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King County Environmental Laboratory Internship Report

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Environmental Laboratory
Internship Report

Kristen Weatherford

HUXLEY COLLEGE
OF
ENVIRONMENTAL STUDIES

Internship Report Adviser:

[redacted]

Dean of Huxley College:

[redacted]

Date: 12/9/00

NTERNSHIP REPORT

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INTERNSHIP REPORT

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HONORS THESIS

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Date 12/7/00

King County
Water and Land Resources Division
Environmental Laboratory
Department of Natural Resources
322 West Ewing Street
Seattle, WA 98119-1507
(206) 684-2300

November 29, 2000

Dear Sir or Madam:

I am writing this letter to confirm that Kristen Weatherford worked as a Student Intern at the King County Environmental Laboratory (Lab) during the summer of 2000. In addition, I want to discuss her work and comment on her performance.

Kristen was hired by King County as a Student Intern and was employed at the Lab from June 12, 2000 through September 15, 2000. Kristen worked full time (40 hours) Monday through Friday and her attendance was perfect. Kristen was on time and used her time effectively.

Kristen was assigned a body of work, which she was expected to track, perform and document under the supervision of a chemist and myself. Specifically, Kristen was trained to perform a variety of physical parameter testing including pH, Dissolved Oxygen, conductivity, alkalinity, color, turbidity and suspended solids. In addition, Kristen assisted chemists in other areas of the unit throughout the summer (filtration of chlorophylls and nutrients). Kristen also worked on a small method development project to evaluate method detection limits for suspended and dissolved solid parameters. Her duties included tracking all incoming samples, performing the analyses within required holding times, conducting the testing using written protocols and by including required quality control samples. Kristen was also responsible for documenting her work using analysis log books and instrument logbooks. She processed raw data and performed all the necessary calculations and data entry into spreadsheets and LIMS.

As a supervisor, with several years of experience working with professional chemists as well as students, I can say with confidence that Kristen's work was well above average for a person in her position. In my evaluation I use several criteria including, productivity and quality of work, aptitude, organizational and communication skills as well as interpersonal skills.

Kristen is a very fast learner. She impressed me with her ability to absorb new information. The training period and supervision for her work was minimal and far less than expected for a person in her position. The quantity, quality and timeliness of her work were very good.

Kristen demonstrated excellent communication and organizational skills. She used all the resources available to her to communicate and understand her workload including e-mail, business team meetings, co-workers, and supervisor, and LIMS. She asked questions at appropriate times and organized her work well. Kristen was able to perform a variety of methods and tasks and her ability to organize, be flexible and multitask were well above average. Kristen documented her work well and followed required protocols.

In my opinion Kristen was very successful this summer partly because of her technical skills but also because of her interpersonal skills. She worked well with all her co-workers and she gained their respect and trust very quickly. She listened to suggestions and advice and was flexible and willing to shift her priorities when necessary. Kristen demonstrated interest in the work of the unit and the Lab as a whole and took every opportunity to learn more about the organization. Kristen shared her views openly with everyone and demonstrated a lot of confidence in expressing her views.

Overall, I rate Kristen's work very high and well above average. She contributed significantly to our mission and I believe she acquired skills that will be beneficial to her in the future.

Sincerely,

[redacted]

Despina Strong, Ph. D.
Conventionals Supervisor

Acknowledgments

I would like to thank Despina Strong and the chemists in Conventionals. I enjoyed working and learning with all of you. Also, thanks to my advisors, Leo Bodensteiner, Huxley College Professor, and George Mariz, Honors Program Director for your guidance and support.

Introduction

The goal of the Water Pollution Control Act of 1972, otherwise known as the Clean Water Act (CWA), is to restore and maintain the chemical, physical, and biological integrity of the waters of the United States. The CWA includes national policies to prohibit discharge of toxic pollutants, and to fund publicly owned wastewater treatment facilities (Cornell 2000).

The CWA prohibits discharge of pollutants by any person through a point source into U.S. waters unless that person has a National Pollution Discharge Elimination System (NPDES) permit. NPDES permits include limits on what can be discharged, and monitoring and reporting requirements. The permits also require facilities to sample their discharges and to notify the Environmental Protection Agency (EPA) and state regulatory agencies of the results (EPA OWM 2000). As a result of the CWA, state governments are responsible for monitoring the quality of the state's waters.

King County Environmental Laboratory

I had the opportunity to work as a student intern in the Conventionals Section of the King County Environmental Laboratory during the summer of 2000. The Conventionals Section performs conventional chemistry tests on a variety of samples (see below). My supervisor was Despina Strong, the Laboratory Supervisor for the Conventionals Section. King County Environmental Laboratory is part of the Water and Land Resources Division of the King County Department of Natural Resources (DNR). The lab provides analytical services and sample collection in support of various DNR programs; the primary purpose of the lab is to support the Waste Water Treatment Division of the DNR

The data collected by the lab are consolidated into a central computer system to form a database that is used to monitor trends in regional waters. The information generated by the lab is used to:

- Design and operate wastewater treatment plants;
- Comply with treatment plant and biosolids permit requirements from the Washington State Department of Ecology;
- Enforce industrial waste regulations;
- Plan for new facilities and wastewater infrastructure;
- Evaluate alternative technologies for wastewater treatment;
- Formulate plans to protect and enhance local water bodies; and
- Participate in cooperative studies with other public agencies (DNR 2000).

The lab includes trace organic, trace metal, conventional chemistry, and microbiological and bioassay determinations for samples. Additionally, lab employees perform data management and analysis, including the upkeep of a long-term historical database. The lab is accredited by the Washington State Department of Ecology and the Washington State Department of Health, and routinely uses performance evaluation samples and performs internal and external audits (DNR 2000).

Chemists in the Conventionals section of King County Environmental Laboratory analyze samples including freshwater, marine, domestic and industrial wastewater, biosolids, and soils and sediments from fresh and marine waters. Chemists in

Conventionals perform tests for parameters such as color, conductance, pH, alkalinity, chloride, ammonia, nitrogen, salinity, silica, total phosphorus, biological oxygen demand, total organic carbon and chlorophyll (DNR 2000).

Internship Experience

As an intern I assisted the chemists in the Conventionals Section of the lab. I was also trained to do many of the tests on my own, which included learning to use automated equipment for several of the parameters that I tested. My experience at the lab began with a week that consisted primarily of safety training and orientation. Safety training is essential for new employees at the lab; it includes watching safety videos, taking a quiz, touring the lab, and discussing safety rules and the reasons behind them with a supervisor or the Safety Officer. Failure of the safety quiz indicates a need for a more extensive explanation and review of safety policies and procedures.

After I completed the safety training I began to observe the chemists and to prepare to learn the analytical procedures. Before I worked on a test in the lab I read the Standard Operating Procedure (SOP), the instrument manuals for any equipment I would use, and the applicable Material Safety Data Sheets (MSDS). The SOPs I worked with are based on EPA methods, such as the EPA Methods for the Chemical Analysis of Water and Wastes and EPA residue methods, as well as Standard Methods for the Analysis of Water and Wastewater, 20th Edition, and Methods Manual for Forest Soil and Plant Analysis. Reading about the procedure in advance allowed me to be informed about any risks associated with that test; it also gave me a better understanding of the test I was going to perform. The SOP includes possible sources of error for the test, preservation and storage requirements of the samples, reagents and standards needed for analysis, documentation and reporting requirements, quality control (QC) requirements, and training procedures.

Before I performed a test on my own I was required to complete cross-training with a chemist who is qualified to perform the test. Cross-training is accomplished by following a checklist of training guidelines. The first section on the training list includes review of the SOP and equipment and/or instrument manuals. This allowed me to become familiar with the test procedure. The second section is a review of safety issues. The qualified chemist and I went over routine safety practices for the test, such as wearing a specific type of gloves, as well as special safety precautions. The safety section also includes a review of hazardous reagents and waste, and the proper procedure for sample and waste disposal. The third section on the checklist covers purchasing. Each chemist is responsible for making sure that the lab has a sufficient supply of any chemicals, equipment and supplies needed for the tests that they perform. The fourth section covers documentation. During analysis I was responsible for maintaining several log books, documenting my actions. After analysis was complete I prepared a data package that contained the results. The next two sections include a review of schedules and projects. Tests include reagents, standards and QC samples that expire after a certain period of time; new batches need to be prepared regularly. Also, some of the instruments and equipment used in analysis require routine maintenance. The trainer and I reviewed the projects that typically require the parameter that I was being trained to analyze and the frequency and number of samples for each project.

The final step in cross-training was to perform the analysis. First, I watched the qualified chemist perform the test. Next, I performed the procedure with the chemist. Finally, I performed the analysis independently. The final analysis included four samples whose qualities were known to the trainer but not to myself. When the test was complete my results for the four blind samples were compared to the actual value. If my results were within the acceptable range my cross-training was complete.

During my first week I also started to learn to use the Laboratory Information Management System (LIMS), software that is used to organize samples and data. When samples are brought to the lab they are given an identification number and logged in to LIMS. Chemists can then create a list that gives them important information about samples, such as the project and due date of the sample. One example of a project is the Lake Monitoring Program. The due date is the date that the customer requires the data from all analysis. The chemist can then put the samples into a workgroup, based on the parameter that they are going to test, such as turbidity. A workgroup is a batch of samples being analyzed for one parameter at the same time; it may include samples from more than one project. Each sample may be tested for more than one parameter. The label on each sample container lists the tests required by that sample. After the samples have been tested, the data from the test are transferred into LIMS from a Microsoft Excel spreadsheet. LIMS is then used to put together the data package that shows the results of the test.

Documentation is required for every test that is performed in the lab. An analytical log book is kept for each test. This book includes: the sample numbers of all samples that are analyzed, the workgroup number assigned by LIMS, the system ID number assigned to each sample if computer software is used in the analysis, the preparation ID number of any reagents and standards used, the date, the name of the chemist performing the analysis, and any observations made during testing. Another logbook is used to record the preparation of any reagents or standards, including the dates of preparation and expiration, the chemical name, the stock and lot number of chemicals used, the volume and concentration prepared and the name of the analyst doing the preparation. Instrument manuals are used to record the data, purpose, and chemists' name each time an instrument is used. Data packages are prepared when analysis is complete. The data package includes, but is not limited to, a copy of the laboratory notebook, a workgroup report that lists the samples being tested, a table showing the results of the analysis, and a QC report that shows whether or not the results were within required ranges.

Quality Control (QC) is an important part of the testing procedure. Customers want to know that the data they are receiving is both precise and accurate. Each parameter tested at the lab has QC requirements, which are included in the SOP for the procedure. QC includes things such as laboratory duplicates (LD), laboratory control samples (LCS) and performance evaluation (PE) samples. When performing a test, a chemist will test two aliquots of the sample; this is a laboratory duplicate. The results of the LD and the original sample must be within a certain range from one another; this tests for precision. Laboratory control samples and performance evaluation samples have known characteristics; for example, a LCS sample could have a known alkalinity. The results that the chemist obtains must be within a certain range of the actual value of the sample. LCS samples test for accuracy. LCS samples are internal controls, while PE samples are obtained from an external source, such as the EPA. For the tests that I performed, LCS samples were usually included at the beginning and end of each batch of samples and after every 20 samples if there were more than 20 samples in the batch. One LD was usually analyzed for every 20 samples; PE samples were usually evaluated once a month.

An important part of working in a laboratory is housekeeping. Although I was primarily responsible for analytical tests, I also washed glassware, moved and disposed of sample, and neutralized acidic waste. Clean glassware is essential because any residue left in the glassware, such as a chemical that was not washed out, can interfere with a test. Acidic waste is neutralized with sodium bicarbonate, alkaline waste is neutralized by mixing it with acidic waste. Samples that are not depleted during analysis are stored in coolers for up to a year. After analysis they are placed in a temporary cooler, then moved to another cooler for storage. When the data package for each sample is finished and approved by a supervisor the sample is placed on a list for disposal.

During the last month of my internship I was assigned a set of parameters and was responsible for performing the tests and preparing the data packages associated with those parameters. The parameters included alkalinity, pH, turbidity, total suspended solids, color and conductivity. In addition to performing the tests, I was responsible for instrument calibration, preparation of reagents and standards, and documentation. Each parameter has a different holding time, so it was important for me to balance the workload carefully. Turbidity, for example, has a holding time of 48 hours, while pH must be tested within 24 hours. Other parameters, such as alkalinity and conductivity, had two or more weeks in which they could be tested. I performed this analysis independently; however, other chemists were always present in the lab. My supervisor and the chemists in my section were available to answer any questions that I had, or to help me when there was a large workload. The data packages that I produced, containing the results of my analysis, were reviewed and approved by another chemist and by my supervisor.

During the internship, I also had the chance to perform several Method Detection Limit (MDL) studies. An MDL study is used to determine to what level the results of a test can be considered significant, and what is just "background noise." I performed MDL studies for Total Suspended Solids (TSS) and Total Dissolved Solids (TDS). I did the analysis using ten crucibles, for TDS, or dishes, for TSS, that included deionized water, and ten that had no water or sample included. In order to determine the MDL, a mathematical equation would then be applied to the results. The MDL study shows how much of the results for a test vary due to the method, instrument and equipment before any samples are added. For example, if empty crucibles were put through the TDS testing process and consistently yielded a result of 0.3 mg/L TDS, the analyst knows that any result obtained in TDS testing that was below 0.3 mg/L could be due to variations in the testing process, and not to the actual sample being tested.

I also compared the results of industrial waste samples that were analyzed for TSS. To homogenize industrial waste TSS samples, the lab usually blends them in a blender prior to analysis. My supervisor wanted to know if there was a difference between the results of the blended samples and samples that had not been blended. After performing the analysis using 20 blended samples and their unblended duplicates, I put the results in the spreadsheet and sent them to Tom Georgianna, a statistician. Tom used a natural log transform on the TSS data. The t-test showed that there was no statistically significant difference between blended and unblended samples. Tom performed a regression on the log transformed data, which resulted in an R square of 0.954 with an intercept of zero and a slope of 1.0. Therefore, blending the industrial waste samples prior to analysis does not affect the results when testing for TSS.

Conclusion

I was fortunate to have worked in the lab the summer before my internship as a lab assistant. Consequently, when I began my internship I was already familiar with the layout of the lab, the location of equipment and, most importantly, the people with whom I would be working. As I progressed through the internship I became more comfortable performing tests independently. The quality of my data packages improved, as did my time management. I was able to use extra time to ask questions of other chemists and learn more about other tests that I did not have time to train for.

I would recommend this type of internship to any environmental science student with an interest in chemistry, or to chemistry majors interested in protecting the environment. In order to improve my experience I would have needed a longer internship. The Conventionals Section performs many more tests that I would like to have learned about and cross-trained for. I would also like to have learned more about the lab in general. This might include spending a day collecting samples for analysis, spending a day with sample managers to learn more about their job, or interviewing laboratory project managers. I would be interested to learn more about the decision making process that goes into deciding things such as what projects to do, what parameters to include, and how often to perform each test.

My internship has proved to be an extremely valuable experience; it has given me the chance to apply knowledge from classes in both my Environmental Science major and my Chemistry minor. I have learned about the daily operations of a laboratory, and about the importance of Quality Control, safety, and communication. King County Environmental Lab is a teamwork-oriented environment; I have learned how to work as a part of a team by helping others and by asking for help when I need it. This internship has allowed me to work with technology such as automated equipment that measures pH, alkalinity, and conductivity, that I would not have had access to otherwise. Most importantly, I have had the chance to connect with people who have given me valuable skills and advice that will help me to advance my career.

Works Cited

Cornell University Legal Information Institute. "Chapter 26 United States Code: Water Pollution Prevention and Control." http://www4.law.cornell.edu/uscode/33/ch26.html.

Department of Natural Resources, King County Environmental Lab. http://dnr.metrokc.gov/wlr/envlab/index.htm.

Environmental Protection Agency, Office of Wastewater Management. "NPDES Permit Program, FAQ." http://www.epa.gov/owm.

Appendix A

Standard Operating Procedure

STANDARD OPERATING PROCEDURE

for

pH of Water, Sediments, and Soils

SOP # 03-01-003-003

Date of Implementation: June 14, 1999

Supersedes SOP # 03-01-003-002

Approved by:

Author: ______________________ Date: ____________

Supervisor: ______________________ Date: ____________

QA Officer: ______________________ Date: ____________

King County Environmental Laboratory
322 W. Ewing Street
Seattle WA 98119-1507

CHRONOLOGY OF SOP MODIFICATIONS

Revision Number	Section Revised	Date Implemented and Initials	Type of Change	Rationale
1	10.1 & 10.2	11/28/99 sm	Buffers 4 and 7 are commercially prepared pH buffers. Observe manufacturer's suggested expiration date. A working aliquot, replaced weekly, is used to calibrate the pH meter.	Clarification
1	10.3	11/28/99 sm	A working aliquot, replaced weekly, is used to verify pH meter calibration and monitor its operation.	Clarification
1	10.3.1 & 10.3.2	11/28/99 sm	LCS Low and LCS High identifications are replaced with LCS Level1 and LCS Level2, respectively.	Implementation of LIMS change.
1	12.3.3	11/28/99 sm	LCS Level2 is analyzed with samples whose pH readings are above pH 7.1.	Clarification QC purposes.
1	12.1.1	11/28/99 ts	1:1 solid to water ratio is recommended. Greater ratios up to 1:3 may be used based on the sample characteristics.	To minimize matrix effects – ionic strength, viscosity, interference… - and to generate sufficient extract for analysis.
1	8.1.2	11/28/99 sm	A holding time of 7 days after collection is utilized in lieu of 24 hours for solid samples.	
1	14.2	11/28/99 sm	Include LCS level and ID (date prepared) in the logbook.	Documentation for tracking and review.
1	12.4.3	11/28/99 sm	To maintain hydration of permeable pH probe tip, clean probe only by rinsing with RO water between samples. Do not blot dry with kimwipe.	Dehydration of probe tip due to wiping can damage the permeable surface or can result in sluggish response.
1	11.1	11/28/99 sm	3m KCl is used instead of 4M KCl	Manufacturer recommendation
1	12.5.1	2/1/00 ts	Remove this line	Combined pH / temperature compensation probe now in use.
1	12.5	2/1/00 ts	Use language from 12.3.4 be replace with method Auto pH to specify automated method. Manual analysis can also be used as desired.	Automated pH method in use for liquid samples in addition to availability of manual method.
1	12.1.1	1/00 ts	Orbital shaker used for solid sample stirring: 10 minutes at 175 RPM	Availability of orbital shaker recently purchased by conventionals

1	12.1.1	1/00 ts	Holding time for solids is 7 days at 4 C	Incorrect in the current SOP
1	8.2.1	1/00 ts	Standard bottle is a 500 mL	Incorrect in current SOP
1	8.1.1	4/17/00 ts	Updated required sample for full QC up to 150 g from 100g	Using approximately 50 g per sample to generate enough liquid for analysis. Duplicate and triplicate would therefore require 150 g for the QC sample.
1	12.1.1	4/00 ts	Updated weighed aliquot to 50 g from 20 g	At a 1:1 ratio, 50 g is needed to provide enough liquid for pH analysis
1	1	4/18/00 ts	Removed "title" section	Redundant

1. SCOPE AND APPLICATION

1.1 This Standard Operating Procedure (SOP) applies to the electrometric measurement of pH in sludges, soils, sediments, drinking water, freshwater, saltwater, ground water, storm water, sewer water, domestic waste, and industrial waste.

2. ASSOCIATED DOCUMENTS

2.1 King County Environmental Laboratory Hazardous Waste SOP # 11-02-003-000.

2.2 Conventionals Unit Sample Disposal SOP# 03-05-006-000.

3. METHOD SUMMARY

3.1 The solid sample is mixed with RO water. pH of the supernatant is determined electrometrically using a combination electrode in association with a potentiometric meter and temperature compensation probe.

3.2 The water sample is stirred and the pH is determined electrometrically using a combination electrode with a potentiometric meter and temperature compensation probe.

4. INTERFERENCES

4.1 Scratches, deterioration and accumulation of debris on the glass surface affect the glass electrode.

4.1.1 The presence of clay may slow the electrode response.

4.1.2 Suspended colloids (e.g., clay particles or organic mater) influence pH through the junction effect, with pH varying up to one unit between the suspension and the supernatant.

4.1.3 Biological activity in field moist samples can affect the pH during storage.

4.1.4 Organic soils and sludges may have insufficient supernatant at the specified solid:water ratio.

4.1.5 Oil in the sample may coat the electrode and cause a sluggish response and errors in pH.

4.2 pH measurements are affected by temperature in two ways: mechanical effects that are caused by changes in the properties of the electrode, and chemical effects caused by equilibrium changes.

5. DEFINITIONS

5.1 pH - the negative log10 of the hydronium ion concentration

6. SAFETY

6.1 General safety- All general laboratory safety practices should be complied with, including wearing a lab coat, safety glasses and gloves. Samples must be treated with regard to possible toxicity and microbiological potential.

6.2 This method involves the use of corrosive and caustic reagents as well as chemicals that pose contact hazards. Care should be taken to avoid skin contact or inhalation of these chemicals. All reagents that involve the use of concentrated acids and bases should be made in a fume hood.

6.3 Sample/Reagent Disposal – For disposal of samples, calibration buffers and standards refer to the King County Hazardous Waste SOP # 11-02-003-000, and the Conventionals Unit Sample Disposal SOP # 03-05-006-000.

6.4 Chemical Handling

6.4.1 Sulfuric Acid (H_2SO_4)- Sulfuric acid is a strong acid. Concentrated sulfuric acid should only be used in a fume hood. Bottles of concentrated sulfuric acid must be transferred to a fume hood from the acid storage cabinet in plastic pails. Neoprene gloves, splash goggles and an apron must be used in addition to other personal safety gear when dealing with concentrated acids. Bottles of concentrated sulfuric acid must be stored in the acid cabinet when not in use.

6.4.2 Nitric Acid (HNO_3)- Nitric acid is a strong acid. Concentrated nitric should only be used in a fume hood. Because we use such a small amount of this reagent (approximately 20 mls/year), bottles of concentrated nitric must be transferred to a fume hood from the metals' section acid storage cabinet in plastic pails. Neoprene gloves, splash goggles and an apron must be used in addition to other personal safety gear when dealing with concentrated acids. Bottles of concentrated nitric acid must be stored in the acid cabinet when not in use.

6.4.3 Anhydrous Alcohol - Anhydrous alcohol is extremely flammable. Avoid contact with skin and eyes, as well as open flame and excessive heat. Bottles are stored in the flammable cabinet when not in use. Gloves, safety glasses and lab coat must be worn when handling this chemical.

6.4.4 Ammonium Hydroxide (NH_4OH) - Ammonium hydroxide is a strong, fuming base. Concentrated ammonium hydroxide should only be used in a fume hood. Gloves, safety glasses and lab coat must be worn when handling this chemical.

7. SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1 Solids

7.1.1 The recommended sample container is a 4oz. glass jar. The minimum sample recommended for full QC is 150 g.

7.1.2 Samples can be frozen for six months or stored at 4 degrees for 7 days. The pH of the extract should be analyzed as soon as possible following the extraction.

7.2 Waters

7.2.1 The recommended sample container is a 500mL clear HDPE wide mouth bottle. Glass is an acceptable alternative to HDPE.

7.2.2 100 mL is the minimum volume required for analysis.

7.2.3 Samples should be analyzed as soon as possible or within 24 hours of sample collection.

8. APPARATUS AND EQUIPMENT

8.1 Metrohm, 674 Sample Changer with 2 lift stations (Brinkman #2.674.0020)

8.2 Metrohm Titrino Model 736/A20 with Magnetic Stirrer Titration Stand model 728, 20 mL buret unit, combination glass electrode with cable (Brinkman #20 87 254-3)

8.3 Brinkman Workcell Titration Software 4.3 (Brinkman #20 01 465-2)

8.4 Magnetic Stir Bars

8.5 Metrohm 50 to 250 mL sample cups

9. REAGENTS AND STANDARDS

9.1 pH 4 Buffer Solution (Fisher catalog # SB101-4) - This solution is commercially prepared and purchased through a vendor. Expiration dates are specified by the manufacturer on the container label. A working aliquot, which is replaced weekly, is used for daily calibration of the pH meter. This solution is stable at room temperature.

9.2 pH 7 Buffer Solution (Fisher catalog # SB107-4) - This solution is commercially prepared and purchased through a vendor. Expiration dates are specified by the manufacturer on the container label. A working aliquot, which is replaced weekly, is used for daily calibration of the pH meter. This solution is stable at room temperature.

9.3 pH 6.86 Laboratory Control Samples (Beckman catalog # BK3006)– Prepare the Laboratory Control Samples (LCSs) according to the manufacturer's instructions. The date of preparation and expiration, the analyst's initials, the lot number for the pouch containing the dry powder, the number of pouches used, and the final volume of LCS prepared must be recorded in the Physicals Reagent Log. The preparation number recorded in the Physical Reagent Log characterizes the LCS. A working aliquot, which is replaced weekly, is used to verify the calibration of the pH meter.

9.3.1 Transfer the pH LCS solution to a 1 Liter, wide mouthed, HDPE bottle. Place a label on the outside of the bottle with the chemical's name and concentration, the analyst's initials, the date of preparation, the expiration date (six months after preparation), and the preparation number. This reagent is stored at room temperature.

9.3.2 Transfer an aliquot of the stock solution (9.3) to a 125mL HDPE container bottle for use as the working aliquot. This container should be labeled with the chemicals name, concentration, date of transfer, expiration date, and preparation number. This reagent is stored at room temperature.

9.4 pH 10.01 Laboratory Control Samples (Beckman catalog # BK3019)- Prepare the Laboratory Control Samples (LCSs) according to the manufacturer's instructions. The date of preparation and expiration, the analyst's initials, the lot number for the pouch containing the dry powder, the number of pouches used, and the final volume of LCS prepared must be recorded in the Physicals Reagent Log. The preparation number recorded in the Physical Reagent Log characterizes the LCS. A working aliquot, which is replaced weekly, is used to verify the calibration of the pH meter.

9.4.1 Transfer the pH LCS solution to a 1 Liter, wide mouthed, HDPE bottle. Place a label on the outside of the bottle with the chemical's name and concentration, the analyst's initials, the date of preparation, the expiration date (six months after preparation), and the preparation number. This reagent is stored at room temperature.

9.4.2 Transfer an aliquot of the stock solution (9.4) to a 125mL HDPE container bottle for use as the working aliquot. This container should be labeled with the chemicals name, concentration, date of transfer, expiration date, and preparation number. This reagent is stored at room temperature.

9.5 Mixed Acid Cleanser – In a fumehood, mix 4 mls H_2SO_4 (Fisher # A510-212) and 4 mls HNO_3 (borrowed from the metals section) in a labeled 50 mL beaker. This solution should be kept in a fumehhod. This solution must be added to an acid waste stream for neutralization prior to disposal.

10. PREVENTATIVE MAINTENANCE

10.1 Monitor the level of KCl in the pH probe. When the level is below three fourths of the length of the probe, remove the plug and fill the reference electrode up with 3M KCl (VWR # BK566467). Never allow the solution in the electrode to dry.

10.2 Immerse the pH probe in RO water or 3M KCl when not in use.

10.3 To rejuvenate the electrode due to poor performance, use the following procedure:

10.3.1 Gently wipe the glass membrane with anhydrous alcohol (VWR# JT9401-11).

10.3.2 Submerge the lower 1-cm portion in the mixed acid cleaner (9.5) for a period of 5 minutes. This procedure must be performed in a fume hood.

10.3.3 Remove the electrode and rinse thoroughly with de-ionized water.

10.3.4 Soak the lower 1-cm in concentrated NH_4OH (VWR # JT9721-01) for 15 minutes (longer if the slope falls below 0.950). This procedure must be performed in a fume hood.

10.3.5 Remove the electrode and rinse thoroughly with de-ionized water.

10.3.6 Store the probe in saturated KCL for at least 10 minutes before use.

11. PROCEDURE

11.1 Solid Sample Preparation

11.1.1 Weigh 50 g +/- 0.5 g of solid sample into 250 mL HDPE containers. Add 50 mL of RO water. Larger or smaller volumes of sample can be used as long as a 1:1 ratio between solid to water is maintained. A ratio of 1:2 or 1:3 (solid:water) may be required for organic soils to minimize matrix affects.

11.1.2 Stir the suspension on the orbital shaker for 10 minutes at 175 RPM.

11.1.2.1 Turn on the orbital shaker by depressing the Power button in the lower right hand corner of the display.

11.1.2.2 Press the "CONT." button. A green light to the right of the button will indicate continuous shaking has been selected.

11.1.2.3 Press the "Speed" button. A green light to the right of the button will indicate speed parameters are being selected. Input 175 followed by the "Enter" button to set the speed at 175 RPM. The orbital shaker will begin rotating at the set speed.

11.1.2.4 Once the shaker is rotating at the set speed press the "Timed" button. Input 10 followed by the "Enter" button. The shaker will continue operating at the current conditions for 10 minutes. The shaker will automatically shut-off after 10 minutes. The digital readout will count backward indicating the time, in minutes, remaining.

11.1.3 Let the soil suspension stand for 1 hour to allow the suspended clay to settle out from the suspension. The sample may be centrifuged prior to decanting the aqueous layer for analysis.

11.2 Water Sample Preparation

11.2.1 Samples are allowed to reach room temperature before analysis.

11.3 Calibration

11.3.1 Open the Titrino program in the computer by double clicking on the Titrino icon. When the User-Password box opens, type "MANAGER" and click enter without entering a password.

11.3.2 Click on the Titrator icon ("T"), and activate the sample table by clicking on the a/e icon.

11.3.3 To build the sample table, select "Silo" from the toolbar, then select "Insert After". This will make spots on the table for the sample numbers and QC to be placed. Selecting the shortcut icon under the toolbar can also do this.

11.3.4 Insert the sample number under the Id#1 column on the screen following the nomenclature shown in 11.4.2. The first two rows should be reserved for data associated with the pH calibration (slope value) and the initial LCS to verify calibration. If more samples need to be added, select the sample add icon located in the toolbar. If any need to be removed, click the sample delete icon.

11.3.5 After the sample table is complete, click the a/e icon to lock the table (should be shaded dark grey in color). Analysis is ready to begin.

11.3.6 Pull the rubber plug from the side, near the top of the electrode. Remove the probe from storage solution, rinse with RO water and gently blot dry with a KimWipe™.

11.3.7 Calibrate the meter with pH buffer solutions 4 and 7 daily or with each use. For samples with pH values greater than 7.5, verify linearity by analyzing the LCS-High (9.4.2) with the samples. Place a magnetic stirrer in the cups and place the cups on the stir plate. Stir the buffers during the calibration procedure by turning the magnetic stirrer on.

11.3.8 Using the 736 GP Titrino keyboard, press the "**User Meth (3)**" button to download the method. "Recall Method" will show on the screen, press "**Enter.**" Press "**Select**" until the "Cal74" appears. Press "**Enter**", and then "**Start.**"

11.3.9 The keyboard will prompt for Temperature, update temperature (enter 25 for 25°C correction) using the numeric keypad and press "**Enter.**" The screen will prompt for the pH of the first calibration standard, which is routinely 7. This first standard can be updated by typing the pH value and pressing "**Enter.**" Press "**Start.**" The meter will start calibrating the first buffer solution. The screen will show readings in mV.

11.3.10 When the first standard is finished, the 736 GP will prompt the analyst for the next calibration standard. Remove the probe from the first calibration standard, rinse the probe with RO water, and blot using a KimWipe™. Place the probe into the 4 buffer solution. Press "**Start.**"

11.3.11 When calibration is complete, the slope will appear on the Titrino screen and be saved by the software as the first data point associated with the analysis. The slope must be 0.95 or greater. If the slope is not greater than 0.95, repour the buffers and recalibrate the pH meter. If the slope is still less than 0.95, follow the steps in section 10 (Preventative Maintenance), and also consult the pH probe instructions for maintenance. If these steps do not resolve the problem, check with a senior chemist or the section supervisor. The slope value must be recorded in the pH logbook and in the pH Instrument Logbook under "Comments."

11.3.12 Verify the calibration curve, by analyzing a laboratory control sample. The pH for the LCS-low is 6.86 and the acceptance window is +/- 0.1 pH units. The pH for the LCS-High is 10.01 and the acceptance window +/-0.1 pH units. If the LCS is out of control, pour and analyze a fresh laboratory control sample. If the LCS is still out of acceptable range, recalibrate the pH meter using fresh calibration buffer solutions.

11.3.12.1 Press "**Start**" on the Titrino keypad to begin monitoring data collection.

11.3.12.2 When the reading has stabilized, press "**Stop**". The pH reading acquired will be displayed on the Titrino screen and saved by the software.

11.4 Solid Sample Analysis

11.4.1 All samples and standards must be stirred during the analysis.

11.4.2 In the "pH Analysis Laboratory Logbook", located above the instrument, construct a sample table resembling the one below (L stands for "Lab" and #####-# for the sample number). Those quality control (QC) requirements listed in section 9.3 should be followed when the analytical sequence is constructed, starting with the low-level laboratory control sample (9.3.2), including the laboratory duplicates, and ending with a laboratory control sample within the range of the samples. Record the date, analyst's initials, sample number, QC samples, and any comments associated with the samples.

Date/Initials	Sample #	Comment
MM/DD/YY	LCS LOW	
Initials	L#####-#	
	L#####-# LD	
	L#####-#	
	L#####-#	
	L#####-#	
	L#####-#	
	L#####-#	
	L#####-#	
	L#####-#	

11.4.3 Following the LCS and each sample, rinse the pH probe with copious amounts of RO water and gently blot dry with a KimWipe™.

11.4.4 Put the probe into the top portion of the sample supernatant. Care is needed to place the electrode in deep enough to establish sufficient electrical contact without disturbing the solid material (samples may be centrifuged and the supernatant poured into a clean cup).

11.4.5 Press "**Start**" on the Titrino keypad to begin monitoring data collection.

11.4.6 When the reading has stabilized, press "**Stop**". The pH reading acquired will be displayed on the Titrino screen and saved by the software.

11.5 Liquid Sample Analysis

11.5.1 All samples and standards must be stirred during the analysis.

11.5.2 Analysis can be conducted manually or automated for liquid samples depending on volume limitations and/or analyst preference. Manual analysis is preferable when limited volume is available. A minimum of 100mL of sample is required for automated analysis.

11.5.3 Follow instructions described above for solids (11.4) if manual analysis is desired. For automated analysis follow the instructions described below.

11.5.4 Loading the Automated Method – follow the instructions for recalling a method described under section 11.3.8. Choose the method "True ALK" when it appears on the on the Titrino LCD screen. This method allows for automated analysis of pH.

11.5.5 Sample setup - Invert the sample bottle slowly several times to homogenize the sample. In a 250 mL, pre-labeled (labeled to know which sample goes to which cup), plastic sample cup, dispense a 100 mL aliquot of the sample. Do not allow time between the shaking and the pouring of the sample, since homogeneity is desired. Line the cups up on the autosampler according to the sample table, following the manufacturer's diagram printed on the Metrohm 674 Sample Changer. Before the run is started, make sure the electrode is resting in blank water in position one on the autosampler. Also confirm that the pH electrode is placed into the holder in position 1 before the analysis.

11.5.6 664 Control Unit - Turn the 664 power unit on; flip the red switch to Titrino; the stirrer to 1; and set the 664 unit counter to the number of samples to be run including all the QC. Of the two stations on the autosampler on which an analysis can take place, pH is analyzed on the right station, which has a number "1" taped to it. The other station is for conductivity.

11.5.7 Starting and Monitoring the Run - When the 664-control unit is set up for analysis, toggle the red switch to start. The autosampler will then begin the analysis and start to advance, and the computer will start to analyze and collect data. Clicking View in the toolbar, and going to Result can monitor the analysis.

11.5.8 When the analysis is complete, the autosampler will stop loading samples. The samples can then be disposed of according to the Conventionals Unit Sample Disposal SOP #

03-05-006-000, and the pH probe can be placed back in storage solution. The data can then be converted to a text format according to section 14.0 (Reporting).

11.5.9 After the data has been successfully transferred into an excel spreadsheet, the Titrino software can be closed. The Titrino 736 stays on and is not shut off.

12. QA / QC REQUIREMENTS

12.1 Laboratory Control Sample – Measure the pH initially, after every 20 samples, and at the end of analysis. The pH for the LCS-Low is 6.86 and the acceptance window is +/- 0.1 pH units. The pH for the LCS-High is 10.01 and the acceptance window is +/-0.1 pH units. If the LCS is out of control, pour and analyze a fresh laboratory control sample. If the LCS is still out of acceptable range, recalibrate the pH meter using fresh calibration buffer solutions.

12.2 Laboratory Duplicate – For liquids, measure one laboratory duplicate per matrix per 20 samples. The acceptance range for the laboratory duplicate is ± 0.2 pH units of the background sample. If the duplicate is out of control, verify the calibration with a LCS and re analyze the sample and it's duplicate.

12.3 Laboratory Triplicate – Measure one laboratory triplicate per solid matrix per 20 samples. The RSD acceptance limit for laboratory triplicates in soils is 2%. Reanalyze if out of control.

13. DOCUMENTATION

13.1 Reagent Logbook – Record date prepared, initials, manufacturer, lot number, volume prepared, concentration, and comment. Each standard or reagent used for this test is prepared in the pH reagent logbook. The identification number for each standard and reagent is the preparation date of the standard and the lot number of the chemical used to make the standard.

13.2 Instrument Logbook – Document calibration of pH meter, include date, slope efficiency, calibration check, and initials of the analyst.

13.3 Laboratory Logbook – For solids, record the date, sample numbers, sample weight, and volume of RO water added and pH results. For liquids, record the date of analysis, and the projects analyzed. For both parameters, record the calibration slope, the calibration check standards, and any observations or unusual sample appearances.

14. REPORTING

14.1 Data Reduction – Calculated values are transferred automatically to the computer via the Brinkmann Titrino Workcell software. Once the analysis of samples is completed, the following steps should be taken to adjust the data to a usable format for LIMS.

14.1.1 After the analysis is terminated, click on "View" and on the pull-down menu click "Results." All the result files that were generated during analysis will be displayed in the top field of the Result Manager screen. Highlight the entire batch by performing the following actions. Click on the first sample and scroll down to the last sample. Hold down the Shift key and click on the last sample.

14.1.2 Click on the "X" icon, which will initiate the Titrino Workcell "Query Tag" function. This allows for the processing of raw data using an Excel template. Double click on "Physicals Excel Format.xlt" and wait for the list of all reporting parameters to display on the screen.

14.1.3 Double click on "System ID", "ID1", "Result1Value", "Units", "Rundate", "Runtime", and "Method ID" in that order. Next click on "OK." Data for each of the selected parameters for each sample is transferred into an Excel worksheet.

14.1.4 Save this file to a personal folder on the K: drive for data reduction and reporting purposes.

14.1.5 Export the data onto the C: drive under LabWare4.3/Titrino/Result/Export/pH. When saving the file, name it with a year/month/ day nomenclature, preceded by the prefix pH (i.e. pH990125). Additionally a letter should be added to the end of the file name if multiple analyses are performed on the same day (i.e. pH990125a for the first run of the day). Exporting is accomplished by first highlighting the data following the same procedure used in 14.1.1 above. Clicking on the "Properties" button followed by "Edit" prior to exporting the data allows the analyst to change the path where data will be sent. Check the path prior to exporting the data to ensure it is transferred to the designated location stated above. Clicking the export icon on the Result Manager screen will transfer all chosen data to the export folder.

14.1.6 Archive the data onto the C: drive under LabWare4.3/Titrino/Result/Archive/pH. When saving the file, name it with a year/month/ day nomenclature, preceded by the prefix pH (i.e. pH990125). Additionally a letter should be added to the end of the file name if multiple analyses are performed on the same day (i.e. pH990125a for the first run of the day). Archiving is accomplished by first highlighting the data following the same procedure used in 14.1.1 above. Clicking on the "Properties" button followed by "Edit" prior to archiving the data allows the analyst to change the path where data will be sent. Check the path prior to archiving the data to ensure it is transferred to the designated location stated above. Clicking the Archive icon (treasure chest) on the Result Manager screen will transfer all chosen data to the archived folder.

14.1.7 Copy the results saved to the K: drive (14.1.4) and paste them into the "pH_Report_Template.xlt" workbook in the "Query Tag" worksheet. Be sure to paste the results under the correct headers that are established in the template. This template is stored under the directory K:\conventi\qamanual\analytical\pH_Report_Template.xlt.

14.1.8 Use the key combination CNTRL+Z to execute a macro that formats the data for printing. In the comments section enter the QC numbers associated with lab control samples and lab duplicates that will be assigned in LIMS.

14.1.9 Use the key combination CNTRL+ X to execute a macro that formats the data for CSV (Comma Separated Variable) transfer into LIMS. The CSV file will be stored in the "CSV" worksheet within the same workbook. Cut the QC sample numbers in the "Comments" field and paste them into their corresponding "Sample ID" fields.

WGXXXXX	CVPH
Sample Number	pH
Date Analyzed	MM/DD/YY
LXXXXX-X	####

14.2 Copy the CSV file to the G: drive. The format above must be followed for correct data transfer to occur into LIMS. Any deviations from this format may result in data not transferring correctly into LIMS.

14.3 The data package shall include: a copy of the laboratory notebook pages, a copy of the Excel.CSV spreadsheet sent to LIMS, LIMS data report, LIMS QC summary report, a completed internal workgroup review sheet and a corrective action form (if needed).

15. REFERENCES

15.1 Solids

15.1.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, Method 4500H-B.

15.1.2 Methods Manual for Forest Soil and Plant Analysis, Forestry Canada, 1991.

15.1.3 SW 846, Soil and Waste pH, Method 9045C, January 1995.

15.2 Waters

15.2.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, Method 4500H^+.

15.2.2 EPA Methods for Chemical Analysis of Water and Wastes, 1979, Method 150.1.

16. TRAINING

16.1 The steps followed for training a new analyst is to demonstrate the calibration of the pH meter and analyzation of pH on samples, explaining each step of the procedure, and using the SOP as an aid. The analyst-in-training then calibrates the pH meter, performs routine maintenance on the pH probe, and analyzes samples while being observed by the trainer, who answers questions, provides feedback on technique and further explains the steps in the procedure. The trainer continues to observe the analyst-in-training on a daily basis until both are comfortable that the new analyst is competent to perform the procedure. All personnel performing pH calibration and analysis will be fully trained by an experienced analyst.

Appendix B

Cross-training Checklist

CONVENTIONAL SECTION
CROSS TRAINING GUIDELINES CHECK SHEET

Test Procedure:	
Trainer:	
Trainee:	
Date Training Started:	
Date Training Completed:	

Item	**Date Completed**	**Trainer initials**	**Trainee Initials**
Review SOP Review equipment and /or instrument manuals			
Review Safety Issues: Routine safety Special precautions Hazardous reagents and waste Sample and waste disposal Housekeeping			
Review Purchasing: Chemicals (how much, how often) Spare parts (vendor, how often) Other consumables			
Review Documentation: Analytical Log Book Instrument Log Book Reagent / Standards Book Data Package			
Review Schedule for: Reagent preparation Standards preparation QC samples preparation Instrument or equipment maintenance			
Review Projects: Routine workload (which customers, LPMs) Special requests Typical ranges for routine samples			
Observe analytical procedure			
Perform analytical procedure together			
Perform analytical procedure independently using one blind set of 4 replicates, prepared by the trainer			

Appendix C

Data Package

METRO Environmental Laboratory

WORK GROUP REPORT (wk02)

Jun 29 2000, 04:12 pm

Work Group: WG49696 (conductivity) for Department: 3 - Conventionals

Created: 29-JUN-2000 Due: Operator: Kristen/Gretchen

Sample	Project Number	Project Description	PKey	C	Product	Matrix	Stat	UA	Workdate	Duedate
L18247-1	423121	Madsen Creek Monitoring		S	COND	FRESH WTR	WKGP	U	19-JUL-00	19-JUL-00
WG49696-1	LCS			S	COND	BLANK WTR	WKGP	U	29-JUN-00	
WG49696-2	LD			S	COND	FRESH WTR	WKGP	U	29-JUN-00	
WG49696-3	LCS			S	COND	BLANK WTR	WKGP	U	29-JUN-00	

Comments:

WG49696-1	LEVEL1
WG49696-2	L18247-1
WG49696-3	LEVEL2

Parameter: Conductivity

List Type: CVCOND
Run ID: R55690
Work Group: WG49696
Method Code: SM2510-B (03-01-002-001)

Col. Date	Anal.Date	Project	Matrix	Site	Locator	Sample#	Value	Units	Valq	Mdl	Rdl	Textvalue
28-JUN-00	29-JUN-00	423121	FRESH WTR	STREAMS	MADSEN1	L18247-1	179	umhos/cm		.5	10	
	29-JUN-00		BLANK WTR			WG49696-1	79.8	umhos/cm		.5	10	
	29-JUN-00		FRESH WTR			WG49696-2	181	umhos/cm		.5	10	
	29-JUN-00		BLANK WTR			WG49696-3	774	umhos/cm		.5	10	

WG49696	CVCOND	
Sample Number	umhos/cm	
Date Analyzed	6/29/00	
LCS LVL 2	774.2	WG49696-3
L18247-1 LD	180.6	WG49696-2
L18247-1	179.4	
LCS LVL 1	79.75	WG49696-1

Summary of Multiple Samples

(Select samples, then select information needed)

SystemId	Id1	Result1Value	Units	RunDate	RunTime	MethodId
1953	monthly QC	360.5	uS/cm	00-06-29	######	COND-MES
1952	LCS LVL 2	774.2	uS/cm	00-06-29	######	COND-MES
1951	L18247-1 LD	180.6	uS/cm	00-06-29	######	COND-MES
1950	L18247-1	179.4	uS/cm	00-06-29	######	COND-MES
1949	LCS LVL 1	79.75	uS/cm	00-06-29	######	COND-MES
1948	Blank	0.827	uS/cm	00-06-29	######	COND-MES

Sample Excel Report #2
Workcell 4.0

Conductivity Analysis

Date/Initials:	Sample #	System I.D #	Comment:
9/19/00 KW/OW	LCS LVL2	1778	prep#23
WG49480	L18135-5	1779	
	-6	1780	
	-7	1781	
	-8	1782	
	-9	1783	
	-10	1784	
	L18133-1	1785	
	-1LD	1786	
	-2	1787	
	-3	1788	
	-4	1789	
	-5	1790	
	-6	1791	
	-7	1792	
	LCS LVL 1	1793	prep#22
6/29/00 KW/OW	Blank	1948	
WG49696	LCS LVL 1	1949	prep# 22
	L18247	1950	
	-1LD	1951	
	LCS LVL 2	1952	prep#23
	monthly QC	1953	lot# 9998

KING COUNTY METR 'IRONMENTAL LABORATORY
Lab QC Repoı 06/30/2000 10:49
Run ID: R55690 Workgroup: WG49696 ()

LCS:WG49696-1 Matrix: BLANK WTR Listtype: CVCOND Method: SM2510-B (03-01-002-001) Project: PKey: STD

Parameter	Mdl	Rdl	Units	SampValue	Truevalue	LCS Value	% Rec.	Qual	Limits	RPD/RSD	Qual	Limits
Conductivity	.5	10	umhos/cm		73.9	79.8	108		90-110			

LD:WG49696-2 L18247-1 Matrix: FRESH WTR Listtype: CVCOND Method: SM2510-B (03-01-002-001) Project: 423121 PKey: STD

Parameter	Mdl	Rdl	Units	SampValue	Truevalue	LD Value	% Rec.	Qual	Limits	RPD/RSD	Qual	Limits
Conductivity	.5	10	umhos/cm	179		181				1		10

LCS:WG49696-3 Matrix: BLANK WTR Listtype: CVCOND Method: SM2510-B (03-01-002-001) Project: PKey: STD

Parameter	Mdl	Rdl	Units	SampValue	Truevalue	LCS Value	% Rec.	Qual	Limits	RPD/RSD	Qual	Limits
Conductivity	.5	10	umhos/cm		717.5	774	108		90-110			

Conventionals Internal Wc roup Review Sheet

Workgroup WG496916 Products COND First Due Date 19-JUL-00

	Analyst KW				Peer	Analyst	Peer
Data Package	Yes	No	NA	Comments	Revise	Revised	OK
1. Workgroup sheet included.	☑	☐	☐	☐	☐	☐	☐
2. Complete and accurate Seedpak3 data sheets and QC report.	☑	☐	☐	☐	☐	☐	☐
3. Complete and legible copies of laboratory notebook pages included.	☑	☐	☐	☐	☐	☐	☐
4. Complete and accurate copies of instrument report/calculation sheets included.	☑	☐	☐	☐	☐	☐	☐
5. Data source is correctly identified on Seedpak3 data report.	☑	☐	☐	☐	☐	☐	☐
6. Corrective Action Form included.	☐	☐	☑	☐	☐	☐	☐
7. Compromised Samples Record Form.	☐	☐	☑	☐	☐	☐	☐
Analytical and Workgroup Batch Information							
1. Instrument calibration was verified and in control.	☐	☐	☑	☐	☐	☐	☐
2. Sufficient CCVs were analyzed and results were in control	☐	☐	☑	☐	☐	☐	☐
3. Sufficient method blanks were analyzed and results less than MDL.	☐	☐	☑	☐	☐	☐	☐
4. Sufficient positive controls were analyzed and recoveries in control.	☑	☐	☐	☐	☐	☐	☐
5. Sufficient replicates were analyzed and RPD/RSD values were in control.	☑	☐	☐	☐	☐	☐	☐
6. Sufficient matrix spikes were analyzed and recoveries in control.	☐	☐	☑	☐	☐	☐	☐
7. All results are reported from analyses within working range.	☑	☐	☐	☐	☐	☐	☐
8. Detection limits adjusted to reflect any required dilutions.	☐	☐	☑	☐	☐	☐	☐
9. Have any data been qualified (other than for detection limits).	☐	☑	☐	☐	☐	☐	☐
10. QCQUAL process is complete.	☑	☐	☐	☐	☐	☐	☐
11. Date analyzed included.	☑	☐	☐	☐	☐	☐	☐
12. Holding time met.	☑	☐	☐	☐	☐	☐	☐

Analyst Comments:

Review Comments:

Workgroup approved and moved on ____________ by ____________

Appendix D

Method Detection Limit and Comparison Studies

TSS MDL study

Analyte: TSS					
Analyst: Kristen Weatherford					
Data Analyzed: 8/10/00					
Sample	Sample Volume, mL	Initial Crucible Weight, g	Final Crucible Weight, g	Weight Difference, g	TSS, mg/L assume 1000 mL sample volume
dish 1	0	1.371	1.3707	0.0003	-0.3
dish 2	0	1.381	1.3807	0.0003	-0.3
dish 3	0	1.3766	1.3764	0.0002	-0.2
dish 4	0	1.3623	1.3621	0.0002	-0.2
dish 5	0	1.3801	1.3800	0.0001	-0.1
dish 6	0	1.3873	1.3874	0.0001	0.1
dish 7	0	1.3570	1.3569	0.0001	-0.1
dish 8	0	1.3629	1.3628	0.0001	-0.1
dish 9	0	1.3648	1.3646	0.0002	-0.2
dish 10	0	1.3685	1.3684	0.0001	-0.1
MB 1	1000	1.3676	1.3673	0.0003	-0.3
MB 2	1000	1.3916	1.3914	0.0002	-0.2
MB 3	1000	1.3680	1.3679	0.0001	-0.1
MB 4	1000	1.3643	1.3644	0.0001	0.1
MB 5	1000	1.3727	1.3725	0.0002	-0.2
MB 6	1000	1.3664	1.3661	0.0003	-0.3
MB 7	1000	1.3751	1.3747	0.0004	-0.4
MB 8	1000	1.3686	1.3685	0.0001	-0.1
MB 9	1000	1.3862	1.3862	0.0000	0
MB 10	1000	1.3727	1.3728	0.0001	0.1

TDS MDL study

Analyte: TDS						
Analyst: Kristen Weatherford						
Data Analyzed: 8/10/00, 8/29/00						
Sample	Sample Volume, mL	Initial Crucible Weight, g	Final Crucible Weight, g	Weight Difference, g	TDS, assuming 100 mL sample volume, mg/L	Date Analyzed
Dish 1	0.0	106.7942	106.7948	0.0006	6.0	8/10/00
Dish 2	0.0	93.5880	93.5902	0.0022	22.0	8/10/00
Dish 3	0.0	102.9827	102.9838	0.0011	11.0	8/10/00
Dish 4	0.0	99.8440	99.8446	0.0006	6.0	8/10/00
Dish 5	0.0	100.0607	100.0608	0.0001	1.0	8/10/00
Dish 6	0.0	105.4058	105.4057	0.0001	-1.0	8/10/00
Dish 7	0.0	95.4451	95.4461	0.0010	10.0	8/10/00
Dish 8	0.0	113.0428	113.0438	0.0010	10.0	8/10/00
Dish 9	0.0	103.7407	103.7402	0.0005	-5.0	8/10/00
Dish 10	0.0	100.2587	100.2581	0.0006	-6.0	8/10/00
MB 1	100.0	104.1508	104.1523	0.0015	15.0	8/10/00
MB 2	100.0	113.7710	113.7714	0.0004	4.0	8/10/00
MB 3	100.0	102.9444	102.9425	0.0019	-19.0	8/10/00
MB 4	100.0	108.8331	108.8328	0.0003	-3.0	8/10/00
MB 5	100.0	98.6327	98.6326	0.0001	-1.0	8/10/00
MB 6	100.0	104.2522	104.2518	0.0004	-4.0	8/10/00
MB 7	100.0	94.7940	94.7896	0.0044	-44.0	8/10/00
MB 8	100.0	95.4346	95.4357	0.0011	11.0	8/10/00
MB 9	100.0	98.6361	98.6356	0.0005	-5.0	8/10/00
MB 10	100.0	102.9555	102.9562	0.0007	7.0	8/10/00
DISH 1	0.0	102.5227	102.5232	0.0005	5.0	8/29/00
DISH 2	0.0	98.2167	98.2162	0.0005	-5.0	8/29/00
DISH 3	0.0	106.7946	106.7952	0.0006	6.0	8/29/00
DISH 4	0.0	100.4932	100.4934	0.0002	2.0	8/29/00
DISH 5	0.0	106.3092	106.3084	0.0008	-8.0	8/29/00
DISH 6	0.0	104.9594	104.9595	0.0001	1.0	8/29/00
DISH 7	0.0	100.2115	100.2113	0.0002	-2.0	8/29/00
DISH 8	0.0	107.3418	107.3430	0.0012	12.0	8/29/00
DISH 9	0.0	103.7401	103.7405	0.0004	4.0	8/29/00
DISH 10	0.0	103.7176	103.7179	0.0003	3.0	8/29/00
MB 1	100.0	102.4338	102.4345	0.0007	7.0	8/29/00
MB 2	100.0	104.2529	104.2528	0.0001	-1.0	8/29/00
MB 3	100.0	104.1520	104.1519	0.0001	-1.0	8/29/00
MB 4	100.0	100.9997	100.9997	0.0000	0.0	8/29/00
MB 5	100.0	98.6317	98.6326	0.0009	9.0	8/29/00
MB 6	100.0	102.9841	102.9834	0.0007	-7.0	8/29/00
MB 7	100.0	100.2586	100.2580	0.0006	-6.0	8/29/00
MB 8	100.0	105.4070	105.4056	0.0014	-14.0	8/29/00
MB 9	100.0	102.9429	102.9420	0.0009	-9.0	8/29/00
MB 10	100.0	95.4469	95.4464	0.0005	-5.0	8/29/00

TSS comparison study

TSS Comparison Study: Blended vs. Unblended Samples						
Units: mg/L						
Analyst: Kristen Weatherford						
Sample #	Unblended	Blended	Date Analyzed	Comments		
L18418-1	700.00	680.00	7/31/00	WG50286		
L18418-3	594.74	564.71	7/31/00	WG50286		
L18422-1	2000.00	1490.91	7/31/00	WG50286		
L18422-3	2133.33	2170.00	7/31/00	WG50286		
L18422-5	2600.00	2880.00	8/2/00	WG50337		
L18425-1	338.00	269.23	7/31/00	WG50286		
L18425-3	510.42	438.30	7/31/00	WG50286		
L18430-1	266.67	273.33	7/31/00	WG50286		
L18430-2	230.43	305.26	7/31/00	WG50286		
L18430-3	175.00	165.22	7/31/00	WG50286		
L18473-1	747.06	1490.91	8/11/00	WG50506		
L18473-3	663.16	652.63	8/11/00	WG50506		
L18473-5	378.26	407.41	8/11/00	WG50506		
L18507-1	2318.18	1263.06	8/11/00	WG50506		
L18473-9	710.53	881.25	8/11/00	WG50506		
L18473-11	262.96	260.87	8/11/00	WG50506		
L18473-13	571.43	612.50	8/11/00	WG50506		
L18505-1	165.00	193.33	8/11/00	WG50506		
L18521-1	1323.53	1109.09	8/11/00	WG50506		
L18473-15	1069.57	858.33	8/11/00	WG50506		

Weatherford, Kristen

From: Georgianna, Tom
Sent: Tuesday, September 12, 2000 2:18 PM
To: Weatherford, Kristen
Cc: Strong, Despina
Subject: RE: TSS comparison study

Kristen:

Here are the results of the statistical analysis of your TSS data. The data were log-normally distributed, so I used a natural log transform. The resultant t-test indicates that there is no statistically significant difference between the blended and unblended data. A subsequent regression on the log transformed data yields an R square of .954, an intercept of zero, and a slope of 1.0. In other words the two data sets are almost identical, statistically speaking.

From: Weatherford, Kristen
Sent: Monday, September 11, 2000 12:25 PM
To: Georgianna, Tom
Cc: Strong, Despina
Subject: TSS comparison study

Tom,

I am doing a study between two different methods for TSS analysis. I would like to know if there is a difference between the two methods of analysis. The study involves industrial waste samples - some samples were blended before analysis, and some were not. The data is in the attached file. My last day is this Friday, 9/15; if you do not have time to analyze the data before Friday, please email the results to Despina.

Thank you,

Kristen Weatherford

<<File: TSS comparison study-Tom.xls>>