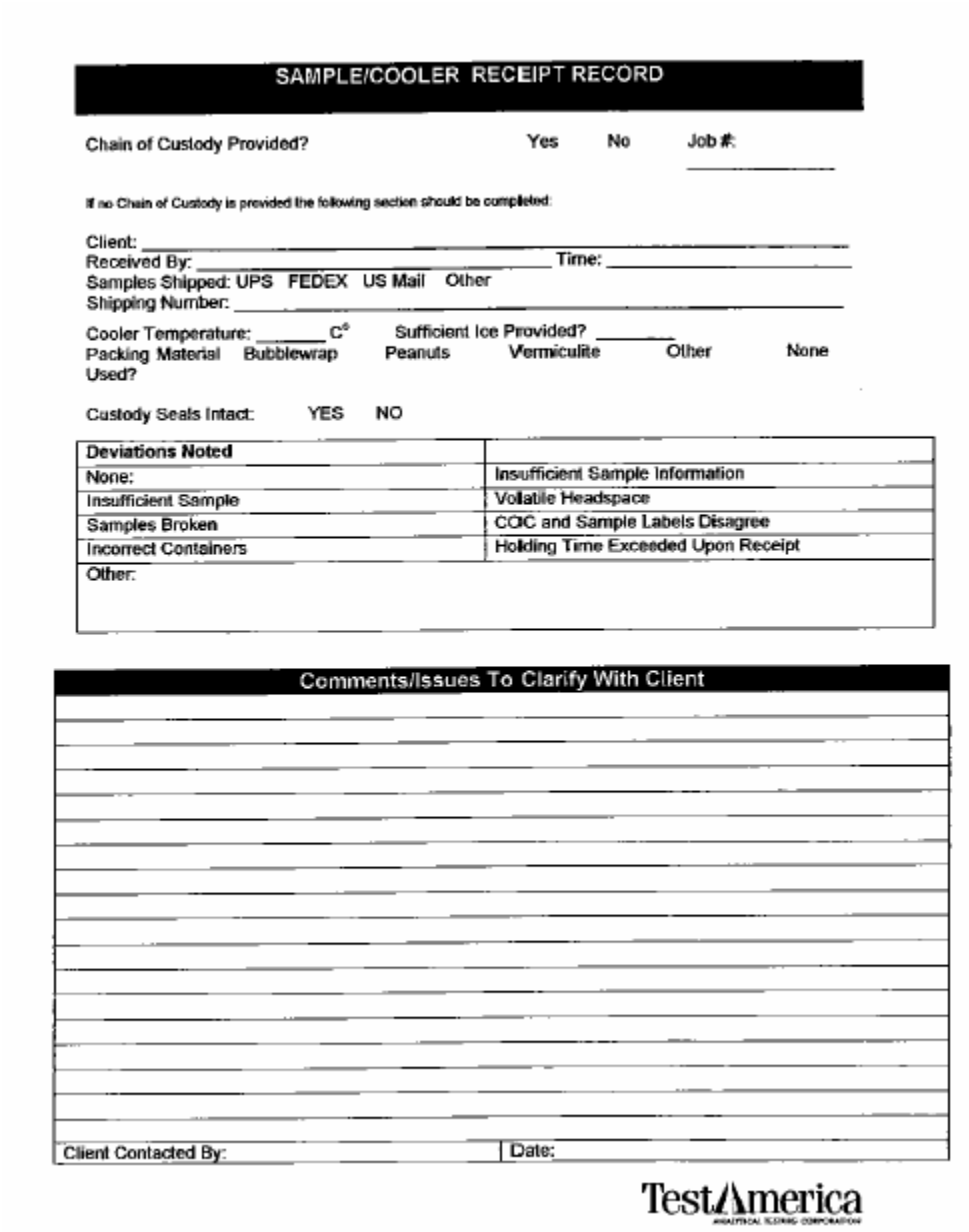


Figure 7-4: Example of Sample Receipt Record



SAMPLE/COOLER RECEIPT RECORD

Chain of Custody Provided? Yes No Job #:

If no Chain of Custody is provided the following section should be completed:

Client: _____
Received By: _____ Time: _____
Samples Shipped: UPS FEDEX US Mail Other
Shipping Number: _____

Cooler Temperature: _____ C° Sufficient Ice Provided? _____
Packing Material Used? Bubblewrap Peanuts Vermiculite Other None

Custody Seals Intact: YES NO

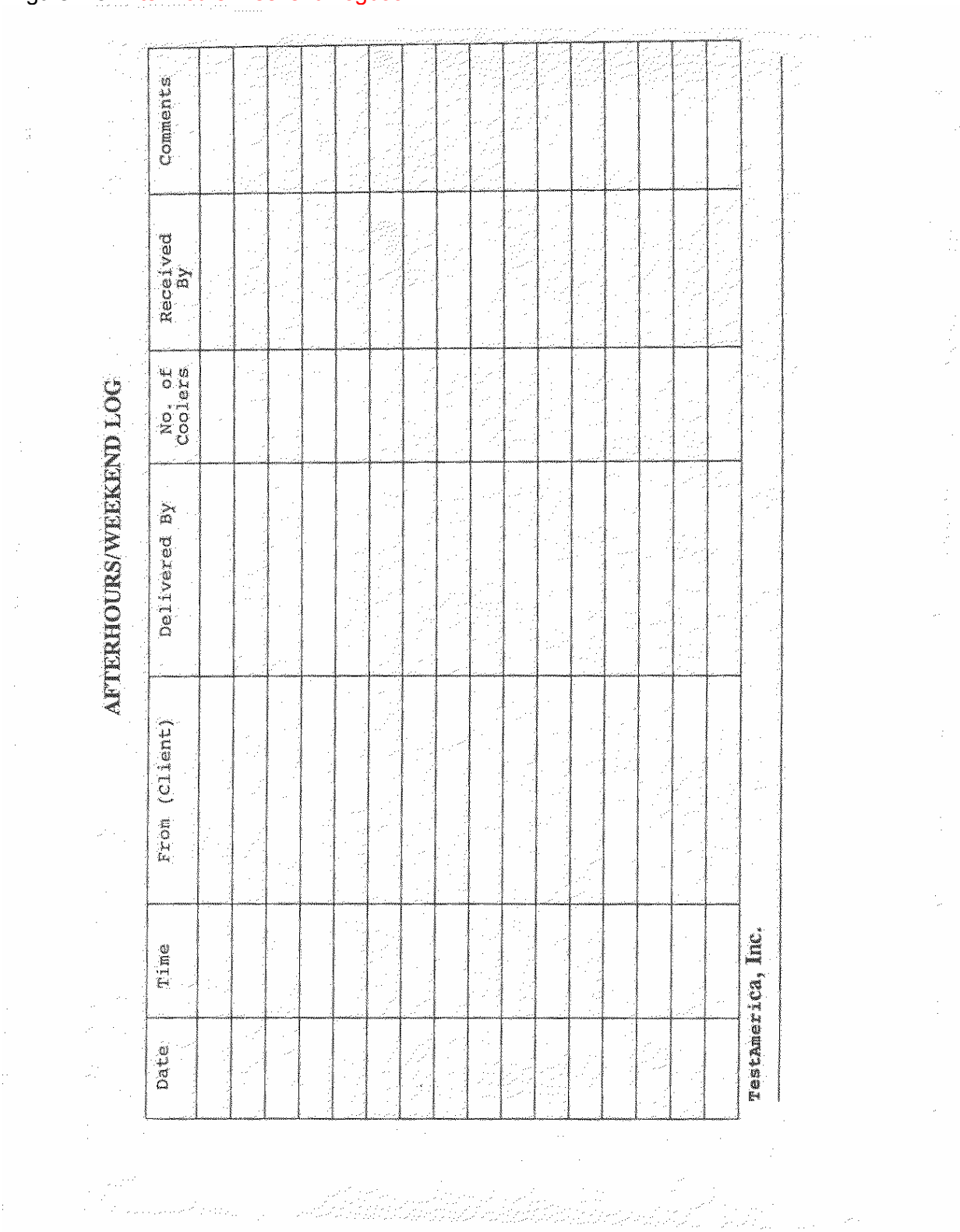
Deviations Noted	
None:	Insufficient Sample Information
Insufficient Sample	Volatile Headspace
Samples Broken	COC and Sample Labels Disagree
Incorrect Containers	Holding Time Exceeded Upon Receipt
Other:	

Comments/Issues To Clarify With Client

Client Contacted By: Date:

TestAmerica

Figure 7-5. After-hours/Weekend Logbook



AFTERHOURS/WEEKEND LOG

Date	Time	From (Client)	Delivered By	No. of Coolers	Received By	Comments

TestAmerica, Inc.

Figure 7-6: An Example of Sub-Contracting Laboratory Approval

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 7 – Quality Assurance Manual

Date: _____

Laboratory: _____

Address: _____

_____

Contact: _____

Phone: Direct _____ Fax _____

Requested Item	Date Received	Reviewed/ Accepted	Date/Initial
QA Manual			
Copy of State Certification[1]			
State Audit with Corrective Action Response (or NELAC or A2LA Audit)			
Most Recent 2 Sets of WP/WS Reports with Corrective Action Response[1]			
Summary List of Technical Staff and Qualifications			
SOPs for Methods to Be Loadshifted[2]			
Insurance Certificate			
Sample Report			

1 - Required when emergency procedures are implemented.
2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable.

On Site Audit Planned: YES NO If yes, Date Completed: _____

Comments:

Lab Acceptable for Sub-Work: YES NO

QA Officer: _____ Date: _____

Division Manager: _____ Date: _____

Section 8.0
Analytical Procedures

8.1 Methods

8.1.1 Method Sources

The analytical methods used are those currently accepted and approved by the U. S. EPA, NIOSH, and the state or territory from which the samples were collected. Reference manuals include:

- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020,
- Standard Methods for the Examination of Water and Wastewater, 18th Edition,
- Federal Register, 40 CFR Parts 136, 141, 143, 172, 173, 178, 179 and 261,
- Test Methods for Evaluating Solid Waste, Physical Chemical Methods, EPA SW-846,
- Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039,
- The most current revision of the NELAC standard.

and selected analytical methods approved and cited by U. S. EPA.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods) or ASTM. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized. Specific analytical procedures used by TestAmerica are listed in the Control Limits Manual (Quality Assurance Objectives).

8.1.2 Standard Operating Procedures

TestAmerica has developed Standard Operating Procedures (SOPs) for all analytical procedures and laboratory operations; these are specifically adapted to our laboratory. The method SOPs derive from the most recently promulgated/approved, published method. All SOPs are controlled in the laboratory: numbered sequentially, approved and signed by the Laboratory Director, Technical Director, and Quality Assurance Officer, dated with an effective date, replaced in controlled manuals or placed in a read only format on the network, and archived when updated. Procedures for preparation, review, revision, and control are incorporated by reference to Corporate SOPs: CP01-01 (Writing a Standard Operating Procedure (SOP)) and CP01-02 (Distribution and Control of Standard Operating Procedures and the QA Manual). All SOPs must be reviewed at least annually.

Each method SOP must contain a minimum of the following: identification of the test method; applicable matrix or matrices; method detection limit (procedure); scope and application, including components to be analyzed; summary of the test method; definitions; interferences; safety; equipment and supplies; reagents and standards; sample collection, preservation, shipment and storage; quality control; calibration and standardization; procedure; calculations; method performance; pollution prevention; data assessment and acceptance criteria for quality control measures; corrective actions for out-of-control or unacceptable data; waste management; references; and any tables, diagrams, flowcharts and validation data.

A general SOP must contain scope/application, definitions, safety issues, procedure, documentation, contingencies, attachments, and references.

Note: Regarding NELAC standards:
If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed.

8.1.3 Requirements for Methods Start-up

Before the laboratory may institute a new method (i.e., new to the TestAmerica division) and begin reporting results, it must write an SOP, demonstrate satisfactory performance, and conduct a method detection limit study. There may be other requirements as stated within the published method or regulations (i.e., retention time window study, IDL). The method may be a recognized and published method or it may be a performance-based method. Procedures for start-up of a performance based method are incorporated by reference to the NELAC website “Quality Systems Guidance” documents.

Methods adopted prior to the effective date of this section may need to be “grandfathered” if initial start-up procedures cannot be found. Proof of continued proficiency must be documented by an annual method detection limit study discussed in Section 11.4.

Note: In some instances a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this Quality Assurance Manual (SOP, MDL, Demonstration of Capability). If the client states that the information is not for legal or regulatory purposes, the result may be reported as long as the following criteria are met: 1) the instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless

an ICV/CCV is not required by the method); 2) the reporting limit is set at or above the first standard of the curve for the analyte; 3) the client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: Reporting Limit based on the low standard of the calibration curve.

8.1.3.1 Demonstration of Capability

A Demonstration of Capability (DOC) must be made prior to using any test method to report results, and at any time there is a significant change in instrument type, personnel or test method.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix, e.g., water, solids and air. However, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last 12 months. In addition, for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

a) The spiking standard used must be prepared independently from those used in instrument calibration.
b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or if unspecified to a concentration approximately 10 times the method stated or laboratory calculated method detection limit.
c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.
d) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.
e) When it is not possible to determine mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.
f) Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there are no mandatory criteria established. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter.
g) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1 or 2:
    1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.
    2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs,

locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c) above.

h) A certification statement (see Figure 8-1) shall be used to document the completion of each demonstration of capability. A copy of the certification is archived in the instrument folder and a copy is archived in the analyst's training folder.

i) Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

8.1.4 Analyst Training

TestAmerica recruits, trains, and maintains an analytical staff with the education and technical knowledge necessary to perform appropriate analytical methods. The training program consists of evidence of an analyst's experience and performance. An experienced analyst or the supervisor trains a new analyst and documents the topics covered during the training (a form such as that found in Figure 8-2 may be used). Prior to the completion of an analyst's demonstration of capabilities, all work must be reviewed and initialed by the trainer. All analysts are required to meet safety-training requirements outlined in the safety manual. Employee training files contain the following information:

8.1.4.1 A completed Personnel Qualifications form or resume (see Attachment 8-3)

8.1.4.2 A statement that he/she has read and understood the latest version of pertinent SOPs.

8.1.4.3 A statement that he/she has read and understood the latest version of the Quality Assurance and Safety Manuals.

8.1.4.4 An annual signing of TestAmerica Incorporated's Ethics Policy (Appendix 1). This policy outlines ethical and legal responsibilities and discusses penalties for improper, unethical or illegal action.

8.1.4.5 A Demonstration of Capabilities for all methods performed – both initial (see Figure 8-1) and continued proof of proficiency. Analysts hired prior to the effective date of this section may summarize their training on the form provided in Figure 8-4. Proof of continued proficiency must be documented by following one of the procedures listed below on an annual basis:

a) Acceptable performance of a blind sample (single blind to the analyst) (see Figure 8-5 for example summary form that may be used);

b) Another demonstration of capability as defined in specific Standard Operating Procedure.
c) Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for 524.2, 624 or 5030/8260) would only require documentation for one of the test methods (see Figure 8-5 for example summary form that may be used);
d) At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;
e) If a-d cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results.

8.1.4.6 As stated in the "Manual for the Certification of Laboratories Analyzing Drinking Water, Fourth Edition", performance of drinking water analysis also requires the following additional training and documentation before an analyst is allowed to independently produce data. Prior to the completion of these items, all work must be reviewed and initialed by the trainer. The following must be available:
a) Satisfactory analysis of an unknown sample.
b) Demonstrate acceptable results for method detection.

8.1.4.7 Documentation from training courses or workshops relevant to the employee's position. Documentation might include a copy of the agenda and any certificate received.

8.1.4.8 The Quality Assurance Department maintains a training summary that tracks which analysts have fulfilled the training requirements listed above for each method. This tracking is performed as part of the Internal Audit program (Section 14 – Performance and System Audits).

8.2 Laboratory Operations

8.2.1 Glassware

8.2.1.1 Glassware Specifications

All volumetric glassware must be Class A. Pyrex glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

8.2.1.2 Glassware Cleaning

The proper technique for cleaning glassware depends upon the intended use of the glassware being cleaned. The goal is to remove all substances from the glassware that might interfere with the analysis. Water-soluble substances can be removed with tap water followed with multiple rinses

with laboratory-grade water. In some instances, detergent may be required. Detergent washings should be followed by three rinses with analyte-free water. Specific guidelines can be found in Table 8-1.

8.2.1.3 Glassware Storage

Once cleaned, glassware is capped, inverted, or covered for storage in a designated cabinet, away from bulk chemicals or reagents.

8.2.2 **Reagents/Standards**

8.2.2.1 **Purchase**

The nature of the analytical laboratory demands that all material used in any of the procedures is of a known quality. The wide variety of materials and reagents available makes it advisable to specify the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The department should determine the vendor and prepare a purchase request. It is the responsibility of the office manager to place the order, receive the shipment, and date the material when received.

Material Safety Data Sheets are kept in a central location known to all personnel. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

8.2.2.2 **Specifications**

a) There are many different grades of analytical reagents available to the analyst. All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.
b) Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.
c) Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable) and expiration date (when applicable).
d) Reagents or working standards that are prepared in house shall be recorded in the Standard and Reagent tracking Log: dated, initialed by the analyst preparing the reagent or standard, and entered in the logbook with a unique designation for tracking purposes. The tracking

procedure for all standards and reagents requires that they be given the identification A-B-C, where A represents the standard logbook number; B represents the page number of the logbook where the entry was made; and C represents the line number. Each standard and reagent must be properly labeled with the reference number, description, concentration (if appropriate) and expiration date.

e) Commercial materials purchased for preparation of calibration solutions, spike solutions, etc. are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

f) Any material used in the preparation of a reagent must meet or exceed the quality of the standard or reagent chemical, e.g., solvents used in the preparation of organic standards.

g) Wherever possible, standards must be traceable to NBS/NIST standards, and records to that effect are maintained in the area in which the standard is to be used.

h) Water used in the preparation of standards or reagents must be of at least laboratory-grade Type II. Type II has been processed through activated carbon to remove organics and a reverse osmosis system; it must have a resulting conductivity of less than <1 micromhos. The conductivity is checked and recorded daily. If the water's conductivity exceeds the specified limit, the Technical Director must be notified immediately in order to notify all departments, decide on cessation of activities, and arrange for correction.

i) The laboratory may purchase reagent-grade water for use in the determination of volatile organics. This water must be certified "volatiles-free."

8.2.2.3 **Storage**

Reagent and chemical storage is important from the aspects of both reagent integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 8-2 details specific storage instructions. Table 9-3 (Calibration Procedures and Frequency) notes storage conditions for standards.

8.2.4 Sample Aliquots/Subsampling

8.2.4.1 Numerous texts describe proper sampling techniques for ensuring that the sample is indicative of the collector's intent, whether it is to represent quality at a moment or an interval in time, flow, or location.

8.2.4.2 It is the laboratory's responsibility to take for analysis a representative subsample or aliquot of the sample provided. In that regard the following guidelines apply to analysts:

a) For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

b) For solid samples, when volatile organics are not requested, if the solid can be mixed, stir with a spatula before removing the aliquot. If the solid cannot be mixed, take several aliquots from various areas of the container to make up the final aliquot. Record all pre-weights and post-weights. For soil samples, try to avoid gravel and grass in the subsample to be analyzed. If the solid is extremely heterogeneous, and the client has given no instructions, utilize the following technique: separate the like materials into groups on a clean surface and take portions of masses from each group, proportional to their contribution to the original sample, to make a composite. Record in detail on the worklist exactly how the composite was created. For very unusual samples, consult with the QA department.

c) For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been improperly collected and is in a common container from which aliquots for other test methods must be taken, login should carry the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out. Only a volatiles department staff person may conduct this subsampling. For this specific case, the analyst would take several aliquots from the various areas of the container to make up the final aliquot, recording all pre- and post-weights.

d) For multiphasic samples, the client should instruct us as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

e) If the client has not specified a sample to be duplicated, the analyst shall select as the duplicate from the analytical batch one sample with sufficient volume/mass.

f) Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and labcoats must be worn when preparing aliquots for analysis.

8.2.3 **Waste Disposal**

TestAmerica collects, stores, packages, labels, ships, and disposes of wastes in a manner which ensures compliance with all federal, state, and local laws, regulations, and ordinances. Procedures are designed to minimize employee exposure to hazards associated with laboratory-generated wastes and to afford maximum environmental protection.

A waste is a hazardous waste if it is listed in 40 CFR Part 261.30-261.33 or fails any of the criteria in 40 CFR Part 261 Subpart C. Personal knowledge of the waste's characteristics must also be considered. Hazardous wastes must be segregated, labeled appropriately, stored in a designated waste disposal area, and disposed of by a commercial waste disposal company. The Hazardous Waste Coordinator is responsible for maintaining the on-site system to prepare the wastes for disposal, scheduling removal by the contractor, maintaining records, and assuring that the contractor is permitted, reputable, and trustworthy. The selection of a waste transporter must be predicated on their being permitted to transport hazardous wastes coupled with an absence of RCRA/DOT violations and a proven record of successful performance.

Waste Disposal procedures are detailed in the Waste Disposal SOP. Waste solvents from organic extractions and glassware cleaning are stored in drums labeled "Waste Flammable Liquid, N. O. S." Organic solvents containing PCBs are segregated for disposal with the appropriate manifest. Extracts are archived for 30-60 days before transferal to the appropriate drum for disposal. On a case-by-case basis, samples may be returned to the client; otherwise, the samples are disposed of in accordance with current waste regulations. Samples known to be very hazardous (e.g., high cyanide) are isolated for special consideration.

Records related to the generation and disposal of hazardous wastes are retained as permanent facility records. Records to be maintained include the following: manifests, inspection reports, waste analysis data, annual reports, certificates of disposal, facility audit reports and documentation of correspondence with federal, state or local regulatory authorities.

Table 8-1: Laboratory Glassware Cleaning Guidelines

Laboratory Glassware Cleaning Procedures

Analysis/Parameter	**Cleaning Procedure** (in specified order)
Extractable Organics (including Pesticides and Herbicides)	Solvents: 13, 1, 2, 3, 4, 5 or 7, (6, 8 optional) Or Muffle: 13, 1, 2, 3, 4, 14 Or Oxidizer: 13, 1, 2, 3, 15, 3, 4
Purgeable Organics	2 (no Soap), 3, 4, (7, 9, 10, 11 optional)
Trace Metals	10, 4 (2,3 optional)
Nutrients, other Wet Chemistry, Minerals, Demand, CN, BOD, and Phenols	1, 2, 3, 4, 16
Phosphorus	1, 2, 3, 4, 9, 4, 16
Residues	1, 2, 3, 4, 12

Key to laboratory glassware cleaning procedures:

1 Remove all labels using sponge or acetone.
2 Wash with hot tap water, a brush to scrub inside glassware and stopcocks and other small pieces, if possible, using a suitable laboratory-grade detergent:
Organics – Liquinox, or equivalent,
Inorganic Anions – Liquinox or equivalent,
Inorganic Cations – Liquinox, Acationox, Micro or equivalent.
3 Rinse thoroughly with hot tap water (3 times)
4 Rinse thoroughly with deionized water (3 times)
5 Rinse thoroughly with pesticide-grade acetone.
6 Rinse thoroughly with pesticide-grade methylene chloride.
7 Rinse thoroughly with pesticide-grade methanol.
8 Rinse thoroughly with pesticide-grade hexane.
9 Rinse or soak with 1:1 HCl.
10 Rinse or soak with 10% HNO_3.
11 Bake at 105°C for 3-4 hours (Note: Class A volumetric glassware should not be baked.)
12 Bake crucibles at 105°C or 180°C for 1 hour (prior to use as per method).
13 After use, rinse with solvent used.
14 Drain, and then heat in muffle furnace for 15-30 minutes.
15 Soak in oxidizing agent: chromic acid or equivalent.
16 Dry in a 75° C to 100° C Dryer

TABLE 8-2

STORAGE OF REAGENTS AND CHEMICALS

1. Concentrated acids and bases are stored in the original containers in acid/base cabinets.

2. Standards for metals are stored at room temperature.

3. Standards for extractable organics are Stored at temperatures below 0^{o} C

4. Standards for volatile organics are stored at temperatures below 0^{o} C.

5. Bulk dry chemicals are stored at room temperature in the reagent storage room of the laboratory.

6. Working solutions containing organic compounds are stored refrigerated at 0-6^{o} C in the departments.

7. Working solutions containing only inorganics are stored at room temperature in the department; refrigeration is parameter dependent.

8. Flammable solvents are stored in flammable cabinets located throughout the laboratory.

9. Non-flammable solvents are stored in cabinets located throughout the laboratory.

Figure 8-1: Example of Demonstration of Capability Documentation

TestAmerica Analytical Testing Corporation

DEMONSTRATION OF CAPABILITY (DOC)

Method: Matrix:
Date: Analyst(s):
Source of Analyte(s):

Analytical Runs (units)

Analyte	Conc. (units)	1	2	3	4	Average % Recovery	%RSD
Analyte Name							

RSD = Percent relative standard deviation = 100s/X percent

Attach raw data or reference location: _____

Certification Statement:

We, the undersigned, certify that:

1. The cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met Demonstration of Capability.
2. The test method was performed by the analyst(s) identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.
4. The data associated with the method demonstration of capability are true, accurate, complete, and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well-organized and available for review by authorized inspectors.

_____	_____	_____
Analyst	Signature	Date
_____	_____	_____
Dept. Supervisor	Signature	Date
_____	_____	_____
Quality Assurance Officer	Signature	Date

Figure 8-2: Example Training Summary Form that may be Used to Document Training Topics

TRAINING SUMMARY

Trainee: _____ **Date:** _____.

Topic(s) of Training (circle): Non-Analytical (General) Analytical Method

SOPs Reviewed: _____

SOPs Signed-off in QA Office: Y / N

I. METHOD/PARAMETER _____
- ___ Detailed Reference Method/SOP
- ___ Basic Method/Instrument Theory
- ___ Safety Precautions
- ___ Waste Handling
- ___ Instrument
- ___ Routine Maintenance
- ___ Interferences
- ___ Extraction/Preparation

II. QUALITY CONTROL
- ___ Calibration Curve, Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
- ___ Precision/Accuracy
- ___ MDL Study
- ___ Review of Chain-of-Custody
- ___ Documentation (sequences, maintenance, logbooks/worksheets, observations, modifications, standards)
- ___ QC Requirements (MS/MSD, LCS, Dups, Blanks, Surrogates, Internal Stds, Interference Checks, etc.)
- ___ Miscellaneous QC (Retention Time Window Studies, IDL)
- ___ Non-Conformance and Corrective Action Documentation

III. DATA HANDLING AND REPORTING
- ___ Review Equations and Calculations (concentrations, dry/wet weight)
- ___ Data Entry or Down-Loading
- ___ Significant Figures
- ___ Reporting Dilutions

IV. OTHER TRAINING
- ___ Attach sheet to describe training.

Results of Start-up QC :

P&A Results Acceptable _____ Attach copy of Demonstration of Capabilities

PE Sample Results Acceptable _____ Attach copy of Performance Evaluation Sample Results (Summary)

Trainee: _____ Date: _____

Trainer: _____ Date: _____

Department Supervisor: _____ Date: _____

QA Officer: _____ Date: _____

Figure 8-3: Example of a Personnel Qualifications Form

PERSONNEL QUALIFICATIONS

Name: _____

Education

Month/Year From - To	Degree	Major	College/University
Number of Hours of Chemistry:			

Month/Year	Training Course Name	Training Provider	Brief Description of Course

Professional Experience

Month/Year From - To	Job Title	Employer - Name and Address

Description
Instruments and Techniques:

Briefly describe your relevant experience:

_____ _____
Signature Date

Figure 8-4:

STATEMENT OF CAPABILITY

(Used to "Grandfather" in Current Analysts)

_____, has been performing the following analyses:

	(Since)
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

*(*If additional *space is needed, attach a separate sheet.)*

The above analyst is proficient in the performance of the above listed analyses due to:

1) Analyst's experience.

2) Analyst has demonstrated the use and understanding of the SOP and referenced methods.

3) Acceptable results on past PT samples such as WP, WS or internal PEs (attach dates of performance).
   OR
   Acceptable Accuracy and Precision on four LCS replicates (attach data or reference location of data).

Approved By:	Date:
Supervisor:	
QA:	

Figure 8-5: Example Form to Summarize PT Results

PERFORMANCE TESTING SAMPLE RESULTS

Sample ID: _____

Division:_____ Instrument:_____ Analyst:_____

SOP/Method:_____ Date of Analysis:_____

PT Sample Source/Reference:_____

Was the PT Analysis: Double Blind_____ Single Blind_____

Attach copy of PT Result Summary or Complete the Following Table:

Analyte	True Value	Lab Result	% Rec.	Acceptance Criteria		Pass Y / N

If a PT sample with a known value (i.e., APG) is the source, then the acceptance criteria is the published range associated with the 99% confidence level. If a PT sample is made from a laboratory stock standard, then the acceptance range is in the method SOP.

_____ _____
Analyst Date:

_____ _____
Quality Assurance Date:

Section 9.0
Calibration Procedures and Frequency

9.1 Instrumentation Lists

Lists of the field and laboratory equipment (including manufacturer's name and model number) used to obtain representative samples and analyze for the parameters listed in the Control Limits Manual (Quality Assurance Objectives) are provided in Tables 9-1 and 9-2 respectively.

9.2 Standard Traceability

Standard sources and preparation for laboratory use are provided in the analytical SOPs and the Standard and Reagent Tracking Logs. Section 8.2.2 (Analytical Procedures) provides additional information regarding the tracking and use of standards. Standard logbooks serve as a source of documentation to trace internal working standards to primary (purchased) standards.

9.3 Calibration

Calibration requirements are divided into two parts: requirements for analytical support equipment and requirements for instrument calibration.

9.3.1 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, thermal/pressure sample preparation devices and volumetric measuring and dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. A summary of support equipment requiring calibration checks can be found in Table 9-3.

Table 9-3 also includes acceptance limits and the proper action in response to unacceptable results. Records of these calibration checks must be documented and include (as appropriate):

9.3.1.1 Instrument Model number or a specific lab identification.

9.3.1.2 Identification of standards used for the calibration check.

9.3.1.3 Performance tolerances.

9.3.1.4 Results of the calibration checks, the initials of the individual making the check, and the date of the check.

9.3.1.5 As appropriate, a reference for the procedure used to perform the calibration check.

9.3.2 Operational Calibration

The frequency, acceptance criteria and corrective action of instrument calibration and standardization are summarized in Appendix 4 by method. Method specific Standard Operating Procedures (SOPs) expand on the general discussion following.

9.3.2.1 Tuning Criteria (Volatiles (BFB)/ and Semi-volatiles (DFTPP) analysis only)

When determining if an instrument is properly tuned prior to analysis, a single scan or a sequential combination of scans at or near the apex at or near the apex of the DFTPP/BFB peak is selected. Background subtraction is generally not required but may be used by acquiring a single scan no more than 20 scans prior to the elution of the DFTPP/BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. No part of the DFTPP/BFB should be subtracted. The spectra/spectrum must meet the criteria outlined in Table 9.54.

9.3.2.2 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

a) For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as summarized in Appendix 4. If a reference or mandated method does not specify the number of calibration standards, the minimum number is 3, not including blanks or a zero standard.
b) The lowest concentration calibration standard that is analyzed during an initial calibration establishes the reporting limit based on the final volume of extract (or sample) described in the appropriate sample preparation SOP.
c) The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags or explained in a case narrative (with the exception of: ICP, ICPMS, or other methods where the referenced method does not specify two or more standards). The lowest calibration standard must be at or above the detection limit (MDL).

d) Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards (see Appendix 4). It is acceptable to drop calibration standards below or above the intended working range
e) All initial calibrations are verified with a standard obtained from a second source (a minimum of a different lot) and traceable to a national standard, when available.

9.3.2.3 External Standard Calibration Procedure

General calibration procedures are described below for GC and HPLC procedures using non-MS detection. The calibration procedures for other techniques are described within the applicable method SOP.

External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

For multi-component analytes, see the appropriate method SOP for information on calibration.

The CF can also be calculated using the concentration of the standard rather than the mass in the denominator of the equation above. However, the use of concentrations in CFs will require changes to the equations that are used to calculate sample concentrations.

9.3.2.4 Internal Standard Calibration Procedure

Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. If specific internal standards are not recommended in the method, then the analyst needs to select one or more internal standards that are similar in analytical behavior to the compounds of interest, and not expected to be found in the samples otherwise. The use of specific internal standards is available in the method SOP.

For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows:

$$RF = \frac{A(s) \times C(is)}{A(is) \times C(s)}$$

where:
A(s) = Peak area (or height) of the analyte or surrogate.
A(is) = Peak area (or height) of the internal standard.
C(s) = Concentration of the analyte or surrogate, in ug/L.
C(is) = Concentration of the internal standard, in ug/L.

Note that in the equation above, RF is unitless, i.e., the units from the two area terms and the two concentration terms cancel out. Therefore, units other than ug/L may be used for the concentrations of the analyte, surrogate, and internal standard, provided that both C(s) and C(is) are expressed in the same units. The mass of the analyte and internal standard may also be used in calculating the RF value.

9.3.2.5 Evaluating the Linearity of the Initial Calibration

To evaluate the linearity of the initial calibration, calculate the mean CF (external standard calibration) or RF (internal standard calibration), the standard deviation (SD), and the RSD as follows:

$$\text{Mean CF} = CF = \frac{\sum_{i=1}^{n} CFi}{N}$$

$$\text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RFi}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CFi - \overline{CF})^2}{n-1}}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RFi - \overline{RF})^2}{n-1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

where n is the number of calibration standards and RSD is expressed as a percentage (%).

If the RSD of the calibration or response factors is less than or equal to the acceptance limit stated in Appendix 4 over the calibration range, then linearity through the origin may be assumed, and the average calibration response factor may be used to determine sample concentrations. Corrective action procedures are outlined in Appendix 4 and expanded on in Section 9.3.2.6.

9.3.2.6 Percent RSD Corrective Action – Additional Information

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

a) The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance,

changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

b) If the RSD for any analyte is greater than the applicable acceptance criteria in Appendix 4, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

c) A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

   NOTE: As noted in Sec. 9.3.2.1, the reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Hence, narrowing the calibration range by changing the concentration of the lowest standard will, by definition, change the method quantitation limit. When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

d) In those instances where the RSD for one or more analytes exceeds the acceptance criteria, the initial calibration may still be acceptable if the following conditions are met:
   1) The mean of the RSD values for all analytes in the calibration is less than or equal to the acceptance criteria. The mean RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. If no analyte has an RSD above the acceptance criteria, then the mean RSD calculation need not be performed.
   2) The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project. In other words, if the target analyte is part of the calibration standard, its RSD value is included in the evaluation.
   3) The data user must be provided with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded the acceptance criteria and the results of the mean RSD calculation.

NOTE: The analyst and the data user should be aware that the use of the approach listed in Sec. 9.3.2.5(d) (i.e., the average of all RSD values </= the acceptance criteria) will lead to greater uncertainty for those analytes for which the RSD is greater than the acceptance criteria. The analyst and the data user should review the associated quality control results carefully, with particular attention to the matrix spike and laboratory control sample results, to determine if the calibration linearity poses a significant concern. If this approach is not acceptable for a particular application, then the analyst may need to employ another calibration approach (see Sec. 9.3.2.6) or adjust the instrument operating conditions and/or the calibration range until the RSD meets the acceptance criteria.

e) If all of the conditions in Sec 9.3.2.5 (d) are met, then the average calibration or response factor may be used to determine sample concentrations.

9.3.2.6 Use of other types of calibration (i.e., Linear Calibration Using a Least Squares Regression or Non-Linear Calibration) may be described in manufacturer's manuals or within a published method. These procedures are included in the analytical SOPs.

9.3.2.7 Retention Time Windows

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

The following subsections describe the approach used to establish retention time windows for GC and HPLC methods.

NOTE: The criteria listed in this section are provided for GC and HPLC procedures using non-MS or FTIR detection. Identification procedures are different for GC/MS.

(a) Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all single component standard mixtures and multi-component analytes (such as PCBs) over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

(b) Record the retention time for each single component analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks (see the determinative methods for more details) and calculate the mean and standard deviation of those peaks.

(c) If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).

(d) The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as +/- 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in (c) is employed, the width of the window will be 0.03 minutes.

(e) Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

(f) The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed.

(g) If the instrument data system is not capable of employing compound-specific retention time windows, then the analyst may choose a window that minimizes false negatives and positives and apply it to all compounds. As noted above, other approaches may also be employed, but must be documented by the analyst. In general you should not use a window greater than 0.2 to 0.3 minutes. If windows larger than this have been determined a cause should be looked for and the windows should be redetermined.

(h) The surrogates are added to each sample, blank, and QC sample and are also contained in each calibration standard. Although the

surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

9.3.2.8 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in Appendix 4. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

NOTE: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration, and is neither appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses. TestAmerica - Dayton does not perform this type of calibration.

As a general rule, the initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more frequent verifications – see Appendix 4). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

If the response (or calculated concentration) for an analyte is within the acceptance limits (summarized in Appendix 4) of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF or RF values from the initial calibration to quantitate sample results. If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable

calibration verification may be reported as qualified data under the following special conditions:

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

In keeping with the approach described for initial calibration, if the average of the responses for all analytes are within that required in Appendix 4, then the calibration has been verified. However, the conditions in Sec. 9.3.2.5(d) also apply, e.g., the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the limit. The effect of using the average of the response for all analytes for calibration verification will be similar to that for the initial calibration - namely, that the quantitative results for those analytes where the difference is greater than the limit will include a greater uncertainty. The analyst and the data user should review the note in Sec. 9.3.2.5(d).
If the calibration does not meet the limit (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within the acceptance criteria, then a new initial calibration must be prepared.

(a) Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP.

$$\%\ \text{Drift} = \frac{\text{Calculated concentration - Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

where the calculated concentration is determined using the mean calibration factor or response factor from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{CF(v) - \overline{CF}}{CF} \times 100$$

OR

$$= \frac{RF(v) - \overline{RF}}{RF} \times 100$$

where CF(v) and RF(v) are the calibration factor and the response factor (whichever applies) from the analysis of the verification standard, and CF and RF are the mean calibration factor and mean response factor from the initial calibration. Except where superseded in certain determinative methods, the % difference or % drift calculated for the calibration verification standard must be within +/- 15% for each analyte, or averaged across all analytes (see Sec 9.3.2.8), before any sample analyses may take place.

(b) Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift calculation described in (a). Calibration verification must be acceptable before any sample analyses may take place. It may also be appropriate to employ two standards at different concentrations to verify the calibration. This is outlined in the method SOP when used.

(c) Regardless of whether a linear or non-linear calibration model is used, if either the percent drift difference criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications in Appendix 4 and those in the method SOP. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

(d) All target analytes and surrogates, including those reported as non-detects, must be included in a periodic calibration for purposes of

retention time confirmation and to demonstrate that calibration verification criteria are being met. The frequency is noted in Appendix 4.

(e) Calibration verification may be performed using both high and low concentration standards from time to time. This is particularly true when the ECD or ELCD is used. These detectors drift and are not as stable as FID or PID, and periodic use of the high and low concentration standards serves as a further check on the initial calibration. The concentrations of these standards should generally reflect those observed in samples.

(f) Samples analyzed using external standards must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The results from these bracketing standards must meet the calibration verification criteria and the retention time criteria. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected were it present.

TABLE 9-1A
FIELD SAMPLING EQUIPMENT (Dayton Laboratory)

Equipment/ Instrument	Manufacturer/ ID
Automatic Sampler (7)	ISCO/2700
Automatic Sampler (2)	ISCO/3700
Automatic Sampler (1)	ISCO/2100
Flow Meter (4)	ISCO/1870
Flow Meter (1)	ISCO/3210
Flow Meter (3)	ISCO/3230
Conductivity Meter	YSI/33
pH Meter (2)	Hanna/9025
Residual Chlorine Kit	Hach/CN66
Water Level Indicator	SINCO/51405301
Pressure Filtration Device	Geotech/0856
Ventilator	Air Systems Int./SVB-G8
Winch	Miller Equipment/ 50 G
Gas Monitors	Ind. Scientific/HMX 271
4 inch Well Pump	Suburban/PO51-2W
2 inch Well Pump	Grandfos/Rediflo 2
Electrical Generator	Pincor/RF-30HM5
Power Auger	Tecumseh Engines/21
2 inch Teflon Bailer	Mod. Ind. Plas./GWE-300

TABLE 9-1B
FIELD SAMPLING EQUIPMENT (Pontiac Laboratory)

Equipment/ Instrument	Manufacturer/ ID
Automatic Samplers (9) Peristaltic Pump	ISCO/2910 Geo Pump, Model 2

TABLE 9-1C
FIELD SAMPLING EQUIPMENT (Indianapolis Laboratory)

Equipment/ Instrument	Manufacturer/ ID
Automatic Samplers (4)	ISCO/3710
Automatic Sampler (2)	ISCO/6700
Automatic Sampler (1)	ISCO/2910

TABLE 9-2A
WET CHEMISTRY LABORATORY EQUIPMENT AND INSTRUMENTATION

Equipment/ Instrument	Manufacturer/ ID	Put In Service	Condition When Received (New/Used)
Dayton Laboratory			
Autoanalyzer w/Autosampler	Bran-Luebbe/Traacs 800	1989	New
Autoanalyzer w/Autosampler	Bran-Luebbe/Traacs 800	1991	Used
Autoanalyzer w/Autosampler	Lachat/Quik Chem	2002	Used
TOC Analyzer	Tekmar-Dohrmann/Phoenix 8000	2001	Used
Spectrometer	Milton Roy 301	2001	Used
Spectrometer	Milton Roy 401	2002	Used
Spectrometer	Milton Roy/301	1990	New
Spectrometer	Milton Roy/501	1989	New
Ion Analyzer	Orion Research/901	Pre-1989	New
pH Meter	Orion Research/SA 520	Pre-1989	New
pH Meter	Accumet/20	1993	New
pH Meter	Orion Research/601A	Pre-1989	New
Turbidimeter	Hach/2100AN	1997	New
Flashpoint Analyzer	Fisher Scientific	2002	Used
Flashpoint Analyzer	Precision Scientific	Pre-1989	New
Oxygen Meter	YSI Scientific/5000	1996	New
Analytical Balance	Mettler/ AE 200	2002	Used
Analytical Balance	Mettler/HK 160	Pre-1989	New
Analytical Balance	Mettler/AE 163	Pre-1989	New
Top Loading Balance	Mettler/PJ6000	1998	Used
Flow Injector Autoanalyzer w/ Autosampler	Lachat/Quik Chem 8000	2002	Used
Block Digestor	Lachat BD-46	2001	New
Rapidstill II	Labconco	1997	New
Ammonia Distiller	Enviro Midi Dist	2001	New
Midi CN Distillers (2)	Labcrest	1998	New
Incubator	VWR/3020	Pre-1989	New
Autoclave	Amsco/57 CR	Pre-1989	Used
Available CN Autoanalyzer	OI Analytical/FS 3000	2002	New
Indianapolis Laboratory			
Lachat 8000 Series • Software- Lachat	Lachat • Omnion FIA	1995 1994	New
Spectronic 301	Milton Roy	1997	Used
Spectronic 301	Milton Roy	Pre-1996	Used
pH meter	Orion	Pre-1990	New
BOD meter	Orion/501	Pre 1990	New
Isotemp Ovens (2)	Fisher Scientific	Not Available	New
Oven (wastewater)	Amer. Scientific DX-58	Pre-1990	New

WET CHEMISTRY LABORATORY EQUIPMENT AND INSTRUMENTATION

Equipment/ Instrument	Manufacturer/ ID	Put In Service	Condition When Received (New/Used)
Indianapolis Laboratory			
Balance	Mettler AE163	Not Available	New
Balance	Mettler PM 600	Not Available	New
Fluoride Meter	ATI Orion Expandable EA940	Pre 1990	New
Conductivity Meter	Fisher Scientific/Accumet AR20	2000	New
Muffle Furnace	Blue M Electric Company	Pre-1990	New
Pontiac Laboratory			
BOD Incubator	Precision Scientific, Model 815	1997	New
Incubator	Precision Scientific	Pre-1990	New
Incubator Bath	Precision Scientific	2003	New
Sterilizer	Barnstead, Model C57835	1989	New
Analytical Balance	Mettler/ AE 200	1997	New
Oxygen Meter	YSI Scientific/33	1994	New
Spectrometer	Milton Roy 401	1989	New
pH Meter	Corning, Model 220	1989	New
Ice Maker	Whirlpool, Gold	2003	New
Refrigerators (3)	Dynasty Model J 653-008A	1992	New
Refrigerator	GE Model TBX18LPJR	1992	New
Gravity Oven	Blue M	1992	New
Mechanical Convection Oven	Precision, Model STM 135	1999	New

TABLE 9-2B
METALS LABORATORY EQUIPMENT AND INSTRUMENTATION

Equipment/ Instrument	Manufacturer/ ID	Put In Service	Condition When Received (New/Used)
Dayton Laboratory			
Graphite Furnace	Perkin Elmer/SIMAA 6000	1995	New
Graphite Furnace	Perkin Elmer/4100 ZL (A)	2000	Used
Graphite Furnace	Perkin Elmer/4100 ZL (B)	2001	Used
ICP	Thermal Jarrell Ash/Enviro 36	1992	Used
ICP	Thermal Jarrell Ash/Enviro 61	2002	Used
Trace ICP	Perkin Elmer/Optima 3000DV	2000	Used
ICP-MS	Perkin/Elmer/ELAN 6000	1995	New
Cold Vapor AA (Hg)	Leeman Labs/PS200	1994	New
Analytical Balance	Mettler/AG 204	1992	New
Microwave Oven	CEM/MDS 2100	1999	Used
Hot Block (2)	Environmental Express/SC100	1999	New
Hot Plates (2)	Thermolyne/Type 2200	1995	New
Water Bath	Precision/270	1999	New

TABLE 9-2C
ORGANICS LABORATORY EQUIPMENT AND INSTRUMENTATION

Equipment/ Instrument	Manufacturer/ ID	Put In Service	Condition When Received (New/Used)
Dayton Laboratory			
GC/MS A	GC – HP/5890 MS – HP/5970 Concentrator – Tekmar/LSC-3000 Autosampler – Archon/5100	1993 1993 1997 1999	Used Used New New
GC/MS B	GC – HP/5890 MS – HP/5970 Autosampler – HP/7673A	1997 1997 1997	Used Used Used
GC/MS C	GC – HP/5890 MS – HP/5970A Concentrator – Tekmar/LSC-3000 Autosampler – Archon/5100	1990 1990 1996 1999	New New New New
GC/MS D	GC – HP/5890 MS – HP/5970 Concentrator – Tekmar/LSC-2000 Autosampler – Tekmar/ALS 2050	1989 1989 1988 1988	Used Used New New
GC/MS E	GC – HP/5890 Series II MS – HP/5970 Autosampler – HP/7673A	1987 1987 1987	New New New
GC/MS F	GC – HP/5890 MS – HP/5970 Autosampler – HP/7673A	1998 1998 1998	Used Used Used
GC/MS G	GC – HP/5890 Series II MS – HP/5970 Concentrator – Tekmar 3000 Autosampler – Archon	1999 1999 1999 1999	Used Used Used New
GC/MS H	GC-HP/6890 MS-HP/5973 Concentrator-Tekmar 3100 Autosampler-Archon	1999 1999 1999 1999	New New New New
GC/MS I	GC-HP/6890 MS-HP/5973 Autosampler-HP/7683	2000 2000 2000	Used Used Used
GCMS J	GC-HP/6890 MS-HP/5973 Autosampler-HP/7683	2002	Used Used Used
GCMS K	GC-HP/6890 MS-HP/5973 Concentrator-Tekmar 3100 Autosampler-Archon	2002	Used Used Used
GCMS L	GC-HP/5890 MS-HP/5970 Concentrator-Tekmar 3000 Autosampler-Archon	2003	Used
GCMS M	GC-HP/6890 MS-HP/5973 Autosampler-HP/7683	2003	Used
GCMS N	GC-HP/6890 MS-HP/6890 Concentrator-Tekmar 3000 Autosampler-Archon	2003	Used

TABLE 9-2C (Continued)
ORGANICS LABORATORY EQUIPMENT AND INSTRUMENTATION

GC 1	GC – HP/5880A Autosampler – HP/7671A Detector – FID Integrator – HP (2)	1985 1985 1985 1985	New New New New
GC 2	GC – HP5890 Autosampler – HP/7673A Detector – ECD (2)	1997 1997 1997	Used Used Used
GC 3	GC – HP5890, Series II Detector – ECD (2)	1999 1999	Used Used
GC 4	GC – HP5890, Series II Detector PID (2), FID Concentrator – Tekmar/LSC-2000 Autosampler – Archon	1999 1999 1988 1996	Used Used New New
GC-5	GC – HP5890 Autosampler – HP/7673A Detector – ECD (2)	1990 1990 1990	New New New
GC-6	GC – HP5890 Autosampler – HP/7673A Detector – ECD (2)	1990 1990 1990	New New New
GC-7	GC – HP5890 Autosampler – HP/7673A (2) Detector – PID (2), FID (2)	1991 1999 1991	New Used New
GC 8	GC – HP5890, Series II Detector PID, FID Concentrator – Tekmar/LSC-2000 Autosampler – Archon	1999 1999 1989 1999	Used Used New New
GC 9	GC – HP5890 Detector FID Autosampler – HP 7673A	2001 2001	Used
GC 10	GC – HP5890 Autosampler – HP 7673B(2) Detectors – ECD (2	2001	Used
GC 11	GC – HP5890 Autosampler – HP 7673C(2) Detectors – FID (2)	2002	Used
GC 12	GC – HP 5890 Autosampler –HP 7673A Detector – FID (2)	2002	Used
GC 13	GC – HP 6890 Autosampler -7683(2) Detector – ECD (2)	2002	Used

TABLE 9-2C (Continued)
ORGANICS LABORATORY EQUIPMENT AND INSTRUMENTATION

HPLC #1	LC – HP 1050 Fluorescence Detector – HP1046A	1999 1999	Used Used
HPLC #2	LC – HP 1100 FLD – HP1046A	2001 2001	Used
FT-IR	Perkin Elmer/1600 Series	1989	New
Analytical Balance	Mettler/AE163	1989	New
Top Loading Balance	Mettler/PE 360	1998	Used
Top Loading Balance	Mettler/BB 330	1995	New
Nitrogen Sample Concentrator (2)	Labconco Rapidvap	1994	New
Automated Soxhlet Extraction	Dionex	1999	New

Table 9-3
Periodic Calibration

(Table 9-3) **Instrument**	**Type of Calibration/ Number of Standards**	**Frequency**	**Acceptance Limits**	**Corrective Action**
Analytical Balance	Accuracy determined using Class S or ASTM Class Type 1 weights. Minimum of 2 standards bracketing the weight of interest. Inspected by Accredited Firm	Monthly Daily Yearly	See Balance Calibration Check SOP	Call for service Recalibrate Balance Remove from Service
Top Loading Balance	Accuracy determined using Class Type 1 or 2 weights. Minimum of 2 standards bracketing the weight of interest.	Monthly Daily	See Balance Calibration Check SOP	Call from service Recalibrate
Class S or ASTM Class Type 1 Weights	Accuracy determined by accredited weights and measurement laboratory.	5 years.	N/A.	Replace.
NIST Traceable Thermometer	Accuracy determined by accredited weights and measurement laboratory.	5 years.	N/A	Replace.
Refrigerator	Temperature checked using NIST traceable thermometer.	Daily	4°C + 2°C	Adjust thermostat and recheck later.
Freezer	Temperature checked using NIST traceable thermometer.	Daily	Below or at Temperature listed on the Temp. Log	Adjust thermostat and recheck later.
Oven	Temperature checked using NIST traceable thermometer.	When in use.	See various SOPs	Adjust thermostat and recheck later.
Incubator	Temperature checked using NIST traceable thermometer.	When in use.	See various SOPs	Adjust thermostat and recheck later.
Water Bath	Temperature checked using NIST traceable thermometer.	When in use.	See various SOPs	Adjust thermostat and recheck later.

(Table 9-3) **Instrument**	**Type of Calibration/ Number of Standards**		**Frequency**	**Acceptance Limits**	**Corrective Action**
Autoclave for Biological Tests	Maximum Temperature Registering Thermometer	Checked by State During Surveys.	N/A	N/A	N/A
Autoclave for Biological Tests	Autoclave External Timer		Checked quarterly	N/A	N/A
	Autoclave External Thermometer		Checked quarterly	N/A	N/A
Volumetric Dispensing Devices (Eppendorf ® pipet, auto dilutor or dispensing devices)	Verify by weight calibration.		Monthly	4 % Error	Remove from Service
Glass Microliter Syringes	Calibrated by manufacturer.		N/A	N/A	N/A
Conductivity Meter	See Conductivity SOP		Daily	See Conductivity SOP	Remove from Service
Volumetric Glassware/ Plasticware	Verify by weight calibration		When received.	4 % Error	Etch/mark correct volume or discard.

TABLE 9-4
DFTPP/BFB Tuning Criteria

BFB Requirements (EPA 624/8260/524.2)

m/z	Requirements)
50	15 to 40 % of m/z 95
75	30 to 60 % of m/z 95 (30 to 80 % of m/z 95 for EPA 524.2)
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of m/z 95
173	Less than 2 % of m/z 174
174	Greater than 50 % of m/z 95
175	5 to 9 % of m/z 174
176	Greater than 95 % but less than 101 % of m/z 174
177	5 to 9 % of m/z 176

DFTPP Requirements (625/8270)

Mass	Requirements)
51	30 to 60 % of Mass 198
68	< 2 % of Mass 69
70	< 2 % of Mass 69
127	40 to 60 % of Mass 198
197	< 1% of Mass 198
198	Base Peak, 100 % Relative Abundance
199	5 to 9 % of Mass 198
275	10 to 30 % of Mass 198
365	> 1 % of mass 198
441	Present but lass than mass 443
442	>40 % of mass 198
443	17 to 23 % of mass 442

Section 10.0
Preventive Maintenance

10.1 Each TestAmerica Division follows a well-defined program to prevent the failure of laboratory equipment or instrumentation during use. This program of preventative maintenance helps to avoid delays due to instrument failure.

10.2 Routine preventative maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to meet one of the quality control criteria.

10.3 An instrument maintenance logbook documenting instrument problems, instrument repair and maintenance activities shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters. The inside cover of the maintenance log can include a schedule or Tables 10.1 and 10.2 can be used to determine the schedule for routine maintenance. It is the responsibility of each section supervisor to ensure that instrument maintenance logs are kept for all equipment in his/her section. Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments. Entries must include the date, name of the person performing the service and when appropriate, a statement that the instrument has returned to control and is available for use (state what was used to determine a return to control – CCV acceptable, etc.). When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be stapled into the logbooks adjacent to pages describing the maintenance performed.

10.4 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted using the procedures outlined within this manual.

10.5 Any item of equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown to be defective shall be taken out of service, clearly posted as “Out of Service” and wherever possible, stored in a different location until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

10.6 Some of the analytical equipment systems used in the laboratory are currently maintained under manufacturer's service contract. These instruments for which the laboratory manager decided to hold a contract; for example the ICP unit, have a full preventative/maintenance service contract which provides instrument adjustment and calibration and priority field service calls for maintenance in the event that equipment failure occurs.

Table 10-1
Preventative Maintenance Procedures
For Field Equipment

Instrument/ Equipment Type	Activity	Frequency	Maintenance
Automatic Sampler – ISCO 3710/3700/2700	Check tubing and connections through pump head	Before and after use	Replace tubing when necessary
	Check battery power and program	Before and after use	Replace battery when necessary
	Clean tubing in pump head	After each use	Replace pump head tubing when necessary
	Clean tubing for sample collection	After each use	---
	Check functionality – manual sample; program sample	Prior to use	---
	Check sample container for breakage, etc.	Prior to use	Replace if needed
YSI 85 – Depth Meter, Temperature, Conductivity, and DO	Check battery	Before and after use	Replace batteries when necessary
	Check cable	Before and after use	Send for repair
	Check probe	Before and after use	Send for repair
	Check LCD	Before and after use	Send for repair
Disposable Bailers – ($1^1/_2$" diameter	Check ball valve for overall condition	Prior to use	Clean/replace accordingly
	Check rope	Before, during and after use	Retie or replace as necessary
Solinst Model (Depth Gauge)	Check battery	Prior to use	Replace batteries when necessary
	Clean line	Prior to use	---
Residual Chlorine – HACH Kit	Check battery	Before and after use	Replace batteries when necessary
	Inspect glass cells	Before and after use	Replace as necessary
	Clean glass cells	Prior to use	---
	Inspect ampules for cracks	Before and after use	Replace as necessary
	Inspect cell holder	Before and after use	Remove obstructions, if present
	Check expiration dates of reagents	Prior to use	Remove and reorder as necessary

Table 10-1 (Continued)
Preventative Maintenance Procedures
For Field Equipment

Instrument/ Equipment Type	Activity	Frequency	Maintenance
pH Meter – Hannah 9025; Orion 230A	Check battery	Prior to use	Replace as necessary
	Inspect probe or tip	Prior and during use	Replace or clean as necessary
	Check expiration dates of reagents	Prior to use	Remove and reorder as necessary

Table 10-2
Preventative Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Gas Chromatograph	Replace Gas line dryers and filters	As needed
	Replace Gas cylinders	As needed
	Check or adjust column gas flow and/or detector make-up flow	As needed
	Replace Injection port Septa	Daily
	Replace Injection port liners/re-silonize liners	GC, As needed; GC/MS, As needed
	Replace injection port liner o-ring	GC, As needed; GC/MS, As needed
	Replace inlet seal and ring	GC, As needed, GC/MS, As needed
	Replace column ferrules	GC, As needed;
	Clip column (injector and detector end)	GC, As needed; GC/MS, As needed
	Replace syringes on autosamplers	As needed
	Replace heated-zones heaters and sensors	As needed
	Replace inlet assembly	As needed
	Empty solvent rinse and solvent rinse-waste vials (on autosampler tower)	Daily or as needed
	Replace column	As needed
Flame Ionization Detector (FID)	Clean/replace jet	As needed
	Clean collector	As needed
	Check and/or adjust gas flows	As needed
	Replace graphite ferrule	After each cleaning (OI detectors only)
Electron Capture Detector (ECD)	Perform wipe test	Bi-Yearly
	Remove and send to authorized agency for cleaning	As needed
	Check and/or adjust gas flows	As needed
	Replace gas supply cylinders	As needed
Photoionization Detector (PID)	Clean window	As needed
	Replace o-ring seat	As needed
	Replace Lamp	As needed
	Check and/or adjust gas flows	As needed
	Adjust Lamp power supply intensity	As needed

Table 10-2 (Continued)
Preventative Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Mass Spectrometer (MS)	Clean source, replace source parts, replace filaments	As needed
	Clean analyzer	As needed
	Replace electron multiplier	As needed
	Clean or replace glass jet separator, replace transfer line from jet separator to MS	As needed
	Change rough pump oil	Yearly
	Refill calibration compound (PFTBA) vial	As needed
Purge and Trap Equipment	Refill rinse water supply/Empty rinse water waste	As needed
	Refill spiking solutions vials	As needed
	Rinse sparge tubes	As needed
	Clean or replace 6-port valve	As needed
	Replace Transfer lines (from Autosampler to LSC and from LSC to GC)	As needed
	Adjust gas flows and pressures	As needed
	Perform leak check	As needed
Graphite Furnace, Atomic Absorption (GFAA)	Change Graphite contact rings	As needed
	Clean quartz windows	As needed
	Refill rinse water	As needed
	Check water cooler water level and filter	Monthly
	Change argon and other gases	As needed
	Clean or replace sampling probe	As needed
ICP-MS	Clean Cones by Sonication	As needed
	Replace Pump Tubing	As needed
	Replace/Clean Sample Probe	As needed
	Replace /Clean Sample Gem Tips	As needed
	Replace Cone O-rings	As needed
	Replace Probe Tubing	As needed
	Replace or Clean Ion Lens A) O rings B) Injector C) Injector support	As needed
	Replace Pump Oil when dark color	As needed
	Replace Electron Multiplier	As needed
	Re-optimize Pulse & Analog Stages of Detector	As needed
	Dual Detector Cross Calibration	As needed
	Clean Spray Chamber/ Replace O-Rings	As needed

Table 10-2 (Continued)
Preventative Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Inductively Coupled Plasma, Atomic Emission Spectrometer (ICP-AES)	Replace Peristaltic pump tubing	As needed
	Clean autosampler, change tubing	As needed
	Clean nebulizer and torch assembly	As needed
	Replace argon tanks	As needed
	Refill rinse water receptacle	Daily
	Empty waste receptacles	Daily
	Operate and check vents	Daily
	Check water level and water filter on cooling unit, refill	As needed
	Replace nebulizer and o-rings	As needed
	Replace torch	As needed
	Replace mixing chambers	As needed
	Clean or replace air filters	Weekly
High Pressure Liquid Chromatography (HPLC)	Replace pre-column filter	As needed
	Refill Solvent reservoirs	As needed
	Reverse column and rinse with solvents	As needed
	Replace column	As needed
	Clean solvent reservoir filters	As needed
	Replace ball-valve cartridges on high pressure pump	As needed
	Replace DAD flow cell windows	As needed
	Check system solvent pressure	Daily
pH Meters	Clean or replace electrode	As needed
	Refill electrode electrolyte	As needed
ISE Meter	Clean or replace electrode	As needed
	Refill electrode electrolyte	As needed

Table 10-2 (Continued)
Preventative Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Balance	Clean pan and platform	After each use
	Check Level bubble	Daily
	Check calibration	Daily
	Cleaning and calibration by authorized service	Annually
Conductivity Meter	Clean probe	Every use
	Clean temperature compensating probe	Every use
	Replace probe	As needed
	Replace temperature compensating probe	As needed
Dissolved Oxygen Meter	Replace membrane	As needed
	Clean probe	As needed
ZHE vessels/TCLP filtration unit	Replace o-rings and screens	As needed
ZHE and TCLP Tumblers	Check Rotation Rate	Monthly
Spectrophotometers	Clean and check tubing	As needed
	Change lamp	As needed
	Align lamp/mirrors	Every time the lamp is changed
Burettes	Drain	Daily
Pipets	Clean and check calibration	Monthly
Thermometers	Check calibration	Semi-annually, Quarterly for Digitals, IR Thermometer, Autoclave External Thermometer
Ovens	Check and/or adjust temperature, record temperature on log sheet	Daily
Incubators	Check and/or adjust temperature, record temperature on log sheet	Daily
	Refill water reservoir	As needed
	Calibrate timer	Quarterly
Refrigerators and Freezers	Check and/or adjust temperature, record temperature on log sheet	Daily
	Defrost freezers	As needed
Accelerated Solvent Extractor	Refill solvent reservoir	Daily or as needed
	Empty waste receptacle	As needed
	Check/ clean solvent receiver-vial needle	Daily
	Replace cell seals	As needed
	Clean or replace cell frits	As needed
	Replace nitrogen gas tank	As needed
Leeman Hg Analyzer	Change Nitrogen supply tank	As needed
	Change drying tube	Daily or as needed
	De-clog drying tube and/or reductant tubing	Daily or as needed
	Clean optical cell	As needed (when aperture is out of line)
	Perform “Clean-out” procedure of entire system	Quarterly
	Change system tubing	2-3 weeks
	Rinse tubing prior to operation and following operation	Daily

Table 10-2 (Continued)
Preventative Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
TOC	Check mist trap and empty	Daily
	Check copper scrubber / change as needed	Daily
	Check gas flow daily	Daily
	Clean mist trap w/ soap and water	Weekly
TRAACS	Change pump tubing	Bi Monthly
	Check gas supply	Daily
	Clean tubes with dilute Clorox solution	Bi-Weekly
	Clean sample probe as needed	As needed
	Change color lamp	As needed
	Cold start	Daily
Ammonia Distiller (Labconco Rapidstill)	Replace Heating Coil	As needed
	Replace water in boiling flask	Daily
	Clean boiling flask with 1:1 HCl	Daily
	DI Blank run through system	At start up and tear down
Flashpoint	Check /Clean analyzer cup, thermometer, and accessories with acetone, soapy water, DI water	Daily
	Check gas flow; replace gas as needed	Daily
	Take down flashpoint instrument; inspect parts and replace as needed, thoroughly clean with acetone, tap water, DI water	Monthly

Section 11.0
Quality Control Checks and Routines to Assess Precision and Accuracy and Calculation of Method Detection Limits

TestAmerica maintains a well-defined internal quality control program. Systems of specific activities are in use in the laboratory to ensure that the analytical data generated is of consistent high quality. Data quality is defined in terms of data quality objectives (DQOs). DQOs are the qualitative and quantitative statements that specify the required level of data quality based on the end use of the data. The laboratory is able to assess data quality by monitoring precision and accuracy. The end user of the data may assess data quality by monitoring completeness, comparability, and representativeness. These terms are defined in Section 5 (QA Objectives for the Measurement of Data).

11.1 Quality Control Checks

The following are definitions of specific quality control checks and other relevant terms. The quality control checks are performed as required by method or regulations to assess precision and accuracy. For specific information regarding frequency, control limits and corrective action, see Appendix 4 and Section 5 (QA Objectives for the Measurement of Data). Section 9 (Calibration Procedures and Frequency) discusses calibration procedures and Section 13 (Corrective Action) discusses corrective action.

11.1.1 **Analysis Duplicate**: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

11.1.2 **Batch**: Environmental samples which 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 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) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples.

11.1.3 **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.

11.1.4 **Blind Sample**: A sample 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 the analyst's or laboratory's proficiency in the execution of the measurement process.

11.1.5 **Calibrate:** To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct

value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

11.1.6 **Confirmation**: A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Calculate the percent difference (RPD) between the results using the formula described in Section 12 where R_1 and R_2 are the results for the two columns and the vertical bars in the equation indicate the absolute value of the difference. Therefore, RPD is always a positive value.

If one result is significantly higher (e.g., > 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or analyst) during peak integration.

If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The data user should be advised of the disparity between the results of the two columns.

11.1.7 **Detection Limit:** The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.

11.1.8 **Dilution Test:** A recommended quality control sample used in metals analysis whenever a new or unusual sample matrix is encountered. It will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within +/- 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

11.1.9 **Duplicate Analyses:** The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (See 11.1.16 – Matrix Spike Duplicate.)

11.1.10 **Internal Standard:** A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method.

11.1.11 **Laboratory Control Sample (LCS)**: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified 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.

An LCS shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance. Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (Also see 11.1.25 – Spike, Section 11.2.4 and Appendix 4.)

11.1.12 **Laboratory Duplicate:** Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

11.1.13 **Limit of Detection (LOD):** The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank.

11.1.14 **Limit of Quantitation (LOQ)**: The level above which quantitative results may be obtained with 99% confidence. This value is statistically determined as 10 times the standard deviation from a MDL Study.

11.1.15 **Matrix Spike (spiked sample, fortified sample)**: Prepared by adding a known mass 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, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (Also see 11.1.25 – Spike, Section 11.2.4 and Appendix 4.)

11.1.16 **Matrix Spike Duplicate (spiked sample/fortified sample duplicate)**: A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

11.1.17 **Method Blank**: A sample of a matrix similar to the batch of associated samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch. Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with the appropriate data qualifier.

11.1.18 **Method Detection Limit**: The minimum concentration of a substance (an analyte) 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.

11.1.19 **Method of Standard Additions (MSA)**: In metals analysis, when the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. An alternative to using the method of standard additions is the internal standard technique. Add one or more elements not in the samples and verified not to cause an interelement spectral interference to the samples, standards and blanks.

11.1.20 **Post Digestion Spike**: A recommended quality control sample used in metals analysis whenever a new or unusual sample matrix is encountered. The spike is added to the sample after digestion. It is a test for matrix interference (positive or negative bias). The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

11.1.21 **Quality Control Check Sample**: An uncontaminated sample with known amounts of analytes from a source independent from the calibration standards.

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.

11.1.22 **Quality Control Check Standard**: In general, these samples are prepared similarly to an LCS, except that the reagent water is spiked with all compounds of interest. It must be from an independent source from the calibration standards. The standard is generally required in 40 CFR Part 136 methods (e.g. EPA 624) due to the long list of analytes and the risk that the spiked sample may have some analytes outside of control limits. Note the required concentration of the standard as described within the published method or laboratory SOP.

11.1.23 **Range:** The difference between the minimum and the maximum of a set of values.

11.1.24 **Reagent Blank (method reagent blank)**: A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

11.1.25 **Spike:** A known mass of target analyte added to a blank, sample or subsample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

11.1.26 **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.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (Also see Section 11.2.4 and Appendix 4.)

11.2 Generating Control Limits for Precision and Accuracy

Historical data that the laboratory generates are used to calculate in-house control limits for matrix spike recoveries, surrogate recoveries and laboratory control sample recoveries. The development of in-house control limits and the use of control charts or similar procedures to track laboratory performance are important.

11.2.1 Accuracy

Accuracy is estimated from the recovery of spiked analytes from the matrix of interest. For each matrix spike sample analyzed, calculate the percent recovery of each matrix spike compound added to the sample, as described in Section 12.1.3 (Data Reduction, Validation and Reporting).

For each collected sample, calculate the percent recovery of each surrogate, as follows:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.2.1.1 Calculate the average percent recovery (p) and the standard deviation (s) for each of the matrix spike compounds after analysis of 15-20 matrix spike samples of the same matrix. Calculate the average percent recovery (p) and the standard deviation (s) for each of the surrogates after analysis of 15-20 collected samples of the same matrix, in a similar fashion.

11.2.1.2 Calculate upper and lower control limit for each matrix spike or surrogate compound, as follows:

Upper control limit = p + 3s
Lower control limit = p - 3s

Calculate warning limits as:

Upper warning limit = p + 2s
Lower warning limit = p - 2s

11.2.2 Precision

Calculation of Precision - Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for matrix spike/matrix spike duplicate pairs, or for duplicate analyses of unspiked samples. For each matrix spike/matrix spike duplicate or sample and sample duplicate analyzed, calculate the relative percent difference, as described in Section 12 (Data Reduction, Validation and Reporting).

(Note: Range is a better measurement of precision than RPD as analytical results approach the MDL (20x the MDL is a reasonable figure). This is especially important for those analyses that do not lend themselves to spiking (i.e., BOD, pH, Solids). For each sample and sample duplicate, calculate range as follows:

$$\text{Range} = |C(_1) - C(_2)|$$

where:

$C(_1)$ = Measured concentration of the first sample aliquot
$C(_2)$ = Measured concentration of the second sample aliquot)

11.2.2.1 Calculate the average (p) and the standard deviation (s) for each of the duplicated compounds after analysis of 15-20 duplicate samples of the same matrix.

11.2.2.2 Calculate control and warning limits for each compound (since RPD or range are expressed as a positive number, there can be no lower control limit, as that value would be a negative number), as follows:

Control limit = p + 3s
Warning limit = p + 2s

11.2.3 Control limits approximate a 99% confidence interval around the mean, while warning limits approximate a 95% confidence interval. Statistically, sixty-eight percent of all results should fall within one standard deviation of the mean. Statistically, seven consecutive results on one side or the other of the mean indicate an anomaly that should be corrected, while three consecutive results exceeding warning limits also indicate an event that should be investigated.

11.2.4 Any matrix spike, surrogate, or LCS result outside of the control limits requires evaluation by the laboratory. Such actions should begin with a comparison of the results from the samples or matrix spike samples with the LCS results. If the recoveries of the analytes in the LCS are outside of the control limits, then the problem may lie with the application of the extraction and/or cleanup procedures applied to the sample matrix or with the chromatographic procedures. Once the problem has been identified and addressed, corrective action may include the re-analysis of samples, or the extraction and analysis of new sample aliquots, including new matrix spike samples and LCS. When the LCS results are within the control limits, the problem may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, and determinative methods. For a further discussion of corrective action, see Appendix 4.

Control (acceptance) limits and warning limits are printed and updated at least semi-annually. Once limits are updated, the new limits are posted in the laboratory (dated and approved by the QA Officer) and entered into the LIMS. The QA Officer maintains an archive of all limits used within the laboratory with

the start and ending effective dates. QC results are added daily, but acceptance limits are updated semi-annually. The control and warning limits used to evaluate sample results are those that are in place at the time of sample analysis.

11.2.6 For methods and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

11.2.7 Results used to develop acceptance criteria must meet all other QC criteria associated with the determinative method. For instance, matrix spike recoveries from a GC/MS procedure are generated from samples analyzed after a valid GC/MS tune and a valid initial calibration that includes the matrix spike compounds. Another example is that analytes in GC or HPLC methods must fall within the established retention time windows in order to be used to develop acceptance criteria.

11.2.8 It is advisable to consider the effects of the spiking concentration on matrix spike control limits, and to avoid censoring of data. The acceptance criteria for matrix spike recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling matrix spike/matrix spike duplicate data to generate control limits. Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup, and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

11.2.9 Another common error in developing acceptance criteria is to discard data that do not meet a preconceived notion of acceptable performance. These results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. Remember that for a 95% confidence interval, 1 out of every 20 observations likely will still fall outside the limits. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. Rather, a statistical test for outlier values is employed (see Section 11.3).

11.2.10 In-house QC limits must be examined for reasonableness. Poor recoveries should not be legitimized due to the incorrect choice of methods or spiking levels. In-house limits are important when considering the objectives of specific analyses. For example, recovery limits that include allowance for a relatively high positive bias (e.g., 70 - 170%) may be appropriate for determining that an analyte is <u>not</u> present in a sample. However, they would be less appropriate for the analysis of samples near but below a regulatory limit, because of the potential high bias.

It may be useful to compare QC limits generated in the laboratory to the performance data that may be listed in specific determinative methods. However, be aware that performance data generated from multiple-laboratory data tend to be significantly wider than those generated from single-laboratory data. In addition, comparisons between in-house limits and those from other sources should generally focus more on the accuracy (recovery) limits of single analyses rather than the precision limits. For example, a mean recovery closer to 100% is generally preferred, even if the ±3 standard deviation range is slightly wider, because those limits indicate that the result is likely closer to the "true value." In contrast, the precision range provides an indication of the results that might be expected from repeated analyses of the same sample.

11.3 Statistical Outlier Tests

It is important to exclude extreme measurements from a data set to eliminate bias in statistical evaluations such as control limit calculations or Method Detection Limit (MDL) studies. Extreme or atypical values are often referred to as outliers because of their location outside the normal distribution curve. When data measurements are normally distributed, certain statistical assumptions can be made about the data: about 68% of the measurements will be within one standard deviation of the mean; about 95% of the measurements will be within two standard deviations of the mean; and about 99% of all measurements will be within three standard deviations of the mean.

Outliers can significantly alter the outcome of an MDL Study. Including outliers in an MDL calculation leads to increased variability, and therefore a larger standard deviation. Theoretically, no result should be rejected, because it may indicate either a faulty technique that casts doubt on all results or the presence of a true variant in the distribution. An MDL calculated using outliers would be inaccurate and higher than the actual detection limit. For this reason, it is important to recognize outliers, and to reject them from the calculation of the MDL. Known outliers, that is, the result of any analysis in which an error is known to have occurred (i.e., a leak during extraction/distillation, incorrect integration), should automatically be rejected from the data set.

11.3.1 Z Score

Z-scores can be calculated for large sample sizes (greater than 30 data points), and thus are useful to determine if a value should be excluded from a calculation of control limits. A Z-score of greater than 4 is an indication that the data point in question is an outlier. The Z-score is calculated as follows:

$$Z = \frac{|x - x_{bar}|}{s}$$

where:
Z = Z-score
x = the measurement in question
x_{bar} = the mean of the measurements

s = the standard deviation of the measurement

11.3.2 Grubbs' T test

The Grubbs' T test is an objective test for determining whether a point is an outlier in a smaller data set (less than 20 data points). The Grubbs' T value is calculated as follows:

$$T = \frac{|x_q - x_{bar}|}{s}$$

where:

T = Grubbs' T value
x_q = the measurement in question (the data point furthest from the mean)
x_{bar} = the mean of the measurements
s = the standard deviation of the measurement

The result of the calculation is compared against the value of T from Table 11-1, using the appropriate number of measurements and the acceptable rejection factor (the 5% rejection factor is presented here). If the Grubbs' T value is greater than the value of T from the table, the data point in question is a statistical outlier, and should be rejected from the data set.

Table 11-1: Critical Values for Grubbs' T

Number of Data Points	Critical Value
7	1.94
8	2.03
9	2.11
10	2.18
12	2.29
14	2.37
15	2.41
16	2.44
18	2.50
20	2.56

11.4 Procedure for the Determination of the Method Detection Limit (MDL)

The MDL defined below is adapted from 40CFR Part 136, Appendix B, Revision 1.11. If another procedure is used to determine a Limit of Detection, it is important that the correct terminology be applied (i.e., Detection Limit, Limit of Detection), as MDL is defined to mean the procedure outlined in 40CFR Part 136. Similarly, Limit of Quantitation is defined on the basis of this MDL study. Reporting Limit and Quantitation Limit are not interchangeable with Limit of Quantitation.

11.4.1 Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent water spiked with the analyte, to wastewater containing analyte, to sand or other sold matrices containing the analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample-processing steps of the analytical method be included in the determination of the MDL. The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample. The MDL procedure was designed for applicability to a broad variety of physical and chemical methods, and should be performed in both aqueous and non-aqueous matrices (where samples are analyzed in both matrix types). MDLs must be determined each time there is a significant change in the test method or instrument type. A MDL study is not required for any component for which spiking solutions or quality control samples are not available, such as odor and temperature.

11.4.2 Procedure

11.4.2.1 Make an estimate of the detection limit using one of the following:

a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.
b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.
c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.
d) Instrumental limitations.
e) It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

11.4.2.2 Prepare a matrix (i.e., reagent water) that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the MDL of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

11.4.2.3 Matrix choice

a) If the MDL is to be determined in reagent water, prepare a laboratory standard at a concentration which is at least equal to or in the same concentration range as the estimated detection limit (recommend between 1 and 5 times the estimated detection limit). Proceed to Step 11.4.2.4.
b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 11.4.2.4. (Note: Clean sand may also be spiked to determine the MDL for solids.)
    1) If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.
    2) If the measured level of analyte is greater than five times the estimated detection limit, there are two options.
        (i) Obtain another sample with a lower level of analyte in the same matrix if possible.
        (ii) This sample may be used as is for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL; hence the MDL determined under these

circumstances may not truly reflect method variance at lower analyte concentrations.

11.4.2.4 Analysis

Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method-reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. Where allowed by the method, the average blank measurement is subtracted from the respective sample measurements.

11.4.2.5 Calculate the standard deviation (s) of the replicate measurements.

11.4.2.6 Compute the MDL, as follows:

$$MDL = t_{(n-1,1-\mu=0.99)} (s)$$

where:

MDL = the method detection limit
$t_{(n-1,\mu-1=0.99)}$ = the Students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (see Table 11-2).
s = standard deviation of the replicate analyses.

11.4.3 Reporting

The analytical method used must be specifically identified by number and method title. The date of the study, instrument ID and the name of the analyst(s) performing the analysis must be included. If the analytical method permits options that affect the MDL, these conditions must be specified with the MDL value (i.e., sample preparation methods, columns, detectors). The sample matrix, date of calibration and the standard (ID# and concentration) used must be documented. The MDL for each analyte must be expressed in the appropriate method-reporting units. Report the mean analyte level with the MDL. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery. If the level of analyte in the samples was below the determined MDL or exceeded 10 times the MDL of the analyte in reagent water, do not report a value for the MDL. An example format for documenting each MDL can be found in Figure 11-1.

Table 11-2: Students' t-Values at the 99 Percent Confidence Level

Number of replicates (n)	Degrees of freedom (n-1)	$t_{(n-1, .99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539

Figure 11-1: Example MDL Reporting Format

TestAmerica MDL Report

Analyst:	aaa	Prep Analyst:	bbb
Date:	10/21/03	Prep Date:	10/20/03
Instrument ID:	SIMAA	Prep Method:	
Method:		Previous MDL:	0.3
Units:		Spike Amount:	1.000
		Reporting Limit:	1.000

Comments:

MDL Replicates

1)	0.800
2)	0.900
3)	1.000
4)	0.950
5)	0.880
6)	1.000
7)	1.000

Average	0.933
Average % Rec.	93 %
STD Deviation	0.077
Calc. MDL	0.241

The MDL is acceptable.

Section 12.0
Data Reduction, Validation and Reporting

12.1 Data Reduction

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method/ per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. If the formulas outlined in this section are not used, the correct formula can be found in the appropriate method SOP.

12.1.1 The analyte concentration in a sample analyzed using external standard calibration can be determined by:

$$\text{Concentration (ppb)} = \frac{(A_s)(V_t)(D)}{(\text{avgCF})\,(V_i)\,(S)}.$$

where

A_s is the area of the peak for the analyte in the sample
V_t is the total volume of the extract in µl (for purge and trap analysis V_t=1)
D is the dilution factor (if no dilution is performed D=1)
avgCF is the mean calibration factor from the initial calibration in area/ng
V_I is the volume of the extract injected in µl (for purge and trap analysis V_I=1)
And S is the sample volume or mass (in mL or g) extracted or purged.

12.1.2 The analyte concentration in a sample analyzed using internal standard calibration can be determined by:

$$\text{Concentration (ppb)} = \frac{(A_s)(C_{is})(V_t)(D)}{(A_{is})(\text{avgRF})(S)}.$$

where

A_s is the area of the peak for the analyte in the sample
C_{is} is the concentration of the internal standard
V_t is the total volume of the extract in ml (for purge and trap analysis V_t=1)
D is the dilution factor (if no dilution is performed D=1)
A_{is} is the area of the internal standard
avgRF is the mean response factor from the initial calibration
And S is the sample volume or mass (in L or kg) extracted or purged.

12.1.3 Calculated values for spiked samples, duplicate analyses, and reference standards are compared with quality control limits to determine data validity. Recovery of any spiked analyte (including surrogate compounds) is calculated as:

$$\%Recovery = \frac{C_s - C_u}{C_n} \times 100$$

where
C_s is the measured concentration of the analyte or surrogate,
C_u is the concentration of the unspiked sample (for LCS and surrogate recoveries $C_u = 0$)
And C_n is the true value or known concentration of the analyte or surrogate.

12.1.4 The precision of duplicate analyses is determined from the relative percent difference (RPD) calculated by:

$$RPD = \frac{|R_1 - R_2|}{\frac{R_1 + R_2}{2}} \times 100$$

where R_1 is the measured concentration of one replicate and R_2 is the measured concentration of the second replicate.

12.1.5 Relative Standard Deviation (RSD) is computed from the standard deviation and mean recovery when the standard deviation is derived from multiple recovery results:

$$RSD = \frac{\text{Standard Deviation}}{\text{Mean Recovery}} \times 100$$

12.1.6 If results are not within acceptance limits, the analysis data is fully reviewed to determine if sample contamination or matrix problems exist. If there is still a problem with the quality of the data, in-depth investigation into the method in question is conducted until the problem is solved.

12.1.7 When comparing results from different columns or detectors, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Calculate the RPD as described above.

12.1.7.1 If one result is significantly higher (e.g.,>40 %), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system or operator during peak integration.

12.1.7.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is a conservative relative to the protection of the environment. The data user should be advised of the disparity between the results on the two columns.

12.1.8 Procedures for manual integration are incorporated by reference to SOP on Manual Integration.

12.2 Corrections

Entries in records shall not be obliterated by methods such as erasures, liquid paper, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria shall also apply to electronically maintained records.

12.3 Logbook Use Guidelines

12.3.1 Use permanent dark ink. No pencils may be used.

12.3.2 Corrections: Use a single line to cross out errors. Date and initial cross out.

12.3.3 Blank pages or blank lines between the last entry and the bottom of page must be "Z 'd" through, initialed and dated.

12.3.4 Data must be entered directly and consecutively into the notebook. It is not to be placed onto scratch paper and entered later.

12.3.5 Entries added to previously signed pages must be dated, initialed and witnessed (if appropriate) below the new material.

12.3.6 Initial and date all pages upon completion.

12.3.7 When pages are added to the notebook, they must be initialed and dated across both the added page and the notebook page.

12.4 Data Verification

Data verification or review is the routine laboratory process through which proper quantification, recording, transcription, and calculations are confirmed. It also confirms that the data is reasonable and complete. The process should be such that errors are minimized and that corrective action steps are taken when errors are detected. The data verification process includes three (3) steps: primary (initial), secondary, and final review.

12.4.1 Primary Review

The analyst performs the initial review of the data. The analyst is responsible for verifying the correctness of the data entered into the Laboratory Information Management System (LIMS). This review includes, but is not limited to, verifying that quality control indicators (QCI) meet protocol criteria, calibration criteria are met, appropriate detection limits were used, data was reduced correctly and that any corrective action was documented properly. The primary reviewer is responsible for verifying any documentation associated with the data, completing review records associated with the process, and compiling QC Reports (level II-IV) requested in the job The analyst must perform primary review on 100% of the data generated.

12.4.2 Secondary Review

The Department Supervisor or a second analyst can be responsible for a secondary review of the data. This step is intended as a verification of the primary review. Secondary review focuses on calibration criteria, QCIs, compound identification, results expression, reporting limits, and level of documentation. All of the data receives a review with 10 % of the calculations being checked for accuracy. If there are errors with the calculations, a 100 % check of the calculations is performed.

12.4.3 Manual Integration Review

Manual Integration Reviews are done as described in the Manual Integrations Procedures SOP (CP01-03).

12.4.4 Final Review

The Project Management Group prior to releasing the final report must perform final review of the completed project. This review ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is completed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

12.4.5 Data found to be of doubtful quality by the analyst, through internal audits or arising from customer concerns, must be reviewed by a member of laboratory management using the procedures outlined in Section 13.

Figure 12-1 is a flow chart for the generation of data. After verification of the data is complete, the results are printed from the LIMS into a final report for transmittal to the client.

Table 12-1 summarizes responsibility for data verification/validation.

12.5 Data Reporting

Analytical results are issued in a format that is intended to satisfy customer requirements. A variety of report formats are available to meet specific needs. The Analytical Report will be printed on laboratory stationary, reviewed, and signed by a designated person. Persons designated to sign reports include: Members of the Project Management Team, the Division Manager, and the Department Supervisors.

12.5.1 Required Report Format and Contents

An example report can be found in Figure 12-3. At a minimum, the following information must be included in all reports:

12.5.1.1 A title;

12.5.1.2 Name and address of laboratory and phone number and a contact name for questions;
12.5.1.3 Unique identification of the report and of each page and the total number of pages. The total number of pages is noted (Page # of Total)
12.5.1.4 Name and address of client and project name if applicable.
12.5.1.5 Description and unambiguous identification of the tested sample including the client identification code.
12.5.1.6 Identification of test results derived from any sample that did not meet NELAC or other regulatory requirements for sample acceptance such as improper container, holding time, or preservation.
12.5.1.7 Date of receipt of sample, date of sample collection, date(s) of sample preparation and analysis.
12.5.1.8 Identification of the test method used, or an unambiguous description of any non-standard method used.
12.5.1.9 If the laboratory collected the sample, reference to sampling procedure a copy of the field notes will be provided upon request.
12.5.1.10 Any deviations from (such as failed QC), additions to or exclusions from the test method (such as environmental conditions), and any non-standard conditions that may have affected the quality of results. Include the use and definitions of any data qualifiers. An example list of typical reporting flags used by the laboratory can be found in Figure 12-3.
12.5.1.11 Measurements, examinations and derived results. Identify whether data are calculated on a dry weight or wet weight basis. Identify the reporting units.
12.5.1.12 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue.
12.5.1.13 A statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory.
12.5.1.14 A statement that the report shall not be reproduced except in full, without prior written approval by the laboratory.
12.5.1.15 Clear identification of numerical results with values outside of the reporting limits.
12.5.1.16 Labs accredited to be in compliance with NELAC standards shall certify that the test results meet requirements of NELAC or provide reasons and/or justification if they do not.
12.5.1.17 Clear identification of all test data provided by a subcontracted laboratory.

12.5.2 TestAmerica offers four (4) levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above

12.5.2.1 Level I-method references, preparation/ analysis dates, surrogate recoveries, and reporting limits.
12.5.2.2 Level II- includes blank, LCS, and precision/accuracy (MS/MSD/duplicates) and CCV information.

12.5.2.3 Level III- documentation for the validation of data, i.e., QC data associated with the preparation, calibration (if applicable), and analysis of samples.

12.5.2.4 Level IV- all information described above in addition to sample raw data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 12.5.4.

12.5.3 Corrected reports

Occasionally a report must be re-issued due to the addition of a test, or the correction of an error. When the report is re-issued, a notation of "Corrected" is to be placed on the page of the report.

12.5.4 Confidentiality and Proprietary Rights

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica Incorporated or any information disclosed to TestAmerica Incorporated by the Client. Report deliverable formats are discussed with each new client. If a client requests that reports be faxed, the reports are faxed with a cover sheet that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the number above.

This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process and copies may be removed from the laboratory for purposes of assessment.

12.5.5 Rounding Convention

When the calculation or instrument gives more figures than needed. It is necessary to round off. The following rules shall be used:

Rule 1: If the next digit beyond the rounding point is less than 5, leave the previous digit unchanged (e.g. 21.4 becomes 21).

Rule 2: If the next digit beyond the rounding point is greater than 5, increase the previous digit by one (e.g. 21.6 become 22).

Rule 3: If the next digit beyond the rounding point is equal to 5, with no digits other than zeros following the 5, round the previous digit to the nearest even number (e.g. 21.5 and 22.5 both become 22).

Rule 4: If the next digit is a 5 followed by other digits, then treat the case as for greater than five as in Rule 2 (e.g. 21.51 becomes 22).

Rule 5: If there are not enough numbers to get to the required number of significant figures, for example 2.3 when working with three significant figures, do not add extra zeros.

Rule 6: When performing calculations, carry at least one extra significant figure through the process and round only the final result. Rounding data before a calculation introduces cumulative errors. Carrying at least one extra digit minimizes this error.

12.6 Data Storage

12.6.1 Records Management

The following records are maintained for a minimum of 5 years unless otherwise designated (Drinking Water – 10 years, Copper and Lead – 12 years, Voluntary Action Program – 10 years (must notify OEPA/Client of intent to dispose.) and to meet the NELAC standards.

12.6.1.1 Correspondence between laboratory and client (including communication logs).
12.6.1.2 Field records of sampling events.
12.6.1.3 Original raw analytical data. This includes, but is not limited to, logbooks, hard copies of chromatograms or computer data printouts of calibration standards, QC samples and analytical samples, MDLs, control limits, standard preparation, method reference and data review records.
12.6.1.4 Copies of final reports including analytical results, log sheets, chain of custody, shipping receipts and where required, copies of the raw data.
12.6.1.5 Quality Assurance records including, but not limited to, archived SOPs, corrective action reports, internal and external audits and responses, Performance Testing sample results and raw data, and employee training records.
12.6.1.6 Business information, including invoices and records pertaining to suppliers.

12.6.2 Record Storage

Hard copies of these records are stored and filed numerically, alphabetically, or chronologically by date or batch as appropriate for the type of record. Periodically, all records are transferred to storage boxes which are labeled with the month(s) and year(s) in which the records were generated. Each box is given a unique number and entered into an archive log that includes a description of the contents of each box and the box location. The archived boxes are stored on-site until transferred to an off-site storage facility. Boxes are stored in such a way to allow easy retrieval of records upon request. Final reports are also maintained electronically on computer hard drives and back-up tapes.

Access to archived information is documented with an access log (see Figure 12-4 for example logs for archiving). Archive areas must be protected against fire, theft loss, environmental deterioration, and vermin. Archive areas are regularly inspected as part of the Internal Audit Program. Representatives of an accrediting authority may have access to archived information.

Sample data relating to known litigation samples and subsamples will be stored in a locked file cabinet maintained by the Division Manager or QA Manager. An Archive Access Log is maintained to document entry into this cabinet.

Should any of The TestAmerica divisions be sold, all records required to document compliance with this QAM will be transferred to the purchaser. Should TestAmerica Analytical Testing Corporation close any of the divisions' offices, all records required to document compliance with this QAM will be transferred to the Corporate Headquarters. Should TestAmerica Analytical Testing Corporation close, all records required to document compliance with this QAM will be offered to the clients to whom they pertain.

12.6.3 Analytical Logbooks

A log is kept of all notebooks (e.g. standard logbooks, instrument logbooks) that are issued. The log is an ACCESS database that is automatically backed up. This log includes:

12.6.3.1 Issuance of Logbooks

(a) Notebook Number. Each notebook is issued a unique number that is determined sequentially.
(b) Notebook Name.
(c) Replaces Notebook Number. Place the number of the notebook that the issued notebook will replace if applicable.
(d) Date of Issue. This is the date that the notebook is released.
(e) Department
(f) Archive.
    1) All notebooks are archived when they are complete and no longer in use. They are turned into the QA Department for archiving.
    2) The date and initials of the person who placed the notebook in the box for archiving.

3) The box number that the notebook is archived in.

Figure 12-1 Analytical Data Review and Reporting Scheme

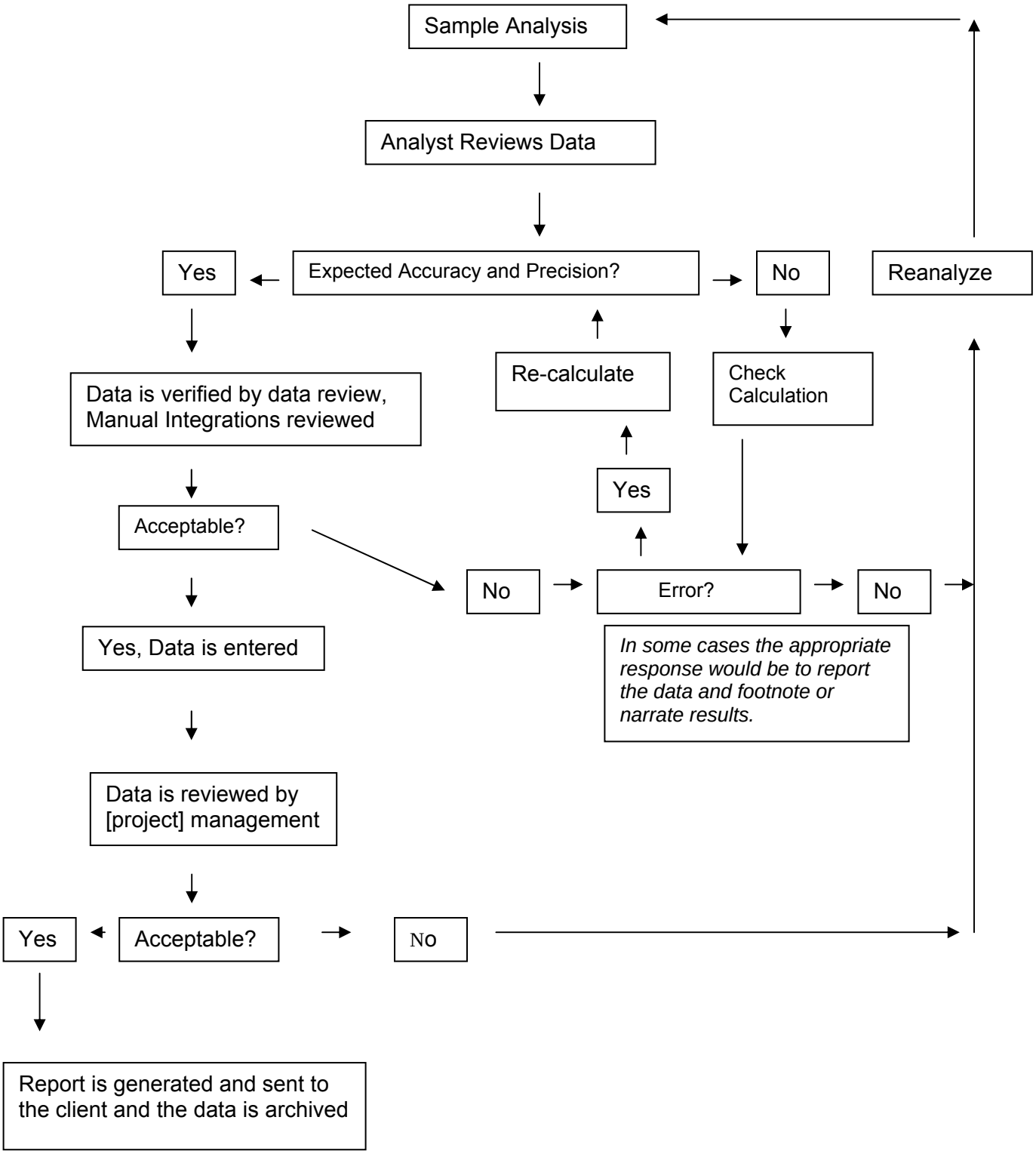
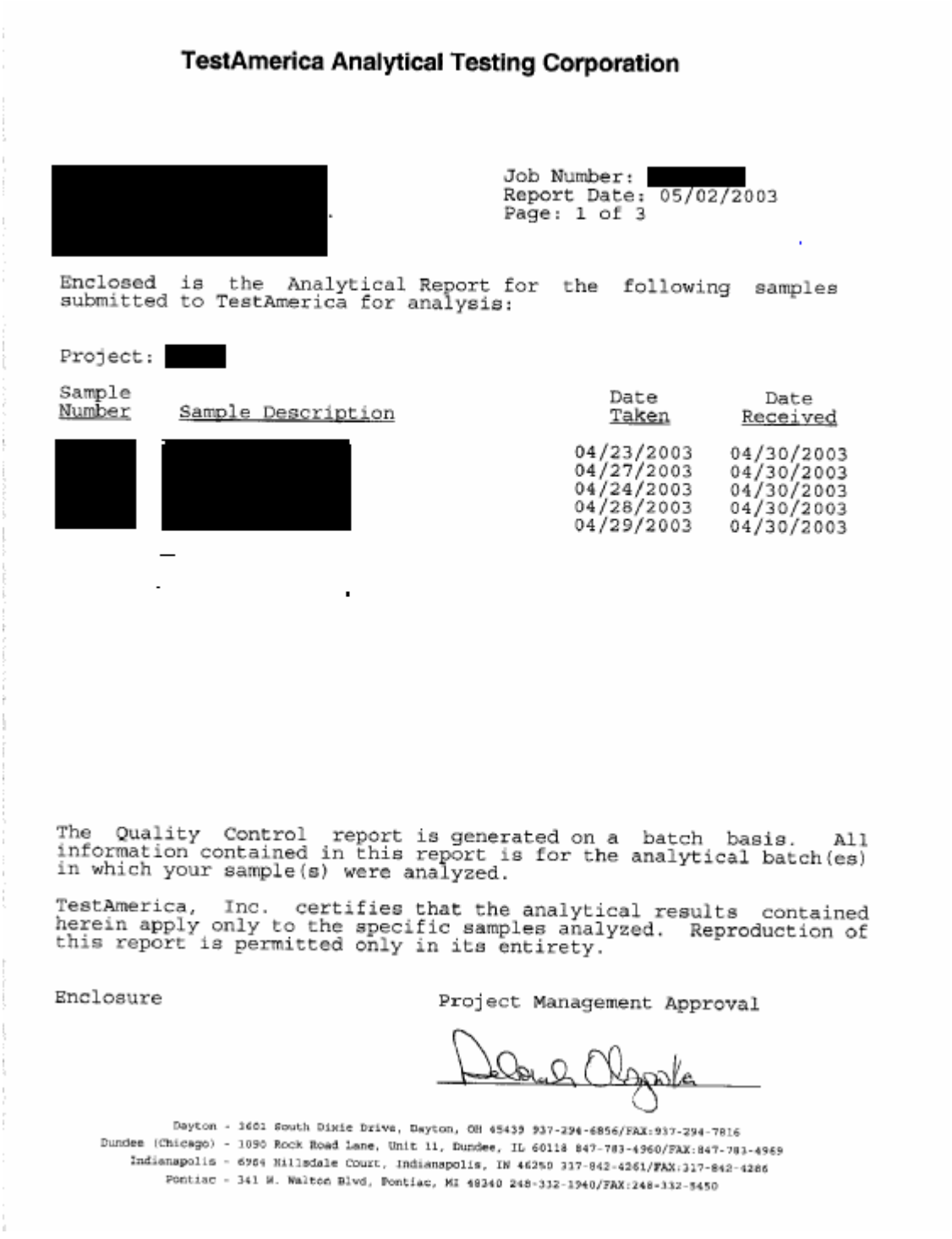


Figure 12-2: Example Report Format



TestAmerica Analytical Testing Corporation

[redacted]

Job Number: [redacted]
Report Date: 05/02/2003
Page: 1 of 3

Enclosed is the Analytical Report for the following samples submitted to TestAmerica for analysis:

Project: [redacted]

Sample Number	Sample Description	Date Taken	Date Received
[redacted]	[redacted]	04/23/2003	04/30/2003
		04/27/2003	04/30/2003
		04/24/2003	04/30/2003
		04/28/2003	04/30/2003
		04/29/2003	04/30/2003

The Quality Control report is generated on a batch basis. All information contained in this report is for the analytical batch(es) in which your sample(s) were analyzed.

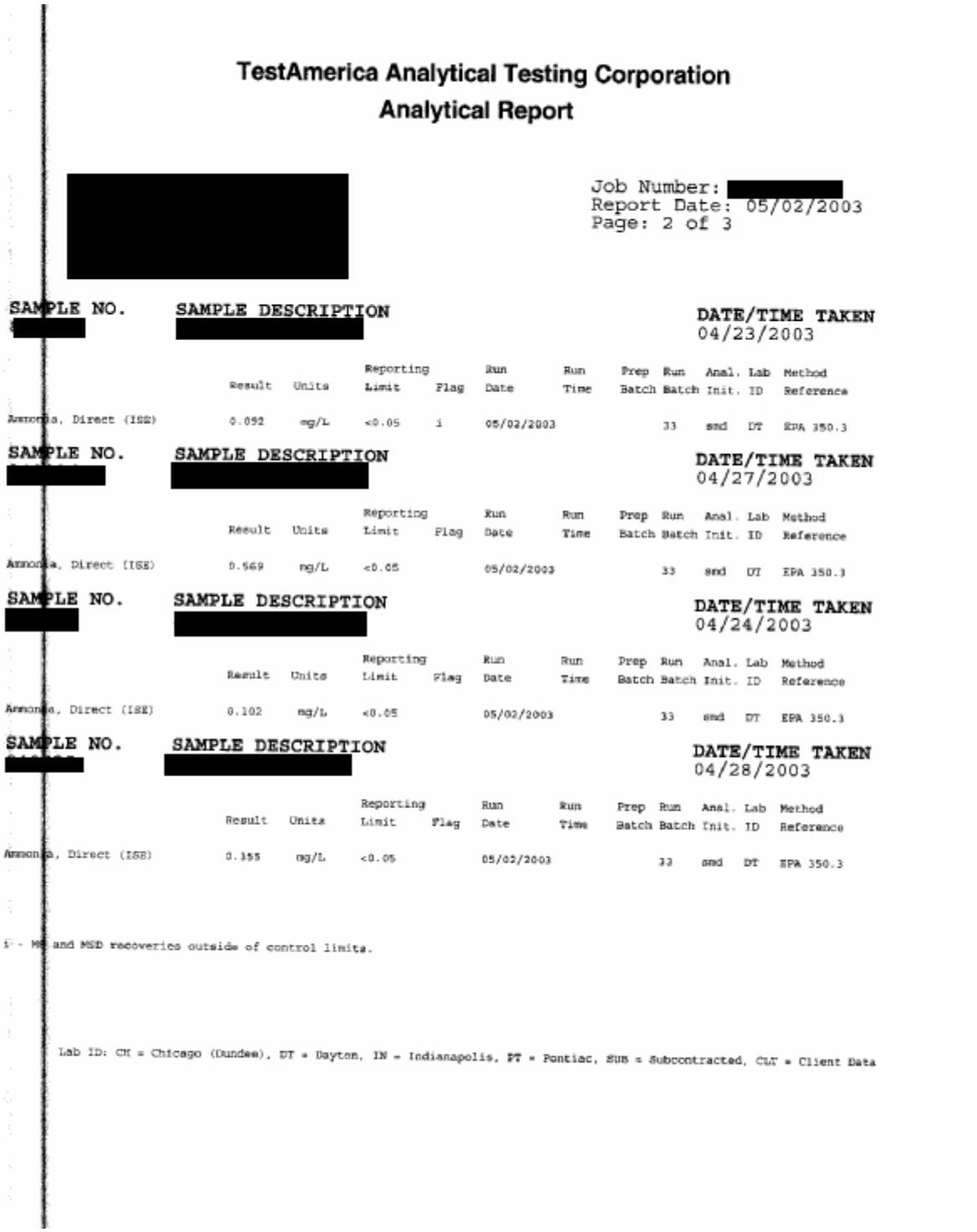
TestAmerica, Inc. certifies that the analytical results contained herein apply only to the specific samples analyzed. Reproduction of this report is permitted only in its entirety.

Enclosure

Project Management Approval

Dayton - 3601 South Dixie Drive, Dayton, OH 45439 937-296-6856/FAX:937-294-7816
Dundee (Chicago) - 1090 Rock Road Lane, Unit 11, Dundee, IL 60118 847-783-4960/FAX:847-783-4969
Indianapolis - 6964 Hillsdale Court, Indianapolis, IN 46250 317-842-4261/FAX:317-842-4286
Pontiac - 341 W. Walton Blvd, Pontiac, MI 48340 248-332-1940/FAX:248-332-5450

Figure 12-2: Example Report Format (Continued)



TestAmerica Analytical Testing Corporation
Analytical Report

[redacted]

Job Number: [redacted]
Report Date: 05/02/2003
Page: 2 of 3

SAMPLE NO.	SAMPLE DESCRIPTION	DATE/TIME TAKEN
[redacted]	[redacted]	04/23/2003

	Result	Units	Reporting Limit	Flag	Run Date	Run Time	Prep Batch	Run Batch	Anal. Init.	Lab ID	Method Reference
Ammonia, Direct (ISE)	0.092	mg/L	<0.05	i	05/02/2003			33	smd	DT	EPA 350.3

SAMPLE NO.	SAMPLE DESCRIPTION	DATE/TIME TAKEN
[redacted]	[redacted]	04/27/2003

	Result	Units	Reporting Limit	Flag	Run Date	Run Time	Prep Batch	Run Batch	Anal. Init.	Lab ID	Method Reference
Ammonia, Direct (ISE)	0.569	mg/L	<0.05		05/02/2003			33	smd	DT	EPA 350.3

SAMPLE NO.	SAMPLE DESCRIPTION	DATE/TIME TAKEN
[redacted]	[redacted]	04/24/2003

	Result	Units	Reporting Limit	Flag	Run Date	Run Time	Prep Batch	Run Batch	Anal. Init.	Lab ID	Method Reference
Ammonia, Direct (ISE)	0.102	mg/L	<0.05		05/02/2003			33	smd	DT	EPA 350.3

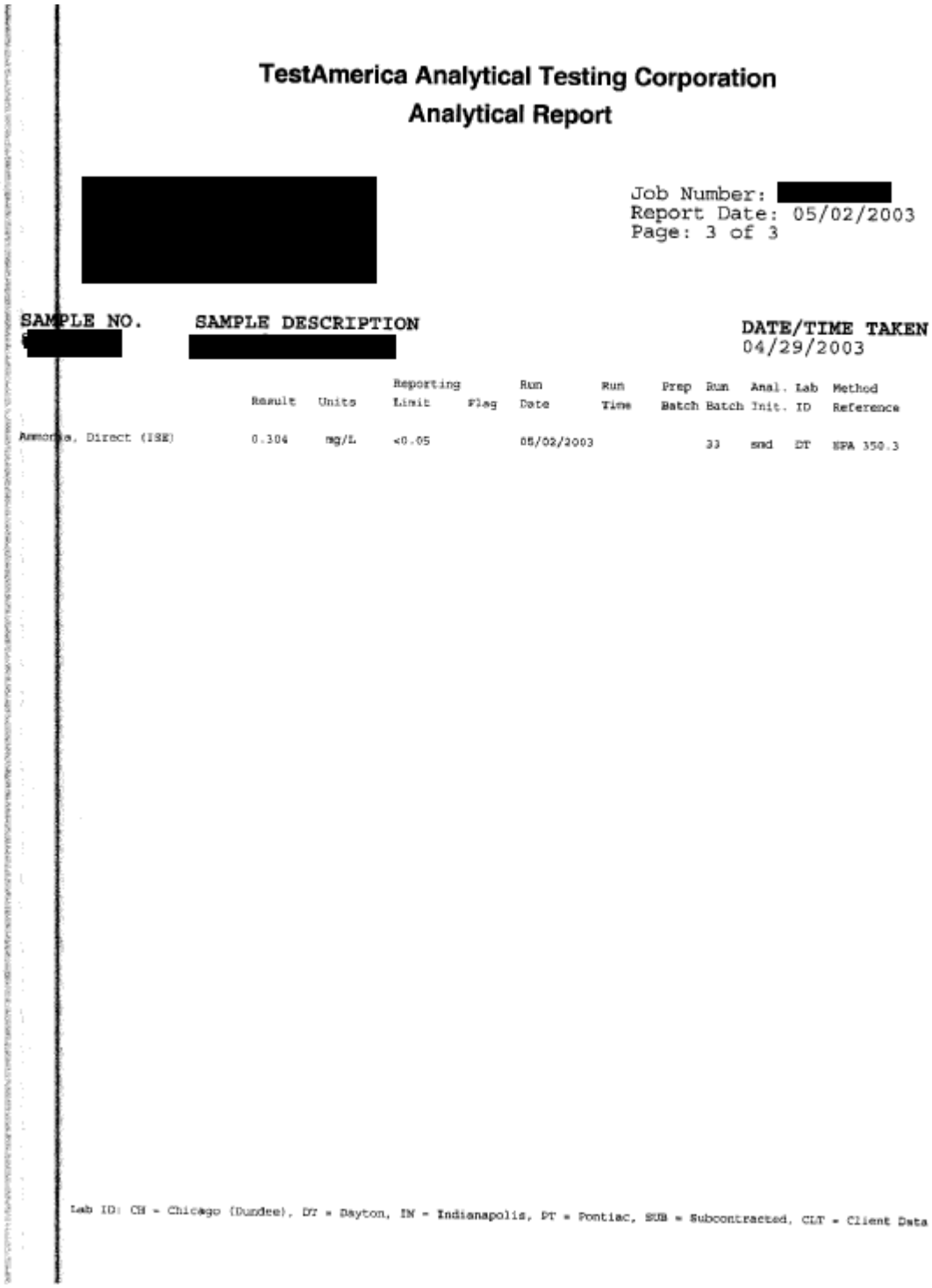
SAMPLE NO.	SAMPLE DESCRIPTION	DATE/TIME TAKEN
[redacted]	[redacted]	04/28/2003

	Result	Units	Reporting Limit	Flag	Run Date	Run Time	Prep Batch	Run Batch	Anal. Init.	Lab ID	Method Reference
Ammonia, Direct (ISE)	0.355	mg/L	<0.05		05/02/2003			33	smd	DT	EPA 350.3

i - MS and MSD recoveries outside of control limits.

Lab ID: CH = Chicago (Dundee), DT = Dayton, IN = Indianapolis, PT = Pontiac, SUB = Subcontracted, CLT = Client Data

Figure 12-2: Example Report Format (Continued)



TestAmerica Analytical Testing Corporation
Analytical Report

[redacted]

Job Number: [redacted]
Report Date: 05/02/2003
Page: 3 of 3

SAMPLE NO.	SAMPLE DESCRIPTION	DATE/TIME TAKEN
[redacted]	[redacted]	04/29/2003

	Result	Units	Reporting Limit	Flag	Run Date	Run Time	Prep Batch	Run Batch	Anal. Init.	Lab ID	Method Reference
Ammonia, Direct (ISE)	0.304	mg/L	<0.05		05/02/2003			33	smd	DT	EPA 350.3

Lab ID: CH = Chicago (Dundee), DY = Dayton, IN = Indianapolis, PT = Pontiac, SUB = Subcontracted, CLT = Client Data

Figure 12-3: Example of Typical Reporting Flags

FLAG	DESCRIPTION	CUSTOMER REPORT MESSAGE
*	RPD out	* - Duplicate analysis not within control limits
A	Not preserved properly	A – Sample was not preserved properly at the time of collection
B	Blank contamination	B – Target analyte detected in blank.
C	Preserved at laboratory	C – Sample was preserved at the laboratory
D	MS out of Control	D – MS recovery outside of control limits
E	Sample over range	E- Target analyte exceeds upper end of curve.
F	MSD out of Control	F – MSD recovery outside of control limits
G	MS and RPD Out	G – MS recovery and RPD outside of control limits
H	Analyzed out of hold time	H – Sample was analyzed outside of holdtime
I	MS, MSD and RPD Out	I – RPD, MS, and MSD recovery outside of control limits
J	Estimated result	J – Reported value is estimated.
K	RPD between MS and MSD is out of control	K – RPD between MS and MSD is outside of control limits
L	Low Spike	L – Spike concentration is less than ¼ analyte concentration
M	Spike Diluted Out	M – Spike concentration is diluted out
N	Elevated RL due to insufficient sample	N – Elevated reporting limit due to insufficient sample
O	Elevated RL due to matrix	O – Elevated reporting limit due to sample matrix.
P	Residual Chlorine detected	P – Res. Chlorine detected. Sample dechlorinated prior to analysis.
Q	Elevated RL due to high non-targets	Q – Elevated reporting limit due to high levels of non-target analytes.
R	Received out of hold time	R – Sample was received outside of hold time
S	Elevated RL due to foaming	S – Elevated reporting limit due to sample foaming
T	Lab not certified	T – Lab not certified for this analyte, not for compliance purposes
U	Result between MDL and RL	U – Reported value is between the MDL and the quantitation limit.
V	Internal Standard out of control	V – Internal Standard is outside of control limits
W	Surrogate out of control	W – Surrogate is outside of control limits
X	LCS out of control	X – Laboratory Control Standard is outside of control limits
Y	Verified by duplicate analysis	Y – Verified by duplicate analysis
Z	Insufficient Sample for MS/MSD	Z – Insufficient sample for MS/MSD
a	See Notes and Comments	a – See Notes and Comments for detailed explanation
b	Diesel Fuel quantitation	b – Quantitated using a diesel fuel standard
c	Kerosene quantitation	c - Quantitated using a kerosene standard
d	Minerals Spirits quantitation	d - Quantitated using a mineral spirits standard
e	Motor Oil quantitation	e - Quantitated using a motor oil standard
f	Elevated RL due to high targets	f – Elevated reporting limit due to high levels of non-target analytes
g	Surrogate Diluted Out	g – Surrogate was diluted out during analysis
h	MSD and RPD out of control	h – MSD recovery and RPD outside of control limits
i	MS and MSD out of control	i – MS and MSD recoveries outside of control limits
j	Collected in an incorrect sample container	j -Incorrect sample container was used for this analysis
K	Residual Chlorine detected	k- Res. Chlorine detected. Sample results may be adversely affected.

Figure 12-4: Example Archive Log and Archive Access Log

ARCHIVE LOG
TestAmerica Analytical Testing Corporation

Box Number	Description of Contents	Location	Date Disposed

Logbook No: _____ Page No: _____

ARCHIVE ACCESS LOG
TestAmerica Analytical Testing Corporation

Date	Time	Initials	Purpose of Access (Item(s) Examined, Item(s) Removed, Item(s) Returned, …)

Logbook No: _____ Page No: _____

Table 12-1 Personnel Responsible for Data Evaluation, Verification and Reporting

Duty	Personnel Responsible
Data Evaluation	
Verification of Sample integrity	Log-in Personnel/Analysts
Chain of Custody Verification	Log-in Personnel
Check of Sample Appropriateness	Analyst/Log-in Personnel
Checking raw data entries and calculations	Peer Review / Dept. Supervisor
Checking instrument/analytical logs	Dept. Supervisor
Checking calibration integrity	Peer Review / Dept. Supervisor / Analyst
Data Validation	
Quality control checks	Analyst, QA officer
Review of laboratory QC data	Dept. Supervisors, QA officer
Review of supporting documentation	Dept. Supervisors/Analyst
Review of data for obviously anomalous values	Dept. Supervisors/Analyst
Manual Integrations	Peer Review / Dept. Supervisor / Analyst/QA Officer
Data Reporting	
Data entry	Analyst
Checking data entry	Analyst, Dept. Supervisor
Final Project Review	Project Management, Division Manager, Quality Assurance,

Section 13.0
Corrective Action

When QC deficiencies or nonconformance situations exist, corrective action procedures provide a systematic approach to assess and restore field or laboratory analytical system integrity.

13.1 Corrective Action Report (CAR)

Corrective actions necessary to obtain acceptable results (i.e. Calibration failed, Blank contaminated) are implemented and documented. Corrective action at the bench level is documented on the raw data. A Corrective Action Report (CAR) (Figure 13-1) documents laboratory quality control and quality assurance issues that warrant further investigation such as across department issues, lab contamination, Performance Testing (PT) result problems, etc. These are retained in the QA Office and a copy included with any applicable data. These reports are approved by the Quality Assurance Officer and Management. An additional type of corrective action documentation is a formally presented report of findings and resolutions for internal and external audits and PT results. These reports are filed in the QA Office with the audit and are distributed to parties interested in the audit findings (see Section 14 – Performance and System Audits).

13.2 Method Suspension/Restriction

In some cases it may be necessary to suspend or restrict the use of a method when a nonconformance exists which constitute a significant risk and/or liability to TestAmerica Incorporated.

13.2.1 Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective action will be stated in writing and presented to the Division Manager.

13.2.2 The Quality Assurance Officer and affected Department Supervisor will be notified.

13.2.3 The Division Manager shall arrange for the appropriate operations people to come to meet with the Quality Assurance Officer the day of notification. This meeting shall be held to confirm that there is a problem, and that suspension/restriction of the method is required.

13.2.4 The meeting will conclude with a discussion of the steps necessary to bring the method or test fully back on line if the method is suspended or restricted. The Quality Assurance Officer will also specify any documentation necessary to verify that corrective action has occurred. A copy of the meeting notes and agreed upon steps should be faxed by the laboratory to Corporate Operations and Corporate Quality Assurance. This fax acts as notification of the incident.

13.2.5 After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. It is the responsibility of the Division Manager to hold all reporting. Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the nonconformance issue.

13.2.6 Within 72 hours, lab management must determine if compliance is now met, and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Division Manager, Quality Assurance Officer, Department Supervisor, Project Management, other), can devise a start-up plan to cover all steps from client notification through compliance of method and release of reports.

13.3 Quality Control Batch Problems

A measurement system may be out of control when QC samples fall outside of the limits described in Sections 5 (QA Objectives for Measurement of Data), 9 (Calibration Procedures and Frequency), 11 (Quality Control Checks and Routines to Assess Precision and Accuracy and Calculation of Method Detection Limits) or Appendix 4.

An entire batch of samples may require corrective action if these quality control criteria are not met. Department Supervisors and/or the Quality Assurance Officer decide if re-analysis, re-extraction, etc. is necessary.

13.3.1 The EPA recommends the following guidelines for assessing acceptable data. If any data is determined to be out of control, one or all of the following should be followed:

13.3.1.1 Review the method with the analyst.

13.3.1.2 Re-analyze the sample batch and evaluate the new results.

13.3.1.3 Recalibrate the instrument with freshly prepared reagents and reanalyze the samples.

13.3.1.4 Re-extract and/or re-analyze the samples per method.

13.3.1.5 Evaluate the data and sample behavior and investigate any possible chemical interferences.

13.3.1.6 Check instrument for possible maintenance requirements.

13.3.1.7 Seek additional help from other analysts or provide additional training for laboratory personnel.

13.3.1.8 Perform a system audit to evaluate corrective action measures.

13.4 Sample Collection Problems

Samples may have to be re-collected if review of the data related to the sample collection, preservation, storage and custody indicates that representative, compliant samples were not obtained.

13.5 Systematic Problems

Those problems of a procedural/system nature generally require the Division Manager's involvement. Examples might include previously reported data that has been affected by a situation requiring correction or if corrective action will impact project schedule or budget. If previous data is affected, the laboratory management staff is responsible for determining the significance of the problem and notifying the customer, of any event that casts doubt on the validity of the data. This notification must be documented.

13.6 Departures from Documented Policies and Procedures

Due to the frequently unique nature of environmental samples, sometimes departures may be needed from documented policies and procedures. When the analyst encounters such a situation, the analyst presents the problem to his/her supervisor for advice. The supervisor may elect to discuss it with the QA Officer or have a member of the Project Management Team representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst so notes it on the raw data. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

13.7 Addressing Complaints

Addressing complaints is a normal function of conducting business and a valuable tool to improve services to and relationships with clients. The goal is expeditious resolution of complaints. At TestAmerica, the supervisor and/or the management team handles systematic problems. Client Services resolves specific complaints concerning container orders, shipping, and expected report dates. These are documented in the individual notebooks. The Project Management Team resolves specific questions about specific sample reports. The staff documents the request on a "Re-Evaluation Request" form (Figure 13-2), researches the situation by reviewing the quote, raw data, Chain of Custody, method, regulations, etc., discusses the details with management who may decide to retain the result, require re-analysis, provide a case narrative and/or reissue the report. This form is filed with the chain of custody and the login sheets.

13.8 Immediate vs. Long-Term Corrective Action

13.8.1 Immediate corrective actions are necessary to correct or repair non-conforming equipment and systems. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis.

13.8.2 Long-term corrective actions are necessary to eliminate causes of non-conformance. The need for such actions will probably be identified by audits. Examples of this type of action include:

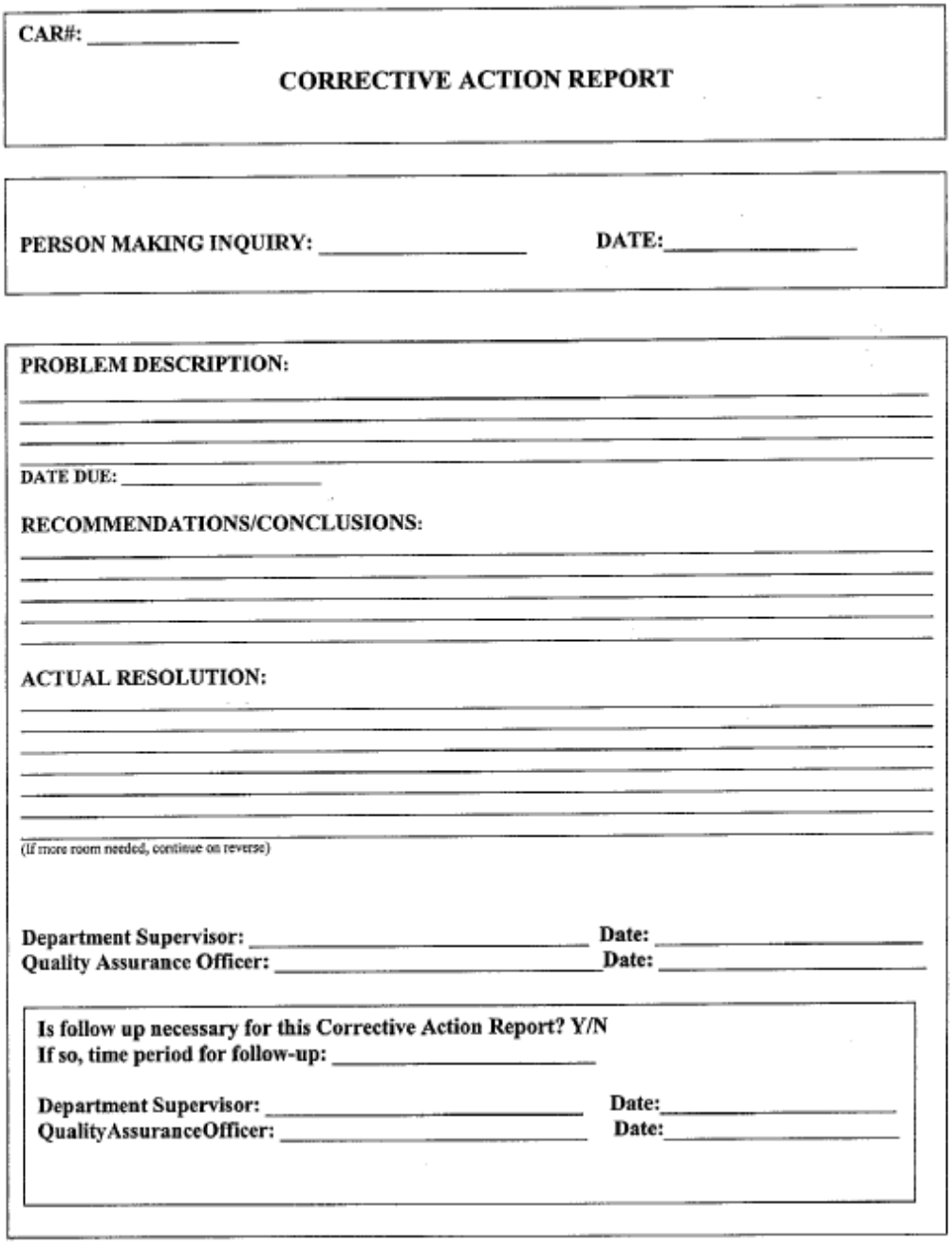
- Staff training in technical skills or in implementing the quality assurance program.
- Rescheduling of laboratory routine to ensure analyses are performed within hold times.
- Identifying vendors to supply reagents of sufficient purity.
- Revision of quality assurance system or replacement of personnel.

13.8.3 Corrective action may also be initiated by various auditing authorities when deemed necessary.

13.8.4 For either immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

- Define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action plan to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

Figure 13-1: Example of a Corrective Action Report



CAR#: _____

CORRECTIVE ACTION REPORT

PERSON MAKING INQUIRY: _____ **DATE:** _____

PROBLEM DESCRIPTION:

_____

_____

_____

_____

DATE DUE: _____

RECOMMENDATIONS/CONCLUSIONS:

_____

_____

_____

_____

_____

ACTUAL RESOLUTION:

_____

_____

_____

_____

_____

_____

(If more room needed, continue on reverse)

Department Supervisor: _____ **Date:** _____

Quality Assurance Officer: _____ **Date:** _____

Is follow up necessary for this Corrective Action Report? Y/N

If so, time period for follow-up: _____

Department Supervisor: _____ **Date:** _____

QualityAssuranceOfficer: _____ **Date:** _____

Figure 13-2: Example of a Re-Evaluation Request Form

Re-evaluation Request

Initiated By:

Date:

Client Name:

Job#:

Sample #	Parameter (s)/Batch	Original Result	Revised Result

Comments:

Action Requested/ Issue to be re-evaluated (checked)

Technical

- ❑ Dilution Error
- ❑ Check raw data vs. LIMS result
- ❑ Units Error
- ❑ Add analyte / test missed on COC
- ❑ Need Lower Dilution
- ❑ Recalculate
- ❑ Lower Reporting Limits
- ❑ Add/Delete Narrative
- ❑ Re-analysis/confirmation
- ❑ Raise Reporting Limits
- ❑ Check QC Data
- ❑ Report to MDL
- ❑ Remove analyte / test not on COC
- ❑ Revise Test Method

Technical Changes Made (Need Ops / Technical Approval Note: (*) – QA Approval Also Required)

- ❑ No change necessary
- ❑ Units Changed
- ❑ Client note was not followed at time of analysis/initial review
- ❑ Reporting Limits Lowered (*)
- ❑ Reporting Limits Elevated
- ❑ Add/ Delete Narrative
- ❑ Added Analytes requested on COC
- ❑ Added Analytes not requested on COC (*)
- ❑ Confirmation Needed
- ❑ Non-target compounds removed
- ❑ Dilution Lowered
- ❑ Flag Corrected / Added /Deleted (circle one and add comment below)
- ❑ Re-analyzed
- ❑ Dilution Error
- ❑ Results Reported at MDL (*)
- ❑ Recalculated
- ❑ Re-analyzed / amend
- ❑ QC Data Edited (requires comment)(*)

Other/Comments:

- ❑ **Report not amended**
- ❑ **Report amended and re-issued** **Date Sent:** _____
- ❑ **Client contacted** **Date:** _____ **Who:** _____
- ❑ **CAR generated** **Date/#** _____

Approval Signature _____ Date: _____
QA Approval Signature (*)_____ Date: _____

Section 14.0
Performance and System Audits

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits are of four main types: external, system, report and blind sample.

14.1 External Audits

External audits are performed when certifying agencies or clients submit samples for analysis and/or conduct on-site inspections. It is TestAmerica's policy to cooperate fully with certifying agencies. It is also TestAmerica's policy to comply fully with system audits conducted by regulatory agencies and clients.

The laboratory is involved in external performance audits conducted semi-annually through the analysis of Performance Testing (PT) samples provided by a third party. In the past these EPA performance-testing studies have been referred to as Water Pollution Study (WP) and Water Supply Study (WS). Additional PTs are analyzed as required by clients and state certifying agencies. See section 14.4 for additional information.

14.1.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend, or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in Section 3.4.5 within the 1999 NELAC standards.

14.2 System Audits

It is the responsibility of the Quality Assurance Department to plan and organize audits as required by a predetermined schedule and as requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. System audits evaluate procedures and documentation in the laboratory. Semi-annual audits are split into smaller audits that are performed within the specified

frequency (see Figure 14-1). The Internal Audit Summary should be completed in January and July. An example audit checklist can be found in Figure 14-2. Additional audits may be necessary throughout the year to address specific project requirements or issues that arise from other audits. If any issues arise which affect client results, the data would be corrected in the LIMS system and a new corrected report will be issued to the client.

14.3 Report Audits

Routine report audits are the responsibility of the laboratory Quality Assurance Officer. The Quality Assurance Officer performs an independent systems review of reports generated by the laboratory. The reviewer is not expected to pursue the correctness of every reference in the file contents, but concentrates on the internal consistency of the data package. Areas for review include chain-of-custody, correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility, and completeness of the project file contents. A list of reports reviewed is maintained in an audit file.

14.4 Blind Sample Audits

Blind sample audits are performed by submitting QC samples to the analyst with true values, which are only made known after the test is complete. Blind sample audits are carried out by the Quality Assurance Officer, Department Supervisor, Corporate Director of Quality Assurance, clients and certifying agencies as necessary to assure the laboratory is capable of achieving success with a blind QC sample. For continuing NELAC accreditation, completion dates of successive proficiency rounds for a given PT field of testing shall be six months. Failure to meet the semi-annual schedule is regarded as a failed study.

In addition to the semi-annual PTs submitted to the laboratory through third party vendors, the laboratory may also participate in a Company wide internal PT program to evaluate methods that are not commonly included in the semi-annual PT studies.

14.4.1 It is recognized that PT samples are often not representative of “real world” samples either in their form (e.g. vials), content (e.g. multiple target analyte hits), or documentation (e.g. no chain of custody) and such present the laboratory with special challenges.

14.4.2 It is the policy of TestAmerica Analytical Testing Corporation that PT samples be treated as typical samples in the normal production process where this is possible. Further, where PT samples present special or unique problems in the normal production process they need to be treated differently, as would any special or unique request submitted by any client.

14.4.3 Holding time begins when the vial is opened. Full volume PTs follow normal hold time procedures and storage requirements.

14.4.4 Login will obtain the normal COC information from the documentation provided with the PTs with review by QA or other designated staff.

14.4.5 Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.

14.4.6 PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample (e.g. If a client requests, as PT clients do, that we split VOA coeluters, than dual analysis IS normal practice).

14.4.7 No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.

14.4.8 Special QC samples can be included in the analytical run IF this is what would be done with normal client samples under similar circumstances.

14.5 Quality Systems Management Review

The senior management team (Division Manager, Department Supervisor, Quality Assurance Officer, Office Manager, and Project Management) conducts an annual review of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. Corporate Operations and Corporate Quality Assurance personnel may be included in this meeting at the discretion of the Division Manager.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (see Section 15) should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation.

14.5.1 Significant issues from the following documentation are summarized by the Quality Assurance Officer prior to the review meeting:

14.5.1.1 Matters arising from the previous annual review.

14.5.1.2 Review of report reissue requests.

14.5.1.3 Minutes from prior management and staff meetings.

14.5.1.4 Minutes from prior Senior Management team meetings, including:

a) Adequacy of staff, equipment and facility resources.
b) Future plans for resources and testing capability and capacity.

14.5.1.5 Prior Customer Service/Business Development meeting information. This include feedback from clients.

14.5.1.6 Internal and External Audits.

14.5.2 The annual review includes the previous 12 months and can occur anytime between December and February to best meet the needs of the Division. Based on the annual review, a report is generated by the Quality Assurance Officer and management. The report is distributed to Senior Management and the Corporate Director of Quality Assurance. The report includes, but is not limited to:

14.5.2.1 The date of the review and the names and titles of participants.

14.5.2.2 A reference to the existing data quality related documents and topics that were reviewed.

14.5.2.3 Quality system changes or improvements that will be made as a result of the review.

14.5.2.4 An implementation schedule including assigned responsibilities for the changes.

14.5.3 The Quality Assurance Manual may be revised at this time to reflect any significant changes made to the quality systems.

14.6 Corrective Action

All deficiencies found during audits are reported to the Division Manager. Audit information is also provided to the Director of Quality Assurance through the monthly report (see Section 15 – Quality Assurance Reports to Management). The Division Manager and Quality Assurance Officer agree upon a time frame for correction. The laboratory's response and corrective action procedures are evaluated by the Quality Assurance Officer and when acceptable, are attached to each audit and filed. If issues arise that may require method suspension or restriction, the procedures outlined in Section 13 (Corrective Action) are followed. If issues arise that affect client results, the client will be notified and a new report with correction will be issued.

External audits often require written reports that include proof of correction. The Quality Assurance Officer coordinates this written response.

Written responses to "Unacceptable" PT results are required. The response must address the reason for any unacceptable result. In some cases, it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

Figure 14-1

INTERNAL AUDITS SUMMARY

Division: _____

Time Period: _____

Number of Required Audits in 6 Month	Date(s) Performed	Description	Date Reported to Lab	Date Responses Complete
2*		Balances: - Use of Standards - Use of Log - Acceptance Criteria		
2*		Standards/Reagents: - Properly Labeled - Notebooks Used - Cert Binder Up To Date		
1		Maintenance Logs: - Preventive Maintenance - Repairs		
1		QA Manual/SOP Binders/Logbooks: - In Location - No Additional Marks - Logged		
2*		Temperature Logs/Thermometers - Logs Up To Date - Thermometers in Place - No Expired Thermometers		
1		Sample Storage/Disposal: - Internal Log Use - Disposal - Storage of Samples (VOA, Trip Blanks, …).		
1		Field Sampling: - Documentation		
1		Miscellaneous: - pH check recordings - Conductivity - Etc …..		

Number of Required Audits in 6 Month	Date(s) Performed	Description	Date Reported to Lab	Date Responses Complete
2*		Review Procedures: - Corrective Action Forms Used - Notebooks Reviewed and Signed Off - Hard Copies of All Data Available - Manual Integration Documentation Procedures Used - Manual Integration – Review on Screen		
1		Archive Procedures: - Hard Copies - System Back-ups		
2*		Training Records: - P&A in Place for all Methods Run - SOPs Signed Off		
2*		MDLs: - All instruments have a current MDL for all methods run on it. - MDL for solid and aqueous where appropriate.		
1		Log-in: - pH Checks - Documentation Procedures - Temperature Checks		
1		Control Limits / Charting		
1		Run Logs: -Proper QC Frequency		
Goal is to review all types of methods on an annual basis. Plan accordingly.		Method Audit: - Review SOP - Review Method - Review Worksheets		

* - The number of audits can decrease to 1 after two audits with no deficiencies.

QA Coordinator _____ Date _____

Attach signature page of each audit to this summary sheet. Send completed copy to Director of QA and file original.

Figure 14-2: Example Internal Audit Checklist

INTERNAL AUDIT

TestAmerica Analytical Testing Corporation – _____

Date(s):	
Area Audited	Archiving
Persons Contacted During Audit:	
Auditor	

Date Reported to Department Management	
Reported To:	
Date Reported to Division Manager:	
Reported To:	

Please review the checklist and comments attached. Comments are identified by the item number in the checklist. Please respond to all comments within one week of the “Date Reported to Department Management”.

Date: _____ QA Signature: _____

Response received and accepted by QA:

Date: _____ QA Signature: _____

Note: Attach a copy of this signature page to the Internal Audit Summary. File original with responses in audit file.

AUDIT CHECKLIST: Archiving

		Yes	No
1.	The archive log(s) include: ❑ A unique box identifier. ❑ A description of the contents of the box. ❑ The location of the box. ❑ The date of disposal.		
2.	Access to archived information is documented with an access log (either per archive area or per box). The log contains the date/time, initials and description of items removed, reviewed or returned.		
3.	Archive boxes are labeled with a unique box identifier and a means for identifying the time for disposal. This would also apply to electronic records.		
4.	Archive locations are protected against fire, theft, loss, environmental deterioration and vermin. In addition, electronic records are protected against electronic or magnetic sources.		
5.	Instructions for the retrieval of electronic records are archived with the electronic records when necessary to facilitate retrieval.		
6.	The laboratory has identified a time period to maintain data records? (_____ years).		
7.	Records that are stored by computers or personal computers (PCS) have hard copy or write-protected back-up copies. Applicable method files are backed up.		
8.	Records stored only on electronic media, are supported by the hardware and software necessary for their retrieval.		
9.	The LIMS is backed up a minimum of once per day.		
10.	A copy of the LIMS back-up is stored off-site.		

AUDIT CHECKLIST: Archiving

	Yes	No
10. The laboratory maintains all information necessary for the historical reconstruction of data. Examples: ❑ Copy of COC. ❑ Log-in Record. ❑ Internal Chain Record (where applicable). ❑ Worksheets/Logbooks/Notebooks ❑ Standard Preparation Log ❑ Calibration Logs – Balance, instrument, pipet, thermometers, …. ❑ Run Logs ❑ Raw Data ❑ Final Report ❑ QA Manual/SOPs ❑ MDLs ❑ QC Limits		

Comments:

Section 15.0
Quality Assurance Reports to Management

15.1 Internal Reports

The Quality Assurance Officer (QAO) is to submit a monthly report regarding QA/QC activities to laboratory management (Division Manager, and Department Supervisors) and the Corporate Director of Quality Assurance. An example format with the minimum required topics for reporting can be found in Figure 15-1.

15.2 External Reports

Certain projects under regulatory review require establishment of explicit quality assurance objectives and quarterly summaries of QA conformance and corrective action. The laboratory technical and quality assurance staff will provide information required to establish quality assurance objectives for particular projects. Once the QA deliverables options are selected for the project, sufficient quality control data will be provided in the individual analytical report to allow a periodic assessment of the overall progress of the project. Upon request, any information/reports needed will be provided by the laboratory management with review by the QAO.

Figure 15-1

QA MONTHLY REPORT TO MANAGEMENT

LABORATORY:
PERIOD COVERED:
PREPARED BY:
TO: x, Division Manager
CC: X, Operations Manager
Director of Quality Assurance

THREE KEY ISSUES:

1.
2.
3.

---

1. **SOPs**
   - 1.1 The following SOPs were finalized (Include updated SOP Summary with Report):
   - 1.2 The following SOPs are in QA for review:
   - 1.3 The following SOPs are due to QA:

2. **Corrective Action Reports (CARs)**
   - 2.1 Total Number of CARs:
   - 2.2 Number of Unresolved CARs:
   - 2.3 Highlights:
     - 2.3.1

3. **MDLs**
   - 3.1 MDLs Completed:
   - 3.2 MDLs Due:

4. **AUDITS**
   - 4.1 INTERNAL AUDITS
     The following internal audits were performed (include method and general):
   - 4.2 EXTERNAL AUDITS
     (Include source, date, highlights, date Corrective Action Package is due, Progress on Corrective Action Packages, …)

5. **PE SAMPLES**
   - 5.1 The following PE samples are now in house (Due Dates):
   - 5.2 The following PE results have been received (Results presented as a percentage by Department, Discuss Corrective Action):

6. **CERTIFICATIONS**
   - 6.1 Certification Packages Being Worked On (Include Due Date):
   - 6.2 Certification Packages Completed (Send any new Certificates):

7. **TRAINING**
   - 7.1 Training Record Issues:

8. **MISCELLANEOUS**
   - 8.1

9. **NEXT MONTH**
   (Items planned for next month)

Appendix 1:

TESTAMERICA ETHICS POLICY AND CODE OF ETHICAL CONDUCT

It is the policy of TestAmerica Analytical Testing Corp. that every employee shall at all times and in all ways comply with both the letter and the spirit of federal, state and local laws, and that every employee shall adhere to the highest standards of ethics, morality, honesty and decency in the performance of the duties of his or her job.

1.1 TestAmerica Code of Ethical Conduct

TestAmerica has adopted a Code of Ethical Conduct, to which each employee must adhere, as follows:

a) To serve human health and environmental interests by performing analytical and testing responsibilities in a manner that justifies the public trust.

b) To present services in a confidential, honest, and candid manner. Facility/location procedures, client names and their results are not discussed outside of the company except with an approved client agent.

c) To produce results that are both accurate and defensible.

d) To comply with all written procedures (i.e., Quality Assurance (QA) Manual, Standard Operating Procedures (SOPs), Safety Manual, Human Resources Manual, etc.). Members of management must comply with all applicable federal, state, and local laws and regulations consistent with accepted professional and analytical practices.

e) To understand and adhere to the guidelines of ethical and quality work that meet the standards required by the environmental testing industry.

1.2 Data Quality Assurance Program

TestAmerica wants to ensure a national standard of quality at all TestAmerica locations.

Each TestAmerica laboratory has a Quality Assurance Manual that focuses on quality related test specifications performed by that laboratory. Documented quality systems are designed to insure that work performed in the laboratory is accurate, precise, complete, comprehensive, and reflects the need of the customer/client.

1.3 Ethics Quality Commitment, Objective, and Policy

TestAmerica wants to ensure quality analytical and data management services to meet the needs of customers/clients while satisfying the requirements of appropriate state and federal regulations. This enables the customer/client to make rational, confident, cost-effective decisions on the assessment and resolution of environmental problems. Protocols and procedures utilized by laboratories, with emphasis on the Quality Assurance/Quality Control (QA/QC) requirements, are based on EPA guidelines.

It is the policy of TestAmerica to incorporate quality into all analytical programs by adhering to the following practices:

a) TestAmerica will not offer any analysis for which we cannot demonstrate consistent quality and defensible analyses;

b) Employees who are aware of falsification or misrepresentation of facts regarding analytical results or the manipulation of data are required to immediately inform the appropriate member of Management;

c) TestAmerica has “Open Door” and “Open Line” Policies which enable every TestAmerica employee to have free access to the respective Manager and Corporate Officers. Such Open Door Policies are intended to foster two-way communications and provide each employee with access to Laboratory and Corporate Management. Such Policies are also intended to encourage each employee to consider it his or her duty and responsibility to “come forward”. Any employee who disagrees with or has a concern or question about any Company practice, process, procedure, or policy, or about any Supervisory/Managerial request, instruction, or directive should come forward. This includes concerns about any undue pressures placed upon an employee which adversely affects the quality of work produced. Such contact should be made to members of Laboratory or Corporate Management. Any contacts with a Manager or representative of Corporate shall be treated as “confidential”, if the employee so requests.

d) No employee of TestAmerica will compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.

1.4 TestAmerica Code of Ethical Conduct Agreement

I. I understand that I am charged with meeting ethical standards in performing all of my duties and responsibilities;

II. I have been formally instructed to consider quality as an important aspect of my job responsibilities. The provisions of the “Ethics Policy and Code of Ethical Conduct” have also been reviewed with me.

III. I also agree to the following:

a) I shall not report data inconsistent with actual values observed or measured.

~~c)~~ I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.

d) I shall not intentionally report dates and times of analyses that do not represent the true and actual dates and times the analyses were conducted.

e) I shall not intentionally represent another individual's work as my own or represent my work as someone else's.

e) I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.

f) I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.

g) I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Officer. The Quality Assurance Officer will initial and date the information and return a copy to me.

I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use.

I understand that if any supervisor, manager, or representative of management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Manager, all supervisors and managers with direct line reporting relationship between me and the Manager, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, Corporate Quality Assurance may be used as a resource.

I should obtain a ruling, in writing, as to whether such practice is or is not improper and will abide by such ruling. However, if I have not received a timely ruling, or if I believe such ruling is incorrect, I may appeal to the Division Manager or President/CEO and will abide by such written ruling.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

I have read and fully understand all provisions of the "Ethics Policy and Code of Ethical Conduct" and realize that even one instance of variance from the above Code of Ethical Conduct may result in discipline, up to and including termination of employment. I have also viewed the Ethics Presentation.

_____ _____

(Dated) (Employee's Signature)

_____

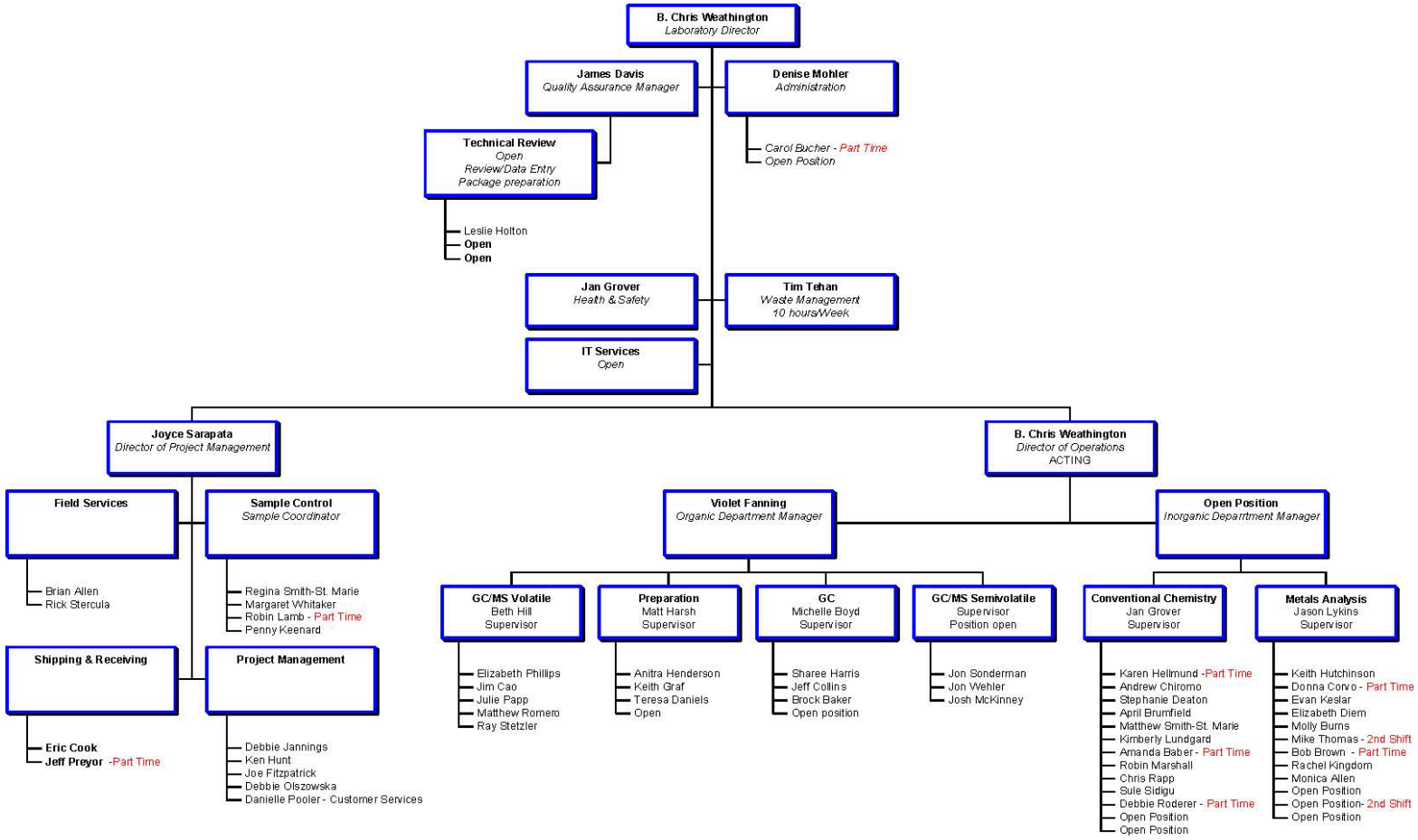
(Print Name)

_____

(If applicable, Employee ID Number)

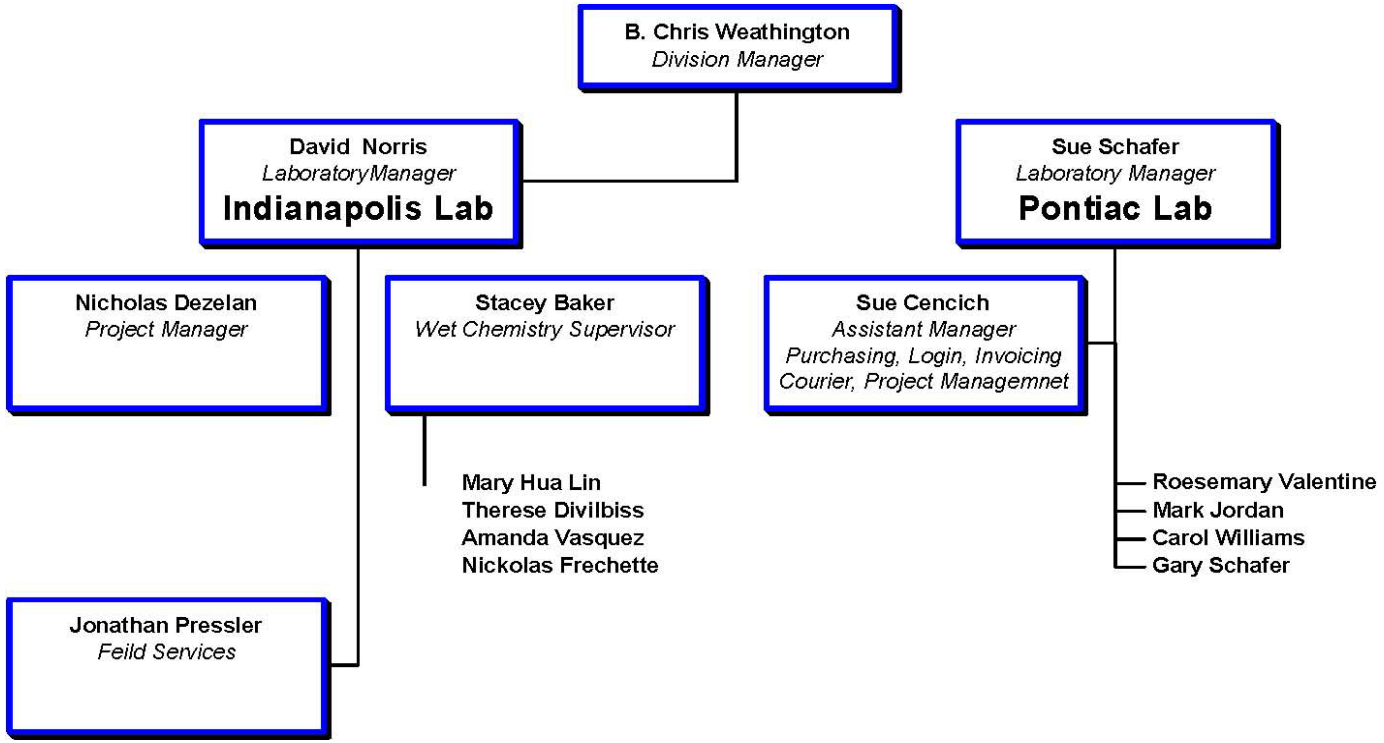
NOTE: This Ethics Policy and Code of Ethical Conduct must be signed at the time of hire (or within 2 weeks of an employee's initial receipt of this Policy, if later) and re-signed between January 1 and January 30 of every year. Such signature is a condition of continued employment. Failure to sign will result in immediate termination of employment.

2/03



Appendix 2-TestAmerica-Dayton Division

Organization- updated 12/15/04



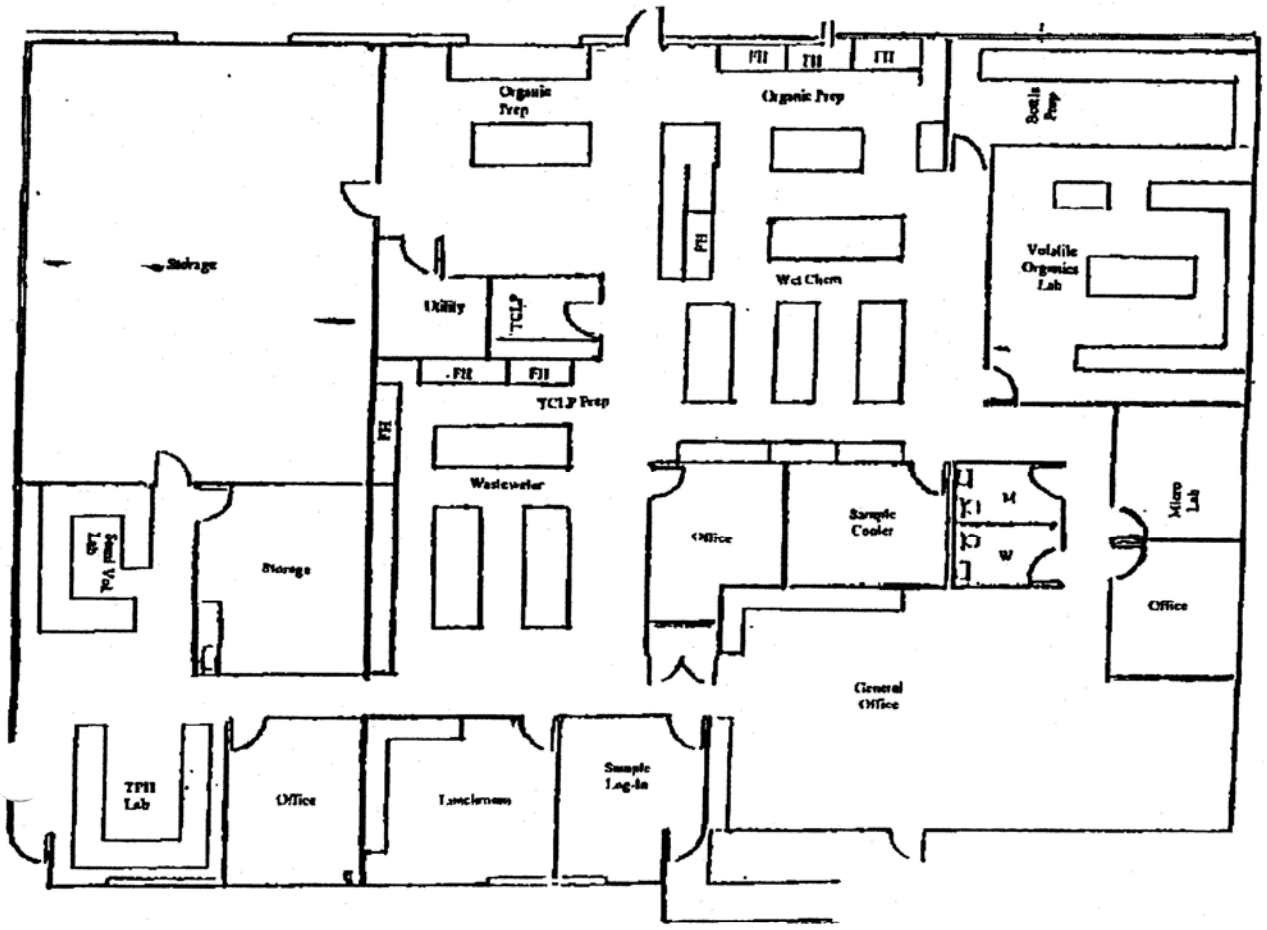
Appendix 2-TestAmerica-Indianapolis and Pontiac Divisions

Organization- updated 12/15/04

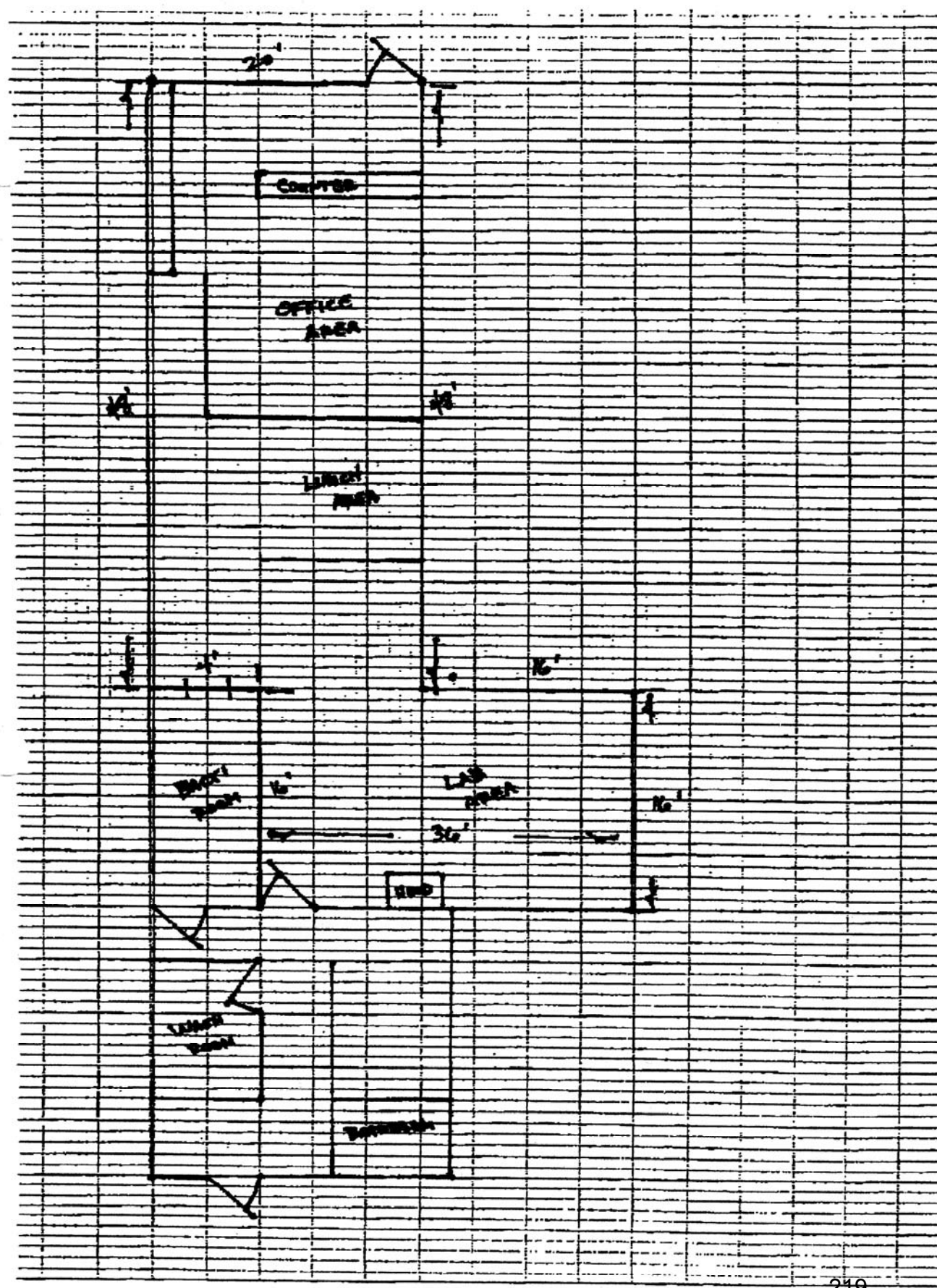
Appendix 3: Laboratory Floor Plans

Main Laboratory

Annex Laboratory



TestAmerica – Indianapolis Laboratory



Testamerica – Pontiac Laboratory

TOTAL

Appendix 4: Summary of Calibration and QC Procedures
GC Organics

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 8081A SW 8082	Five-point initial calibration[3]	Initial calibration prior to sample analysis	RF = 20% CF = 20% Linear – least squares regression r ≥ 0.99	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per five-point initial calibration	± 30% of expected value, advisory.	Correct problem then repeat initial calibration if necessary. Repeat ICV
	Retention time window calculated for each analyte	System set-up	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then re-analyze all samples analyzed since retention time check
	Continuing calibration verification	Before sample analysis, every 12 hours, and at the end of the analysis sequence	All analytes average within 15% of expected value	Correct problem then repeat initial Continuing calibration verification and re-analyze all samples since last successful Continuing calibration verification[4]
	Breakdown check (Endrin and DDT) [1]	Daily prior to analysis of samples	Degradation ≤15%	Inlet/column maintenance; repeat breakdown check
	Method blank	One per analytical prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples with detects processed with the contaminated blank
	LCS for all analytes	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS
	Surrogate spike	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Check system, re-inject, re-extract
	MS/MSD	One per prep batch, per matrix	See Control Limits Manual	None
	Second-column confirmation[2]	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis

1 --8081A only
2 – excludes Chlordane and Toxaphene for SW8081A.
3 – For SW 8081A, Chlordane and Toxaphene employ a single point calibration for identification purposes; A five point calibration is required if a pattern is detected. SOPs
4 – If the ending CCV fails high and there is no detects in the samples, they do not need repeated

Appendix 4: Summary of Calibration and QC Procedures
GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 8260A SW 8260B SW 8270C	Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration and Continuing calibration verification, every 12 hours	Refer to criteria listed in the method description	Retune instrument and verify
	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 (8270) [3] and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration
			Option 1 (average response factor) mean RSD for all analytes ≤15% with no individual ccc RSD >30%	
			Option 2 linear – least squares regression r ≥ 0.99	
	Second-source calibration verification (ICV)	Once per five-point initial calibration	± 30% of expected value, advisory.	Correct problem then repeat initial calibration if necessary. Repeat ICV
	Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT	Correct problem then re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs - Same as initial calibration CCCs ≤ 20% difference (when using RFs) or drift (when using least squares regression. All ccc within 20% of expected value 20% NOT REQUIRED FOR EVERYTHING	Correct problem then repeat
	Internal Standards	Every sample/standard	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning

1 – SW8260 requires BFB; SW8270 requires DFTPP
2 Post runs are not required for CG MS
3 – RF ≥ 0.30 for chlorobenzene and 1,1,2,2-Tetrachloroethane, RF ≥ 0.10 for chloromethane and 1,1-Dichloroethane, RF > 0.10 for Bromoform

Appendix 4: Summary of Calibration and QC Procedures
GC/MS Organics (Continued)

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 8260A SW 8260B SW 8270C	Method blank	One per analytical prep batch (one per run batch for SW 8260).	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples with detects processed with the contaminated blank
	LCS for all analytes	One per prep batch (one per run batch for SW 8260).	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS.
	MS/MSD	One per prep batch per matrix	See Control Limits Manual	None
	Surrogate spike	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Check system, re-inject, re-extract

Appendix 4: Summary of Calibration and QC Procedures
Method SW 6010B

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 6010B	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value.	Repeat calibration and re-analyze all samples since last successful calibration
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No analytes detected ≥ RL	Repeat calibration and re-analyze all samples since last successful CCB
	Spectral Interference Check solution (SICs)	At the beginning of an analytical run	No detects ≥ RL or ≤ - RL for non-spiked metals.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 75-125% of expected results	Correct problem then re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Section 5	None

Appendix 4: Summary of Calibration and QC Procedures
Method SW 6020

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 6020	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value.	Repeat calibration and re-analyze all samples since last successful CCV
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No analytes detected ≥ RL	Repeat calibration and re-analyze all samples since last successful CCB
	Interference check solution (ICS)	At the beginning of an analytical run	Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 85-115% of expected results	Correct problem then re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits Manual	None
	Internal Standards	Every sample and standard		Dilute and repeat analysis

Appendix 4: Summary of Calibration and QC Procedures
GFAA Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 7041 SW 7060A SW 7131A SW 7191 SW 7201 SW 7421 SW 7740 SW 7761 SW 7841	Initial calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per initial daily calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ RL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value	Correct problem then repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution Test	Each preparatory batch	Five times dilution sample result must be within 10% of the undiluted sample result	Perform post digestion spike addition
	Recovery test	When dilution test fails	Recovery within 15% of expected results	Dilute the sample; re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
Method SW 7196A

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 7196A	Initial calibration (minimum five standards)	Initial calibration yearly.	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	After each new stock standard preparation	Within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification	Beginning and after every 10 samples and at the end of the analysis sequence	Within 10% of expected value	Correct problem then if necessary, repeat initial calibration and re-analyze all samples since last successful calibration
	Continuing calibration blank	Beginning and after every 10 samples and at the end of the analysis sequence	Less than reporting limit.	Correct problem then if necessary, repeat initial calibration and re-analyze all samples since last successful calibration
	Method blank (Soil samples)	One per prep batch	Less than reporting limit.	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS (Soil samples)	One per batch	See Control Limits Manual	Re-prep, re-analyze all affected samples.
	MS/MSD	One per 20 samples per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
Method SW 7470A/SW 7471A

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 7470A SW 7471A	Initial calibration (minimum 5 standards and a blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Second-source calibration verification (ICV)	Once per initial calibration	Within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No detects ≥ RL	Correct problem then repeat calibration and re-analyze all samples since last successful CCB
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Within 20% of expected value	Correct problem then repeat calibration and re-analyze all samples since last successful CCV
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
Method SW 9012A

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 9012A	Initial calibration (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per calibration	Within 10% of true value	Correct problem then repeat distilled standards
	Second-source calibration verification (ICV)	Once per initial daily calibration	Within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification	Beginning and after every 10 samples and at the end of the analysis sequence	Within 10% of expected value	Correct problem then repeat calibration ICV and re-analyze all samples since last successful CCV
	Continuing calibration blank	Beginning and after every 10 samples and at the end of the analysis sequence	No detects ≥ RL	Correct problem then repeat calibration ICV and re-analyze all samples since last successful CCB
	Method blank	One per prep batch	No detects ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per batch per matrix	See Control Limits Manual	Re-prep, re-run affected samples
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
GC Organics

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 552.2	Five-point initial calibration	Initial calibration prior to sample analysis	RF = 20% CF = 20% Linear – least squares regression r ≥ 0.99	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per five-point initial calibration	± 30% of expected value, advisory.	Correct problem then repeat initial calibration if necessary. Repeat ICV
	Retention time window calculated for each analyte	System set-up	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then re-analyze all samples analyzed since retention time check
	Continuing calibration verification	Before sample analysis, every 10 sample injections, and at the end of the analysis sequence	All analytes average within 30% of expected value	Correct problem then repeat initial Continuing calibration verification and re-analyze all samples since last successful Continuing calibration verification[1]
	Internal Standards	Injected with every sample	Should not deviate > 30 % from mean	Repeat, dilute if necessary
	Breakdown check (Endrin and DDT)[1]	Daily prior to analysis of samples	Degradation ≤15%	Inlet/column maintenance; repeat breakdown check
	Method blank	One per analytical prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples with detects processed with the contaminated blank
	LPC (Laboratory Performance Check)for all analytes	Prior Calibration Standards or sample analysis set.	See Analytical SOP for Detail	Correct Problem and Re-inject
	Surrogate spike	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Check system, re-inject, re-extract
	MS/MSD	One per prep batch, or 10 % of samples.	See Control Limits Manual	None
	Second-column confirmation[2]	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis

1 – If the ending CCV fails high and there is no detects in the samples, they do not need repeated

Appendix 4: Summary of Calibration and QC Procedures
GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 624 EPA 625	Check of mass spectral ion intensities[1]	Prior to initial calibration and Continuing calibration verification	Refer to criteria listed in the method description	Retune instrument and verify
	Three-point initial calibration for all analytes (minimum)	Initial calibration prior to sample analysis	%RSD < 35% or linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per initial calibration	± 30% of expected value, advisory.	Correct problem then repeat initial calibration if necessary. Repeat ICV
	Retention time window calculated for each analyte	Each sample	Retention time (RT) of the analyte within 30 seconds of the RT	Correct problem then re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification	Daily, before sample analysis and every 12 hours	All calibration analytes within 20% of expected value (EPA 625). Please see the SOP for EPA 624.	Correct problem then repeat initial calibration if necessary
	Method blank	One per prep batch for 625/run batch for 624	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch for 625/run batch for 624	See Control Limits Manual	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per prep batch per matrix	See Control Limits Manual	None
	Surrogate spike	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Recalibrate and confirm.

1 – 624 requires BFB; 625 requires DFTPP

Appendix 4: Summary of Calibration and QC Procedures
GFAA Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 204.2 EPA 206.2 EPA 213.2 EPA 218.2 EPA 219.2 EPA 239.2 EPA 270.2 EPA 272.2 EPA 279.2	Initial calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per initial daily calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ RL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value	Correct problem then repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution Test	Each preparatory batch	Five times dilution sample result must be within 10% of the undiluted sample result	Perform post digestion spike addition
	Recovery test	When dilution test fails	Recovery within 15% of expected results	Dilute the sample; re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
Mercury

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 245.1 EPA 245.5	Initial calibration (minimum 5 standards and a blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Once per initial calibration	Within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No detects ≥ RL	Correct problem then repeat calibration and re-analyze all samples since last successful CCB
	MS/MSD	One per batch per matrix	See Control Limits Manual	None
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analyte within 10% of expected value (except for potable samples where analyte within 5%) before sample analysis; within 10% after sample analysis has begun	Correct problem then repeat calibration and re-analyze all samples since last successful calibration
	LCS	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Matrix Spike	One per every 10 samples	All analytes within 30% of expected value	None
	Duplicate	One per every 10 samples		None

Appendix 4: Summary of Calibration and QC Procedures
ICP Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 200.7	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A
	5% Standard Verification (same source as the calibration standards)	Daily after initial calibration	All analytes within 5% of expected value	Correct problem, repeat initial calibration
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value.	Repeat calibration and re-analyze all samples since last successful calibration
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No analytes detected ≥ RL	Repeat calibration and re-analyze all samples since last successful CCB
	Spectral Interference Check solution (SICs)	At the beginning of an analytical run	No detects ≥ RL or ≤ - RL for non-spiked metals.	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 75-125% of expected results	Correct problem then re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures
ICP-MS

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 200.8	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value.	Repeat calibration and re-analyze all samples since last successful CCV
	Continuing calibration blank	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	No analytes detected ≥ RL	Repeat calibration and re-analyze all samples since last successful CCB
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze LCS and all samples processed with the LCS
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition MS/MSD	When dilution test fails One per batch per matrix	Recovery within 85-115% of expected results See Control Limits Manual	Correct problem then re-analyze post digestion spike addition None
	Internal Standards	Every sample		Dilute and repeat analysis

3
Appendix 4: Summary of Calibration and QC Procedures
GFAA Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 200.9	Initial calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration
	5% Standard Verification (same source as the calibration standards)	Daily after initial calibration	All analytes within 5% of expected value	Correct problem, repeat initial calibration
	Second-source calibration verification (ICV)	Once per initial daily calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ RL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value	Correct problem then repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch	See Control Limits Manual	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution Test	Each preparatory batch	Five times dilution sample result must be within 10% of the undiluted sample result	Perform post digestion spike addition
	Recovery test	When dilution test fails	Recovery within 15% of expected results	Dilute the sample; re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits Manual	None

Appendix 4: Summary of Calibration and QC Procedures for Gravimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 160.1 (TDS) EPA 160.2 (TSS) EPA 160.3 (TS) EPA 160.4 (TVS) EPA 160.5 (Settleable) [1] SM 2540 B (TS) SM 2540 C (TDS) [2] SM 2540 D (TSS) SM 2540 E (TVS) SM 2540 F (Settleable) SM 2540 G (Fixed Solids) SM 2710 F (Density) [1]	Verification standard–single standard (if available)	Each batch	See Control Limits Manual	Repeat if sufficient sample volume exists, note on report otherwise.
	Method blank	Each batch	Less than report limit	Repeat if sufficient sample volume exists, note on report otherwise.
	Duplicate	Every 10 samples	± 20% RPD	Repeat if sufficient sample volume exists, note on report otherwise.

[1]: Density and Settleable Solids do not have a method blank analyzed.
[2]: Potable TDS has an RPD of 5%.

Appendix 4: Summary of Calibration and QC Procedures for Titrimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 310.1 Alkalinity SM 2320B Alkalinity EPA 325.3 Chloride SM 4500 Cl B Chloride SM 4500 Cl C Chloride SW 9253 Chloride EPA 130.2 Hardness SM 2340C Hardness EPA 376.1 Sulfide SM 4500 S^{-2} E Sulfide EPA 377.1 Sulfite[1] SM 2330 Calc. Carb. Stability	Verification standard–single standard (if available)	Each batch	± 10%	Repeat, check
	Method blank	Each batch	Less than report limit	Repeat batch
	Duplicate	Every 10 samples	± 20% (Potable analyses must be within ± 10%)	None
	Matrix Spike/Matrix Spike Duplicate	One every batch of 20 samples or less	± 20% (Potable analyses must be within ± 10%)	Use LCS to assess acceptability;

[1] Sulfite has a duplicate run every 10 samples only and no MS/MSD pair is analyzed. All other methods listed analyze the matrix spike/matrix spike duplicate.

Appendix 4: Summary of Calibration and QC Procedures for Spectrophotometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 350.1: NH_3. SM 4500 NH_{3-H} EPA 410.4: COD. Hach 8000: COD SM 4500Cl G: Cl_2 Res. SM 3500 Cr-D: $Cr^{+6.}$ SW 7196A:$Cr^{+6.}$ EPA 335.2 MOD: CN^-. EPA 335.4: CN^-. EPA 335.2 CLP-M: CN^-. SW 9012A: CN^- SM 4500 CN^-C: CN SM 4500 CN^-G: CN Amen. EPA 335.1: CN^-Amen. EPA 335.2 MOD: CN^-.Amen. EPA 425.1: MBAS SM 5540C: MBAS EPA 353.2: NO_3, NO_2. SM 4500 NO_3^- F: NO_3, NO_2 EPA 351.2: TKN. SM 4500 NH_3-H: TKN EPA 365.2: Phos. SM 4500 P-E: Phos. EPA 365.2: O-Phos. SM 4500 P-E: O-Phos. EPA 420.1: Phenolics. SW 9065: Phenolics. SWSec8.3:Rx. CN^-. EPA 375.4: SO_4^{-2}. SM 4500 SO_4^{-2}-D: SO_4^{-2} SW 9038: SO_4^{-2}. SM 5310B: TOC.[1]	Calibration curve – minimum: See individual SOP	Per analytical run (TOC done monthly or if change to instrumentation or reagents)	Correlation Coefficient $\geq$ 0.995	Recalibrate
	Independent calibration verification – mid-level, second-source required (ICV)	Initial calibration	± 10%	Recalibrate
	Continuing calibration verification	Each use	± 10%	Correct, recalibrate
	Method blank	Each use	Less than report limit	Reprep entire batch
	LCS	Each batch, less than 20	See Control Limits Manual	Reprep entire batch
	MS/MSD	Each batch, less than 20	RSD < ± 20%	Use LCS to assess acceptance

Potable Nitrate MS/MSD must be analyzed 1 per 10 samples or less.

Appendix 4: Summary of Calibration and QC Procedures for Electrometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 405.1: BOD[1] SM 5210B: BOD[1] SM 5210B: CBOD[1]	Calibration Curve – See individual SOPs for criteria	Initial Calibration	Correlation Coefficient ≥ 0.995	Recalibrate
EPA 120.1: Cond. SW 9050A: Cond. SM 2510: Cond.	Independent calibration verification (second source) (ICV)	Immediately after initial calibration	±10%	Recalibrate
EPA 160.1: DO. SM 4500 O-G: DO	Continuing calibration verification	Beginning, every 10 samples, and end of batch	±10%	Rerun
EPA 340.1: F^-. EPA 340.2: F^-. SM 4500 F^-. -B: F^-. SM 4500 F^-. -C: F^-. EPA 150.1: pH. SW 9040B: pH. SW 9041A: pH. SM 4500 H^+: pH. EPA 180.1: Turbidity. SM 2130B: Turbidity.	Method blank	Each batch	Less than or equal to the report limit	Reprep
	LCS	Each batch	See Control Limits Manual	Rerun batch
	MS/MSD	Each batch	± 20%	None
	Duplicate	When spike not available	± 20%	None

[1]Calibration curve does not apply.

Appendix 4: Summary of Calibration and QC Procedures for Oil & Grease Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA 1664A.	Method blank	Each batch of 20 samples or less	Less than report limit	Repeat batch
	LCS	Each batch of 20 samples or less	See Control Limits Manual	Repeat batch
	MS/MSD	Each batch of 20 samples or less	± 20%	None
	LCS/LCS duplicate[1]	Each batch of 20 samples or less	See Control Limits Manual	Repeat batch

[1] The LCS/LCS duplicate is analyzed to calculate the precision of the analysis only when there is insufficient sample for a MS/MSD pair to be run

Appendix 4: Summary of Calibration and QC Procedures for Physical Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW 1010: Flash Point. SM 2120B: Color SM 2150B: Odor. SW 9095A: Paint Filter. EPA 170.1: Temperature	Method blank	Each batch	Less than report limit	Repeat, rerun
	Single standard[1]	Each batch	+/- 10%	Rerun batch
	Duplicate	Every 10 samples per batch	+/- 20%	None

[1] Flash Point only.

Appendix 5: Glossary/Acronyms

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analytical Detection Limit:
The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)

Assessor Body:
The organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, performs on-site assessments, etc., whether EPA, the State, or contracted private party. (NELAC)

Accrediting Authority Review Board (AARD):
Five representatives from the Territories, States, EPA, and/or other Federal Agencies, appointed by the NELAP Director, in consultation with the NELAC Board of Directors, for the purpose of reviewing the processes and procedures used by EPA to approve accrediting authorities in accordance with NELAC standards. (NELAC) [1.6.3]

Analyst:
The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Assessment Criteria:
The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit:
A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch:
See Section 11. (NELAC Quality Systems Committee)

Blank:
See Section 11. (ASQC)

Blind Sample:
See Section 11. (NELAC)

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 should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
See Section 11. Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures

(NELAC)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Contributor:
A participant in NELAC who is not a Voting Member. Contributors include representatives of laboratories, manufacturers, industry, business, consumers, academia, laboratory associations,

laboratory accreditation associations, counties, municipalities, and other political subdivisions, other federal officials not engaged in environmental activities, and other persons who are interested in the objectives and activities of NELAC> (NELAC) [Art III, Const]

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
See Section 11. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:
See Section 11. (EPA-QAD)

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media, which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):
The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Interdependent Analytes:
Analytes analyzed using methods in which the ability to correctly identify and quantitate a series of analytes is indicative of the laboratory's ability to correctly determine the presence or absence of similar analytes. (NELAC) [2.C5.1]

Internal Standard:
See Section 11. (NELAC)

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
See Section 11. (NELAC)

Laboratory Duplicate:
See Section 11. (NELAC)

Limit of Detection (LOD):
See Section 11. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Manager (however named):
The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: 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.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with, 15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):
See Section 11. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
See Section 11. (QAMS)

Method Blank:
See Section 11. (NELAC)

Method Detection Limit:
See Section 11. (40 CFR Part 136, Appendix B)

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

Non-interdependent Analytes:
Analytes that are analyzed using methods in which the ability to correctly identify and quantitate a series of analytes in a sample is not indicative of the laboratory's ability to correctly identify and quantitate similar analytes. (NELAC) [2.C.5.2]

Objective Evidence:
Any documented statement of fact, other information, or records, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified. (ASQC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes 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. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

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. (NELAC)

Primary Accrediting Authority:
The agency or department designated at the Territory, State, or Federal level as the recognized authority with responsibility and accountability for granting NELAC accreditation for a specified field of testing. (NELAC) [1.5.2.3]

PT Fields of Testing:
NELAC's approach to offering proficiency testing by regulatory or environmental program, matrix type, and analyte. (NELAC)

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. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol:
A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed. (EPA-QAD)

Pure Reagent Water:
Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method. (NELAC)

Quality Assurance:
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. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities that purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. 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. (EPA-QAD)

Quality 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. (NELAC)

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 (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
See Section 11. (EPA-QAD)

Reagent Blank (method reagent blank):
See Section 11. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)

Resume:
The summary (usually written) of an individual’s relevant technical and management experience, including training. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Secondary Accrediting Authority:
The Territorial, State, or Federal Agency that grants NELAC accreditation to laboratories, based upon their accreditation by a NELAP-recognized Primary Accrediting Authority. See also Reciprocity and Primary Accrediting Authority. (NELAC) [1.5.2.3]

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
See Section 11. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
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. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate:
See Section 11. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Testing Laboratory:
A laboratory that performs tests. (ISO/IEC Guide 2-12.4)

Test Sensitivity/Power:
The minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, Section 2.4.a). (NELAC)

Toxic Substances Control Act (TSCA):
The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)
NOTE:
In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell:
A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Acronyms:

CAR – Corrective Action Report
CCV – Calibration Verification
CF – Calibration Factor
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
GFAA – Graphite Furnace Atomic Absorption
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICPMS - Inductively Coupled Plasma Mass Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample or Laboratory Control Standard
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS: Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
NIST – National Institute of Standards and Technology
PT – Performance Testing
QAM – Quality Assurance Manual
QAO – Quality Assurance Officer
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

STANDARD OPERATING PROCEDURE

TestAmerica Incorporated
Dayton Division

Title: **Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry**

SOP No.: **DT05-57**
Revision: **0**
Effective: **January 25, 2002** **Page 1 of 9**

Computer File Name: SOPs/Wet Chemistry/DT05-57.0 Available Cyanide

Quality Assurance Approval Date	Division Approval Date

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe, nonabsorbent shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

Method Reference: Method OIA-1677, United States Environmental Protection Agency, Office of Water, EPA-821-R-99-013, August 1999.

Modifications:

Item	Method	Modification
7.8.3	OIA-1677	KCN solution is purchased
7.9	OIA-1677	Only 10 mg/L secondary standard is required to prepare Calibration Standards
7.12.2	OIA-1677	LCS concentration is 0.050 mg/L so that it is within Calibration Range
9.3.4	OIA-1677	If the MS/MSD is outside of acceptance limits, the data is reported with a flag indicating matrix interference
10.2.1	OIA-1677	Instrument is stabilized with a 5 mg/L solution. 10 mg/L is beyond the capacity of the detector.
12.2.2	OIA-1677	Blanks are reported as less than the reporting limit (ML)

1. SCOPE AND APPLICATION

1.1 This method is for determination of available cyanide in water and wastewater by flow injection, ligand exchange, and amperometric detection. The method is for use in EPA's data gathering and monitoring programs associated with the Clean Water Act, Resource Conservation and Recovery Act, Comprehensive Environmental Response, Compensation and Liability Act, and Safe Drinking Water Act.

1.2 Cyanide ion (CN^-), hydrogen cyanide in water (HCN_{aq}) and the cyano-complexes of zinc, copper, cadmium, mercury, nickel, and silver may be determined by this method.

1.3 The presence of polysulfides may prove intractable for application of this method.

1.4 The method detection limit (MDL) is 0.5µg/L and the minimum level (ML) is 2.0µg/L. The dynamic range is approximately 2.0µg/L (ppb) to 5.0mg/L (ppm) cyanide ion using a 200µL sample loop volume. Higher concentrations can be determined by dilution of the original sample or by reducing volume of the sample loop.

1.5 The current reporting limit for available cyanides is 5.0 ug/L.

2. SUMMARY OF METHOD

2.1 The analytical procedure employed for determination of available cyanide is divided into two parts: sample pretreatment and cyanide detection. In the pretreatment step, ligand exchange reagents are added at room temperature to 100 mL of a cyanide-containing sample. The ligand-exchange reagents form thermodynamically stable complexes with the transition metal ions listed in section 1.2.

2.2 Cyanide detection is accomplished using a flow-injection analysis (FIA) system. A 200-µL aliquot of the pre-treated sample is injected into the flow injection manifold of the system. The addition of hydrochloric acid converts cyanide ion to hydrogen cyanide (HCN) that passes under a gas diffusion membrane. The HCN diffuses through the membrane into an alkaline receiving solution where it is converted back to cyanide ion. The cyanide ion is monitored amperometrically with a silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter electrode, at an applied potential of zero volt. The current generated is proportional to the cyanide concentration present in the original sample.

3. INTERFERENCES/COMMENTS/DEFINTIONS

3.1 Solvents, reagents, glassware, and other sample-processing hardware may yield artifacts that affect results. Specific selection of reagents or purification of these reagents may be required.

3.2 All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running a method blank.

3.3 Glassware is cleaned by washing in hot water containing detergent, rinsing with tap water and deionized water, and drying in an area free from interferences.

3.4 Interferences extracted from samples will vary considerably from source to source, depending upon the diversity of the site being sampled.

3.5 Sulfide is a positive interferent in this method, because an acidified sample containing sulfide liberates hydrogen sulfide that is passed through the membrane and produces a signal at the silver electrode. In addition, sulfide ion reacts with cyanide ion in solution to reduce its concentration over time. To overcome this interference, the sulfide ion must be precipitated with lead ion immediately upon sample collection. Sulfide ion and lead sulfate react with cyanide ion to form thiocyanate that is not detected in the analytical system. Tests have shown that if lead carbonate is used for sulfide precipitation, the supernate containing cyanide must be filtered immediately to avoid loss of cyanide through reaction with precipitated lead sulfide.

3.6 Though not interferences, substances that react with cyanide should also be removed from samples at time of collection. These substances include water-soluble aldehydes that form cyanohydrins and oxidants such as hypochlorite and sulfite. Water-soluble aldehydes react with cyanide to form cyanohydrins that are not detected by the analytical system; hypochlorite and sulfite oxidize cyanide to on-volatile forms. Procedures for the removal of these substances are provided in Section 6.

3.7 Available Cyanide consists of cyanide ion (CN^-), hydrogen cyanide in water (HCN_{aq}) and the cyano-complexes of zinc, copper, cadmium, mercury, nickel, and silver.

3.8 Minimum level (ML)—The level at which the entire analytical system shall give a recognizable signal and acceptable calibration point, taking into account method specific sample and injection volumes.

4. EQUIPMENT AND SUPPLIES

4.1 The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

4.2 Flow injection analysis (FIA) – ALPKEM Model 3000 consisting of the following:

4.2.1 Injection valve capable of injecting 40 to 300 µL samples

4.2.2 Gas diffusion manifold with a microporous Teflon® or polypropylene membrane

4.2.3 Amperometric detection system with:

4.2.3.1 Silver working electrode

4.2.3.2 Ag/AgCl reference electrode

4.2.3.3 Pt/stainless steel counter electrode

4.2.3.4 Applied potential of 0.0 volt

4.3 Miscellaneous laboratory glassware.

5. REAGENTS AND STANDARDS

5.1 The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

5.2 Deionized Water.

5.3 Sodium Hydroxide, ACS reagent grade.

5.4 Potassium cyanide, ACS reagent grade.

5.5 Mercury (II) cyanide, ≥ 99% purity.

5.6 Potassium nickel (II) cyanide.

5.7 Silver nitrate, ACS reagent grade.

5.8 Hydrochloric acid – approximately 37%, ACS reagent grade.

5.9 Silver Nitrate solution (0.0192 N): Weigh 3.27 g of $AgNO_3$ into a 1-L volumetric flask and bring to the mark with deionized water.

5.10 Rhodanine solution, 0.2 mg/mL in acetone: Weigh 20 mg of p-dimethylaminobenzalrhodanine in a 100-mL volumetric flask and dilute to the mark with acetone.

5.11 Potassium cyanide stock solution, 1000 mg/L

5.11.1 Dissolve approximately 2 g (approximately 20 pellets) of sodium hydroxide in approximately 500 mL of reagent water contained in a one-liter volumetric flask. Add 2.51 g of potassium cyanide, dilute to one liter with reagent water and mix well. Store KCN solution in an amber glass container at 0-4^{o} C. The stock can also be purchased pre-made.

5.11.2 Standardize the KCN solution monthly by adding 0.5 mL of rhodanine solution and titrating with $AgNO_3$ solution until the color changes from canary yellow to a salmon hue. Based on the determined KCN concentration, dilute the KCN solution to an appropriate volume so the final concentration is 1.00 g/L.

5.12 1M Sodium Hydroxide: Dissolve 40g of sodium hydroxide pellets in approximately 500 mL of reagent water in a 1-liter volumetric flask and let cool to room temperature. Dilute to one liter with reagent water. Store in an amber bottle at room temperature.

5.13 Secondary Standards

5.13.1 Cyanide, 10 mg/L: Dilute 1.0 mL of cyanide stock solution and 1 mL of 1 M sodium hydroxide solution to 100 mL with reagent water. Store in an amber glass bottle at 0-4^{o} C.

5.13.2 Cyanide working calibration standard solutions for CCVs (2 – 100 µg/L as cyanide): Working calibration standards may be prepared by adding the appropriate volumes of secondary standards

(See Table 14.1) to 100 mL volumetric flasks that contain 40 mL of reagent water and 1 mL of 1 M sodium hydroxide. Dilute the solutions to 100 mL with reagent water.

5.14 Sample Preservation Reagents

5.14.1 The presence of sulfide may result in the conversion of cyanide to thiocyanate. While lead acetate test paper has been recommended for determining the presence of sulfide in samples, the test is generally unreliable and is typically not usable for sulfide concentrations below approximately 1 ppm. The use of lead carbonate, followed by immediate filtration of the sample is required whenever sulfide ion is present. If the presence of sulfide is suspected but not verifiable from the use of lead acetate test paper, two samples may be collected, one without lead carbonate addition and another with lead carbonate addition followed by immediate filtration. Analyze both samples. If sulfide is present, the preserved sample should contain higher levels of cyanide than the unpreserved sample. Lead acetate test paper may be used, but should be tested for minimum level of sulfide detection by spiking reagent water aliquots with decreasing levels of sulfide and determining the lowest level of sulfide detection attainable. The spiked samples are tested with lead acetate test paper moistened with acetate buffer solution. The buffer solution is prepared by dissolving 146 g anhydrous sodium acetate, or 243 g sodium acetate trihydrate in 400 mL of reagent water, followed by addition of 480 g concentrated acetic acid. Dilute the solution to 1 L with reagent water. Each new batch of test paper and/or acetate buffer should be tested to determine the lowest level of sulfide ion detection prior to use.

5.14.2 Ethylenediamine solution—In a 100 mL volumetric flask, dilute 3.5 mL pharmaceutical-grade anhydrous ethylenediamine with reagent water.

5.14.3 Ascorbic acid—Crystals

5.15 FIA Reagents

5.15.1 Carrier and acid reagent (0.1M hydrochloric acid): Dilute 8 mL of concentrated HCL acid to one liter with deionized water.

5.15.2 Acceptor reagent (0.1 M sodium hydroxide): Dilute 100 mL of 1 M sodium hydroxide solution to 1000 mL with deionized water.

5.15.3 Ligand-exchange reagent A-ALPKEM (WAD) Reagent A part number A001416, or equivalent.

5.15.4 Ligand-exchange reagent B–ALPKEM (WAD Reagent B part number A001417, or equivalent.

5.16 Quality Control Standards

5.16.1 Mercury (II) cyanide stock solution (1000 mg/L as cyanide): Weigh 0.486 g of mercury (II) cyanide in a 100 mL volumetric flask. Add 10-20 mL of deionized water and 1 mL of 1 M sodium hydroxide. Swirl to mix. Dilute to the mark with deionized water.

5.16.2 Mercury (II) cyanide, 10 mg/L: Dilute 1.0 mL of Mercury (II) cyanide stock solution and 1 mL of 1 M sodium hydroxide solution to 100 mL with reagent water. Store in an amber glass bottle at 0-4^{o} C.

5.16.3 Laboratory Control Standard (LCS): Place 0.50 mL of the mercury (II) cyanide stock and 1 mL of 1 M sodium hydroxide solution to 100 mL with reagent water to provide a final cyanide concentration of 0.050 mg/L.

6. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

6.1 Samples are collected using manual (grab) techniques and are preserved immediately upon collection.

6.1.1 Grab sampling—Collect samples in amber glass bottles with PTFE-lined caps. Immediately after collection, preserve the sample using any or all of the preservation techniques listed in this section followed by adjustment of the sample pH to <12 by addition 6 NaOH pellets per L and refrigeration at 0-4^{o}C.

6.1.2 Compositing—Compositing is performed by combining aliquots of grab samples only. Automated compositing equipment may not be used because cyanide may react or degrade during the sampling period. Preserve and refrigerate each grab sample immediately after collection until compositing.

6.2 Preservation Techniques

6.2.1 Samples Containing Sulfide Ion

6.2.1.1 Test the sample with lead acetate test paper to determine the presence or absence of sulfide ion. If sulfide ion is present, the sample must be treated immediately (within 15 minutes of collection) with sufficient solid lead carbonate to remove sulfide (as evidenced by the lead acetate test paper), and immediately filtered into another sample bottle to remove precipitated lead sulfide.

6.2.1.2 If sulfide ion is suspected to be present, but its presence is not detected by the lead acetate paper test, two samples should be collected. One is treated for the presence of sulfide and immediately filtered, while the second is not treated for sulfide. Both samples must be analyzed. (Tests conducted prior to the interlaboratory validation of this method showed significant and rapid losses of cyanides when lead sulfide was allowed to remain in contact with the sample during holding times of three days or less. As a result, the immediate filtration of samples preserved with lead carbonate is essential

6.2.1.3 If the sample contains particulate matter that would be removed upon filtration, the sample must be filtered prior to treatment with lead carbonate to assure that cyanides associated with the particulate matter are included in the measurement. The collected particulate matter must be saved and the filtrate treated using the sulfide removal procedure above. The collected particulate and treated filtrate must be recombined and homogenized, and then sent to the laboratory for analysis.

6.2.2 Samples containing water soluble aldehydes—Treat samples containing or suspected to contain formaldehyde, acetaldehyde, or other water soluble aldehydes with 20 mL of 3.5% ethylenediamine solution per liter of sample.

6.2.3 Samples known or suspected to contain chlorine, hypochlorite, and/or sulfite—Treat with 0.6 g of ascorbic acid (Section 7.10.3) per liter of sample.

6.3 Aqueous samples are collected in either glass or plastic. At a minimum, 500mL should be collected.

6.4 Samples shall be stored at $4^{o}C \pm 2^{o}C$ prior to analysis.

6.5 Preserved samples must be analyzed within 14 days of collection.

7. QUALITY CONTROL

7.1 Demonstration of Capabilities

7.1.1 Prior to the analysis of samples, a Demonstration of Capabilities (DOC) as described in Section 8 of the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

7.2 Method Detection Limit Study

7.2.1 A Method Detection Limit (MDL) study, as described in Section 11 of the QA Manual, must be performed initially, annually and any time a significant change is made to the analytical system.

7.3 Initial Precision and Recovery (IPR)—To establish the ability to generate acceptable precision and accuracy, the laboratory shall perform the following operations:

7.3.1 Analyze four LCS samples as described in Section 8.

7.3.2 Using the results of the set of four analyses, compute the average percent recovery (x) and the standard deviation of the percent recovery (s) for cyanide.

7.3.3 Compare s and x with the acceptance criteria specified in Table 14.2. If s exceeds the precision limit or x falls outside the range for recovery, system performance is unacceptable and the problem must be found and corrected before analyses can begin.

7.4 Matrix spike/matrix spike duplicate (MS/MSD)—The laboratory shall spike, in duplicate, a minimum of 10 percent of all samples (one sample in duplicate in each batch of ten samples).

7.4.1 Analyze one sample aliquot out of each set of ten samples according to the procedure beginning in Section 8 to determine the background concentration (U) of cyanide.

7.4.2 Spike two additional 100 mL aliquots of this sample with 0.5 mL of 10 mg/L mercury (II) cyanide secondary stock solution necessary to produce a cyanide concentration in the sample of 0.050 mg/L and analyze these aliquots to determine the concentration after spiking (S).

7.4.3 Calculate the Percent recovery as described in Section 9.

7.4.4 Compare the recovery to the QC acceptance criteria in Table 14.2. If recovery is outside of the acceptance criteria, and the recovery of the LCS in the ongoing precision and recovery test for the analytical batch is within the acceptance criteria, an interference is present. The sample shall be reported with a flag indicating matrix interference.

7.4.5 If the results of both the MS/MSD and the LCS test fail the acceptance criteria, the analytical system is judged to be out of control. In this case, the problem shall be identified and corrected, and the analytical batch reanalyzed.

7.4.6 Calculate the relative percent difference (RPD)(Section 9) between the two spiked sample results (not between the two percent recoveries).

7.4.7 Compare the precision to the RPD criteria in Table 14.2. If the RPD is greater than the acceptance criteria, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch reanalyzed.

7.4.8 Method precision and accuracy for samples may be assessed using the following procedure. After the analysis of five spiked samples in which the recovery passes the test, compute the average percent recovery (P_a) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery p interval from $P_a - 2s_p$ to $P_a + 2s_p$. For example, if P $P_a = 90\%$ and $s_p = 10\%$ for five analyses, the accuracy interval is expressed as 70 – 110%. Update the accuracy assessment on a regular basis (e.g., after each five to ten new accuracy measurements).

7.5 Laboratory Blanks - Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.

7.5.1 Analyze a reagent water blank with each analytical batch. The blank must be subjected to the same procedural steps as a sample.

7.5.2 If cyanide is detected in the blank at a concentration greater than the ML, analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination.

7.6 Calibration verification—Verify calibration of the analytical equipment before and after each analytical batch of 14 or fewer measurements. (The 14 measurements will normally be 10 samples, 1 reagent blank, 1 LCS, 1 MS, and 1 MSD). Verification is accomplished by analyzing the mid-range calibration standard (Continuing Calibration Verification Standard, CCV) and verifying that it is within the QC acceptance criteria for recovery in Table 14.2. Failure to verify calibration within the acceptance criteria requires recalibration of the analysis system.

7.7 Laboratory control sample (LCS)—To demonstrate that the analytical system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the laboratory shall perform the following operations:

7.7.1 Analyze an LCS with each analytical batch according to the procedure in Section 8.

7.7.2 If the results for the LCS are within the acceptance criteria specified in Table 14.2, analysis of the batch may continue. If, however, the concentration is not within this range, the analytical process is not in control. In this event, correct the problem, repeat the LCS test, and reanalyze the batch.

7.7.3 The laboratory should add results that pass the specification in Table 14.2 to IPR and previous LCS data and update QC charts to form a graphic representation of continued laboratory performance.

7.7.4 The laboratory can also develop a statement of laboratory data quality for cyanide by calculating the average percent recovery (R) and the standard deviation of the percent recovery (s_r). Express the r accuracy as a recovery interval from $R - 2s_r$ to $R + 2s_r$. For example, if R = 95% and s_r = 5%, the accuracy is 85% to 105%.

8. PROCEDURE

8.1 Instrument setup

8.1.1 Set up the FIA system and establish initial operating conditions necessary for determination of cyanide. Establish a method for multi-point calibration and for determining the cyanide concentration in each sample.

8.1.2 Verify that the reagents are flowing smoothly through the FIA system and that the flow cell is purged of air bubbles.

8.2 Instrument Stabilization

8.2.1 Start the pump, allowing the reagents to flow through the entire system.

8.2.2 Verify that the flow cell of each detector is purged of all bubbles and that her flow is stable and free from surging.

8.2.3 Load a 5-mg/L-KCN into the sampling valve and inject it into the FIA system.

8.2.4 Continue to inject 5-mg/L KCN until three successive peak heights results are within 2% RSD, indicating that the electrode system is stabilized.

8.2.5 Following stabilization, inject the highest concentration calibration standard until three successive peak heights or area results are within 2% RSD, indicating stabilization at he top of the calibration range.

8.3 External Standard Calibration

8.3.1 Set up instrument per manufacturer instructions.

8.3.2 Inject a series of at least 3 calibration standards by preparing them as outlined in Table 14.1. One of the standards should be at the minimum level (ML) unless measurements are to be made at a higher level. The other concentrations should correspond to the expected range of concentrations found in samples or should define the working range of the FIA system.

8.3.3 Using injections of constant Volume, analyze each calibration standard. Record peak height against standard concentration.

8.3.4 The instrument will construct a calibration curve and calculate a correlation coefficient. The correlation coefficient of 0.995 or greater must be achieved using the calibration standards. Repeat calibration if the correlation coefficient requirements cannot be met.

8.3.5 Each calibration curve should be verified by analysis of a LCS.

8.4 Sample Pretreatment/Ligand Exchange Reagent Treatment

8.4.1 Verify with pH paper that the pH of the samples is $\geq$12.

8.4.2 Add 50 µL of the Ligand exchange reagents WAD Reagent A and 100µL of WAD Reagent B to 100 mL of sample. Mix well.

8.4.3 Analyze the treated samples within 2 hours of adding the two Ligand exchange reagents.

8.5 Analysis

8.5.1 Place all reagents on-line and allow to pump at least 10-15 minutes. Verify that there are no bubbles in the flowcell. Obtain a stable baseline and auto zero the baseline before beginning the analysis.

8.5.2 Load the sampler tray with calibrants, blanks, samples and QC samples.

8.5.3 Using the Method and Sample Table created for the analytical batch to be analyzed and with the baseline verified to be stable, begin the analysis by selecting the "Fast Forward" button on the left side of the Data Analysis Window in the WinFLOW. This will initiate the sequential analysis of samples as defined in the Sample Table.

8.5.4 When analysis is complete, pump reagent water through the system for at least 10-15 minutes. Stop the pump; release the tension on all pump tubes, disconnect the electrodes and power off the system.

9. CALCULATIONS/DATA REDUCTION AND INTERPRETATION

9.1 The calibration curve allow for accurate quantitation of the concentration in each sample.

9.2 Results for Available Cyanide in mg/L:

Result (mg/L) = (Reading) x (Dilution Factor)

9.3 Relative Percent Difference (RPD) between Duplicates:

$$\text{RPD} = \frac{(\text{Larger Value} - \text{Smaller Value}) \times 100}{(\text{Average of the Values})}$$

9.4 Spike Recovery of Laboratory Control Standards, Matrix Spikes, and Matrix Spike Duplicates:

$$\%\,\text{Recovery} = \frac{(S - U) \times 100)}{T}$$

Where:

S = Spiked result

U = Unspiked result (use 0 for LCS)

T = Spike True Concentration

10. METHOD PERFORMANCE

10.1 The supervisor has responsibility to ensure that an analyst who performs this procedure is properly trained in its use and has the required experience. Performance is monitored through internal QC and outside performance evaluation samples. Please refer to Section 5 of the QA Manual for additional information concerning Precision and Accuracy.

11. POLLUTION PREVENTION

11.1 This procedure will be carried out in a manner consistent with all applicable federal, state, and local regulations regarding pollution control and prevention. Specific procedures in regards to accidental release of hazardous materials can be found in the TestAmerica Safety Manual.

12. WASTE MANAGEMENT

12.1 Waste generated in this procedure is segregated and disposed of in accordance with all applicable federal, state, and local regulations. The Safety Officer or Department Supervisor should be contacted if additional information is required.

13. REFERENCES/CROSS-REFERENCES

13.1 Method OIA-1677, United States Environmental Protection Agency, Office of Water, EPA-821-R-99-013, August 1999.

13.2 CN Solution 3000 Operation Manual, Part Number A002370, Revision B, January 1998, ALPKEM, A Division of OI Analytical.

13.3 WinFLOW V4 Operator's Manual – Advanced Technology in Ion Analysis, Part Number A001965, Revision 1.1 April 1999, ALPKEM, A Division of OI Analytical.

13.4 Method OIA-1677: Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA), Part Number A002966, ALPKEM, A Division of OI Analytical.

14. ATTACHMENTS/TABLES

14.1 Working Calibration Standard Concentration Table

Standard	**Volume**	**Of Solution**	**Diluted with 1 mL of 1M NaOH and DI water to**	**= Concentration in µg/L**
Standard 1	0.020 mL	10 mg/L CN Secondary Std	100 mL	2 µg/L
Standard 2	0.10 mL	10 mg/L CN Secondary Std	100 mL	10 µg/L
Standard 3/CCV	0.50 mL	10 mg/L CN Secondary Std	100 mL	50 µg/L
Standard 4	1.0 mL	10 mg/L CN Secondary Std	100mL	100 µg/L

14.2 Quality Control Acceptance Criteria Table

Criterion	**Required Recovery Range (%)**	**Precision**
Initial Precision and Recovery	92-122	<5.1% RSD
Ongoing Precision and Recovery	82-132	N/A
Calibration Verification	86-118	N/A
Matrix Spike/Matrix Spike Duplicate	82-130	<11% RPD

15. CONTINGENCIES

15.1 If for any reason a part of this SOP cannot be followed, seek the guidance of the Department Supervisor or Quality Assurance Department. Document all deviations on a Corrective Action Report and submit to the QA Quality Assurance Department.