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• ENVIRONMENT • ENERGY • ENGINEERING

June 13, 2008

Ms. Annette Weissbach
Wisconsin Department of Natural Resources
2984 Shawano Ave.
Green Bay, WI 54313

Subject: Revisions to Kewaunee Field Trials Workplan and Response to Comments

Dear Ms. Weissbach:

Attached is the revised Workplan for Bioreduction Field Trials at the Kewaunee Marsh. We have incorporated the comments in your June 9 letter into the workplan. The comments are also addressed below.

Comments on Section 4:

The sides of the test plot boxes have been increased to 4 feet by 8 feet, so that we have 2 feet below grade and 2 feet above. In addition, there will be 2-inch by 4-inch corner posts that will be pushed 4 feet into the soil to secure the corners of the test plot sides. This should be enough to secure the plywood through 2009. There is a sketch of the test plot construction in the workplan.

The workplan has been upgraded to include the initial screening sample analysis and the pesticide application by the U.S. Fish and Wildlife Service and to correct the typos that slipped through.

Comments on Section 5:

5.1 Soil Sampling. The workplan has been amended to incorporate monthly sampling for both soil and gas through October 2008.

Analysis for ethanol and sucrose in the soil/pore water has not been incorporated into the approach. The additives will be spread on the surface of the marsh, and not mixed into the soil to ensure a uniform concentration. The purpose is to create highly reducing conditions favorable to methane formation at the top of the marsh soil and thus prevent oxygen flux into the marsh soil. Some of the sugar/ethanol will also migrate into the underlying material and enhance the anaerobic conditions deeper in the marsh. However, the additives are designed to enhance the degradation on the natural material in the marsh, rather than providing the main carbon source for the methanogens. Since the initial additive concentration in the soil will be heterogeneous, since both additives are highly biodegradable and soluble and should not remain for long, and since the bacteria are not dependent

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on the additives for their food supply, the effectiveness of the additives can be better monitored by looking at the reduction in arsenic levels rather than monitoring the concentrations directly.

"Compositional" arsenic is the same as total arsenic, and includes arsenate, arsenite, organic arsenicals and arsenic incorporated into plant matter. Analysis of the pore water may not provide much additional information, since the bulk of the arsenic is incorporated in the solid matter in the marsh.

5.2 Arsine Gas Sampling. We have added an arsine gas screening analysis to the initial phase of work, using the Hach Company arsine gas test strips in their field kit. The gas will be collected in a plastic beaker over the soil sample locations for a day, and then analyzed using the test strips. Please note that there is no pore gas in the marsh (except in the cap), so the soil gas sampling procedures are not relevant.

Gas will be generated by the marsh material, and directed to the tedlar bags for collection. We do not anticipate any problem in filling the bags using the pressure from the marsh itself. (Note that we are not pulling the gas out of the marsh, as is done in soil gas sampling, but rather directing the flow of gas from the marsh to the tedlar bags.) As noted in the workplan, the arsine gas collection is not a standard method, and may need to be modified based on the initial findings.

The arsine gas concentrations will be converted to an arsine flux from the marsh by assuming that the gas is generated over a period of 24 hours, and using the flux in a calculation. The flux will be used to estimate arsine concentrations in the breathing zone of someone standing in the marsh and on the bike trail. These calculations will involve dispersion modeling, which will be done by people in RMT's Milwaukee Office who are familiar with dispersion modeling. We may also need to incorporate arsine oxidation rates in the model, depending on the concentrations found in the modeling.

5.3 Bioreductant Application. The purpose of the multiple applications of the bioreductant is to see if more than one application provides enhanced treatment. We are assuming that any enhancement for the sucrose application would also apply to ethanol.

Comments on Section 6:

The deliverables have been modified to include the information for a brochure/poster in the first progress report, and a discussion of the potential health threats from arsine gas in the final report.

We are looking forward to working on the field trials, and to seeing if the idea of bioreduction is feasible and safe.

Sincerely,

RMT, Inc.

Robert Stanforth
Senior Chemist

Attachments

cc: Richard Fish – RMT, Inc.
Alyssa Sellwood – RMT, Inc.

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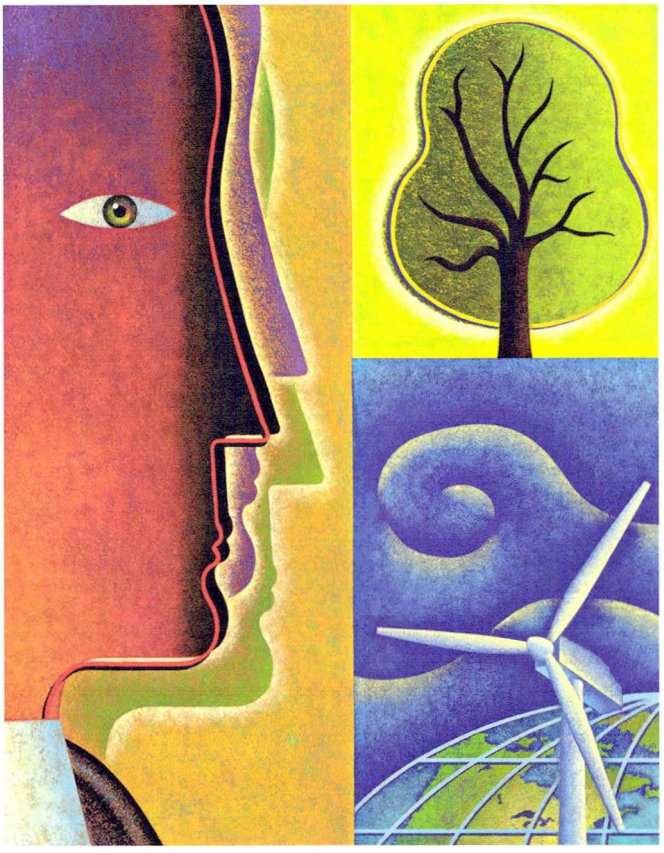
Workplan for Bioreduction Field Trials

Kewaunee Marsh

May 2008/Revised June 2008



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Workplan for Bioreduction Field Trials

Kewaunee Marsh

May 2008/Revised June 2008

Alyssa Sellwood
Staff Engineer

Richard P. Fish
Senior Project Manager

Robert Stanforth, Ph.D.
Senior Chemist

RMT, Inc. | Wisconsin Department of Natural Resources
Final
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Section 1
Background

Approximately 15 acres of the Kewaunee Marsh Besadny Wildlife Area, in Kewaunee, Wisconsin, are contaminated with arsenic (Figure 1). The source of the arsenic is likely a spill that occurred from the adjacent railroad in the 1940s. An interim action was completed in 1996 to limit the threat of direct contact to the arsenic in which approximately 4 acres of the marsh were capped, and the 15 acres were enclosed in a fence. In 2002, STS Consultants completed a Phase II Site Investigation (SI), and found that the arsenic contamination is limited to the shallow groundwater and the upper 2 feet of the marsh sediment (soil), and that arsenic is entering the Kewaunee River through two surface water sloughs. On the basis of the results of the SI, site-specific cleanup standards of 19 milligrams/kilograms (mg/kg) for soil and 148 micrograms/liter (µg/L) for groundwater/surface water were established for the site.

RMT, Inc. (RMT), completed a treatability study and remedial options analysis for the marsh to evaluate alternatives for achieving the site-specific cleanup standards. The investigations and studies showed that the concentration of arsenic in the marsh soil has decreased over time, and that the majority of this decrease can be attributed to volatilization of arsenic to arsine gas under reducing conditions in the marsh. Based on this finding, enhanced bioreduction of arsenic was evaluated as a remedial option for the marsh soil using laboratory-based treatability testing. The results of the testing demonstrated that introduction of specific bioreductants enhanced methane formation and arsenic volatilization from the marsh soil.

Based on the results of the laboratory testing and an evaluation of the cost and feasibility of implementing enhanced bioreduction (as compared to excavation of the contaminated material), RMT recommended that bioreduction be pursued as the remedial option of the marsh soil for the site. Prior to full-scale implementation of this option, RMT strongly recommended that field trials be completed to confirm the effectiveness of enhanced bioreduction outside of the laboratory setting, and to determine the best approach for full-scale implementation of the alternative, if it was evaluated to be effective.

The WDNR has retained RMT to further evaluate enhanced bioreduction of arsenic in the marsh soil by completing a field trial study. The purpose of this submittal is to present RMT's plans for conducting the field trials.

Section 2
Project Objectives and Approach

2.1 Project Objectives

The objectives of the field trials are as follows:

- To demonstrate and evaluate the performance of enhanced bioreduction in the field.
- To evaluate design criteria:
  - Approaches to cattail management (mechanical versus chemical)
  - Differences in the effectiveness of two bioreductants (sugar versus ethanol)
  - Effect of single versus multiple applications of bioreductant

2.2 Approach

The bioreduction field trials will be completed using seven 8-foot by 8-foot test plots, which will be constructed immediately south of the capped area on the western half of the 15-acre site (near the former railroad) in the area shown on Figure 2. This area was selected for the test plots because the soil in this area has high arsenic concentrations and the location can be accessed relatively easily (Figure 1). In order for bioreduction to occur, reducing conditions must be present. Because cattails oxygenate the marsh within their root zone, the cattails within the test plots must be removed in order for bioreduction to occur in the upper 2 feet of soil at the site.

Each test plot will be constructed to prevent flow across the test plot so as to minimize the effects of overland flow and mixing between test plots. This will allow RMT to evaluate the loss of arsenic over time due solely to volatilization in each plot. The seven test plots will be set up as follows:

1. Control plot (no alteration to existing conditions)
2. Cattail removal using mechanical means
3. Cattail removal (mechanical) and one application of ethanol as the bioreductant
4. Cattail removal (mechanical) and one application of sugar (sucrose) as the bioreductant
5. Cattail removal (mechanical) and multiple applications of sugar (sucrose) as the bioreductant
6. Cattail removal using an herbicide
7. Cattail removal (herbicide) and one application of sugar (sucrose) as the bioreductant

Section 3
Field Trial Project Organization

The project organization for the field trials is similar to that implemented by RMT for the treatability and feasibility study.

The organizational chart (Figure 3) presents the key members of the project team for the field trials. The primary roles and responsibilities of the key members of the team are as follows:

RMT, Inc.

Mr. Richard Fish, Senior Project Manager

- Coordinate the work and provide status reports to the WDNR Project Manager.
- Review and approve the Site-specific Health and Safety Plan.
- Review and approve the procurement of equipment and supplies.
- Track the progress of field trials.
- Review the data from field trials.

Mr. Paul Turpin, Quality Assurance Provider

- Provide overall quality assurance for the field trials.

Mr. Robert Stanforth, Senior Chemist

- Develop the scope for the field trials.
- Coordinate and oversee the laboratory analysis of the soil samples.
- Analyze the data from the field trials.
- Provide conclusions and recommendations based on the results of the field trials.
- Report the results of the field trials to the WDNR.

Ms. Alyssa Sellwood, Staff Engineer

- Prepare a Site Health and Safety Plan.
- Coordinate activities of RMT personnel in conducting the field trials.
- Assist with test plot construction and performance monitoring.
- Prepare Progress Reports and the Field Trial Documentation Report.

Mr. Jason Schoephoester, Environmental Scientist

- Collect background and performance monitoring soil samples for laboratory analysis.
- Procure the equipment, material, and supplies required to construct test plots.
- Construct the test plots, including an impermeable barrier, and cut and chop cattails.
- Add bioreductant sugar (sucrose) during each monitoring event to Test Plot 5.

Mr. Kent McCord, Environmental Scientist

- Develop the arsine gas collection method.
- Collect the arsine gas samples.
- Assist with test plot construction and performance monitoring.

Pace Analytical Services

- Provide sediment/soil analysis as necessary to verify background levels of arsenic in the test plots and the effectiveness of the bioreductants. The Quality Manual for Pace is included as Appendix A of this Workplan.

Section 4
Background Sampling and Test Plot Construction

The seven test plots will be constructed in June of 2008 as follows:

1. Test plots for the field trials, will be constructed in the area of higher arsenic contamination as shown on Figure 2. The location of the test plots will be determined using a global positioning system (GPS) unit and the coordinates from previous investigations. The locations to be treated with herbicide have been offset from the five other test plots to ensure that the herbicide does not affect the control test plot or the test plots that will use mechanical methods to remove the cattails.

2. RMT will mobilize to the site to construct the test plots. The first step will be to collect soil samples from throughout the test plot area and perform a rapid screening compositional arsenic analysis to ensure that arsenic concentrations are in the desired range (>200 mg/kg As). These results will be used to select the final test plot locations. Once the locations have been established, the cattails will be cut down in Test Plots 2 though 5 using garden shears. The cattails will be cut into pieces and stockpiled for later use.

3. RMT will provide the WDNR with the location of the plots to be treated with a herbicide (Test Plots 6 and 7). The specific herbicide and approach used to apply the herbicide will be developed by the WDNR, but discussed with RMT for documentation purposes. The herbicide (most likely Rodeo) will be applied by a representative of the U.S. Fish and Wildlife Service after completion of the test plots.

4. For those test plots in which the cattails will be removed by mechanical methods (Plots 2 through 5), the root structure of the cattails will be removed using a rototiller and/or shovel.

5. An impermeable barrier will be constructed to define the boundaries of each test plot, and to prevent the mixing of materials between each plot. The impermeable barrier will be constructed using 4-foot by 8-foot sheets of marine-grade plywood (see Sketch 1). The plywood will be trenched a minimum of 2 feet into the marsh soil using a mechanical trencher and/or hammer. The corners of each plot will be reinforced with 2 x 4 posts, pushed 4 feet into the soil, and sealed with silicone caulk or a similar material. A stake will be placed in each test plot and labeled with the test plot number and bioreductant application approach.

6. Five baseline soil samples will be collected from each test plot using the sampling protocol described in Appendix B, and submitted to Pace for arsenic analysis. These samples will provide a baseline concentration of arsenic in each test plot. The approximate locations of the samples are shown on Figure 2.

7. After baseline soil samples have been collected, the bioreductants will be applied to Test Plots 3, 4, 5, and 7. A concentrated solution of either ethanol or sugar (described below) will be prepared using tap water brought to the site by RMT. The solution will be applied evenly across each test plot using a hand-held garden sprayer or similar device, as follows:
   - **Test Plot 3:** An approximate 250-gram per liter (g/L) solution of ethanol will be prepared by combining 40 liters (10 gallons) of water with 12 liters of ethanol.
   - **Test Plots 4, 5, and 7:** An approximate 250-g/L solution of sucrose will be prepared by combining 40 liters (10 gallons) of water with 20 pounds of sucrose for each test plot.
8. A gas collection device will be constructed at each test plot as described in Subsection 5.2.
9. The chopped-up cattails that were stockpiled in Step 6 will be spread across Test Plots 2 though 5.

Section 5
Performance Monitoring and Follow-on Activities

5.1 Soil Sampling

Starting in July 2008 (or 1 month after completion of the test plots), bioreduction will be monitored on a monthly basis, through October, 2008. During each site visit, five soil samples will be collected from each test plot and submitted to Pace for laboratory analysis for compositional (total) arsenic. The samples will be collected from locations similar to those collected during the baseline sampling, and as shown on Figure 2. Soil sampling and decontamination procedures will follow those described in Appendix B. In addition, any new vegetative growth in Test Plots 2 through 7 will be documented and then removed using a shovel.

5.2 Arsine Gas Monitoring

Enhancing the natural conversion of arsenic to arsine gas is a new approach for remediating arsenic-contaminated soil. Arsine gas is toxic, and it is therefore critical to quantify the concentration of arsine gas being produced by the enhanced bioreduction. Arsine gas generated during the field trials at Kewaunee Marsh will be collected and analyzed as described in Appendix C. In addition, samples of the gas generated at the initial soil sample locations will be monitored while the soil samples are being analyzed (i.e., overnight). This will provide an initial indication of whether arsine is being generated, and if so, provide some comparison with soil arsenic concentrations. Arsine gas analysis will be done by placing an inverted plastic beaker over the soil sample locations. The gas will be analyzed using an arsine test strip from Hach Company. Preliminary calculations have indicated that the concentrations of arsine generated will be well below levels of concern; however, the gas data collected during the field trials will be used to verify this preliminary calculation and specifically evaluate health and safety concerns. In addition, quantifying arsine gas generation will provide documentation of the formation of arsine, and provide input for a mass balance calculation for the amount of arsenic lost according to the compositional analysis and the amount of arsine generated.

The proposed approach for completing the gas collection is described in Appendix C. It should be noted that the collection and measurement of arsine in marsh gas is not a standard laboratory procedure, and both the collection and analytical methods may be modified to optimize the process during the periodic sampling events.

5.3 Bioreductant Application

During monthly monitoring events in July, August, and September, 2008, additional bioreductant (sugar) will be applied to Test Plot 5, per the protocol described in Section 4.

5.4 Contingency for Additional Monitoring

If bioreduction is occurring more slowly than expected, two additional sampling events will occur between May and September 2009. Up to five soil samples will be collected from each test plot and analyzed for arsenic by Pace during each of the two 2009 site visits. In addition, if new vegetative growth is observed in Test Plots 2 through 7 during the site visits, RMT will evaluate the growth and develop an approach to remove it by mechanical or chemical means, as necessary. Additional bioreductant will be applied to Test Plot 5 during each of the site visits in 2009.

Section 6
Deliverables

The following deliverables will be provided to WDNR:

- Up to three progress letter reports after test plot construction and the initial soil analysis, after the soil sampling has been completed, and after arsine gas monitoring. The first progress report will include a synopsis of the pilot study and other proposed remedial options that may be used in a brochure or to update the sign at the site.
- A final Field Trial Documentation Report, which will include a description of the field trials, a summary of the performance monitoring data, an evaluation to determine whether bioreduction can effectively remediate the site, and recommendations for further action (e.g., full-scale implementation). If the air sampling shows that arsine gas is being generated, the Field Trials Documentation Report will discuss the health threats and relative risks.

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KEWAUNEE RIVER

LEGEND

SOIL ARSENIC CONCENTRATIONS (1994 - 2006)
(MG/KG)

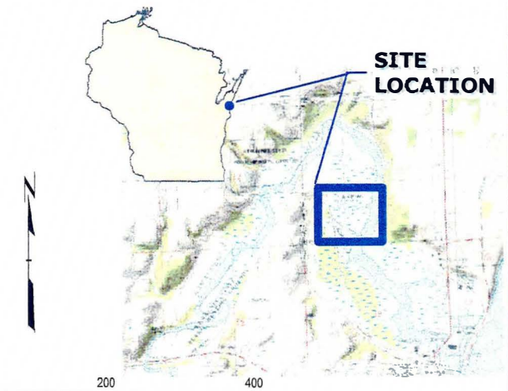
- < 200
- 200 - 1,000
- > 1,000

FENCE

CAPPED AREA

NOTES

1. ARSENIC CONCENTRATIONS AND SAMPLE LOCATIONS FOR SAMPLES FROM STS CONSULTANTS AND RMT.



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CHECKED BY: SELLWOOD A		FILE NO.	72010701.mxd
APPROVED BY: STANFORTH B	DATE PRINTED: 5/27/2008	FIGURE 1	
DATE: MAY 2008			

RMT

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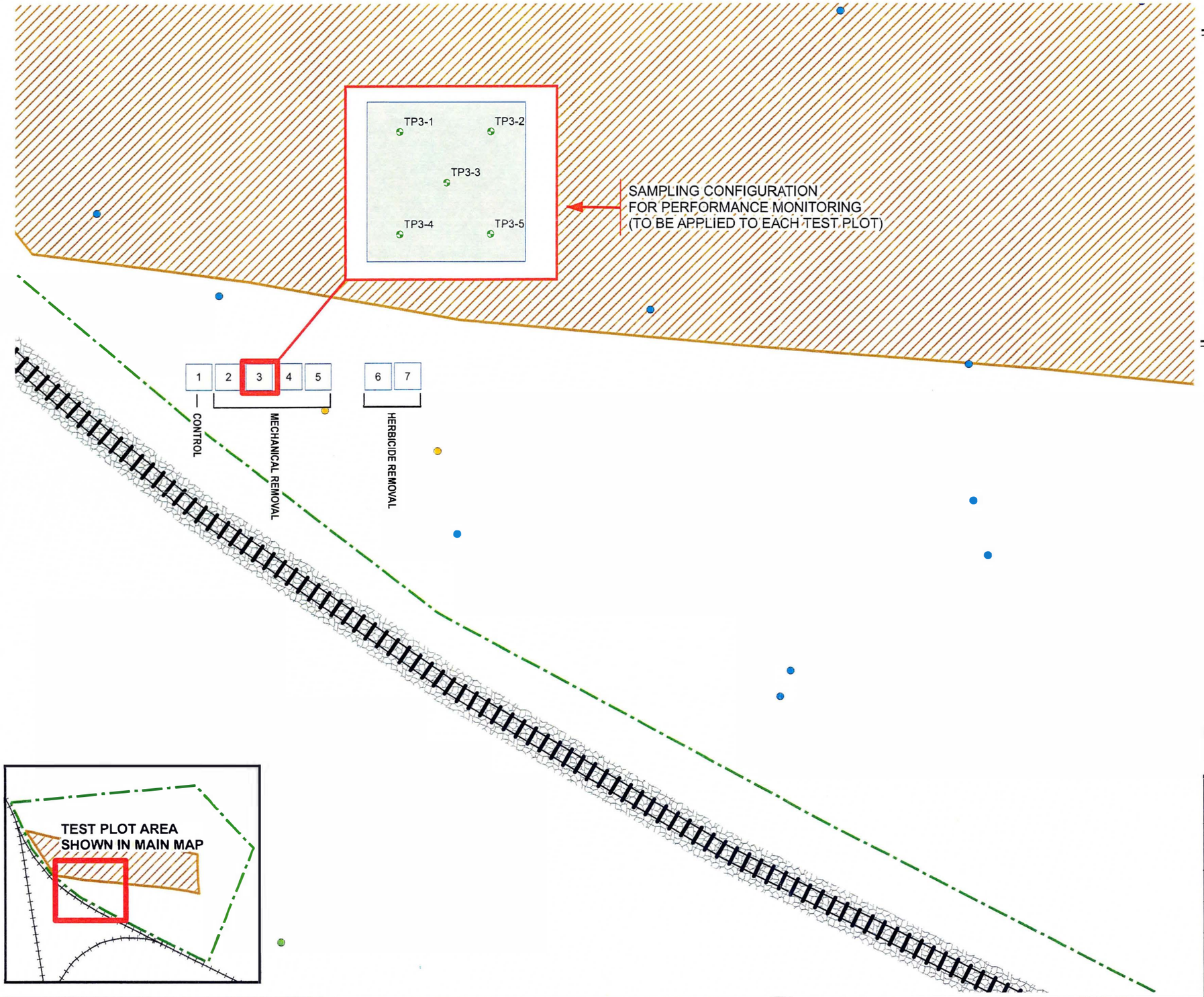


LEGEND

SOIL ARSENIC CONCENTRATIONS (1994 - 2006)
(MG/KG)

- < 200
- 200 - 1,000
- > 1,000

FENCE

CAPPED AREA

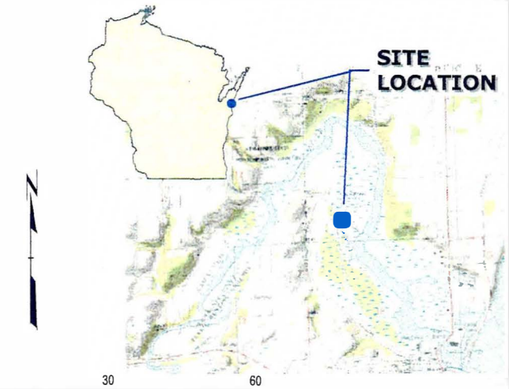
FORMER RAILROAD LOCATION

TP3-1 PROPOSED SAMPLE LOCATIONS

3 8' X 8' TEST PLOT (LOCATION APPROXIMATE)

NOTES

1. ARSENIC CONCENTRATIONS AND SAMPLE LOCATIONS FOR SAMPLES FROM STS CONSULTANTS AND RMT.
2. TEST PLOT NUMBER CORRESPONDS TO CONDITIONS DESCRIBED IN THE APPROACH SECTION OF THE WORKPLAN.



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SHEET TITLE:	BIOREDUCTION FIELD TRIAL LAYOUT

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CHECKED BY:	SELLWOOD A	AS NOTED	FILE NO. 72010702.mxd
APPROVED BY:	STANFORTH B	DATE PRINTED:	FIGURE 2
DATE:	MAY 2008	5/19/2008	

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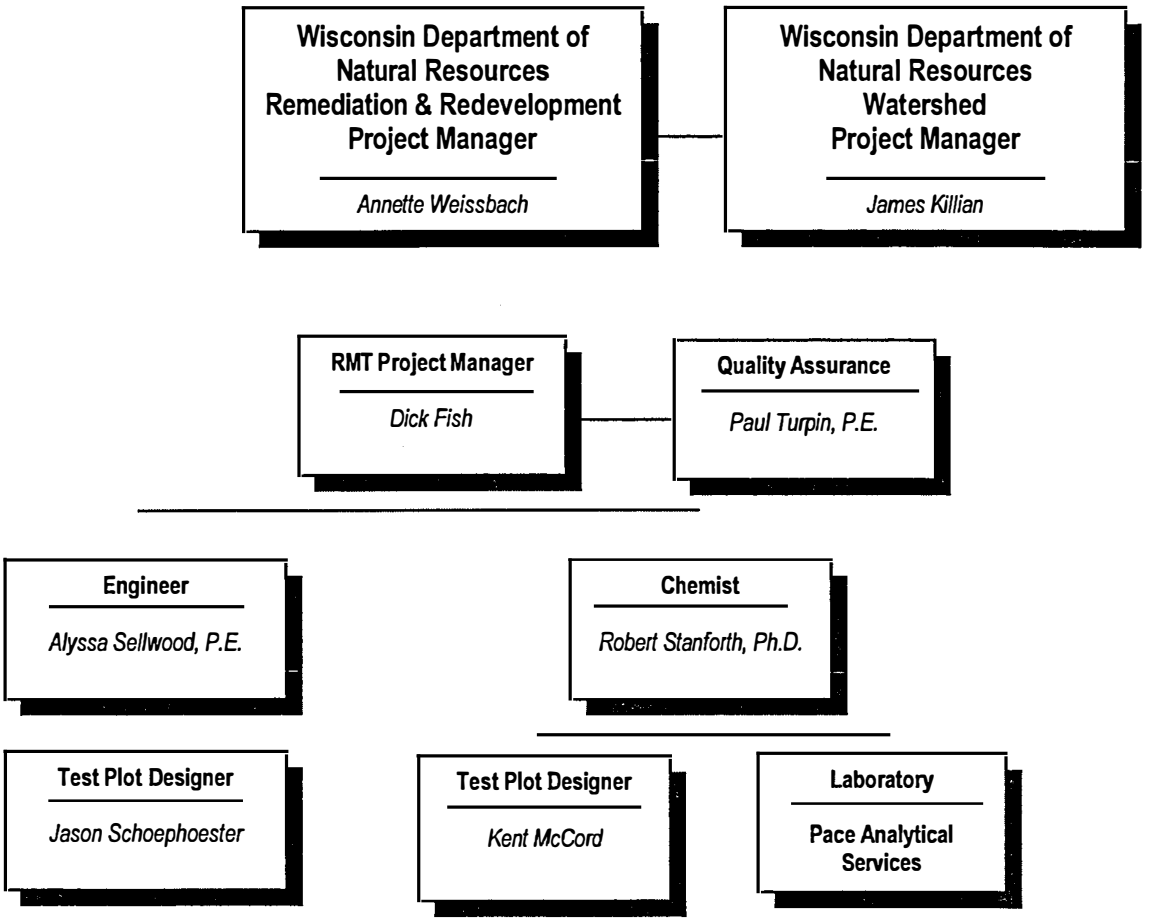
Figure 3
Project Organization Chart
Kewaunee Marsh Field Trial Study



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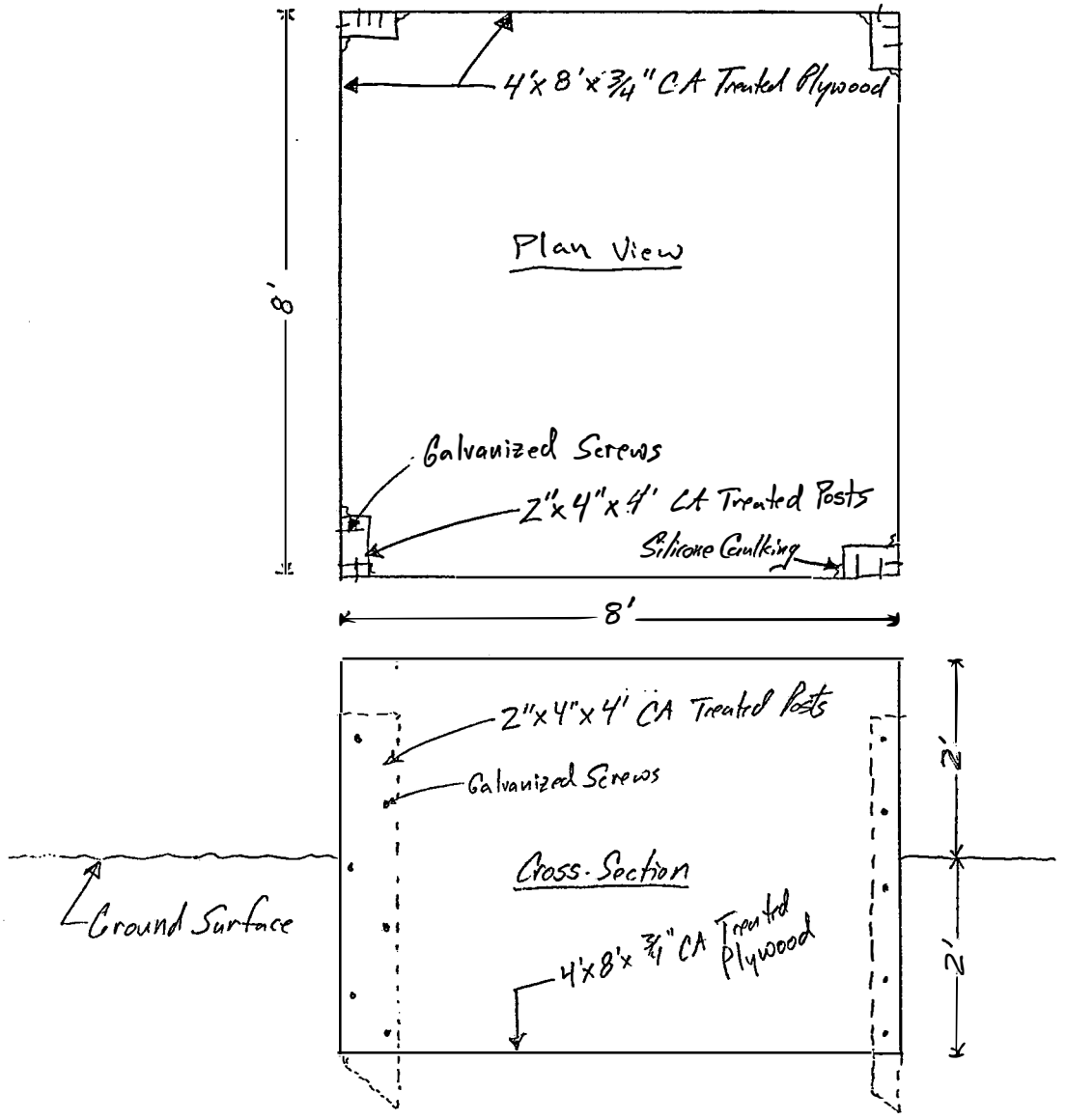
SUBJECT: Bio-Reduction Field Trials Test Plot Construction		7201.07
PREPARED BY: JRS	DATE: 6/10/08	FINAL ☐
CHECKED BY:	DATE:	REVISION ☐

Not to Scale



Sketch 1

Appendix A
Pace Laboratory Quality Manual

QUALITY MANUAL

Quality Assurance/Quality Control
Policies and Procedures

Prepared by Pace Analytical Services, Inc.
1700 Elm Street S.E., Suite 200
Minneapolis, Minnesota 55414
(612) 607-1700

for:

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Approval

Steve A. Vanderboom — 11-21-05
President, CEO — Date
(612) 607-6409

John P. Dullaghan — 11-11-05
COO — Date
(612) 607-6328

Signature Page

This document, with the necessary addenda, has been accepted as the Quality Manual.

[signature]	7/10/06	Green Bay Laboratory
Laboratory General Manager	Date	Laboratory
[signature]	7/6/06	
Quality Manager	Date	
[signature]	7/6/06	
Technical Director	Date	
[signature]	7/6/06	
Technical Director	Date	
[signature]	7/6/06	
Technical Director	Date	
[signature]	7/6/2006	
Technical Director	Date	
[signature]	7-10-06	
Technical Director	Date	

Table of Contents

1.0 INTRODUCTION & ORGANIZATIONAL STRUCTURE

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

Pace Analytical Statement of Purpose

1.1 Introduction to Pace

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

Pace Analytical's goal is to continue to combine its expertise in laboratory operations with customized solutions to meet the specific needs of its clients.

1.2 Statement of Purpose

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

1.3 Quality Policy Statement and Goals of the Quality System

Pace Analytical is committed to providing the highest quality product to our clients, while maintaining good professional practices.

The management of Pace Analytical is responsible for maintaining the highest possible standard of service for our clients by following a documented quality system. The overall objective of this quality system is to provide reliable data by adhering to rigorous quality assurance policies and quality control procedures as documented in this Quality Manual.

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

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

1.4 Pace Analytical Core Values

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

1.5 Code of Ethics

Pace Analytical's fundamental ethical principles are as follows:

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

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

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

1.6 Standards of Conduct

1.6.1 Data Integrity

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

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

1.6.2 Confidentiality

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

Confidential or proprietary information, belonging to either Pace Analytical and/or its clients, includes but is not limited to test results, trade secrets, research and development matters, procedures,

methods, processes and standards, company-specific techniques and equipment, marketing and client information, inventions, materials composition, etc.

1.6.3 Financial Responsibility

Pace Analytical employees must accurately keep all books, records and accounts for which they are responsible. Employees are responsible for maintaining and safeguarding all company funds and/or assets in their possession or control. Additionally, all employees are responsible for submission of accurate reporting documents pertaining to payroll (timesheets, etc.) and reimbursement requests (expense reports, tuition reimbursement, etc.).

1.6.4 Drug-free Workplace

Pace Analytical recognizes that alcoholism and other drug dependencies are a significant social problem with a potential for causing severe detriment to the workforce. Employees have the right to work in an alcohol and drug-free environment. Employees are not to report to work under the influence of alcohol or controlled substances as prescribed in the Drug-free Workplace Program. The possession, use and sale of alcohol and controlled substances as well as detailed information regarding drug testing is described in the Drug-Free Workplace Program Policy handout provided to all new employees. Additional copies are available from Human Resources. Employees are obligated to conform to the strict Drug-Free Workplace Program Policy.

1.6.5 Conflict of Interest

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

- Participation in activities that conflict or appear to conflict with Pace Analytical responsibilities.
- Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced. This includes bribes, kickbacks or illegal payments.

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

1.6.6 Non-Harassment

Pace Analytical endeavors to provide a workplace free of both harassment and discrimination. Harassment of an employee or job applicant on the basis of race, color, creed, religion, national origin, sex, disability, age, marital status, sexual orientation, or any characteristic protected by applicable municipal, state, and federal laws is both illegal and a violation of company policy. Pace Analytical maintains a Sexual Harassment Prevention policy that clearly defines the company's stance against sexual harassment and provides a mechanism for reporting infractions. The Sexual Harassment Prevention Policy is addressed in employee orientation, and employees are provided the Policy handbook. Additional copies are available from Human Resources.

1.6.7 Proper and Professional Conduct

Employees are bound to use fairness, honesty and regard for the law in their business relationships with Pace Analytical investors, clients, suppliers, employees, and applicants as well as all local, national and international communities and governments.

1.6.8 Protection of Property

Pace Analytical employees have an obligation to protect all company and client property against loss, theft and misuse. Employees are responsible for maintaining an orderly, clean workplace. Employees are also liable for using company and client property for intended purposes only. Employees are prohibited from using company property for their personal use without the expressed permission of their supervisor or General Manager. No such use of property may be made after termination of employment with Pace Analytical. Employees must also make every effort to prevent the misuse of company and client property by other persons. Misuse includes selling, loaning or giving away company or client property.

1.6.9 Communication

Each employee is responsible for obtaining the information necessary to follow directives in the Code of Ethics and the Standards of Conduct, and for reporting to their management or Human Resources representative any observed deviations from these policies. The identity of the employee reporting the infraction will not be disclosed without his/her permission unless disclosure is unavoidable during an investigation. No adverse action will be taken against a Pace Analytical employee because he/she has reported a suspected impropriety. These reports will be treated in confidence to the maximum extent consistent with the fair and rigorous enforcement of the Code of Ethics and Standards of Conduct.

1.6.10 Compliance

All employees are required to read, understand and comply with the various components of the standards listed in this document. As confirmation that they understand this responsibility, each employee is required to sign an acknowledgment form annually that becomes part of the employee's permanent record.

1.7 Laboratory Organization

The Pace Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety and training activities. Pace Analytical's Director of Quality, Safety & Training is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Figure 1.1 for the Corporate Organizational structure.

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

A General Manager supervises each regional laboratory. Quality Managers at each lab report directly to their General Manager but receive guidance and direction from the Director of Quality, Safety & Training.

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

- Organic Sample Preparation
- Wet Chemistry Analysis

- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Product Testing
- Equipment Maintenance
- Microbiology

Appropriate support groups are present in each laboratory. Figure 1.2 represents a typical organizational structure for a laboratory operation. The organizational structure for a specific laboratory is part of each laboratory's addendum to this Quality Manual.

1.8 Laboratory Job Descriptions

1.8.1 General Manager

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

1.8.2 Quality Manager

1. Oversees the laboratory Quality Systems while functioning independently from laboratory operations. Reports directly to the General Manager.
2. Monitors Quality Assurance policies and Quality Control procedures to ensure that the laboratory achieves established standards of quality.
3. Maintains records of quality control data and evaluates data quality.
4. Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or client representatives.
5. Reviews and maintains records of proficiency testing results.
6. Maintains the document control system
7. Assists in development and implementation of appropriate training programs.
8. Provides technical support to laboratory operations regarding methodology and project QA/QC requirements.
9. Maintains certifications from federal and state programs.
10. Ensures compliance with all applicable state, federal and industry standards.

1.8.3 Project Manager

1. Coordinates all aspects of specific projects.
2. Focal point for client contact pertaining to project requirements and project status.
3. Arranges bottle orders and shipment of sample kits to clients.
4. Verifies login information relative to project requirements and field sample Chains-of-Custody.
5. Communicates with operations staff to update and set project priorities.
6. Provides results to clients in the requested format (verbal, hardcopy, electronic, etc.).
7. Works with clients, laboratory staff, and other appropriate Pace Analytical staff to develop project statements of work or resolve problems of data quality.

1.8.4 Operations Manager or Department Manager/Supervisor

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

1.8.5 Group Supervisor/Leader

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

1.8.6 Analyst

1. Analyzes samples according to published methods and laboratory procedures.
2. Monitors quality control data. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.7 Sample Management Personnel

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

1.8.8. Systems Administrator or Systems Manager

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

1.8.9. Safety/Chemical Hygiene Officer

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

1.9 Training and Orientation

Additional information can be found in SOP ALL-Q-020 ***Training Procedures.***

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

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

The data integrity system of each lab contains four elements:

- Data integrity training
- Signed data integrity documentation for all employees
- In-depth, periodic monitoring of data integrity –including but not limited to:
  - logbook checks
  - raw data review
  - final report review
  - manual integration checks
- Documentation of review and investigations regarding data integrity issues.

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

The Quality Systems orientation provides the new employee with information through an introduction to the Quality Manual and SOPs, acceptable record keeping practices, and the individual's responsibility to data quality.

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

Supervised training uses the following techniques:

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

Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability (see Section 3.4 for details on Demonstration of Capability requirements). Training documentation files for each person are kept in a central location in each laboratory. The Quality Manager is responsible for maintaining the training files.

All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated annually by lab management.

1.10 Laboratory Safety

It is the policy of Pace Analytical to make safety and health an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety by complying with established company rules and procedures.

Sample receiving areas and laboratories are equipped with suitable hoods, protective clothing and eye wear, gloves, barrier creams and any other appropriate measures to prevent or minimize staff contact with hazardous substances. Other appropriate safety equipment such as eyewash stations, drench showers, spill absorbents and neutralizers, fire extinguishers, and first aid materials are available.

Each laboratory has a designated Safety/Chemical Hygiene Officer and Safety Committee that meets regularly and discusses agenda topics and addresses action items. The Safety/Chemical Hygiene Officer facilitates the preparation and maintains the Chemical Hygiene Plan/Safety Manual, provides safety and occupational health orientation to new employees, conducts safety training and review sessions as required, and maintains up-to-date familiarity with safety and occupational health issues pertinent to the laboratory.

1.11 Security and Confidentiality

Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace Analytical staff. Keyless door-lock combinations (and computer access codes/logins) are changed on a regular basis. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facilities must sign the Visitor's Logbook maintained by the receptionist and/or a staff member will accompany them during the duration of their stay on the premises. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. Prior to departure of the last staff member at the close of each day, the facility is checked for security.

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

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

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

All information pertaining to a particular client, including national security concerns will remain confidential. Data will not be released to outside agencies without written authorization from the client.

Figure 1.1

Corporate/Management Structure

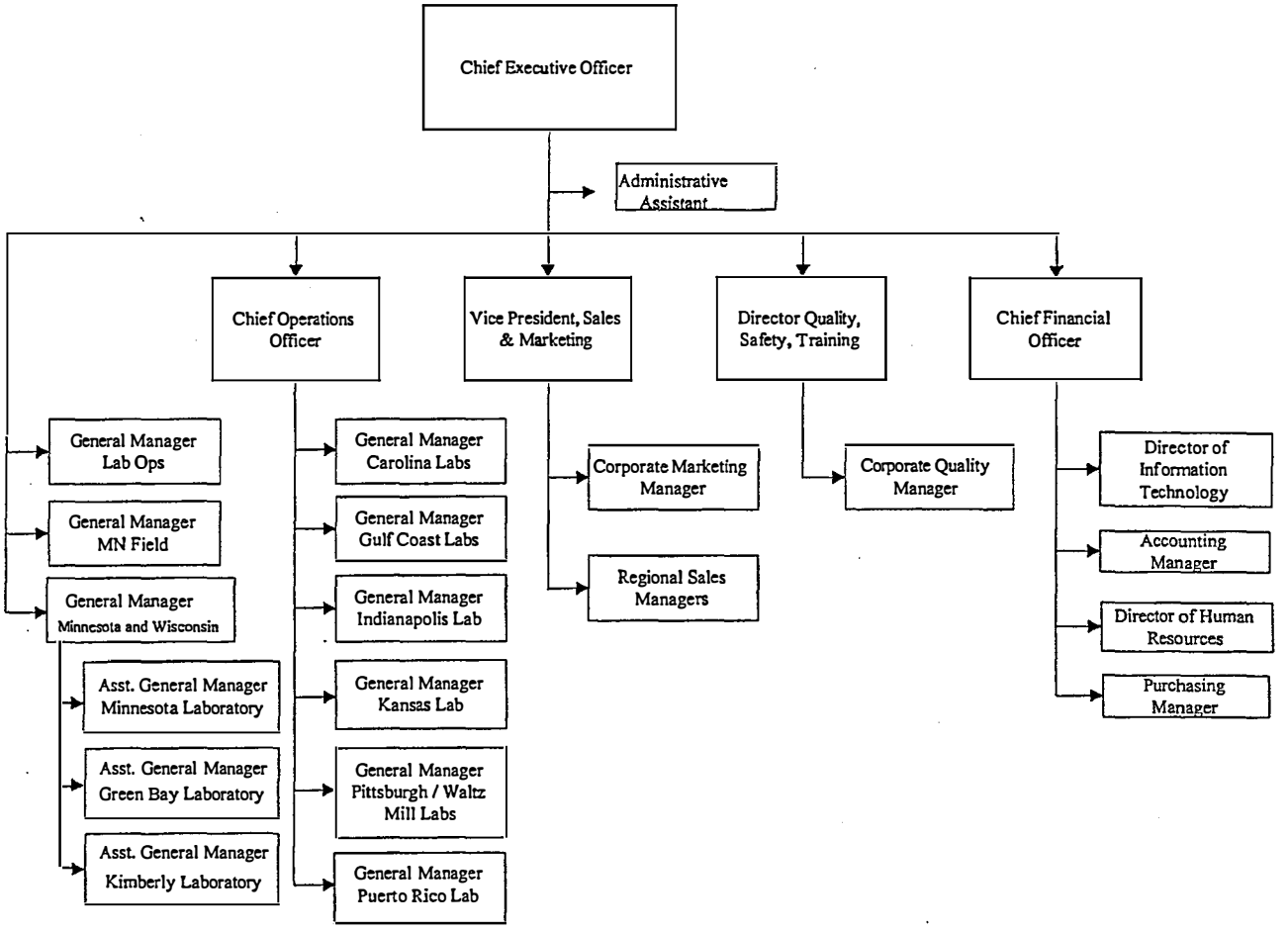
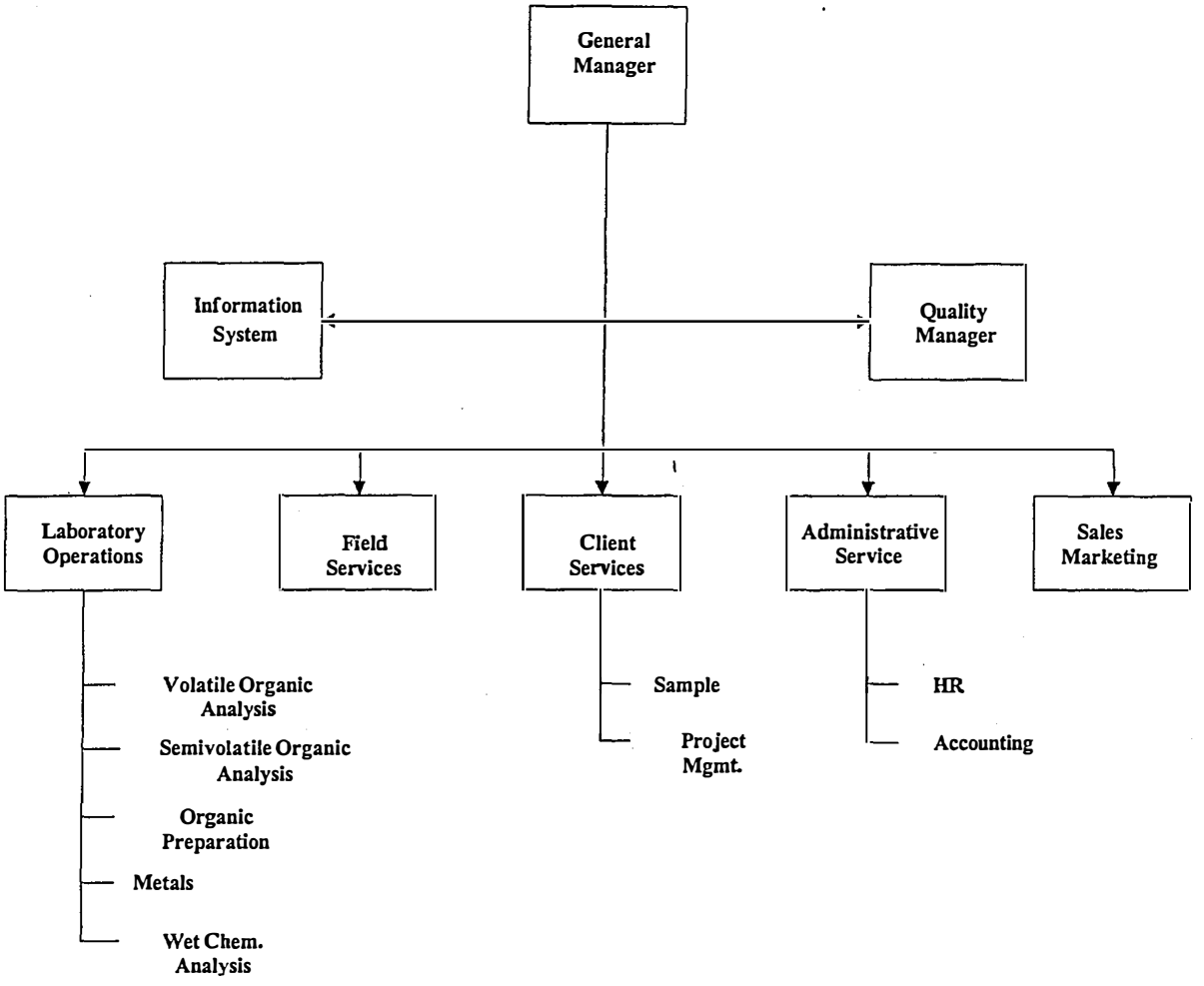


Figure 1.2

Typical Laboratory Organizational Chart



2.0 SAMPLE CUSTODY

Additional information can be found in SOP ALL-C-001 *Sample Management.*

2.1 Sampling Support

Each individual Pace Analytical laboratory provides shipping containers, sample containers (including applicable chemical preservatives), custody documents, and field quality control samples (e.g., trip blanks) to support field-sampling events. Tables 2.1, 2.2, 2.3 and 2.4 list general guidelines for sample container types, preservatives and holding times for a variety of methods. Note that all analyses listed are not necessarily performed at all Pace Analytical locations and there may be additional laboratory analyses performed that are not included in these tables. Pace laboratories may provide pick-up and delivery services to their clients when needed.

2.2 Project Initiation

Prior to accepting new work, the lab reviews its capability to perform this work. The purpose of this review is to establish that the lab has sufficient resources (personnel, equipment capacity, analytical method capability, etc.) to complete the required work. Once client needs and data quality objectives are defined, client services personnel or sales representatives contact the laboratory management. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, laboratory certifications and projected sample load. Management will then inform the sales and client services personnel whether the lab can accept the new projects. This communication is preferably via written correspondence or email, although it may occur during daily operations meetings.

The laboratory maintains records of all such reviews, including discussions with clients. The lab also maintains records of sub-contracted work and keeps a file of all sub-contractor information including current certifications.

2.3 Sample Acceptance Policy

In accordance with regulatory guidelines, Pace Analytical Services has compiled the following sample acceptance policy for all samples received at our laboratories.

If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory shall document all non-compliances and contact the client and either reject the samples or fully document any decision to proceed with the analyses of samples which do not meet these criteria. Any results reported from samples not meeting these criteria will be appropriately qualified on the final report.

All samples must:

- Have unique client identification that are clearly marked with durable waterproof labels on the sample containers and that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature
- Have the requested analyses clearly marked
- Have clear documentation of any special analysis requirements (data deliverables, etc.);
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless method allows for laboratory preservation.

- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior client permission.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without client approval.
- Be received within appropriate temperature ranges, (for samples requiring cooling to 4 °C, the acceptable range is just above freezing to 6°C, as defined by NELAC). The cooler temperature is recorded directly on the COC. Samples that are delivered to the lab immediately after collection are considered acceptable if there is evidence that the chilling process has been started, for example by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the client will be notified. The analysis will NOT proceed unless otherwise directed by the client. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the client is contacted to avoid missing the hold time. Data will be appropriately qualified on the final report.

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

2.4 Chain-Of-Custody

A chain-of-custody (COC) (see figure 2.1) document provides the legal documentation of samples from time of collection to completion of analysis. It is important that these documents be as complete as possible. Pace Analytical has implemented standard operating procedures to ensure that sample custody objectives of traceability and responsibility are achieved for every project.

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

If sample shipments are not accompanied by the correct documentation, the Sample Receiving department will notify the Project Manager. The Project Manager is then responsible for obtaining the correct documentation/information from the client so that analysis of samples can proceed.

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

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

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

2.5 Sample Receipt

Sample receiving personnel inspect each sample shipment upon arrival. The following items are checked:

- Presence of custody seals or tapes on the shipping containers
- Presence of Chain-of-Custody or similar documentation
- Presence of sample tags or labels
- Agreement between the sample tags or labels, Chain-of-Custody, and any client documentation.
- Condition of the samples when received, including:
  - Sample temperature: samples are acceptable if the arrival temperature is within 2°C of the required temperature, except as specified in the applicable test method or other state or federal regulation. Samples that are hand-delivered directly from the field on the same day that they are collected are acceptable if there is evidence that the chilling process has begun (arrival on ice).
  - Sample condition: Intact, broken/leaking
  - Headspace in VOA vials
  - Sample holding time
  - Sample pH when required
  - Adequate sample volume
  - Appropriate containers/preservatives

If discrepancies are found, the Pace Analytical Project Manager is contacted immediately. If the Project Manager is not available, the Quality Manager is contacted for further directions. Discrepancies are documented and reported with analytical results.

2.6 Sample Log-in

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

Sample data must include, at a minimum:

- Client name and contact
- Client number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested

All samples received are logged into the LIMS system within one working day of receipt. Sample login may be delayed due to client clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances.

All sample containers are assigned a unique laboratory identification code that is unequivocally linked to the field identification code. This code is placed on the sample container as a durable label.

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

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

2.7 Sample Storage

2.7.1 Storage Conditions

Samples are stored away from all standards, reagents, food or other potential sources of contamination. Samples are stored in a manner that prevents cross-contamination (e.g. volatile samples are stored separate from other samples). All sample fractions, extracts, leachates and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method

2.7.2 Temperature Monitoring

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

The temperature of each refrigerated storage area is maintained at 4°C ± 2°C unless state or program requirements differ. The temperature of each freezer storage area is maintained at <0°C unless state or program requirements differ. The temperature of each storage area is monitored and recorded each workday. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

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

2.7.3 Hazardous Materials

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

2.7.4 Foreign Soils

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

2.8 Sample Protection

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

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

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

2.9 Subcontracting Analytical Services

Additional information can be found in SOP ALL-Q-017 ***Subcontracting Samples.***

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

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

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

The contact and preliminary arrangements are made between the Pace Analytical Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

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

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

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

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

Any Pace Analytical work sent to other labs within the Pace network is handled as subcontracted work (also known as inter-regional) and all final reports are labeled clearly with the name of the laboratory performing the work.

2.10 Sample Retention and Disposal

Additional information can be found in SOP ALL-S-002 *Waste Handling*.

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

Unused portions of samples are retained by each laboratory based on program or client requirements for sample retention and storage. The typical sample retention time is a minimum of 30 days past the submission of the report. Any Pace laboratory not following this retention/disposal time must have a specific policy for retention and disposal of samples in their Quality Manual addendum.

After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste.

The preferred method for disposition of hazardous samples is to return the excess sample to the client. It may not be feasible to return samples, or the client may require Pace Analytical to dispose of excess samples. In that case, Pace Analytical will arrange for proper disposal by an approved contractor.

Table 2.1 Inorganic Parameters in Aqueous Samples

Parameter	Method			Container	Volume Needed (mL)	Preservative	Max Hold Time
	EPA Water	Standard Methods	EPA Waste SW-846				
Acidity	305.1	2310B		P, G	100	4°C	14 Days
Alkalinity	310.1/310.2	2320B		P, G	250	4°C	14 Days
Anions by IC, including Br, Cl, F, NO_2, NO_3, PO_4, SO_4, SO_3	300.0		9056	P, G	100	4°C	By anion
Bacteria, Total Plate Count		9221D		WK/P	100	4°C, $Na_2S_2O_3$	24 Hours
BOD/cBOD	405.1	5210B		P, G	500	4°C	48 hours
COD	410.1/410.2/ 410.4	5220C		P, G	250	pH<2 H_2SO_4, 4°C	28 Days
Chloride	325.2/325.3	4500-Cl	9250/9251/9252	P, G	100	none required	28 Days
Chorine, Residual	330.1/330.5/ 330.2	4500-Cl		P, G	500	none required	Immediate
Color	110.3/110.2	2120B,C,E		P, G	250	4°C	48 Hours
Cyanide, Reactive			Chapter 7	P, G	100	none required	28 Days
Cyanide, Total and Amenable	335.2/335.3/335.4	4500-CN	9010/9012	P, G	500	4°C; pH>12 NaOH, ascorbic acid if chlorine present	14 Days (24 hrs if sulfide present)
Flashpoint/Ignitability			1010/1030	P, G	50	none required	28 Days
Fluoride	340.1/340.2	4500-Fl C,D		P	500	none required	28 Days
Hardness, Total ($CaCO_3$)	130.2/130.1	2340B or C		P, G	250	pH<2 HNO_3, 4°C	6 Months
Nitrogen, Ammonia	350.1/350.2/ 350.3	4500-NH_3		P, G	500	pH<2 H_2SO_4, 4°C	28 Days
Nitrogen, Kjeldahl	351.2/351.3	4500-N_{org}		P, G	1000	pH<2 H_2SO_4, 4°C	28 Days
Nitrogen, Nitrate	352.1/353.2/ 353.3	4500-NO_3		P, G	100	4°C	48 Hours
Nitrogen, Nitrite	354.1	4500-NO_2		P, G	100	4°C	48 Hours
Nitrogen, Nitrate & Nitrite	353.2	4500-NO_3		P, G	100	pH<2H_2SO_4, 4°C	28 Days
Nitrogen, Organic	351.3	4500-N_{org}		P, G	100	pH<2H_2SO_4, 4°C	28 Days
Odor	140.1	2150B		G	1000	4°C	24 Hours
Oil and Grease/HEM	1664A	5520B,D	9070	G	1000	pH<2 H_2SO_4, 4°C	28 Days
Oxygen, Dissolved	360.1	4500-D		G	500	none required	Immediate
pH	150.1/150.2	4500-H	9040/9041	P, G	100	none required	Immediate
Phenol, Total	420.1/420.2		9065/9066	G	1000	pH<2 H_2SO_4, 4°C	28 Days
Phosphorus, Orthophosphate	365.1/365.2/ 365.3	4500-P		P	100	Filter, 4°C	48 Hours
Phosphorus, Total	365.1/365.2/ 365.4	4500-P		P, G	100	pH<2 H_2SO_4, 4°C	28 Days
Silica, Dissolved	370.1	4500-Si D		P	100	4°C	28 Days
Solids, Total	160.3	2540B		P, G	100	4°C	7 Days
Solids, Total Volatile	160.4	2540E		P, G	100	4°C	7 Days
Solids, Total Dissolved	160.1	2540C		P, G	100	4°C	7 Days
Solids, Total Suspended	160.2	2540D		P, G	100	4°C	7 Days
Solids, Settleable	160.5	2540F		G	1000	4°C	48 Hours
Specific Conductance	120.1	2510B	9050	P, G	100	4°C	28 Days
Sulfate	375.4/375.2	4500-SO_4	9035/9038	P, G	100	4°C	28 Days
Sulfide, Reactive			Chapter 7	P, G	100	none required	28 Days
Sulfide, Total	376.1, 376.2	4500-S	9030	P, G	500	pH>9 NaOH and ZnOAc	7 Days
Sulfite	377.1	4500-SO_3		P, G	500	none required	Immediate
Surfactants	425.1	5540C		P, G	250	4°C	48 Hours
Total Organic Carbon (TOC)	415.1/415.2	5310B,C,D	9060	G	100	pH<2 H_2SO_4 or HCl,4°C	28 Days
Total Organic Halogen (TOX)	450.1	5320	9020/9021	G, no headspace	500	4°C	14 Days
Turbidity	180.1	2130B		P, G	100	4°C	48 Hours
Metals (and other ICP elements)	200.7/200.8		6010/6020	P, G	500	pH<2 HNO_3	6 Months
Mercury	245.1		7470	P, G	250	pH<2 HNO_3	28 Days
Low Level Mercury	1631			G		BrCl, 4°C	90 days (if preserved and oxidized)
Hexavalent Chromium	218.4	3500-Cr	7196	P, G	500	4°C	24 Hours
Paint Filter Liquid Test			9095	P, G	100	none required	N/A
Ferrous Iron		3500-Fe-D		G	100	none required	Immediate

Table 2.2 Organic Parameters in Aqueous Samples

Parameter	Method			Container	Volume Needed (mL)	Preservative	Max Hold Time
	EPA Drinking Water	EPA Water	EPA Waste SW-846				
Aromatic and Halogenated Volatiles		601/602	8021	40-mL vial	3 vials	pH<2 HCl, 4°C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Volatiles	524.1/524.2			40-mL vial	3 vials	pH<2 HCl, 4°C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Volatiles		624		40-mL vial	3 vials	pH<2 HCl, 4°C, $Na_2S_2O_3$ if Cl_2 present	14 Days (7 unpreserved)
Volatiles			8260	40-mL vial	3 vials	pH<2 HCl, 4°C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Gas Range Organics			8015M	40-mL vial	3 vials	pH<2 HCl	14 Days
EDB & DBCP	504.1		8011	40-mL vial	3 vials	4°C, $Na_2S_2O_3$ if Cl_2 present	14 Days
Base/Neutrals and Acids		625	8270	G	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.1/525.2			G	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	7/30 Days
Organochlorine Pesticides and PCB's		608	8081/8082	G	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Organophosphorous Pesticides			8141	G, amber	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Polynuclear Aromatic Hydrocarbons		610	8310	G	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	7/40 Days
Chlorinated Herbicides	515.1		8151	G, amber	1000	4°C, $Na_2S_2O_3$ if Cl_2 present	14/28 Days**
Haloacetic Acids	552.1/552.2			40-mL vial, amber	3 vials	NH_4Cl, 4°C	14/7 Days
Diesel Range Organics			8015M	G	1000	4°C	7/40 Days
Explosives			8330	G	1000	4°C	7/40 Days
2, 3, 7, 8-TCDD	1613B			G	1000	none required	90/40 Days
Methane, Ethane, & Ethene			3810M	20-mL vial	3 vials	pH<2 HCl, 4°C	14 Days

Table 2.3 Inorganic and Organic Parameters in Solid Samples

Parameter	EPA Method	Container	Weight Needed (g)	Preservative	Max Hold Time
Metals	6010 or 6020	G	100	4°C	6 months
Mercury	7471	G	100	4°C	28 days
Aromatic and Halogenated Volatiles	5035/8021	5035 vial kit	1 kit	See Note	14 days
Volatiles	5035/8260	5035 vial kit	1 kit	See Note	14 days
Gasoline Range Organics	5035/8015M	5035 vial kit	1 kit	See Note	14 days
Base/Neutrals and Acids	8270	G	100	4°C	14/40 Days
Organochlorine Pesticides and PCBs	8081/8082	G	100	4°C	14/40 Days
Organophosphorous Pesticides	8141	G	100	4°C	14/40 Days
Polynuclear Aromatic Hydrocarbons	8310	G	100	4°C	14/40 Days
Chlorinated Herbicides	8151	G, amber	100	4°C	14/40 Days
Diesel Range Organics	8015M	G	100	4°C	14/40 Days
Explosives	8330	G	100	4°C	14/40 Days
2, 3, 7, 8-TCDD	1613B	G	100	none required	90/40 Days

Note: 5035 vial kit contains
2 vials water, preserved by freezing **or**
2 vials aqueous $NaHSO_4$, preserved at 4°C **and**
1 vial methanol preserved at 4°C **and**
1 vial unpreserved stored at 4°C

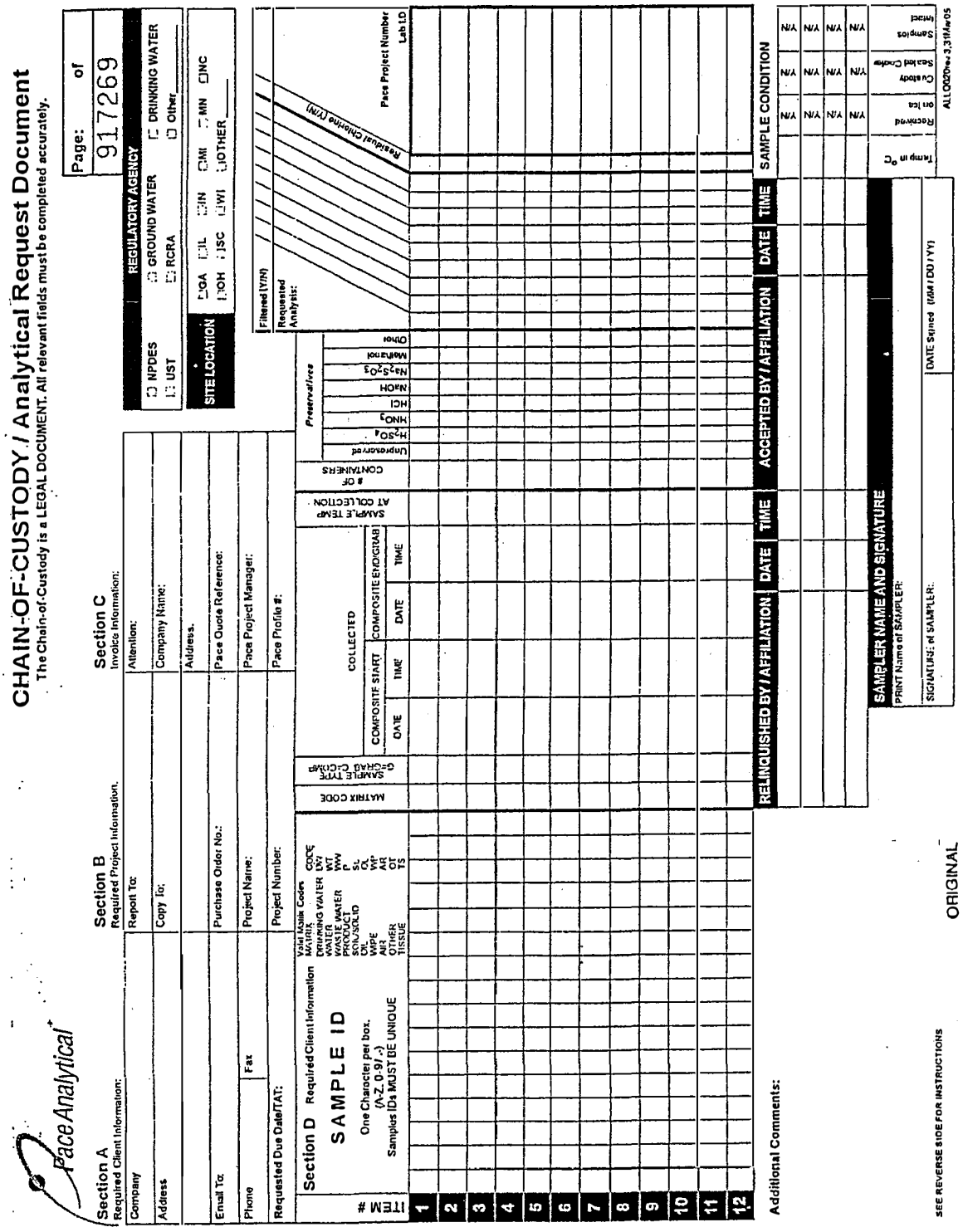
Table 2.3 Inorganic and Organic Parameters in Air Samples

Parameter	EPA Method	Container	Recommended Max Hold Time
Permanent Gases	3C	Summa Canister	14 Days
Permanent Gases	3C	Tedlar Bag	48 Hours
Methane, Ethane, Ethene	3C-M	Summa Canister	14 Days
Methane, Ethane, Ethene	3C-M	Tedlar Bag	48 Hours
Non-Methane Organics	25C	Summa Canister	14 Days
Non-Methane Organics	25C	TedlarBag	48 Hours
BTEX/Total Hydrocarbons	TO-3	Summa Canister	14 Days
BTEX/Total Hydrocarbons	TO-3	Tedlar Bag	48 Hours
Organochlorine Pesticides & PCBs	TO-4	PUF	7/40 Days
Dioxins & Furans	TO-9	PUF	30/45 Days
Polynuclear Aromatic Hydrocarbons	TO-13	PUF	7/40 Days
Volatiles	TO-14	Summa Canister	14 Days
Volatiles	TO-14	Tedlar Bag	48 Hours
Volatiles	TO-15	Summa Canister	14 Days
Ozone Precursors	TO-15	Summa Canister	14 Days
Particulates	PM10	Filters	6 Months
Metals	IO-3.5	Filters	6 Months
Stationary SourceParticulates	5	Filter/Solutions	6 Months
Lead Emissions	12	Filter/Solutions	6 Months
Stationary Source Dioxins & Furans	23	XAD Trap	30/45 Days
Stationary Source Metals	29	Filters	6 Months, 28 Days for Hg
Stationary Source Mercury	101	Filters	6 Months, 28 Days for Hg
Stationary Source PM10	201A	Filters	6 Months
Condensable Particulate Emissions	202	Solutions	6 Months
Hydrogen Halide & Halogen Emissions	26	Solutions	6 Months

Table 2.4 Rad Chem Parameters

Parameter	Method			Container	Volume Needed (mL)	Preservative	Max Hold Time
	EPA Water	Standard Methods	EPASW-846				
Gross Alpha and Gross Beta	900.0		9310	P, G	1000	pH<2 HNO_3	180days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6			P, G	1000	pH<2 HNO_3	48 Hrs
Gamma Emitting Radionuclides	901.1			P, G	1000	pH<2 HNO_3	180days
Alpha Emitting Radium Isotopes	903.0		9315	P, G	1000	pH<2 HNO_3	180days
Radium-226 Radon Emanation Technique	903.1			P, G	1000	pH<2 HNO_3	180days
Radium-228	904.0		9320	P, G	1000	pH<2 HNO_3	180days
Radioactive Strontium	905.0			P, G	1000	pH<2 HNO_3	180days
Tritium	906.0			G	1000	pH<2 HNO_3	180days
Uranium Radiochemical Method	908.0	D5174-97		P, G	1000	pH<2 HNO_3	180days

Figure 2.1 CHAIN-OF-CUSTODY FORM (Example)



3.0 ANALYTICAL CAPABILITIES

3.1 Analytical Method Sources

Pace Analytical laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH and, State agencies. Section 11 is a representative listing of general analytical protocol references. In some situations, Pace Analytical develops and validates methodologies that may be more applicable to a specific problem or objective. Pace Analytical discloses in writing to its clients and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

3.2 Analytical Method Documentation

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

The SOPs may be supplemented by Work Processing and Training Documents that further detail how methods are specifically performed with detailed training information.

3.3 Analytical Method Validation

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

3.4 Demonstration of Capability (DOC)

Analysts complete an initial demonstration of capability (IDOC) study prior to starting a method or when there is a change in instrument type, personnel or test method (when a defined 'work cell' is in operation, the entire work cell must meet the criteria). The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard (analyzed at 1-4 times the Limit of Quantitation), is calculated and compared to method criteria (if available) or established lab criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability, and corresponding raw data, for future reference and must document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are renewed on an annual basis.

Additional information can be found in SOP ALL-Q-020 ***Training Procedures.***

3.5 Regulatory and Method Compliance

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

Pace Analytical will make every effort to detect regulatory or project plan inconsistencies as they arise and communicate them to the clients to aid in the decision-making process.

It is Pace Analytical policy to disclose in a forthright manner any detected noncompliance that may affect the usability of data produced by our laboratories.

4.0 QUALITY CONTROL PROCEDURES

4.1 Data Integrity System

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

- A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics within this training include:
  - Need for honesty in analytical reporting
  - Process for reporting data integrity issues
  - Specific examples of unethical behavior and improper practices
  - Documentation of non-conforming data that is still useful to the data user
  - Consequences and punishments for unethical behavior
  - Examples of monitoring devices used by management to review data and systems
- Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual
- In-depth, periodic monitoring of data integrity: including peer data review and validation, internal data audits, proficiency testing studies, etc.
- Documentation of any review or investigation into possible data integrity infractions. This documentation must be available for review for lab assessors.

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

The management also provides a mechanism for confidential reporting of data integrity issues that includes confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern.

4.2 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system. The method blank is processed through all preparation and analytical steps with its associated samples. Any affected sample associated with a contaminated method blank will be re-analyzed if possible or reported with an appropriate data qualifier.

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

The method blank consists of a matrix similar to the associated samples that is known to be free of the analytes of interest.

Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater then 1/10 of the amount of that analyte found in any associated sample. Corrective actions may include re-analyzing the samples with a clean blank or reporting the data with the appropriate data qualifiers.

4.3 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis. The LCS results are compared to established acceptance criteria and if the results are outside of the criteria, then the system is out-of-control. Any affected sample associated with a failing LCS will be re-analyzed if possible or reported with an appropriate data qualifier.

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

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

The LCS contains all analytes specified by a specific method or by the client or regulatory agency. In the absence of specified components, the lab will spike with the following compounds:

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

The LCS is evaluated against the method or laboratory-derived acceptance criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier.

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

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

A matrix spike can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria (this is a NELAC allowance). When this happens, full documentation must be made available to the data user.

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

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

A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific client. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 client samples per matrix per method.

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

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

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

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

4.5 Surrogates

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

Surrogates are added to each client sample (for organics), method blank, LCS and MS prior to extraction or analysis. The surrogates are evaluated against the method or laboratory-derived acceptance criteria. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results.

4.6 Sample Duplicate

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

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

4.7 Internal Standards

Internal Standards are analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

4.8 Field Blanks

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

4.9 Trip Blanks

Trip blanks are blanks that are prepared in the laboratory before the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the lab. These blanks are routinely analyzed for volatile sample methods.

4.10 Limit of Detection (LOD)

Additional information can be found in SOP ALL-Q-004 ***Method Detection Limit Studies.***

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

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

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

4.11 Limit of Quantitation (LOQ)

Additional information can be found in SOP ALL-Q-004 ***Method Detection Limit Studies.***

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

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

4.12 Proficiency Testing (PT) Studies

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

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

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

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

Additional information can be found in SOP ALL-Q-010 ***PE/PT Program.***

5.0 DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1. Document Management

Additional information can be found in SOP ALL-Q-002 ***Document Management***.

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

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

Each document related to the quality system is uniquely identified to include the date of issue, the revision identification, page numbering, the total number of pages and the issuing authorities. For complete information on document numbering, refer to the company-wide Standard Operating Procedure Document Numbering (ALL-Q-003).

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

- Ready accessibility to all laboratory staff
- A complete description of the computerized aspects, including security
- A provision to explicitly indicate that all printed copies are uncontrolled and expire on the date printed.

5.1.1 Quality Manual

The Quality Manual is the company-wide document that describes all aspects of the quality system for Pace Analytical laboratories. It is document-controlled by the corporate quality office and signed copies are distributed to each of the regional Quality Managers. The regional management personnel sign the Quality Manual and the local Quality Manager is then in charge of distribution to employees and external clients or regulatory agencies and maintaining a controlled list of distributed copies. Each laboratory may attach a lab-specific addendum to the Quality Manual as needed. The Quality Manual is reviewed on an annual basis by all of the Pace Quality Managers and revised accordingly.

5.1.2 Standard Operating Procedures (SOPs)

SOPs fall into two categories: company-wide documents (starting with the prefix ALL-) or individual lab documents (starting with the individual lab abbreviation). Company-wide SOPs (ALL SOPs) are document-controlled by the corporate quality office and signed copies are distributed to each of the laboratory Quality Managers. Laboratory management personnel sign the company-wide (ALL) SOPs and the local Quality Manager distributes to employees and external clients or regulatory agencies and maintains a controlled list of distributed copies. Each laboratory may attach a lab-specific addendum to any of the company-wide (ALL) SOPs as needed. Individual lab specific SOPs are controlled by the local Quality Manager in the same manner.

SOPs are reviewed every two years at a minimum (a more frequent review may be required by state or federal agencies or clients). Documentation of this review and any applicable revisions are made in the last section of each SOP. This provides a historical record of all revisions.

All copies of superseded SOPs are removed from general use and at least one copy of each SOP is archived for audit or knowledge preservation purposes. This not only ensures that all Pace employees use the most current version of each SOP but also supplies the Quality Manager with a historical record of each SOP.

Additional information can be found in SOP ALL-Q-001 ***Preparation of SOPs***.

5.1.3 Training documents

The training documents are more detailed documents that describe lab-specific details such as computer and equipment set-up, sample preparation and analysis steps, data validation steps, documentation requirements, and data packet preparation. Documentation that an analyst has completed their training via these training documents is maintained in their training file.

5.2 Document Change Control

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

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

6.0 EQUIPMENT AND MEASUREMENT TRACEABILITY

Additional information can be found in SOP ALL-Q-013 *Support Equipment.*

Each Pace lab is equipped with instrumentation and support equipment to perform the required analyses. Support equipment includes chemical standards, thermometers, balances, pipettes, etc. This section will detail some of this equipment and instrumentation and the procedures necessary for its proper calibration.

6.1 Standards and Traceability

Laboratories must retain all pertinent information for all standards, reagents and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation and use.

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

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

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

If a second source standard is required to verify an existing calibration or spiking standard, this standard is purchased from a different supplier. If no second source is available, a second standard may be purchased from the same supplier but the lab is required to receive a certificate of warranty or similar documentation noting that both standards were prepared from different raw materials. Obtaining two standards from the same parent material is not acceptable for satisfying this requirement.

6.2 General Calibration Procedures

All types of support equipment and instrumentation are calibrated before use to ensure that they function properly. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards, or reference standards whose values have been statistically validated. Instrumentation or support equipment that cannot be calibrated to specifications, or is otherwise defective, is taken out of service. They are clearly labeled as out-of-service until they have been repaired and tested to meet specifications. In the event that recalibration of a piece of test equipment casts doubt on the validity of test results already transmitted to the client, the client is notified in writing by the laboratory within 3 business days from the time of discovery. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or replaced.

Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers (e.g. J flag) or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be

reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers (e.g. E flag) or explained in the narrative.

Calibration standards are prepared at a minimum of three concentration levels for inorganic analyses and a minimum of five concentrations for organic analyses. A calibration blank is also included for some inorganic analyses. Any specific method requirement for number and type of calibration standards supersedes the general requirement.

Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial curve.
During the course of analysis, the calibration curve is verified by the analysis of a calibration verification standard. This verification standard must also pass acceptance criteria for sample analysis to proceed. Concentrations of calibration verification standards must be varied periodically to evaluate the entire range of the initial calibration curve.

6.3 Calibration Procedures for GC/MS (Gas Chromatograph/ Mass Spectrometer)

More detailed calibration information can be obtained from Pace Standard Operating Procedures or other similar documentation (e.g. SOP ALL-O-001 ***GC/MS Semi-volatiles***).

6.3.1 GC/MS Tuning

The first step in preparing a GC/MS instrument for sample analysis is to tune the instrument. This is accomplished through the analysis of 4-bromofluorobenzene (BFB) for volatile analysis or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. The tune standard must pass the method acceptance criteria for mass spectral abundance for these compounds before calibration standards are evaluated.

6.3.2 Initial Calibration

The GC/MS system is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. The number of calibration standards used depends on the specific method criteria or client project requirements, although normally a minimum of five standards is used.

The response factor (RF) for each compound at each concentration is calculated and an average RF is obtained for each compound in the initial calibration curve. The percent relative standard deviation (%RSD) is calculated for each compound. This RSD value should be $\leq$ 15% for each reported compound and must be $\leq$ 30% for those compounds in the method identified as calibration check compounds (CCCs). For compounds with an RSD >15%, a least squares regression can be applied (the best fit value must be >0.99 to confirm linearity) or data for these compounds must be reported with the appropriate data qualifiers. A non-linear calibration curve (quadratic) may be constructed if at least 6 standards are included in the initial calibration.

The mean RF from the initial calibration curve is used to calculate the sample concentration of the compound of interest when the %RSD demonstrates linearity. Otherwise, the sample concentration is determined using the calibration curve equation generated from the initial calibration standards.

6.3.3 Calibration Verification

A calibration verification standard is analyzed with each analytical batch to verify that the initial calibration is still valid. This standard must contain all target analytes and is typically analyzed at a concentration around the midpoint of the initial calibration curve. For linear calibration using the

average response factor, the RF data from this standard is compared to the average RF from the initial calibration. Other calibration models compare the calculated concentration of the calibration verification standard to the expected concentration of the calibration verification standard. If the % Difference is greater than 20% then either the calibration verification standard must be re-analyzed with acceptable results or any data reported for those compounds that exceeded the acceptance criteria are reported with the appropriate data qualifiers.

6.4 Calibration Procedures for GC and HPLC (Gas Chromatograph and High Performance Liquid Chromatograph)

More detailed calibration information can be obtained from Pace Standard Operating Procedures or other similar documentation (e.g. SOP ALL-O-006 ***Organochlorine Pesticides*** and SOP ALL-O-007 ***Polychlorinated Biphenyls***).

6.4.1 Initial Calibration

The GC or HPLC system is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. The number of calibration standards used depends on the specific method criteria or client project requirements, although normally a minimum of five standards is used.

The response factor (RF) for each compound at each concentration is calculated and an average RF is obtained for each compound in the initial calibration curve. The percent relative standard deviation (%RSD) is calculated for each compound. This RSD value should be $\leq$ 20% for each reported compound. For compounds with an RSD >20%, a least squares regression can be applied (the best fit value must be >0.99 to confirm linearity) or data for these compounds must be reported with the appropriate data qualifiers.

The mean RF from the initial calibration curve is used to calculate the sample concentration of the compound of interest.

6.4.2 Calibration Verification

A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid (this standard is also run at the end of each batch). This standard must contain all target compounds except for multi-component analytes where a representative substance or mixture may be used. The calibration verification standard is typically analyzed at a concentration around the midpoint of the initial calibration curve. The RF data from this standard is compared to the average RF from the initial calibration. If the RF from any compound differs from the average RF from the initial calibration by more than ±15%, then either the calibration verification standard must be re-analyzed or any data reported for those compounds that exceeded the acceptance criteria are reported with the appropriate data qualifiers. Reported sample results must be bracketed by acceptable calibration verification standards.

6.5 Calibration Procedures for ICP, ICP/MS and AAS (Inductively Coupled Plasma, Inductively Coupled Plasma/Mass Spectrometer and Atomic Absorption Spectrometer)

More detailed calibration information can be obtained from Pace Standard Operating Procedures or other similar documentation (e.g. SOP ALL-M-002 ***ICP Metals***)

6.5.1 Initial Calibration

The ICP, ICP/MS or AAS system is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. The number of calibration standards used depends on the specific method criteria or client project requirements, although normally a minimum of three standards is used (ICP and ICP/MS calibration can be performed with a single standard and a calibration blank if annual linear range studies are conducted).

6.5.2 Calibration Verification

A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid (this standard is also run at the end of each batch). If the response from this standard differs from the initial calibration standard by more than 10% for ICP and ICP/MS or by more than 20% for AAS, then the instrument must be re-calibrated before proceeding with sample analysis. A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system.

Interference check standards are also run per method requirements at the beginning and end of each batch and must pass method acceptance criteria.

6.6 General Equipment Calibration Procedures

6.6.1 Analytical Balances

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

6.6.2 Thermometers

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

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

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

6.6.3 pH/Electrometers

The meter is calibrated before use each day, and once after each four hours of continuous use using fresh buffer solutions.

6.6.4 Spectrophotometers

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

6.6.5 Pipettes

Mechanical hand pipettes are calibrated on a quarterly basis.

6.7 Instrument/Equipment Maintenance

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

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

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

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

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

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

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

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

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

6.8 Spare Parts

Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that:

- Are subject to frequent failure,
- Have limited useful lifetimes, or
- Cannot be obtained in a timely manner should failure occur.

7.0 CONTROL OF DATA

Analytical results processing, verification and reporting are the processes that result in the delivery of defensible analytical data to the data user. These processes include calculation of raw data into final concentration units, reviewing results for accuracy and assembly of the technical report for delivery to the data user.

All analytical data undergo a well-defined, well-documented multi-tier review process before being reported to the client. The following information describes procedures employed at Pace Analytical for translating raw analytical data into accurate, finished sample reports and describes data storage policies.

7.1 Analytical Results Processing

When "raw data" is manually generated by an analyst, it is recorded in either a bound notebook (run logbook), or copies of the computer printouts are appropriately labeled and filed. Logbooks and other bench records are kept in accordance with each laboratory's Standard Operating Procedure on documentation practices (this may also include the use of electronic logbooks). The primary analyst is responsible for the initial reduction and review of the data. This includes confirming compliance with required methodology; checking the calculations used; checking quality control data against known criteria; noting any discrepancies that occurred both in the necessary logbooks and as a footnote or narrative in LIMS; and entering analytical data into the LIMS system. The primary analyst then compiles the initial data package for data verification. This compilation must include sufficient documentation to review the data. It may include chromatograms or strip-chart recordings, before and after printouts of manual integrations, other computer printouts, chain-of-custody copies if available, and logbook copies. Some agencies or clients require different levels of data reporting. For these special levels, the primary analyst may also need to compile additional project information such as initial calibration data or extensive spectral data before the data package goes to the verification step.

7.2 Data Verification

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

Analysts performing the analysis and subsequent data reduction have the primary responsibility for the quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data provided QC criteria have been met for the samples being reported. Data review checklists are used to document the data review process.

The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. All data that are manually entered into the LIMS is reviewed at a rate of 100%. This involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer also reviews analyst-generated calculations.

For results that are processed via computer, calculations are checked by the analyst (or designee) assigned to this task at a frequency designed to assure that the data reductions are valid. The results are either manually transferred to a standard reporting form or reported via computer generation of forms.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data.

The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

Use of checklists ensures that all data are systematically handled and no steps are omitted. Checklists are reviewed, retained, and made accessible should they need to be referenced at a later date.

7.3 Data Reporting

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

Final reports are prepared according to the level of reporting required by the client. A standard Pace Analytical final report consists of the following components:

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

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

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

7.4 Data Archiving

All records compiled by Pace Analytical labs are maintained, stored and secured by the Quality Manager or by a designated Data Archivist for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. These records can include client data reports, certificates pertaining to calibration and maintenance of equipment, raw data from instrumentation, quality control documents and logbooks. These records are retained in order to provide for possible historical reconstruction of data. Access to archived data is kept to a minimum, with the Data Archivist maintaining the archive documentation in a secure, fireproof (if possible) location. Some laboratories archive their data in an off-site facility and the Data Archivist will keep a record of this archival as well. Records that are computer-generated have either a hard copy or electronic backup copy.

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

7.5 Resolution of Client Complaints or Questions

Pace Analytical is committed to providing superior service to our customers including cooperation to clarify and resolve questions or complaints pertaining to completed analytical work. Each Pace Analytical laboratory maintains written or electronic documentation of questions and complaints received from clients or the client's authorized representative regarding work performed. The resolution or answers to the questions and complaints are also included in the documentation. In the event that an error is found when investigating the question or complaint, a revised or supplemental report is issued as necessary.

8.0 QUALITY SYSTEM AUDITS AND REVIEWS

In an effort to assess the effectiveness of the Quality Systems, all Pace Analytical laboratories are subject to internal and external audits and reviews.

8.1 Internal Audits

8.1.1 Responsibilities

The Quality Manager is responsible for designing and/or conducting internal audits. Since internal audits represent an independent assessment of laboratory functions, the auditor must be functionally independent from laboratory operations to ensure objectivity. The auditor must be familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The Quality Manger evaluates audit observations and verifies the completion of corrective actions. In addition, an annual corporate audit is conducted by the Director of Quality, Safety & Training and/or designee.

8.1.2 Scope and Frequency of Internal Audits

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

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

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

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

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

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

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

8.1.3 Internal Audit Reports and Corrective Action Plans

Additional information can be found in SOP ALL-Q-011 ***Audits and Inspections***.

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

Once completed, the internal audit report is issued jointly to the Laboratory General Manager and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds with a plan to correct all of the deficiencies cited by the due date specified in the audit report. Each response must include timetables for completion of all proposed corrective actions.

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

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

8.2 External Audits

Pace Analytical laboratories are audited regularly by regulatory agencies, to maintain laboratory certifications, and by clients to maintain appropriate specific protocols.

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

The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the Quality Manager. The Laboratory General Manager provides the necessary resources for staff to develop and implement the corrective action plans. The Quality Manager collates this information and provides a written report to the audit team. The report contains the corrective action plan and expected completion dates for each element of the plan. The Quality Manager follows-up with the laboratory staff to ensure corrective actions are implemented.

8.3 Quarterly Quality Reports

Additional information can be found in SOP ALL-Q-014 ***Quality System Review***.

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

- Results of internal systems or performance audits
- Corrective action activities
- Discussion of QA issues raised by clients

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

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

8.4 Annual Managerial Review

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

The managerial review must include the following topics of discussion:

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

This managerial review must be documented for future reference by the Quality Manager and copies of the report are distributed to laboratory staff.

9.0 CORRECTIVE ACTION

Additional information can be found in SOP ALL-Q-012 ***Corrective Action/Preventative Action Process***.

During the process of sample handling, preparation and analysis, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace Analytical provides systematic procedures for documentation and completion of corrective actions. This can be done using a Corrective Action Tracking Log that lists the deficiency by ID, along with the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

9.1 Corrective Action Documentation

The following items are examples of occurrences that warrant some form of documented corrective action:

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

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

The person who discovers the deficiency or non-conformance initiates the corrective action documentation. The documentation must also include the affected projects and sample numbers, the name of the applicable Project Manager, the client name and the sample matrix involved.

The person initiating the corrective action documentation must also list suspected or known causes of the deficiency or non-conformance as well as any corrective actions that they have taken. They would then sign and date the form and pass it to their immediate supervisor or the Project Manager. The supervisors and PMs add in their observations and further corrective actions, sign and date the form and pass to any other applicable lab employee. The Quality Manager is responsible for final review and signoff of all formal corrective action forms. A copy of the form is archived with each project and a copy is kept in the quality office.

9.2 Corrective Action Completion (Specific Examples)

9.2.1. Quality Control outside of acceptance criteria: the analyst that is generating or validating Analytical data is responsible for checking the results against established acceptance criteria (quality control limits). The analyst must immediately address any deficiencies discovered. Method blank, LCS or matrix spike failures are evaluated against method, program, and client requirements and appropriate footnotes are entered into the LIMS system. Some deficiencies may be caused by matrix interferences. Where possible, matrix interferences are confirmed by re-analysis. Quality control deficiencies must be made known to the client on the final report for their review of the data for usability. If appropriate, the supervisor is alerted to the QC failure and if necessary a formal

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

9.2.2. **Sample Acceptance Policy deviations:** any deviation from the Sample Acceptance Policy listed in this Manual must be documented on the Chain-of-Custody or other applicable form by the sample receiving personnel or by the Project Manager. The client must be notified of these deviations as soon as possible so they can make decisions on whether to continue with the sample analysis or re-sample. Copies of this documentation must be included in the project file. Analysts or supervisors that discover such deviations must contact the sample receiving personnel or appropriate Project Manager so they can initiate the proper documentation and client contact. If a more formalized corrective action must be documented, the Quality Manager should be made aware of the situation.

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

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

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

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

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

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

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

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

10.0 GLOSSARY

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity and Performance. Best Practices are identified that can be used by all Pace labs.
Accuracy	The agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	A portion of a sample taken for analysis.
Analyte	The specific chemical species or parameter an analysis seeks to determine.
Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	A sample for submitted for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test analyst or laboratory proficiency in the execution of the measurement process.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
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.
Calibration Curve	The graphic representation of known values, such as concentrations for a series of calibration standards and their instrument response.
Chain-of-Custody (COC)	A record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, collector, time of collection, preservation, and requested analyses.
Confirmation	Verification of the identity of a component through the use of an alternate scientific approach from the original method. These may include, but are not limited to: • second-column confirmation • alternate wavelength • derivatization derivative • mass spectral interpretation • additional cleanup procedures

Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Calibration Verification	The process of verifying a calibration by analysis of standards and comparing the results with the known amount.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of nonconforming data.
Corrective Action	The action taken to eliminate the causes of a nonconformity, defect, or other undesirable situation in order to prevent recurrence.
Data Quality Objective (DOQ)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate acceptable accuracy.
Detection Limit (DL)	General term for the lowest concentration or amount of the target analyte that can be identified, measured and reported with confidence that the analyte concentration is not a false positive value. See definitions for Method Detection Limit and Limit of Detection.
Document Control (Management)	Procedures to ensure that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled (managed) to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate or Replicate Analysis	The identically performed measurement on two or more sub-samples of the same sample within a short interval of time
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Surface Water and Ground Water • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.

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

Matrix	The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions are 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 a potable or potentially potable water source. • **Saline/Estuarine:** any aqueous sample from an ocean or estuary, or other saltwater source. • **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 sample can be grouped according to origin. • **Solids:** includes soils, sediments, sludges, and other matrices with >15% settleable solids. • **Chemical Waste:** a product or by-product or 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 vapor that are collected with a sorbent tube, impinger solution, filter, or other device.
Matrix Spike (MS)	A sample prepared by adding a known quantity of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency. (sometimes referred to as Spiked Sample or Fortified Sample)
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte. (sometimes referred to as Spiked Sample Duplicate or Fortified Sample Duplicate)
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures: and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Limit of Detection (LOD); defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.

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

Standard	A substance or material with properties known with sufficient accuracy to permit its use to evaluate the same property in a sample.
Standard Blank	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Operating Procedure (SOP)	A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks
Surrogates	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Traceability	The property of a material or measurement result defining its relationship to recognized international or national standards through an unbroken chain of comparisons.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory pure water; any preservative used in the sample is added and then the blank is stored, shipped, and analyzed with its group of samples.
Validation	The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	Confirmation by examination and provision of evidence that specified requirements for instruments have been met. 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.
Warning Limits	The limits (typically 2 standard deviations either side of the mean) shown on a control chart within which most results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.
Work Cell	A 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.
Working Range	The range of results between the Limit of Quantitation and the upper limit of measurement system calibration.

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UPPER MIDWEST REGION
QUALITY MANUAL ADDENDUM To Revision 9.1

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(Effective date of addendum is the last date signed above)

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TABLE OF CONTENTS

The information has been added to the corporate quality manual through this addendum for the Upper Midwest region:

1.0 INTRODUCTION & ORGANIZATIONAL STRUCTURE

1.1 Introduction to Pace

The Upper Midwest region of Pace Analytical Services has three analytical laboratories, performing a wide variety of tests. Its capabilities include:

- Volatiles and semi-volatiles by Gas Chromatography/Mass Spectrometry (GC/MS);
- Metals analysis by Inductively Coupled Plasma spectrometry (ICP) and ICP/MS;
- Pesticide, polychlorinated biphenyls (PCB), and volatile (BTEX) analyses by gas chromatography (GC);
- Dioxin/furan analysis by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) and LRMS
- Volatile and semivolatile air analysis by GC and GCMS
- Polyaromatic hydrocarbon analysis by High Pressure Liquid Chromatography (HPLC);
- Gasoline, diesel and Total Petroleum Hydrocarbon analyses by Gas Chromatography (GC);
- Biota analyses for all major tests
- Mercury by cold vapor analysis
- Low level mercury analysis by Method 1631E
- And numerous inorganic wet chemical analyses

NOTE: All methods are listed in Attachment III and may be subject to change prior to addenda update. The Quality Assurance Office maintains a complete list of each method and analyte approved by NELAC and pertinent state agencies.

The Upper Midwest region is capable of analyzing a variety of environmental and non-environmental samples from different media including surface, drinking and ground waters, air, biota, soils, sediments, waste products, sludge and concretes. All analyses follow written Standard Operating Procedures that are kept on file in the Quality Assurance office. A listing of the major equipment in each facility is available through the quality assurance office in each facility.

The upper Midwest region holds certifications with approximately 40 state and federal agencies. (A complete listing may be obtained through the quality assurance office.) Pace Minneapolis, Pace Green Bay and Pace Kimberly laboratories hold accreditation by NELAC, the National Environmental Laboratory Accreditation Conference, through the accrediting authority of the Florida Department of Health. Copies of the certificates and fields of testing lists may be forwarded to you from the Quality Assurance department.

This addendum, combined with the preceding corporate quality manual, comprises the complete Laboratory Quality Manual for Upper Midwest region of Pace Analytical Services. This addendum contains information specific to the Upper Midwest region, whereas the corporate section details practices and policies that are common to all the member Pace laboratories. The Regional Quality Manager when necessary updates the addendum.

1.7 Laboratory Organization

Figure 1.1 has been expanded to show the detail of the organizational structure in the Minnesota Laboratory (see Attachment I).

1.9 Training and Orientation

Refer to the Standard Operating Procedure *Training Procedures* (ALL-Q-020) for specific information on acceptable means of documenting initial and continuing demonstrations of capability.

1.10 Laboratory Safety

The designated safety/chemical hygiene officer is Melanie Ollila for the Minneapolis laboratory and Steve Mleczko for the Pace Green Bay and Kris Burns for the Pace Kimberly laboratories. All specific information on laboratory safety is located in each facility Safety Manual/Chemical Hygiene Plan.

1.11 Security and Confidentiality

All information pertaining to a particular client and their analytical results will remain confidential including national security concerns. Data will not be released to outside agencies without the written authorization from the client.

2.0 SAMPLE CUSTODY

2.1 Sampling Support

Additions to Table 2.1 through 2.4 include but may not be limited to:

Method/Matrix	Container	Preservative	Maximum Hold Time
CARB 429 W (HRMS PAH)	1 Liter	4°C	1 Year to Extraction*
CARB 429 S (HRMS PAH)	4 or 8 oz. jar	4°C	1 Year to Extraction*
CARB 429 T (HRMS PAH)	4 or 8 oz. jar	4°C	1 Year to Extraction*
SM9221D (Fecal-col)	100mL	4°C, 10% sodium thiosulfate	6 hours
SM9223B (Total-col)	100mL	4°C, 10% sodium thiosulfate	24 hours
1614 Water	1 Liter	4°C	1 Year to Extraction*
1614 Soil	4 or 8 oz. jar	4°C	1 Year to Extraction*
1614 Tissue	4 or 8 oz. jar	4°C	1 Year to Extraction*
1653 Water	2 Liter	4°C, pH<2 H_2SO_4	30 Days to Extraction; 30 days to analysis
1668 Water	1 Liter	4°C	1 Year to Extraction*
1668 Soil	4 or 8 oz. jar	4°C	1 Year to Extraction*
8015 (MeOH, eOH)	3 40 ml vials	HCl	14 Days
8280 Water	1 Liter	4°C	30 Days to Extraction*
8280 Soil	4 or 8 ounce	4°C	30 Days to Extraction*
8290 Solid	4 or 8 ounce	4°C	30 Days to Extraction*
8290 Water	1 Liter	4°C	30 Days to Extraction*
8290 Waste	2 ounce	4°C	30 Days to Extraction*
WI GRO Water	3 40 ml vials	HCl	14 Days
WI GRO Soil	5035 vial kit	See *Note*	14 Days
WI DRO Water	1 Liter	HCl	7 Days to Extraction*

WI DRO Soil	Tared 4 oz. jar	4°C	14 Days to extraction*
Method 23/TO9	Sampling Head	4°C	30 Days to Extraction*
Method 1631E	500mL Glass	None	Oxidized in bottle within 28 days.

**40 Days from Extraction to Analysis. (EPA 1613,WI DRO)*
45 Days from Extraction to Analysis (SW8290, 1668, 8280 and Method 23/TO9)
Note: 5035 kit contains 2 vials water, preserved by freezing or 2 vials aqueous $NaHSO_4$ preserved at 4°C and 1 vial MeOH preserved at 4°C and 1 vial unpreserved at 4°C.

2.3 Sample Acceptance Policy

For samples collected in the State of Wisconsin, Pace Green Bay will document whether the samples were received on ice (ROI) instead of documenting the temperature at the time of receipt.

2.4 Chain of Custody

Clarification: The external chain of custody (COC) is signed by the client for relinquishment of samples. The samples may be transported by the client or through the use of a ground or air carrier (e.g. Fed Ex, UPS, SpeeDee) The carrier will assign a tracking number to each cooler and this number may be traced. When the coolers are received at Pace Analytical Services, Inc., the COC is signed by sample management personnel to signify receipt.

2.5 Sample Receipt

All correspondence pertaining to samples not meeting the acceptance criteria will be fully documented and retained with the project file. All affected data will be footnoted appropriately in the final report.

2.6 Sample Log-in

The Minneapolis laboratory utilizes an "On-Hold" logbook for samples that require further clarification. The logbooks are associated with each sample refrigerator and freezer. The client ID, date, etc are entered into the book, the samples placed into the corresponding refrigerator or freezer, and the project manager associated with the affected client is contacted. The Wisconsin laboratories utilized a master log that is filled in by hand to assign a unique project number to projects. WI sample labels are printed form a program separate from LIMS using the Project Number assigned in the Master Log.

2.7 Sample Storage/Staging

2.7.2 Temperature Monitoring

The Wisconsin laboratories take the samples to the storage locations after the sample containers have been labeled and prior to entry in the LIMS.

Refrigerators are at 4°C ± 2°C unless state or program requirements differ.

2.7.2.1 Freezer Monitoring

All freezers are maintained at <0°C unless otherwise noted in analytical method SOPs. The temperature is monitored and recorded each working day. If the

temperature is greater than 0°C, corrective action is taken and appropriately documented.

2.9 Subcontracting Laboratory Services

Unless holding times will be affected and client is not available for resolution, the client will be advised in writing prior to shipment of subcontracted samples. All laboratories that perform subcontracted work shall provide documentation regarding certification status with state and federal agencies. This documentation is retained in the Quality Assurance Office.

The chain of custody forms generated will not contain any specific client information to protect client confidentiality.

2.10 Sample Disposal

Air canisters will be retained for 14 days after submission of the analytical report. At that time, the cans will be cleaned and certified per the standard operating procedures unless otherwise stated by the project contract or client request.

3.0 ANALYTICAL CAPABILITIES

3.4 Demonstration of Capability (DOC)

Alternate procedures than those outlined in NELAC Quality System Chapter 5 (current standard) are referenced in the corporate standard operating procedure, ALL-Q-020 (*Training Procedures*).

4.0 QUALITY CONTROL PROCEDURES

4.2 Method Blank

The State of Wisconsin requires the concentration of any target analyte detected in the method blank to be less than 1/20 (5%) of the amount found in any associated sample.

4.3 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

4.9 Trip Blanks

The collection date of a trip blank is generally the collection date of the associated samples. The closer the collection date of a trip blank is to its preparation date, the more reliable and valuable is the data obtained by analyzing it. In order to ensure the relevancy of trip blank data, Pace confirms that trip blanks are contaminant free prior to shipment to the client and recommends that trip blanks always accompany original bottle orders back to Pace for analysis. Identifications in such trip blanks can probably be ascribed to contamination during collection or transport.

4.10 Limit of Detection (LOD)

Further information on detection limit studies are outlined in the corporate SOP ALL-P-104 (*Method Detection Limit Studies*) and the relationship to the Limit of Quantitation.

4.11 Limit of Quantitation (LOQ)

The state of Wisconsin requires a 10/3 relationship between the Limit of Detection (LOD) and the Limit of Quantitation (LOQ).

5.0 DOCUMENT CHANGE CONTROL

5.1 Document Management

The Upper Midwest has no further changes to this section of the corporate quality manual. More detailed information on document change control may be found in MN-Q-229 (*Document Change Control*).

5.1.1 Quality Manual

The Upper Midwest Region has developed a single regional addendum instead of each laboratory attaching a lab-specific addendum.

6.0 EQUIPMENT AND CALIBRATION

6.2 General Calibration Procedure

The State of Wisconsin requires laboratories to report to the MDL (LOD) for m ny compounds. Results between the LOD and LOQ (see 4.11 above) are qualified as having less certainty.

6.3 Calibration Procedures for GC/MS (Gas Chromatograph/ Mass Spectrometer)

6.3.3 Calibration Verification

Clarification: If the RF from any CCC compound differs from the average RF from the initial calibration by more than 20%, then either the calibration verification standard must be reanalyzed or recalibrate. All non-CCC compounds should be within 40% from the average RF from the initial calibration unless alternate criteria are specified in the laboratory SOP.

6.4 Calibration Procedures for GC and HPLC (Gas Chromatograph and High Performance Liquid Chromatograph)

6.4.2 Calibration Verification

Method criteria supersede the information in the corporate quality manual.

Table 6.1 Summary of Calibration Requirements

Instrument	Calibration Standard	Acceptance Limit	Corrective Action
Glass Syringes, except autosampler syringes which inject a constant amount.	Prior to use an initial demonstration must be performed, at a minimum	Acceptable based on application	Replace
Automatic Dispensing Devices	Quarterly	Varies	Remove from service and fix, replace
Dial Thermometers	Quarterly	Dependent on Method	Remove from service and fix, replace
Oven	Daily with NIST traceable reference	Acceptable based on application	Remove from service and fix, replace
Refrigerator	Daily with NIST traceable reference	Acceptable based on application	Remove from service and fix, replace
Freezer	Daily with NIST traceable reference	Acceptable based on application	Remove from service and fix, replace
Instrument	Calibration Standard	Acceptance Limit	Corrective Action
Incubator	Daily with NIST traceable reference	Acceptable based on application	Remove from service and fix, replace
Waterbath	Daily with NIST traceable reference	Acceptable based on application	Remove from service and fix, replace
Bottle Top Dispensers	Biannually when the dispenser is used at variable amounts	Varies, See appropriate SOP	Remove from service and fix, replace

6.5.2 Calibration Verification for ICP, ICP-MS

The Kimberly laboratory analyzes the Interference Check Standards at the beginning of the run and every 12 hours thereafter if the run is sufficiently long to require analysis of subsequent Interference Check Standards. This practice follows the requirements of Method 6020. Pace Green Bay analyzes the Interference Check Standard at the beginning of the run and every 12 hours thereafter for Method 6020. It is analyzed at the beginning of the run only for Method 6010B.

6.6 General Calibration Procedures

6.6.1 Analytical Balances

The Manual for the Certification of Laboratories Analyzing Drinking water specifies that Class 1 or 2 weights may be used in the laboratory.

6.6.3 pH/Electrometers

Pace Green Bay calibrates the pH meter before use each day. Recalibration is only performed if CCV becomes out of control.

7.0 CONTROL OF DATA

7.5 Response to Inquiries

The project manager initiates the resolution of customer complaints. A Client Data Review Request Form is used to document complaints, project requests, or comments. The form is forwarded to the laboratory and the actions taken are recorded. When a complaint raises doubt concerning the laboratory policies, procedures, or data quality, the area in question will be internally audited. All records are maintained within the project file or Quality Assurance Office depending on the course of action taken. More detailed information on this process may be found in MN-Q-245 (*Documentation of Client Requests and Complaints*).

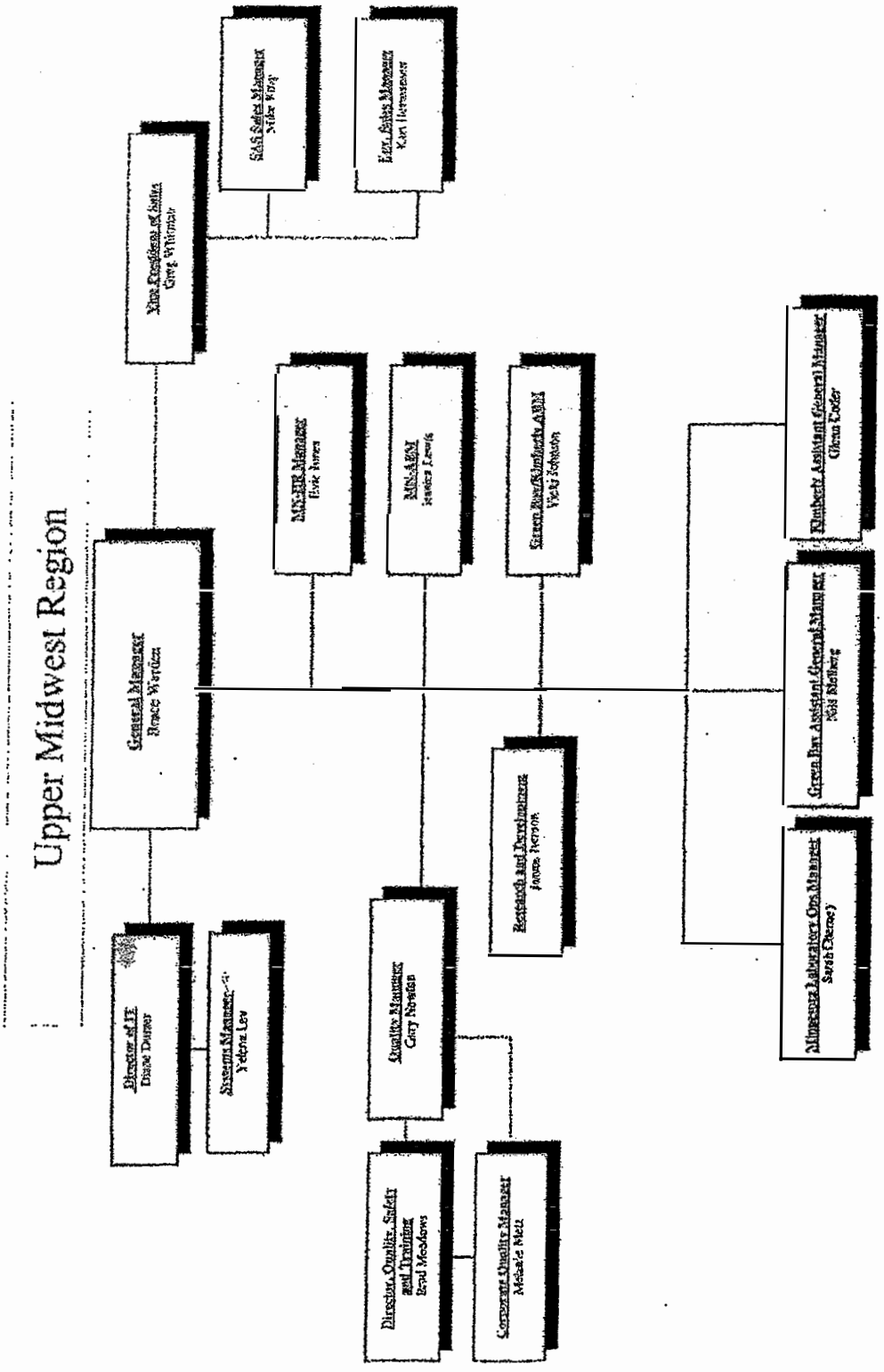
8.0 QUALITY SYSTEM AUDITS AND REVIEWS

The Upper Midwest region has no further changes to this section of the corporate quality manual.

9.0 CORRECTIVE ACTION

The Upper Midwest region has no further changes to this section of the corporate quality manual. More detailed information on the corrective action and investigation process may be found in MN-L-133 (*Out of Specification Investigation*) and ALL-Q-012 (*Corrective Action/ Preventative Action Process*).

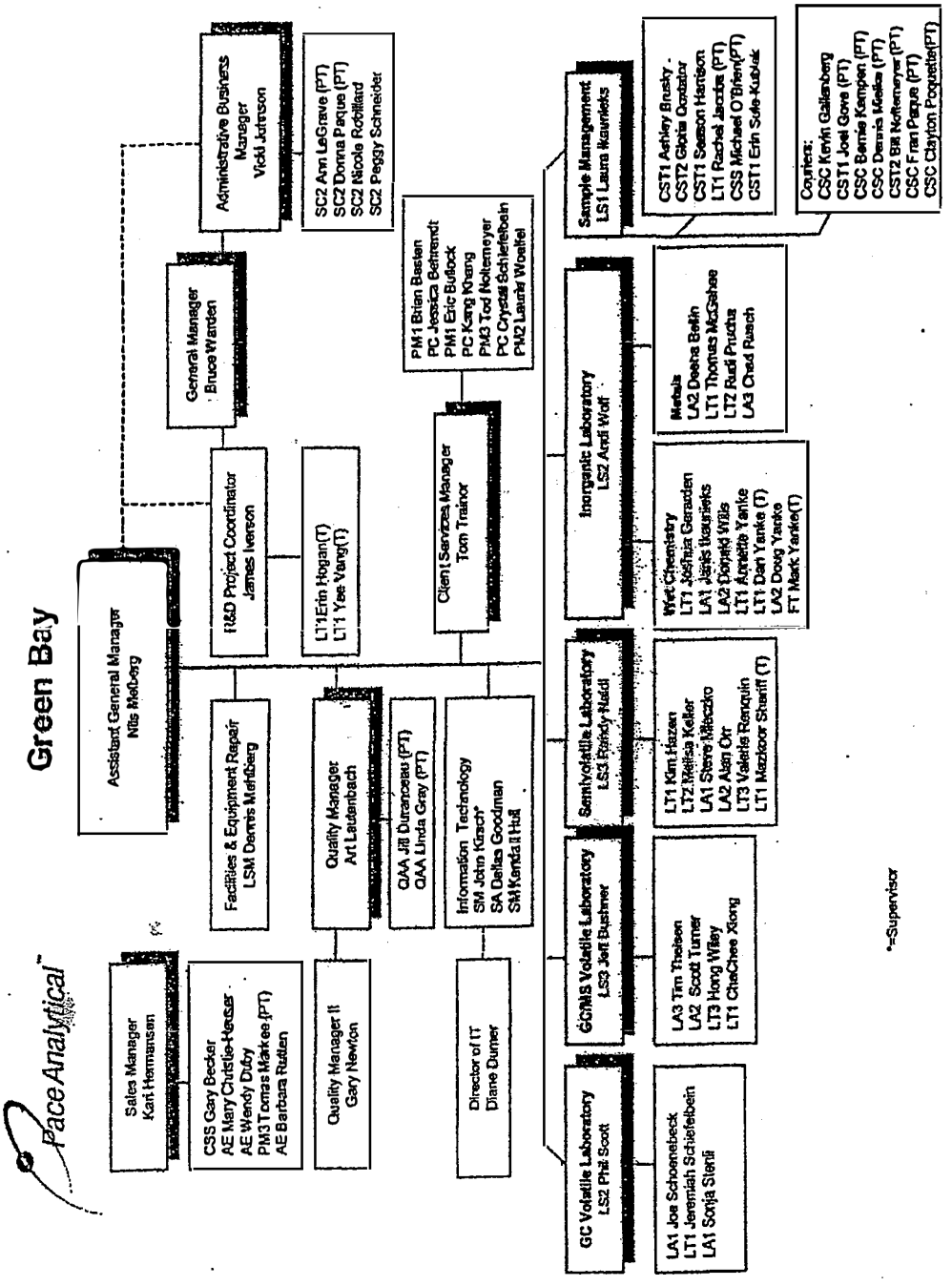
Attachment 1



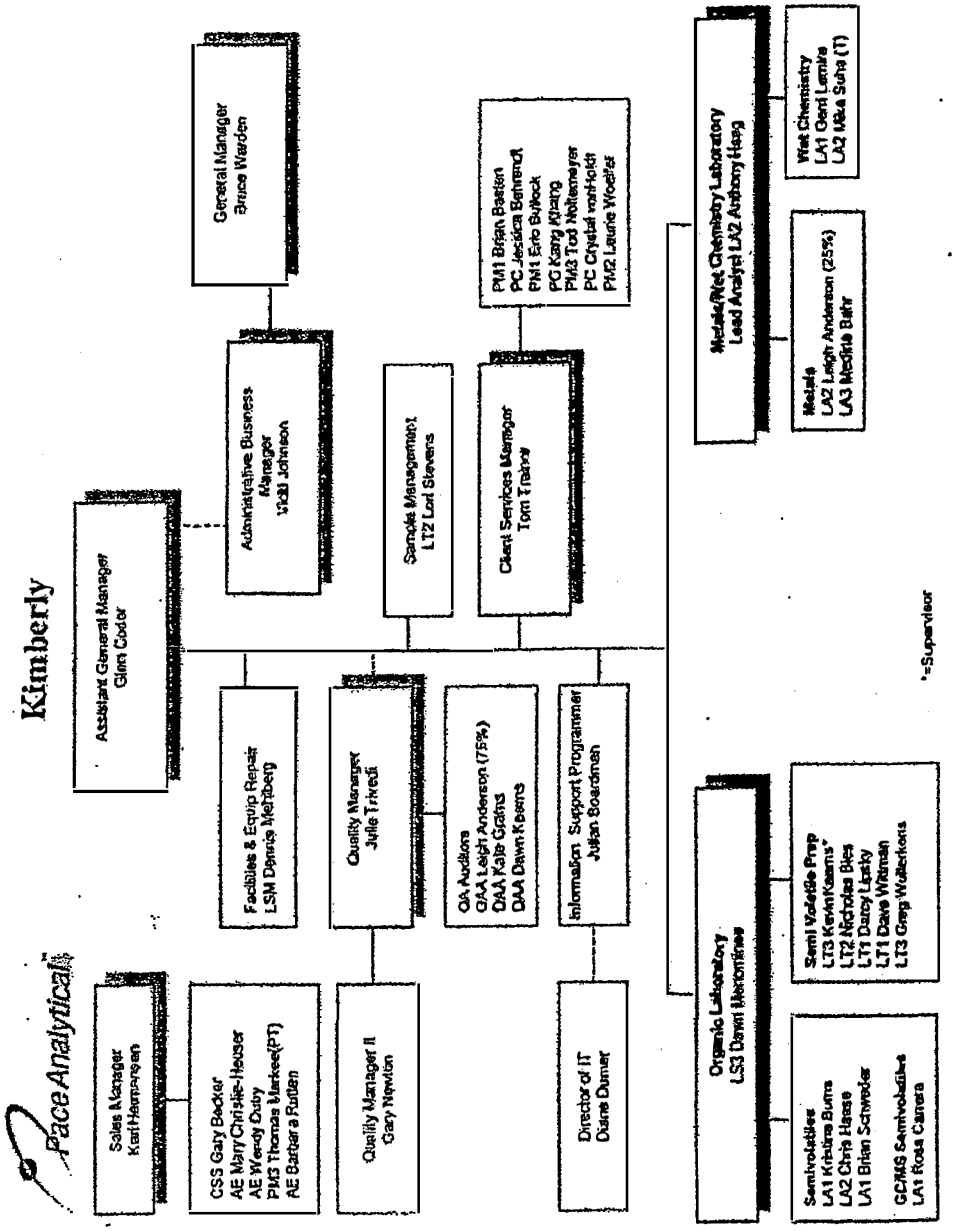
Attachment 2

Minnesota Laboratory

Attachment 3



Attachment 4



Attachment 5 (page 1 of 3)

Method	Description	MN	GB	KM
%MOISTURE	Percent Moisture			
Ag-List	Organophosphorus Compounds by GC/MS: Capillary Column Technique			
ASTM D1125	Conductivity		x	
ASTM D2974	Moisture, Ash, and Organic Matter of Peat and Other Organic Soils		x	
ASTM D5057	Density by ASTM			
ASTM D3987	Neutral Leaching Procedure, Neutral pH, Aqueous Extraction		x	
ASTM G57	Permeability		x	
CARB 429	Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions from Stationary Sources			
EPA 110.2	Color		x	
EPA 120.1	Physical Properties: Conductance, Specific Conductance		x	
EPA 150.1	Physical Properties: pH, Electrometric		x	
EPA 160.1	Physical Properties: Residue, Filterable, Gravimetric, Dried at 180 C o		x	
EPA 160.2	Physical Properties: Residue, Non-Filterable Gravimetric, Dried at 103-105 C o		x	
EPA 160.3	Physical Properties: Residue, Total Gravimetric, Dried at 103-105 C o		x	
EPA 160.4	Physical Properties: Residue, Volatile, Gravimetric, Ignition at 550 C o		x	
EPA 160.5	Physical Properties: Settleable Matter, Volumetric, Imhoff Cone			
EPA 180.1	Turbidity, Nephelometric		x	
EPA 1613	Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS			
EPA 1613B-DW	2,3,7,8-Tetrachlorodibenzo-p-Dioxin in Drinking Water by Isotope Dilution HRGC/HRMS			
EPA 1614	Brominated diphenyl ethers in Water, Soil, Sediment and Tissue by HRGC/HRMS			
EPA 1653	Chlorinated Phenolics in Wastewater by in Situ Acetylation and GC/MS			
EPA 1664 Rev A	N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum			
EPA 1668	Chlorinated Biphenyl Congeners in Water, Soil, sediment, and Tissue by HRGC/HRMS			
EPA 170.1	Physical Properties: Temperature, Thermometric			
EPA 180.1	Determination of Turbidity by Nephelometry			
EPA 200.7	Determination of Metals and Trace Elements in Water and Wastes by ICP			
EPA 200.8	Determination of Trace Elements in Water and Wastes by ICP-MS			
EPA 245.1	Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry			
EPA 300.0	Determination of Inorganic Anions by Ion Chromatography - Revision 2.1		x	
EPA 305.1	Acidity (Titrimetric)		x	
EPA 310.1	Inorganic, Non-Metallics: Alkalinity, Titrimetic (pH 4.5)		x	
EPA 310.2	Alkalinity, Colorimetric, Automated Methyl Orange		x	
EPA 314.0	Determination of Perchlorate in Drinking Water by Ion Chromatography			
EPA 325.2	Chloride (Colorimetric, Automated Ferricyanide AA II)		x	
EPA 330.2	Inorganic, Non-Metallics: Chlorine, Total Residual, Titrimetric, Back, Iodometric			
EPA 335.2	Inorganic, Non-Metallics: Cyanide, Total, Titrimetric, Spectrphotometric			
EPA 335.4	Determination of Total Cyanide by Semi-Automated Colorimetry		x	
EPA 340.2	Inorganic, Non-Metallics: Fluoride, Potentiometric, Ion Selective Electrode			
EPA 350.1	Determination of Ammonia Nitrogen by Semi-Automated Colorimetry -Revision 2.0		x	
EPA 351.2	Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry -Revision 2.0		x	
EPA 354.1	Inorganic, Non-Metallics: Nitrite, Spectrophotometric		x	
EPA 365.2	Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Single Reagent			
EPA 365.3	Phosphorus, All Forms, Colorimetric, Ascorbic Acid, Two Reagents		x	
EPA 365.4	Phosphorus, Total, Colorimetric, Automated Block Digester AA II)		x	
EPA 376.1	Inorganic, Non-Metallics: Sulfide, Titrimetric, Iodine		x	
EPA 3810 (mod)	Methane,Ethane, Ethene in Water by Headspace Analysis			
EPA 410.2	Organics: Chemical Oxygen Demand, Titrimetric, Low-Level			
EPA 410.4	Determination of Chemical Oxygen Demand by Semi-Automated Colorimetry - Revision 2.0		x	
EPA 415.2	Total Organic Carbon (UV Promoted, Persulfate Oxidation)			
EPA 420.1	Organics: Phenolics, Total Recoverable, Spectrophotometric, Manual 4-AAP with Distillation			
EPA 420.2	Phenolics (Colorimetric, Automated 4-AAP with Distillation)		x	
EPA 420.4	Determination of Total Recoverable Phenolics by Semi-Automated Colorimetry			
EPA 524.2	Measurement of Purgeable Organic Compounds in Water by GC/MS		x	

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Method	Description	MN	GB	KM
EPA 602	Purgeable Aromatics		X	
EPA 610	Polynuclear Aromatic Hydrocarbons	X		
EPA 624	Chlorinated Hydrocarbons-Purgeables by Low Resolution GC/MS	X	X	
EPA 625	Base/Neutrals and Acids by Low Resolution GC/MS	X	X	
EPA 1631E	Mercury in Water by Oxidation, Purge and Trap and CVAFS			X
HACH 8146	Chlorine, Free		X	
Method 23	Determinatin of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air	X		
Method 26	Determination of Anions and Cations from Ambient Air	X		
Method 29	Determination of Metals Emissions from Stationary Sources	X		
Method 308	Methanol and Ethanol from Stationary Sources	X		
Method 3C	Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources	X		
Method 5	EPA5, Air Filter, Cyclone Wash, Wet Catch, Probe Wash	X		
Molds	Various molds identification	X		
NIOSH 1500	Hydrocarbons	X	X	
NIOSH 7300 (mod)	Metals, ICP, NIOSH 7300 - MOD	X		
NWTPH-Dx	Semi-volatile Petroleum Products Method for Soil and Water	X		
NWTPH-Gx	Volatile Petroleum Products Method for Soil and Water	X		
OA1	Gasoline Range Organics in Water and Soil		X	
OA2	Diesel Products in Water and Soil		X	
PM10	PM 10, Air Filter, Cyclone Wash, Wet Catch, Probe Wash	X		
REDOX	Oxidation/Reduction Potential	X		
SM 2110	Appearance		X	
SM 2160	Taste		X	
SM 2320B	Alkalinity, Water	X	X	
SM 2340B	Hardness, Ca & Mg as Carbonates by ICP	X	X	X
SM 2540	Solids		X	
SM 2580	Oxidation-Reduction Potential (ORP)		X	
SM 2710F	Sludge Specific Gravity		X	
SM 3500-Cr-D	Chromium, Hexavalent (Colorimetric)	X	X	
SM 4500	Chloride (Silver Nitrate)	X		
SM 4500-CN-E	Cyanide (Colorimetric Method)	X		
SM 4500-CN-G	Cyanides Amenable to Chlorination after Distillation	X	X	
SM 4500-CN-I	Weak Acid Dissociable Cyanide	X	X	
SM 4500-CL	Chlorine		X	
SM 4500-CN	Cyanide		X	
SM 4500-F-C	Fluoride by Ion-Selective Electrode Mode	X		
SM 4500-O	Dissolved Oxygen		X	
SM 4500-P-E	Phosphorous- Ascorbic Acid Method	X		
SM 4500-SO3-B	Sulfide	X		
SM 5210B	5-Day Biochemical Oxygen Demand (BOD) Test	X	X	
SM 5220C	Chemical Oxygen Demand (COD) by Closed Reflux, Titrimetic Method	X		
SM 5220D	Chemical Oxygen Demand (COD) by Closed Reflux, Colormetric Method	X		
SM 9215A	Heterotrophic Plate Count	X	X	
SM 9222D	Fecal Coliform in Water	X	X	
SM 9223B	Total Coliform in Water	X	X	
SM2540C	Total Dissolved Solids	X		
SM2540D	Total Suspended Solids	X		
SPC	Standard Plate Count	X		
SW 1311	Toxicity Characteristic Leaching Procedure	X	X	
SW 1312	Synthetic Precipitation Leaching Procedure	X	X	
SW 3005A	Total Recoverable or Dissolved metals		X	X
SW 3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy	X	X	
SW 3015	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts			X

Attachment 5 (page 3 of 3)

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Method	Description	GRS	GB	KN
SW 3020A	Total Metals for GFAA		x	
SW 3050B	Acid Digestion of Sediments, Sludges, and Soils		x	
SW 3051	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils			
SW 3510C	Separatory Funnel Liquid-Liquid Extraction		x	
SW 3520C	Continuous Liquid-Liquid Extraction			
SW 3535	Solid-Phase Extraction (SPE)			
SW 3540C	Soxhlet Extraction			
SW 3541	Automated Soxhlet Extraction			
SW 3545	Pressurized Fluid Extraction (PFE)		x	
SW 3550B	Ultrasonic Extraction			
SW 3580A	Waste Dilution		x	
SW 3640A	GPC			
SW 3620B	Florisil Cleanup			
SW 3630C	Silica Gel Cleanup			
SW 3660B	Copper Cleanup			
SW 3665A	Sulfuric Acid Cleanup			
SW 5030B	Purge-and-Trap for Aqueous Samples		x	
SW 5035	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples		x	
SW 6010B	Inductively Coupled Plasma-Atomic Emission Spectrometry		x	
SW 6020	Determination of Trace Elements in Water and Wastes by ICP-MS		x	
SW 7196A	Chromium, Hexavalent (Colorimetric)		x	
SW 7470A	Mercury in Liquid Waste (Manual Cold-Vapor Technique)		x	
SW 7471A	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)		x	
SW 8015B	Nonhalogenated Organics Using GC/FID		x	
SW 8021B	Aromatic and Halogenated Volatiles by Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors		x	
SW 8081A	Organochlorine Pesticides by Gas Chromatography			
SW 8082	Polychlorinated Biphenyls (PCBs) by Gas Chromatography			
SW 8260B	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)		x	
SW 8270C	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)		x	
SW 8270C SIM	Semivolatile Organic Compounds by GC/MS-utilizing selective ion monitoring		x	
SW 8280A	The Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by HRGC/LRMS			
SW 8290	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by HRGC/HRMS			
SW 8310	Polynuclear Aromatic Hydrocarbons		x	
SW 8330	Nitroaromatics and Nitramines by HPLC			
SW 9010	Total and Amenable Cyanide: Distillation			
SW 9012A	Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation)		x	
SW 9040B	pH Electrometric Measurement		x	
SW 9045C	Soil and Waste pH		x	
SW 9050A	Conductivity		x	
SW 9056	Determination of Inorganic Anions by Ion Chromatography		x	
SW 9071B	HEM in Soil			
SW 9095A	Paint Filter Liquids Test		x	
SW 9251	Chloride (Colorimetric, Automated Ferricyanide AAII)		x	
TO-13	Determination of Semi-Volatile Organics in Ambient Air			
TO-14	Determination of VOCs in Ambient Air utilizing a Summa Canister and GC/MS			
TO-15	The Determination of VOCs in Air Collected in Summa Canisters and Analyzed by GC/MS			
TO-15 SIM	The Determination of VOCs in Air Collected in Summa Canisters and Analyzed by GC/MS utilizing SIM			
TO-3	TO3, In Air Source			
TO-4 (PCB)	Determinatin of Polychlorinated Biphenyl in Ambient Air			
TO-4 (Pesticides)	Determinatin of Polychlorinated Pesticide in Ambient Air			
TO-9A	Determinatin of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air			
WIDRO	Determination of Diesel Range Organics in water and soil		x	
WIGRO	Determination of Gasoline Range Organics in water and soil		x	

Appendix B
Soil/Sediment Sampling and Decontamination Procedures

Soil/Sediment Sampling

1. Collect sample from the upper 1 to 1.5 feet of soil using a shovel and/or a posthole digger.
2. Place the sample in the bottle provided by Pace.
3. Fill sample location with excess sample.
4. Clean sampling equipment with phosphate-free detergent and water, using a brush to scrub dirt from the equipment.
5. Mark the sample container with the sample number that corresponds to the test plot number, along with the time and date of the sample collection. Identify the sample container on both the side and on the lid using sample labels showing the project name and number, the sample number, and the collection date. Fill out Chain-of-Custody Records, and transport samples to Pace Analytical.

Decontamination Procedures

A decontamination area will be established at the site for cleaning the sampling equipment. A plastic sheet will be placed on the ground on which two 5-gallon buckets will be placed, one containing a non–phosphate-based detergent and the other used for collecting rinse water. Sampling equipment (spades and trowels) will be washed in the detergent solution using a brush to remove large particles, and then rinsed with clean water from a spray bottle. The water will be collected in the second bucket. The buckets will be capped, and the solutions will be disposed in a municipal sewer system after sampling is completed. The City of Kewaunee Sewerage Utility gave verbal approval in a telephone conversation on November 16, 2005, for the disposal of the wash water in the local sewer system. Given the small volumes of water involved, the City said that disposal in the sewer system would be acceptable without testing.

Appendix C
Gas Collection Approach

Marsh gas collection with analysis for arsine is not a standard laboratory procedure. Of necessity, the method described below is experimental, and may be changed as needed to improve the collection and analysis procedures.

1. A gas collection device will be placed over a 285 cm^2 area of the test plot. The device consists of a gas impermeable cap and wall which channels the gas to a central collection tube, which is attached to a Tedlar gas collection bag (Figure 1).

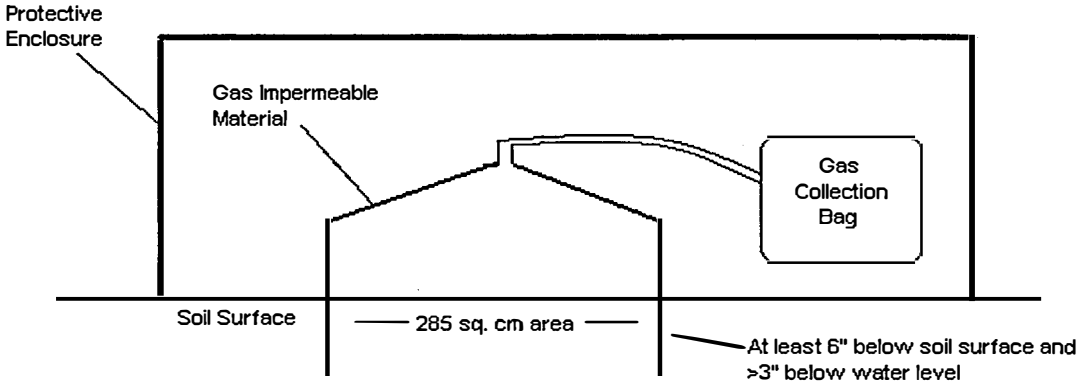

Figure 1: Gas Collection Apparatus

2. Gas samples will be collected from all test plots except the control. Since the cattails have not been removed in the control plot, to do so could alter the redox status and gas generating potential of the soil under the collection device.

3. The gas collection devices including the Tedlar bag will be protected by an enclosure to shelter them from the weather, e.g. high winds or thunderstorms.

4. Samples will be collected periodically throughout the field trials. The gas collection bags will be collected initially after one week of initial application of bioreductants and thereafter during each monthly performance monitoring event. The first few sampling periods will be used to test and optimize the method. Sampling periods may change as needed to obtain sufficient (but not too much) volume. Samples will be returned to the RMT Applied Chemistry laboratory for analysis preparation and total volume measurement. Gas volume will be measured by water displacement.

5. Gas samples will be sent to a commercial laboratory (e.g. the Wisconsin State Laboratory of Hygiene) for arsine analysis using NIOSH method 6001. This method involves passing a known amount of gas sample through a solid sorbent tube with analysis using graphite furnace atomic absorption spectrometry.