Analytical Chemistry Quality Assurance/Quality Control (QA/QC) Process for NEON Reaeration Analysis Version 1.0

I. Background

This protocol describes general procedures in documenting and assuring quality of analytical chemistry results that are produced at the USU Aquatic Biogeochemistry Laboratory. Please see protocols for specific analyses for additional information on analytical procedures, expected detection limits, sources for certified reference materials, etc. Strict adherence to this and analysis-specific protocols will be compensated by increased confidence in the data we produce. Stakeholders and manuscript reviewers are increasingly attentive to information on laboratory detection limits and QA/QC procedures.

II. Potential Hazards

a. Laboratory Safety

Analytical chemistry procedures are conducted in a chemical laboratory. Exposure to reactive, toxic, flammable, and/or corrosive chemicals is possible, as are chemical spills. All laboratory personnel must have up-to-date Chemical Laboratory Safety Training as mandated by OSHA and implemented by the USU Environmental Health and Safety Office. Please review the ABL Chemical Hygiene Plan for specific laboratory emergency plans, details on chemical spill clean-up, chemical inventory lists and MSDS for specific chemicals. Chemical laboratory personnel must wear appropriate attire including closed-toe shoes, lab coats, gloves, and safety glasses.

III. Quality Assurance

a. Policy Statement

The Utah State University Aquatic Biogeochemistry Laboratory (USU-ABL) is committed to provide scientifically valid analytical data for university-sponsored research projects as well as for outside customers, including government agencies and local stakeholders. We aim to produce data in a timely manner following established protocols for analyses of chemical constituents in fresh and saline waters, such as those approved by the Environmental Protection Agency (EPA). Aside from EPA-approved methods, and depending on user needs, we use more state-of-the-art protocols that have been vetted through the scientific peer review process and are in published literature.

Although the USU-ABL is not formally certified by any accreditation program, the lab does follow process used by most certified labs including the following: Lab director and manager have achieved advanced educational degree with background in analytical chemistry, technicians are typically students in a science field with some lab experience before they receive specific training on an analytical instrument, the lab has an up-to-date

Chemical Hygiene Plan as well as a lab manual that contains documents describing QA/QC process and specific standard operating procedures (SOPs) for each analysis/measurement. Instruments are calibrated using these SOPs and analyses are verified using certified reference materials appropriate for each analysis. Method detection limits are calculated for each analysis and updated biannually. Data below the method detection limit are reported with a flag. The lab conducts self-audits to ensure compliance with OSHA and university regulations and is inspected annually by USU's safety office and irregularly by EPA.

b. Organizational Structure

The USU-ABL is staffed by a Director (Dr. Michelle Baker) who has nearly 20 years of experience in aquatic biogeochemistry research. A lab manager (Lisa Ward, M.S.) coordinates laboratory analyses, oversees QA/QC process, ensures compliance with OSHA and university rules and regulations, trains and supervises graduate and undergraduate student technicians. During any given semester the lab is staffed by 2-3 part-time undergraduate student technicians, as well as 2-3 graduate students.

c. Staff Responsibilities

All personnel employed by the USU-ABL are required to be familiar with policies and procedures outlined in this document, as well as individual standard operating procedures (SOPs) used for specific analyses.

All USU-ABL employees must abide by USU's Personnel Policies. These are available in detail from the Human Resources website (http://www.usu.edu/hr/htm/policies). USU conducts background checks on all new employees. USU operates a strict drug- and alcohol-free workplace.

All USU-ABL employees must complete the following training upon appointment, with renewal at the specified rates:

Training	Administrator	Renewal Period
Laboratory Safety	Environmental Health and Safety Office	Annual
Specific SOPs	USU-ABL	Annual
Drivers Training	Motor Pool	Every other year
Purchasing Card	Purchasing Office	Every three years

Students, post-docs, and classified staff sponsored in whole or in part by funds from the National Science Foundation must complete training in the Responsible Conduct of Research. This training is offered annually by the Office of Compliance Assistance, and satisfies requirements of the America COMPETES Act.

All USU-ABL employees must support scientific integrity as outlined in USU Policy #306. Scientific misconduct defined in USU policy as "any incident of fabrication, falsification, or plagiarism in proposing, conducting, or reporting research. It does not include honest error or honest differences in interpretations or judgments of data. Fabrication refers to the making up of data that were not observed as purported. Falsification includes the changing of data or the way in which observations are reported, and spans a broad spectrum, from omitting observed data points from reported data sets to wholesale changing of data to fit the investigator's hypothesis. Plagiarism is the claiming as one's own material that is the product of someone else's work." Scientific misconduct by USU-ABL employees will not be tolerated and will be grounds for immediate dismissal.

d. Training and Performance Requirements

Employees of USU-ABL must maintain training records as outlined in the table above. Performance will be documented initially and on an on-going basis as outlined below.

1) Initial Demonstration of Capability
   Before performing any chemical analysis, USU-ABL employees will conduct an initial demonstration of capability (IDC) to demonstrate that the employee is proficient in the analysis. The IDC includes comparing the calibration standard curve of the new analyst (or method if modified) to that of a previous analyst. Reagent blanks and check standards are also compared within a sample run. Calibration curves should differ by no more than 10%. Reagent blanks should contain no contamination at concentrations ½ of the method detection limit. Percent recovery for check standards should be within acceptance criteria for the given method, typically within ±5%. IDC information should be recorded in the USU-ABL Laboratory manual at the end of the appropriate SOP.

2) Ongoing Demonstration of Capability
   Ongoing capability for a given SOP is demonstrated using reagent blanks, check standards, and quality-control standards prepared from certified reference materials. Check standards made from calibration stock solutions and reagent blanks must be included every 20 samples in a run. Certified reference materials should be analyzed quarterly (or about every 500 samples). ~~Check standards and certified reference materials are analyzed semi-annually for each analyst and method~~. Each analyst must update signatures on specific SOPs annually as part of their annual performance evaluation.

e. Analytical Capabilities of the ABL

1) Analytical capability includes ion chromatography, total organic carbon and total nitrogen analyses, inorganic and total nitrogen and

phosphorus analyses, chlorophyll, biochemical oxygen demand, and gas chromatography with electron capture detection (GC-ECD), flame ionization detection (GC-FID), and thermal conductivity detection (GC-TCD).

2) Additional services include method development, sample processing for stable isotope analysis, dry mass and ash-free dry mass, total suspended solids, volatile suspended solids.

f. Receiving and Handling Samples

1) Receiving samples and storage
Samples are typically received from the NEON domain in a cooler with an ice pack. Upon receipt at ABL, user should verify the sample identifications match with the sample manifest received via email from the NEON domain. Use a barcode scanner to scan each sample identification to a spreadsheet, saved to lab box folder. User will also fill out a paper Analysis Order Form with name and contact information, sample matrix, number of desired analyses, estimated concentration range, data deadline, and billing information. During intake, user should note any problem such as missing samples, loose sample labels, etc. Samples for ion analysis are stored at room temperature in ziplock bags in which they were received. Samples for dissolved gas analysis are stored at room temperature under water in lock-n-lock containers in which they were received.

2) Sample receipt form
After samples are received and verified, user should submit a receipt form using the NEON data ingest page. Save a copy of the receipt form to the lab box folder.

3) Hold NEON samples for 90 days following data return.

4) Sample tracability
USU-ABL calibrates instruments using NIST-certified standards as outlined in method-specific SOPs. This flow chart shows sample tracability related to these standards.

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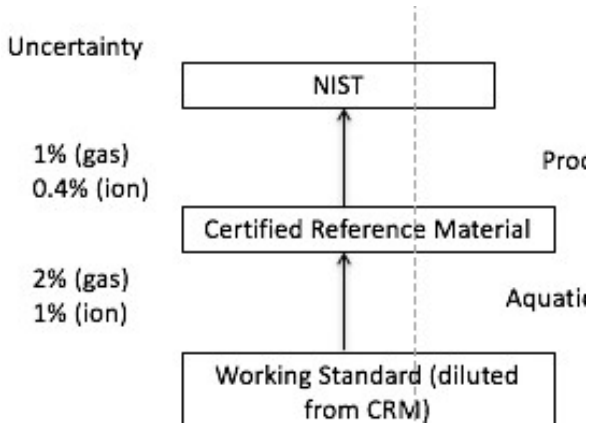

IV. Quality Control

a. Calibration, Verification, and Maintenance of Analytical Instrumentation

1) Calibration

Instrument calibration is conducted according to instrument manufacturer's instruction manual and SOP instructions. Initial calibrations are conducted with a minimum of 5 concentrations for linear curves, and 7 concentrations for non-linear curves, each time the instrument is started up for analysis. The calibration range varies by analysis but includes a low standard at or below the method detection limit, and a high concentration at the maximum expected by the end-user as reported on the Analysis Order Form. Calibration is acceptable by linear or nonlinear regression when a minimum correlation coefficient of 0.995 is achieved. For most analyses the correlation coefficient is 0.999. Initial calibration procedure is repeated if calibration criteria are not met within a run or if new reagents are added in the middle of a run.

2) Verification

Calibration verification within a run is assessed using laboratory-fortified blanks (check standards) prepared in a mid-range concentration. Check standards should be conducted on a 5% basis, or after every 20 samples. Method blanks are run at a minimum at the beginning and end of the run, and more often for certain analyses (see specific SOPs). Calibration verification is calculated as % difference in response factor for check standard compared to initial standard, and as % recovery of check standard. For SF_6 and bromide, deviations between 2-5% are flagged with deviations greater than 5% requiring re-run. For chloride, deviations >10% require re-run. If contamination is present in reagent blanks (greater than detection limit), source of contamination should be identified and samples re-run.

3) Maintenance/Troubleshooting

Analysts must follow instrument users manual for maintenance schedules. Typically if initial calibration is not good, try the following: remake reagents, remake calibration standards, check tubing/plumbing integrity and replace as needed. Follow more detailed troubleshooting as recommended by specific instrument manual. Record maintenance conducted in instrument log book.

b. Quality Control Parameters

1) Method Detection Limit

The method detection limit (MDL) is determined at least annually for each method from a minimum of seven replicates of low-level check standard that have been processed through the entire method, across three or more runs. The MDL is calculated as the standard deviation times the t-value from a one-sided t-distribution at the 99% level. For example for 7 replicates, the t-value is 7-1= 6 degrees of freedom = 3.14. The MDL is reported to the end user with each data set.

2) Repeatability
Sources of variation and bias in analytical measurement include but are not limited to sampling error and preparation, matrix effects, calibration errors, differences among analysts, reagent impurities, and instrument errors (hardware and software). It is usually not possible to minimize all of these errors simultaneously. We have found that sampling error – particularly field filtering of samples can be very large. Contamination of samples in the field, sample bottle, and from the atmosphere can be a large source of error. USU-ABL reports repeatability of measurements from analytical duplicates. End-users are advised to include field duplicates and field blanks with their samples. Analytical replicates that deviate more than 5% are re-run.

c. Data Reporting

1) Data are reported in an excel spreadsheet with these minimum paramaters: analysis date, analyst name, sample identification, concentration, calibration information, reagent blanks, check standards. USU-ABL will work with end-users to provide data in optimal format for their needs.
2) Data flags
In some instances reported data are qualified by flags indicating that quality control was not achieved. Flags include the following:
BDL = below method detection limit
CON = possible contamination because analyte found in reagent blank or reported value exceeds expected value by more than 5%.
E = value exceeds high concentration calibrant. Sample is usually diluted and re-run.
RERUN = sample concentration rejected because of contaminant, method performance, etc.

d. Records Archiving

1) Each analyst is responsible for maintaining electronic copies of each analytical run in the appropriate instrument folder. These are duplicated and archived on the lab box, and stored for a minimum of 3 years.

2) Hard copies of Analytical Order Forms and other data sheets are stored in BNR 257 for a minimum of 3 years.
3) Lab notebooks should be scanned quarterly and the images stored on the lab box folder. Hard copies of lab notebooks should be stored indefinitely in BNR 257.

V. References

1) American Public Health Association (APHA). 1998. Standard methods for the examination of water and wastewater 20th edition. APHA, Washington DC.