



Colorado State University

**Soil, Water, and Plant Testing Laboratory
200 W. Lake St
A320, NESB
Fort Collins, CO 80523**

**Water Quality Assurance Plan
Soil, Water, and Plant Testing Laboratory
2015**

Experience of all performing operators

James Self, Ph.D.

1988-present, Director, CSU Soil Water, and Plant testing Lab
1984-1988, Technician, CSU Soil, Water, and Plant Testing Lab
1977-1984, Graduate Research Assistant (Ph.D.), University of Arizona Secondary Plant Metabolism
1974-1977, Graduate Research Assistant (MS), University of Missouri-Columbia, Carbohydrates In Soybeans
1970-1974, Colorado State University (BS), Agronomy, Crop Science
Forty years of experience in laboratory settings.
Experience related to water inorganic analysis in ICP, IC, titrations, pH, EC, total digestion procedures, hydride analysis, total N and NPOC, Hg analysis, and QA/QC reporting

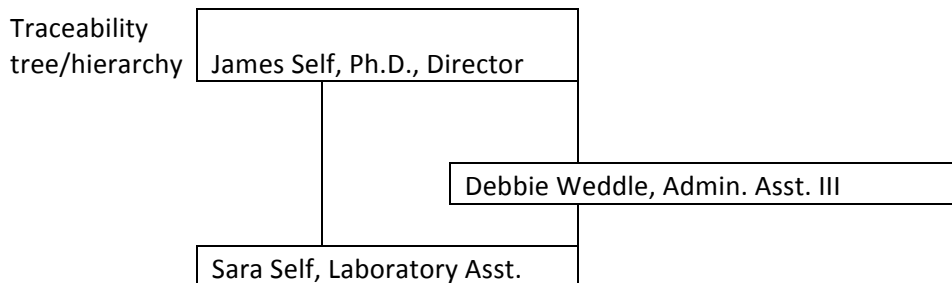
Sara Self

Laboratory Assistant
Twelve years experience at the CSU Soil, Water, and Plant Testing Lab.
Proficient in the use of pH and EC meters, ion chromatography, titration procedures digestion processes, and QA/QC reporting

Debbie Weddle

Administrative Assistant III
Twenty years experience at the CSU Soil, Water, and Plant Testing Lab
Experience in water sample handling, including laboratory check in, tracking samples through the analytical process, and sample storage.

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Sampling Procedures:

The Soil, Water, and Plant Testing Laboratory does not collect water samples. This procedure must be completed by the individual or company requesting the analysis.

Sample Handling Procedures:

Booking In:

Samples are received by the laboratory in room A320 of the Natural and Environmental Sciences Building along with an information sheet from the client indicating what type of analysis is needed. Samples are given a laboratory number with a "W" prefix. Information including laboratory number, date received, customer name, and type of analysis requested is written in the "book-in" notebook. The sample is labeled with the lab number and then placed in the refrigerator at 4° C in the main laboratory (Rm A334). Samples are filtered through a 0.45 µ filter and split. One split is acidified to pH <2.0. The other split is non-acidified. Samples requiring total analysis are not filtered.

Note: NEON samples are received from the NREL laboratory after NREL removes their aliquots for their analyses. Samples are transferred to the SWPTL the same day as they are received at NREL. NEON samples have already been split into filtered and non filtered samples.

Sample Storage Prior To Analysis:

Samples are kept in the water sample refrigerator at 4° C until an analytical procedure is started. Analytical procedures are done within the time frame given in table 1 of EPA 6004-79-020 (Methods For Chemical Analysis of Water and Waste). Samples are removed from the refrigerator when an analysis is performed and returned to the refrigerator when the sample is no long needed

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for analysis. If an analysis requires that the sample be at room temperature, then an aliquot is removed from the original container and allowed to equilibrate to an ambient temperature.

Storage after analysis:

After completion of analysis, the sample is placed in the laboratory storage area for two weeks.

Disposal:

After two weeks of storage, the sample is disposed by use of the sewage system or the hazardous waste removal service at Colorado State University.

Instrument Calibration Procedures & Frequency of Their

Use:

Inductively coupled plasma-Optical Emission Spectrometer (ICP)for metals analysis

The Inductively Coupled Plasma (**Perkin Elmer Optima 7300 DV Optical Emission Spectrometer**) unit is profiled daily or after every new start-up of the instrument. Profiling consists of aligning the detectors of the unit with a reference using a mercury lamp. The ICP is routinely standardized before every batch of samples. Standardization is performed after every 10-15 samples, or when needed. A scan of all elements is performed with each batch of samples using a standard with all elements included. Scanning data is collected and compared to the previous scan to determine the reliability of the ICP.

Ion Chromatograph, (F, Cl, NO₂, Br, NO₃, PO₄, SO₄)

The ion chromatograph (**Dionex ICS-1100**) is standardized daily when an analytical run is set up. Standards are analyzed prior to running samples to develop a standard curve. A standard is then run as a sample to validate the standard curve. If the standard sample is within 10% of expected values, then the unknown samples are analyzed. A duplicate analyses, blanks, and checks are included in the batch to be analyzed.

pH meter and Electrode (pH)

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The pH meter (**Oakton PC2700**) is standardized daily as needed. Standards are evaluated according to the pH range of the samples. A two point curve is established by either standardizing with a pH standard of 7 and 10 or with 7 and 4. An acceptable slope for calibration is 0.99 or better. A check sample from the North American Proficiency Program (NAPT) is analyzed to check the pH output. An acceptable range is $\pm 10\%$.

Conductivity Meter and Electrode (Electrical Conductivity)

The conductivity meter (**Orion Model 420A**) is calibrated daily or as needed. Known conductivity standards (RICCA Chemical Company) are evaluated to check for accuracy. The concentration of the standard used to evaluate the meter will depend on the contraction of the standard. For low level conductivities, a 20 μ S/cm standard is usually tested. An acceptable range is $\pm 10\%$

Titration (Alkalinity/ANC)

The titrating devices (**BrandTech Scientific, Inc Titrette or Metrohm 809 Titrand**) are calibrated daily or as needed. Calibration consists of checking volumes dispensed against volume indicated on the device. A NAPT check sample is also used to evaluate accuracy. An acceptable range of accuracy is $\pm 10\%$.

Colorimeter (ortho-P, TDP, tannins)

The colorimeter (**Thermo Scientific Spectronic D+**) is calibrated according to specifications of the instrument. In the transmittance mode the wavelength is first established, then the instrument is set to zero. Then a blank is placed in the holder and the instrument is set to 100% T. To read absorbance, switch the output to absorbance.

Analytical Procedures:

pH:	Method 150.1
Conductivity:	Method 120.1
Alkalinity:	Method 310.1
Anions:	Method 300.0
Metals:	Method 200.7
TSS:	Method 160.2
Total P:	Method 365.4
Turbidity	Method 180.1
Mercury	Method 245.1

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Ammonium-N	Method 350.1
Total Kjeldahl N	Method 351.1
Dissolved O ₂	Method 360.1
Ortho P (TDP)	Method 365.2
Total Organic Carbon	Method 415.1

Types of QC Checks & Frequency of Their Use:

Internal QC Check (Internal Audit): A blind sample is prepared by the QC/QA analyst and is run through all the normal procedures and instruments.

Reagent blanks: (Method blank). Defined as a solution that has not been mixed or contaminated by the samples or other external influence. It usually consists of DI water or a digestion matrix that was carried through the preparation sequence. At least one is analyzed with every batch of samples.

Laboratory Fortified Blank: (Spiked blank or laboratory control sample). A sample of known concentrations of analytes. This may be a prepared QC sample or certified samples such as NIST 1640a. It may also be a blank spiked with a known concentration of analyte. Sometime referred to quality control check

Matrix Spike: The matrix of the sample is spiked with known concentration of analytes to determine their recovery. It is usually used for difficult matrixes such as samples with very high TDS or samples contaminated with organic solvents.

Calibration Standards: These are used for calibrating the instruments. They may be pre-made purchased standards or standards that are prepared from stock solutions.

Replicate analysis: Also referred to as duplicate analysis. At least one randomly selected duplicate sample is analyzed with each batch of samples. They are usually analyzed with each set of 10-30 samples depending on the availability of sufficient sample.

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Corrective Action Contingencies:

Obtaining Unacceptable QC Checks or Performance Evaluation

Samples: The problem is analyzed and remedied if possible by maintenance and/or cleaning of the instrumentation. The samples since the previous QC check are then reanalyzed along with the new QC checks.

Preventive Maintenance Procedures and Schedules:

ICP:

Tubing is replaced weekly or as needed depending on sample load.
The torch, tip, and spray chamber are removed weekly for cleaning.
Filters are cleaned daily prior to starting the machine.
Hoses and fitting are checked for leaks daily.
The lens is cleaned weekly or as needed.

IC:

Column bed supports are replaced every 3-4 weeks.
The guard column and separatory columns are cleaned monthly
Columns are replaced annually or as needed.
The system is flushed and primed weekly to avoid clogging.

pH meter and electrode

The meter is checked for damage due to spills, acids, etc. weekly.
The electrode is checked daily for damage.

EC meter

The meter is checked for damage due to spills, acids, etc. weekly.
The electrode is inspected daily for damage.

Titration

The pipette is checked daily for smooth operation and clogs.
Pipette volumes are checked daily

Colorimeter

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Checked for damage from spills, etc. weekly
Lamps are replaced as needed.

Digestion blocks

Cleaned after use.

Temperature accuracy is checked monthly.

Generally, all instruments are checked according to the instrument's maintenance checklist once per week or more frequently as needed.

Data Precision and Accuracy:

Precision:

Precision is based on the results of replicate analyses. A 5%-10% deviation between the replicate samples is acceptable depending on the analyte. Precision is reviewed, evaluated, and recorded monthly by the QC/QA analyst.

Accuracy:

Accuracy is determined by analyzing known check samples such as NIST 1640a and prepared reference standards. Deviations up to 10% are considered acceptable

Reporting:

Sample reports are in different formats depending upon the type of sample. There are four different styles of water reports: Domestic, irrigation, livestock and research. After the report is transcribed into one of the formats it is printed and checked by the QC/QA analyst for accuracy and quality of both the data and the transcription. Furthermore, the QC/QA analyst will suggest recommendations (comments) to accompany the report. The Laboratory Supervisor then reviews the report including recommendations. Changes to the report are completed and a photocopy is made. The final original copy is then sent to the customer.

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