

University of Maine
Agricultural & Forestry Experiment Station
Analytical Laboratory
Quality Control/Quality Assurance Plan

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Quality Assurance Project Plan Identification

Document Title: Quality Assurance Plan for MAFES Analytical Laboratory

Organization Title: MAFES Analytical Laboratory

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Plan Coverage: This document describes the quality control measures to be followed by the MAFES Analytical Laboratory (Analytical Lab). The quality control measures will apply to all analytical data measurements run on regulated materials for the purpose of environmental monitoring.

Table of Contents

- 1.0 Introduction
- 2.0 Program Description
 - 2.1 General description
 - 2.2 Intended use of data
- 3.0 Program Organization and Responsibility
 - 3.1 Program organization and line of authority
 - 3.2 Identification of key QA personnel
- 4.0 Quality Assurance Objectives.
 - 4.1 Precision and accuracy for each parameter
 - 4.2 Data quality objectives
- 5.0 Sampling Procedures
 - 5.1 Techniques or guidelines used to select sites
 - 5.2 Specific procedures to be used
 - 5.3 Charts, diagrams or table of sampling operations
 - 5.4 Containers and tools
 - 5.5 Sampling equipment and container preparation
 - 5.6 Sample preservation and holding times prior to delivery
 - 5.7 Chain-of-Custody procedures
 - 5.8 Record keeping (forms, notebooks and documentation procedures)
- 6.0 Sample Custody
 - 6.1 Field sampling documentation
 - 6.2 Laboratory operations
 - 6.2.1 Sample rejection criteria
 - 6.2.2 Monitoring of sample holding times
 - 6.2.3 Identification of sample custodian
 - 6.2.4 Sample custody log
- 7.0 Calibration / Standardization Procedures and Frequency
- 8.0 Analytical Procedures
 - 8.1 Method reference
 - 8.2 List of MDL's
- 9.0 Data Reduction, Validation and Reporting
 - 9.1 Data reduction scheme
 - 9.2 Equations used to calculate concentrations
 - 9.3 Data validation criteria
 - 9.4 Identification and treatment of outliers
 - 9.5 Data flow on reporting scheme

- 10.0 Internal Laboratory QC Checks and Frequency
 - 10.1 Quality control charts
 - 10.2 Blanks
 - 10.3 Reference samples
 - 10.4 Duplicate analysis
 - 10.5 Matrix spike
 - 10.6 Matrix spike duplicate
 - 10.7 QC check samples
 - 10.8 Calibration standards
 - 10.9 Reagent checks
 - 10.10 Blind field spikes and duplicates
- 11.0 Performance and System Audits
 - 11.1 Schedule
- 12.0 Equipment
 - 12.1 Decontamination procedures
 - 12.2 QC monitoring (Temperatures, etc.)
 - 12.3 Preventive maintenance schedule, procedures, and logs
 - 12.4 Critical components
- 13.0 Assessment Procedures for Data Acceptability
 - 13.1 Precision acceptance limits
 - 13.2 Accuracy acceptance limits
- 14.0 Corrective Action
 - 14.1 Limits
 - 14.2 Procedures
 - 14.3 Identification of responsible personnel
- 15.0 Quality Assurance Reports to Management
 - 15.1 Schedule
- 16.0 List of Recipients of Quality Assurance Plan
- 17.0 Appendices
 - 17.1 Appendix A - General Quality Assurance Goals for Analytical Parameters
 - 17.2 Appendix B - Individual Method SOP's

1.0 Introduction

This plan is intended to provide a formal guideline to ensure accurate and precise analytical results generated by the MAFES Analytical Laboratory. The primary function of the Analytical Lab is the analysis of horticultural and agricultural-related products such as soils, plants, feed, compost, manure, and other waste samples for nutrient content. Environmental and nutrient management rules require monitoring of many of these materials for potentially toxic or environmentally sensitive elements. A quality assurance/quality control program is essential to ensuring accuracy and precision in environmental analysis. The outlined quality control measures are primarily directed toward environmental and regulated materials analysis, but the applicable techniques for ensuring accurate and precise results will also be applied towards general nutrient analysis for horticultural and agricultural applications.

2.0 Program Description

2.1 General description

The Analytical Lab is the general service lab of the Maine Agriculture & Forestry Experiment Station and is administratively within the School of Food and Agriculture at The University of Maine. The Analytical Lab exists to serve the needs of research programs at the University of Maine, the agricultural/horticultural community, and any other government agency, company, or individual with regard to chemical analysis of soil, water, plants, feed, and waste materials such as manure, sewage sludge, and processing waste. Interpretation of soil fertility and plant tissue test results is provided. Recommendations for soil and foliar amendments are made in conjunction with the University of Maine Cooperative Extension, where appropriate. General consultations on test results and testing methods are provided upon request.

2.2 Intended use of data

Data generated at the Analytical Lab is intended to determine the quality of soil and waste products for use in plant growth or crop production, to determine the nutrition of plants, and to assess whether soil or waste materials could pose a hazard to plants, animals, humans, or the environment.

3.0 Program Organization and Responsibility

3.1 Program organization and line of authority

Organization of Analytical Lab is presented below with the highest level of responsibility being represented at the top of the diagram.

Laboratory Organization:

Assistant Chemist/Supervisor:	Suzanne Perron
Assistant Scientist/Supervisor:	Bruce Hoskins
Scientific Technician:	J. Elmer
Scientific Technician:	A. Chase
Scientific Technician:	R. Robey
Scientific Technician:	K. Senter
Data Management Technician:	S. Cashman

3.2 Identification of key QA personnel

Both Laboratory Supervisors function as Quality Control Officers within the laboratory and are responsible for ensuring that the data produced by the technicians meet the specified QA/QC plan.

4.0 Quality Assurance Objectives

4.1 Precision and accuracy for each parameter

General data quality objectives for accuracy and precision for all analytes are listed in appendix 17.1.

Specific data quality objectives for accuracy and precision for each method are listed within individual method SOP's, appearing in appendix 17.2.

4.2 Data quality objectives

The basic quality assurance objectives are to produce accurate and precise analytical results. Each client will ensure that their sampling techniques will yield results that are representative of the system being measured.

5.0 Sampling Procedures

5.1 Techniques or guidelines used to select sites

Guidelines used to select sites are determined by clients.

5.2 Specific procedures to be used

Specific sampling procedures used are determined by clients. These will vary with the type of material and specific objectives. It is the responsibility of the client to ensure proper sampling of materials submitted to Analytical Lab, which must be addressed within each client's respective Quality Assurance Project Plan.

5.3 Charts, diagrams or tables of sampling operations

Sampling operations are performed by clients and should be addressed in their respective Quality Assurance Project Plans.

5.4 Containers and tools

The Analytical Lab provides cardboard sample containers for soil samples only. These are available at any county office of Cooperative Extension or by request on the lab's web site: <http://maine.edu/soiltestinglab>. Sample container recommendations are as follows:

- A. Manure, sludge, processing waste - sealed pint or quart plastic container.
- B. Compost - sealed plastic gallon bag.
- C. Soil sampling for nutrients or metals - avoid galvanized or brass tools or containers. Use steel, stainless steel or plastic tools and buckets. Use cardboard or plastic shipping containers.

5.5 Sampling equipment and container preparation

The choice, decontamination, and proper use of sampling equipment and container preparation is the responsibility of the client and should be addressed in their respective Quality Assurance Project Plans.

5.6 Sample preservation and holding times prior to delivery

- A. Manure, sludge, processing waste: keep cool at 4°C or frozen at -18 °C
- B. Compost: keep cool at 4 °C.
- C. Soil nitrate analysis: deliver to the lab or completely dry on the same day sampled.
- D. Nitrate, ammonium, or orthophosphate in undried samples: keep cool at 4 °C and deliver to the lab within 24 hours or freeze at -18 °C and deliver to the lab frozen.

5.7 Chain-of-Custody procedures

The chain-of-custody is initiated by the client. If the client does not have access to chain-of-custody forms, the general Analysis Request form for the Analytical Lab may be used. Analysis Request/COC forms may be downloaded from the lab's website: <http://umaine.edu/soiltestinglab>. All chain-of-custody samples will be received by a Supervisor. Chain-of-custody form(s) will be stored with the Analysis Request form in 407 Deering.

5.8 Record keeping (forms, notebooks and documentation procedures)

The client is responsible for record keeping concerning sampling.

6.0 Sample Custody

6.1 Field sampling documentation

Sample custody during field operations is the responsibility of each client and should be addressed in their respective Quality Assurance Project Plans.

6.2 Laboratory operations

6.2.1 Sample rejection criteria

Upon arrival at the laboratory, the samples are checked for appropriate appearance. Any apparent problems will be documented. If a problem appears to exist, the client will be contacted before the samples are analyzed. The laboratory retains the right to reject any sample that, in the opinion of a QA officer, has not been properly handled prior to arrival at the laboratory or may contain substances harmful to laboratory personnel.

6.2.2 Monitoring of sample holding times

A computerized sample-tracking database is maintained for all samples submitted to the laboratory, containing the date the samples are received, the analysis requested, the expected date of completion, and the date the analysis was actually completed. Sample holding times can be determined from the “date received” in the computer record and recorded on the Analysis Request form, stored in 407 Deering.

6.2.3 Identification of sample custodian

Each of the Supervisors functions as a sample custodian. An available Supervisor will log in the sample(s). After sample archival times are exceeded, samples are disposed of with the approval of one of the Supervisors.

6.2.4 Sample custody log

Upon arrival at the laboratory the sample(s) should be accompanied by an Analysis Request form, which will be stored in a notebook (along with any chain-of-custody form or other documentation) in 407 Deering. A parallel record is kept in a computerized sample-tracking database. The Analysis Request form and database contain the client's name, client's sample identification, date received, assigned laboratory job number, laboratory analyses to be performed, report date, and billing information. All identification and sample collection information provided with the sample will be kept in the notebook.

7.0 Calibration / Standardization Procedures and Frequency

Instrument calibration will be completed with each batch of analyses following the instrument manufacturers' recommended procedures. The number and concentration of standards for each parameter is specified within each method SOP, in Appendix 17.2. Calibrated instrument response will be checked by running a QCCS a minimum of every 10 samples. Control limits are 90 - 110 % recovery of known content for all analytes. On QC failure, the instrument will be recalibrated and all samples rerun since the last QCCS in control.

Calibration standards will be prepared whenever possible in the same matrix as the prepared samples.

8.0 Analytical Procedures

8.1 Method reference

Analytical procedures will follow published and approved methodologies. Method references are listed at the end of each SOP, appearing in Appendix 17.2. Methods are adapted from EPA, AOAC, USDA-NRCS, Standard Methods for the Analysis of Water & Wastewater (SM), Test Methods for the Evaluation of Composting and Compost (TMECC), Recommended Methods for Manure Analysis, and regional plant and soil testing methods manuals. Methods are periodically reviewed by Supervisors, with any changes documented within the written SOP.

8.2 List of MDL's

The method detection limit (MDL) is 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. It is statistically determined as 3 times the standard deviation of at least 10 repeat measurements of a method blank or matrix blank. Method detection limits are specific to calibration standard concentrations and the sample matrix and are given within each SOP, appearing in Appendix 17.2.

9.0 Data Reduction, Validation and Reporting

9.1 Data reduction scheme

The data reduction scheme depends on the analytical procedure. Final results for sample pH or EC are read directly from the calibrated meter. Data output from combustion analysis is reported directly as the final concentration for each sample.

Data generated from solution analysis on ICP, IC, or ion analyzer are output as concentrations in the analyzed solution. Further calculations to factor in any

blank correction and extraction/digestion dilution factor are completed by a Supervisor.

For other analyses, which do not compute final concentrations, measured values, such as titration volumes, are used by a Supervisor to calculate final reported values.

9.2 Example equation used to calculate concentrations

For solution analysis:

$$\text{Final sample conc} = (\text{measured soln conc} - \text{reagent blank conc}) * (\text{dilution factor})$$

For additional equations, refer to individual method SOP's in appendix 17.2.

9.3 Data validation criteria

Data validation and integrity during sample collection are the responsibility of the client and should be addressed in their respective Quality Assurance Project Plans.

Analytical data will be assessed for precision within each batch of samples run, by replicating ten percent of all samples run for each parameter, unless otherwise specified in method SOP. Replicate percent differences (RPD's) will be calculated for each replicated analysis. Control limits on replicated samples will be 10 % RPD, unless concentration is < 2 x MDL or otherwise specified in Appendix 17.1 or in the method SOP.

Precision will also be assessed by repeat measurement of internal reference samples, run with each batch of samples. Results from ongoing reference sample analysis will be compared using quality control charts and appropriate statistical analyses. Precision control limits will be the mean +/- 3 SD based on a population of at least 20 data points. The mean and standard deviation will be recalculated with every batch of samples.

Data accuracy will be assessed by running at least one internal reference and/or one SRM (standard reference material) with every batch of samples. Percent recoveries of known content will be calculated for SRM's. Control limits will be 90 - 110 % recovery of known content for all analytes, unless otherwise specified in Appendix 17.1 or in the method SOP. Accuracy control limits for internal reference samples will be 90 - 110 % recovery of statistical mean content for all analytes. Means will be based on a population of at least 20 data points, recalculated with every batch of samples.

Any data exceeding control limits will initiate corrective action outline in section 14.0.

9.4 Identification and treatment of outliers

Outliers will be determined according to the criteria listed in section 9.3. Samples in analysis runs containing outliers will be reanalyzed. The analyst will try to determine the cause of the outlier prior to reanalysis.

9.5 Data flow on reporting scheme

The laboratory file server is divided into several subdirectories, specific to each analytical process, which are archived at the end of each working day. Titration, EC, and miscellaneous pH data will be recorded by the technician on laboratory data sheets and in the appropriate spreadsheet or database on the server. Soil pH and all sample weights are computer-captured and automatically stored in the appropriate database or spreadsheet on the server. Data output from ICP, IC, combustion analysis, and ion analyzer is generated in batch files by each operating system. These batch files are also stored in the appropriate subdirectory on the server. All data are referenced to analytical job number and by sample ID or lab number.

All reports are generated, validated, and reviewed by one of the Supervisors before being sent to the client. Data accuracy and analytical precision are checked based on reference sample and replicate analysis, respectively. Related parameters are cross-checked to further validate results. Any potential problems will be checked before the analytical results are released. A paper copy of the report will be held in the files for five years. Electronic copies of all reports will be held for five years or longer, as space permits.

10.0 Internal Laboratory QC Checks and Frequency

10.1 Quality control charts

Quality control data will be kept for routinely analyzed parameters on internal reference samples and SRM's. Individual analyses of reference samples will be plotted on quality control charts versus the cumulative mean and two and three times the standard deviation.

10.2 Blanks

A calibration blank is an aqueous solution that is as free of analyte as possible. It is prepared in the same matrix used in the preparation of calibration solution(s). The calibration blank is used to give the null reading for the instrument response versus concentration on the calibration curve. One calibration blank should be analyzed as part of each calibration procedure, where normal sample concentration range approaches MDL.

A reagent blank is an aqueous solution that is as free of analyte as possible and contains all the reagents used in processing the samples. The reagent blank must be

carried through the complete sample preparation procedure and contains the same reagent concentrations in the final solution as in the sample solution used for analysis. The reagent blank is used to document and correct for any systematic contamination from reagents, containers, or the lab environment in the preparation or processing of the samples. Two reagent blanks should be prepared for every sample batch or for every 20 samples, whichever is more frequent, unless otherwise specified in method SOP.

10.3 Reference samples

To verify extraction or digestion efficiency, a standard reference material (SRM) will be analyzed with every batch of 20 samples, where an appropriate material exists. Accuracy will be documented as percent recovery of known or guaranteed content for each parameter.

Where no SRM is available, an internal reference samples will be carried through the digestion or extraction procedure with every batch of 20 samples. Accuracy will be documented as percent recovery of statistical average content for each parameter. Where possible, internal reference sample content will be validated by parallel analysis with one or more SRM's for all parameters.

Repeat analysis of an internal reference material is also used to document precision. Cumulative mean and standard deviation statistics are generated to document long-term method precision. The data will be documented on quality control charts for each parameter.

10.4 Duplicate or replicate analysis

A duplicate sample analysis will also be performed with every sample batch or every 10 samples, unless otherwise specified in method SOP. Precision data will be documented as replicate percent difference (RPD) for each parameter.

10.5 Matrix spike

A matrix spike is employed to provide a measure of bias for the method used within a given matrix. A matrix spike analysis is performed by adding a predetermined quantity of stock solutions of certain analytes to a duplicate sample matrix prior to sample extraction/digestion and analysis. Where required, a matrix spike sample will be analyzed with every batch or every 20 samples, whichever is more frequent.

10.6 Matrix spike duplicates

When required, a duplicate of the matrix spike sample will be analyzed with the matrix spike sample, if a matrix spike is required.

10.7 Quality control check samples

A quality control check sample (QCCS) is a sample prepared from an independent standard at a concentration within the calibration range. An independent standard is defined as a standard derived from a different source than that used in the preparation of calibration standards. QCCS solutions can be purchased ready-made from several suppliers. A QCCS is intended as an independent check of technique, methodology, and standards and should be run with every solution analysis batch.

10.8 Calibration standards

Calibration standards will be prepared according to instrument manufacturer's suggested guidelines. Two-point calibrations are used for ICP analysis. Multi-point calibrations are used for IC, ion analyzer, and combustion analysis. Standards will be checked against previous standards and validated by running a QCCS. If QCCS analysis does not meet 90 - 110% recovery control limit for any analyte, the calibration standards will be remade. The standard curve and instrument response will be verified by analysis of at least one calibration standard or QCCS every 10 samples.

For matrices other than water, matrix-matched calibration standards shall be used.

10.9 Reagent checks

Reagents used in sample and calibration standard preparation shall be verified as free of analyte (below MDL) by analysis of the reagent blanks. If one or more analytes are consistently detectable in the reagent blanks and the source cannot be eliminated, the average reagent blank content is subtracted from each of the samples run in the same batch with the blank(s). This will correct for any systematic error, due to reagent content of each analyte.

10.10 Blind field spikes and duplicates

Blind field spike and duplicates are the responsibility of the client and should be addressed in their respective Quality Assurance Project Plans.

11.0 Performance and System Audits

11.1 Schedule

The Analytical lab participates quarterly in the North American Proficiency Testing (NAPT) program for soil fertility analysis, soil metals analysis, and plant nutrient analysis.

System audits will be carried out by the Supervisors upon repeated QC failure of any method, to evaluate relevant components of the laboratory QA/QC system.

12.0 Equipment

12.1 Decontamination of equipment

All labware used for digestion, extraction, or solution storage is processed for decontamination between each use. All containers are rinsed with deionized water after use and any adhering particulates or colloid are removed by scrubbing with a laboratory detergent and tap water. All containers, stoppers, and caps are then soaked in 0.2 % $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ cleaning solution overnight to desorb any adhering ions. All labware is then rinsed 3 times with deionized water. Open containers are then allowed to dry upside down on a plastic drying rack and then stored in a closed cupboard. Capped or stoppered containers are filled with deionized water and stored full after sealing. Containers which are filled with deionized water before storage are emptied and shaken out just before use.

For equipment used in low-level determinations of Al or Cl, substitute 5 % HNO_3 as a decontaminating solution, to avoid possible labware contamination with these elements.

12.2 QC Monitoring (Temperatures, etc.)

Drying oven temperatures will be monitored daily

The conductivity of the deionized water supply is checked continuously by the deionizing system. Deionized water is not dispensed to storage carboys unless resistance reading is > 17 megaohms. If resistance reading repeatedly drops below 17 megaohms, ion exchange cartridges are replaced.

12.3 Preventive maintenance schedule, procedures, and logs

Preventive maintenance scheduling and procedures will be completed according to manufacturers' recommendations. Routine instrument problems can be corrected by the technician in most cases. Any serious or repeated equipment failure will be reported to the Supervisors.

12.4 Critical components

The analytical capabilities of the MAFES Analytical Laboratory include:

Ametek/Spectro model Genesis SOP ICP-OES
Thermo Electron model iCAP 6300 ICP-OES
Dionex model ICS-1000 Ion Chromatograph
LECO Tru-Mac Carbon/Nitrogen combustion analyzer
LECO 928 Carbon/Nitrogen combustion analyzer
OI Analytic model 3700 dual-channel Flow Injection Analyzer

OI Analytic model 3700 single-channel Flow Injection Analyzer
Milestone MDA-80 direct mercury analyzer
Labfit AS-300 automated pH analyzer
AIM-600 Automated Block Digestion System
CEM MARS-6 Microwave digestion system
Barnstead/Thermolyne E-pure water deionizer
Miscellaneous equipment including pH and EC meters, dispensers,
balances, drying ovens, muffle furnaces, grinders, shakers, sievers, etc.

13.0 Assessment Procedures for Data Acceptability

13.1 Precision acceptance limits

Data will be assessed for precision each day or after each run. Ten percent of all parameter analyses for samples requiring EPA QA/QC protocols will be replicated for precision evaluation. Precision control limit will be 10-20 % RPD for all parameters above 2 x MDL. A general listing of precision control limits by parameter appears in Appendix 17.1 and specifically by method in Appendix 17.2.

Precision will also be assessed daily by running an internal reference sample with each batch or every 20 samples, whichever is fewer. The results will be compared using quality control charts and appropriate statistical analyses. The precision control limits will be mean $\pm$ 3 SD based on a population of at least 20 data points. Warning limits, indicating a possible emerging problem, will be mean $\pm$ 2 SD. The mean and standard deviation will be recalculated daily or after every 10 new data points.

13.2 Accuracy acceptance limits

Data accuracy will be assessed daily or after each run. At least one SRM (where available) or internal reference sample will be analyzed with every batch of samples or every 20, whichever is fewer. Results will be compared to known content for SRM's or cumulative mean values for internal reference samples. Percent recovery will be calculated for SRM's or internal reference samples. The control limits for reference samples recovery will be $\pm$ 10-20 % of known or mean content. A general listing of accuracy control limits by parameter appears in Appendix 17.1 and specifically by method in Appendix 17.2.

Data accuracy will also be evaluated, where required, by running a spiked sample and a spike duplicate with every batch or every 20 samples, whichever is fewer. Percent recovery will be calculated for spiked samples, with a control limit of 90-110 % of known addition.

Any data exceeding control limits will initiate corrective action outlined in Section 14.0.

14.0 Corrective Action

14.1 Limits

Corrective actions will be initiated according to the following criteria: (1) daily data assessment found to be beyond control limits as stated in Section 13.1; (2) unacceptable results on performance evaluation or system audits; (3) unacceptable results on interlaboratory comparison studies or proficiency sample analysis.

14.2 Procedures

Corrective action will be initiated by the analyst. The analyst will identify and define the problem. The analyst, with a Supervisor if necessary, will then investigate and determine the cause of the problem. The analyst, with a Supervisor if necessary, will then determine a corrective action to eliminate the problem and implement the corrective action. The analyst and a Supervisor will evaluate the effectiveness of the corrective action and verify that the corrective action has eliminated the problem.

Corrective actions will be initiated by the analyst in the following sequence: 1) Determine if the instrument is functioning properly (for example: proper flow of gases and/or solutions, proper function of sample introduction system). Recalibrate the instrument and reanalyze QCCS and reference sample(s). 2) Remake calibration standards and reanalyze QCCS and reference sample(s). 3) Re-extract or re-digest the affected batch of samples and reference material(s) and reanalyze. If the analyst cannot determine the reason for unacceptable results, he/she will obtain the aid of a Supervisor.

14.3 Identification of responsible person

The analyst will initiate corrective actions. A Supervisor will ensure all corrective actions have been implemented and were effective, during the data validation process prior to report generation.

15.0 Quality Assurance Reports to Management

15.1 Schedule

All QA/QC problems which develop will be reported to a Supervisor.

16.0 List of Recipients of Quality Assurance Plan

- Laboratory Director
- Analytical lab Staff
- Any client requesting a copy of the Quality Assurance Plan

17.1 Appendix A - Quality Assurance Goals for Analytical Parameters

Analyte (method)	Method Reference	Precision	Accuracy
METALS			
Aluminum (ICP)	EPA 200.7	± 20%	± 20%
Arsenic (ICP)	EPA 200.7	± 10%	± 10%
Cadmium (ICP)	EPA 200.7	± 30%	± 10%
Chromium (ICP)	EPA 200.7	± 10%	± 10%
Cobalt (ICP)	EPA 200.7	± 10%	± 10%
Copper (ICP)	EPA 200.7	± 10%	± 10%
Iron (ICP)	EPA 200.7	± 10%	± 10%
Lead (ICP)	EPA 200.7	± 10%	± 10%
Mercury (AAS)	EPA 245.1	± 20%	± 10%
Manganese (ICP)	EPA 200.7	± 10%	± 10%
Molybdenum (ICP)	EPA 200.7	± 50%	± 10%
Nickel (ICP)	EPA 200.7	± 15%	± 10%

Selenium (ICP)	EPA 200.7	± 25%	± 10%
Silver (ICP)	EPA 200.7	± 25%	± 25%
Tin (ICP)	EPA 200.7	± 20%	± 20%
Titanium (ICP)	EPA 200.7	± 10%	± 10%
Vanadium (ICP)	EPA 200.7	± 10%	± 20%
Zinc (ICP)	EPA 200.7	± 15%	± 10%

Analyte (method)	Method Reference	Precision	Accuracy
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MINERALS

pH	SM 4500-H	± 10%	± 10%
Elec. Cond.	SM 2510A	± 10%	± 10%
Barium (ICP)	EPA 200.7	± 15%	± 10%
Beryllium (ICP)	EPA 200.7	± 15%	± 15%
Calcium (ICP)	EPA 200.7	± 10%	± 10%
Chloride (Ion chromatography)	SM 4500-Cl	± 15%	± 15%
Lanthanum (ICP)	EPA 200.7	± 15%	± 15%
Lithium (ICP)	EPA 200.7	± 15%	± 15%

Magnesium (ICP)	EPA 200.7	± 10%	± 10%
Potassium (ICP)	EPA 200.7	± 20%	± 20%
Sodium (ICP)	EPA 200.7	± 15%	± 15%
Total Alkalinity (Titrimetric)	SM 2320B	± 20%	± 20%
Total Solids (Gravimetric)	SM2540B	± 10%	± 10%
Total Volatile Solids (Gravimetric)	SM2540E	± 10%	± 10%
Analyte (method)	Method Reference	Precision	Accuracy

NUTRIENTS

Ammonium N (Colorimetric)	SM 4500-NH3-G	± 10%	± 10%
Boron (ICP)	EPA 200.7	± 15%	± 15%
Total C (Combustion)	EPA 440.0	± 5%	± 10%
Nitrate (Colorimetric)	SM 4500-NO3-F	± 10%	± 10%
Total Kjeldahl N (Block digestion)	EPA 351.2	± 10%	± 10%
Total N (Combustion)	EPA 440.0	± 5%	± 10%
Orthophosphate (Colorimetric)	EPA 365.1	± 10%	± 10%

Total Phosphorus (ICP)	EPA 200.7	$\pm 15\%$	$\pm 15\%$
Sulfur (ICP)	EPA 200.7	$\pm 15\%$	$\pm 15\%$

17.2 Appendix B - Individual Method SOP's

Individual SOP's for environmental sample analysis are available on request.