QUALITY ASSURANCE PLAN

CCAL WATER ANALYSIS LABORATORY

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Revision 4, April 2018
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Acronyms/Abbreviations

AAII	Technicon Auto-Analyzer II
AD	analytical duplicate
ASTM	American Society for Testing and Materials
AP	Astoria Pacific Analyzer
BB	bottle blank
CCAL	Cooperative Chemical Analytical Laboratory
CFR	Code of Federal Regulations
cm	centimeter
DIC	Dissolved Inorganic Carbon
DIW	deionized water
DL	detection limit
DOC	Dissolved Organic Carbon
DQO	Data Quality Objectives
DSOL	Dissolved Solids
EPA	Environmental Protection Agency
FAAS	Flame Atomic Absorption Spectrophotometer
FD	field duplicate
FY	fiscal year
HDPE	High Density Polyethylene
IC	Ion Chromatograph
IDL	instrument detection limit
L	Liter

MDL	method detection limit
µeq	microequivalent
µg	microgram
µm	micrometer
µS	microsiemen
mg	milligram
mL	milliliter
MDL	Method Detection Limit
ML	Minimum Level of Quantification
ng	nanogram
NIST	National Institute of Standards and Technology
NPS	National Park Service
NTU	Nephelometric Turbidity Units
PPE	Personal Protective Equipment
ppb	parts per billion
ppm	parts per million
psi	pounds per square inch
QA	Quality Assurance
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCCS	Quality Control Check Sample
QMP	Quality Management Plan

RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedure
SRP	Soluble Reactive Phosphorus
SRS	Standard Reference Sample
SSCS	Second Source Check Standard
SSED	Suspended Sediment
TDN	Total Dissolved Nitrogen
TDP	Total Dissolved Phosphorus
TDS	Total Dissolved Solids
TN	Total Nitrogen
TOC	Total Organic Carbon
TP	Total Phosphorus
TS	Total Solids
Turb	Turbidity
TV (tv)	Transition Value
USDA	United States Department of Agriculture
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UTN	Unfiltered Total Nitrogen
UTP	Unfiltered Total Phosphorus
UV-vis	Ultraviolet-visible
v/v	volume to volume ratio

CCAL Water Analysis Laboratory
Quality Assurance Plan

1.0 Introduction

The Cooperative Chemical Analytical Laboratory (CCAL) began in the early 1970's as a combined endeavor of the Oregon State University Department of Forest Science and the United States Department of Agriculture (USDA) Forest Service, Pacific Northwest Forest and Range Experiment Station, Forestry Sciences Laboratory. CCAL, now predominantly Oregon State University Department of Forest Ecosystems and Society in the College of Forestry, specializes in analysis of lake, stream, precipitation and groundwater environmental samples, with samples coming primarily from the Pacific Northwest and Alaska. Sample preparation services provided by the laboratory include sample filtration, preservation, digestion and extraction. CCAL uses standard analytical procedures that have been modified and adapted to meet specific needs of multidisciplinary research.

The CCAL Quality Assurance Plan (QAP) describes protocols and procedures used in the Laboratory. Methods, detection limits and acceptance parameters are tabulated for all analytical procedures. This report replaces IBP Report #160, January 1975, revised February 1991, *CCAL Procedures Manual*. This is a living document that will be updated and revised as new methods and procedures are developed and qualified. See specific method for historical modifications. Standard Operating Procedures (SOPs) for CCAL are listed in the appendix, and are available as separate documents.

2.0 Project Organization and Personnel

Current CCAL staff consists of two chemists, one lab technician and three student interns. Personnel and their primary responsibilities include:

Kathryn Motter, Senior Chemist and Lab Manager: Dionex IC, Shimadzu TOC, Shimadzu FAAS, instrument maintenance and repair, documentation, sample tracking, data reporting, invoicing, customer service, budget, database management, data quality reports, laboratory organization, web page management and quality assurance monitoring

Laura Hartley, Chemist and Lab Supervisor: Astoria Pacific Segmented Flow Analyzer, Technicon AutoAnalyzer II, alkalinity, pH, conductivity, instrument maintenance and repair, laboratory logistics, sample tracking, sample preparation, quality assurance monitoring, customer service, delegation of responsibilities to students

Laboratory Technician (TBD): Sample preparation, sample setup and digestion, alkalinity, pH, conductivity, gravimetric analyses, quality assurance monitoring, reagent preparation, sample log-in, sample tracking, chemist support

2 or 3 Student Lab Aides: sample log-in, sample preparation, reagent preparation, sample setup, Total Dissolved Solids, Total Suspended Solids, alkalinity, pH, conductivity, sample storage and organization, cleaning of glassware and bottles, general laboratory maintenance, chemist support

3.0 Methodology

Instruments used, method descriptions and detection limits are outlined in Table 1. Project specific working ranges may supersede those listed.

Table 1: CCAL Water Analysis Laboratory: Methods and Detection Limits

Analyte	CCAL Method Number[1]	Reference Method[2]	Instrument and Method Description	Detection Limit[3]	Working Range[4]
Determination of pH and Alkalinity	CCAL 10C.1	Alkalinity; APHA 2320	ManTech PC-Titrate	0.2 mg $CaCO_3$/L	NA
		pH; APHA 4500 H		NA	0 - 14 pH units
Determination of Specific Conductance	CCAL 11A.2	APHA 2510	YSI 3200 & YSI 3256 probe with temperature correction	0.4 µS/cm	0 µS - 3 S
Determination of Suspended Sediments	CCAL 12A.3	APHA 2540 B; EPA 160.2	Gravimetric	2 mg/L	NA
Determination of Total and Total Dissolved Solids	CCAL 13A.2	APHA 2540 C; EPA 160.3	Gravimetric	5 mg/L	NA
Determination of Color by Color Kit	CCAL 15A.1	APHA 2120	Visual Comparison	0 CU	0 - 100 CU
Determination of Turbidity	CCAL 16A.1	APHA 2130	HACH 2100A	0.05 NTU	0.1 – 1000 NTU
Analysis of Dissolved and Total Organic Carbon	CCAL 20A.3	APHA 5310 B; EPA 415.1	Shimadzu TOC-VCSH Combustion Analyzer	0.05 mg/L	0.05 – 5.00 mg/L
Analysis of Dissolved Inorganic Carbon	CCAL 21A.1	Shimadzu Methods Manual	Shimadzu TOC-VCSH Combustion Analyzer	0.05 mg/L	0.05 – 5.00 mg/L
Analysis of Ammonia in Fresh Waters	CCAL 30B.1	APHA 4500-NH3 G; EPA 350.1	Astoria Pacific Segmented Flow Analyzer	0.010 mg N/L	0.010 – 0.200 mg N/L
Analysis of Nitrate/Nitrite and Nitrite in Fresh Waters	CCAL 31B.1	APHA 4500-NO3 F; EPA 353.3	Technicon Auto-Analyzer II Cadmium Reduction Method	0.001 mg N/L	0.001 – 0.100 mg N/L

Analyte	CCAL Method Number[1]	Reference Method[2]	Instrument and Method Description	Detection Limit[3]	Working Range[4]
Analysis of Silicon in Fresh Waters	CCAL 32B.1	APHA 4500-SiO2 E	Astoria Pacific Segmented Flow Analyzer	0.20 mg Si/L	0.20 - 10 mg Si/L
Digestion and Analysis of Fresh Water Samples for Total Nitrogen and Total Dissolved Nitrogen	CCAL 33A.4	APHA 4500-NO3 F; APHA 4500-P J;	Persulfate Digestion with subsequent analysis by Technicon Auto-Analyzer II Cadmium Reduction Method	0.01 mg N/L	0.01 – 0.40 mg N/L
Analysis of Fresh Water Samples for Orthophosphorus	CCAL 34C.1	APHA 4500-P F; EPA 365.2	Astoria Pacific Segmented Flow Analyzer	0.001 mg P/L	0.001 - 0.200 mg P/L
Digestion and Analysis of Fresh Water Samples for Total Phosphorus and Total Dissolved Phosphorus	CCAL 35B.2	APHA 4500-P F; APHA 4500-P J;EPA 365.2	Persulfate Digestion with subsequent analysis on Technicon Auto-Analyzer II	0.002 mg P/L	0.002 - 0.300 mg P/L
Analysis of Chloride, Bromide and Sulfate in Fresh Waters by Ion Chromatography	CCAL 50B.2	APHA 4110 B; EPA 9056A	Dionex 1500 Ion Chromatograph with Chemical Suppression of Eluent Conductivity	Cl; 0.01 mg/L F; 0.01 mg/L Br; 0.01 mg/L S; 0.01mg/L	Cl; 0.01 – 5.00 mg/L F; 0.01 – 5.00 mg/L Br; 0.01 – 5.00 mg/L S; 0.01 – 5.00 mg/L
Analysis of Cations in Fresh Waters by Atomic Absorption Spectrometry	CCAL 60B.1	APHA 3111; EPA 7000B	Shimadzu AA-7000 Flame Atomic Absorption Spectrometer	Ca: 0.06 mg/L Fe: 0.06 mg/L K: 0.03 mg/L Mg: 0.02 mg/L Mn: 0.02 mg/L Na: 0.01 mg/L	Ca: 0.06 - 10.00 Fe: 0.06 - 2.00 K: 0.03 - 3.00 Mg: 0.02 - 3.00 Mn: 0.02 - 2.00 Na: 0.01 – 6.00

[1]Standard Operating Procedures for CCAL

[2]Method References *(note: CCAL procedures developed primarily from APHA methods; comparable EPA reference included for informational purposes only)*

- *APHA 2005. Standard Methods for the Examination of Water and Wastewater; 21st Edition; American Public Health Association, Washington, D.C.*
- *U.S. EPA Office of Solid Waste (OSW) Methods Team; Ariel Rios Bldg. (5307W); 1200 Pennsylvania Ave. NW; Washington, DC 20460; Phone: 703-308-8855; Fax: 703-308-0511; URL http://www.epa.gov/epaoswer/hazwaste/test/index.htm*
- *U.S.EPA National Exposure Research Laboratory (NERL); Microbiological and Chemical Exposure Assessment Research Division (MCEARD); [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH]; 26 West Martin Luther King Drive; Cincinnati, Ohio 45268-0001; Fax: 513-569-7757*

[3]Determination of method specified detection level based on a one-sided 99% confidence interval (t-value at a significance level of 0.01 and n-1 degrees of freedom) from multiple replicates of a low concentration standard measure within an analysis run.

[4]For IC, FAAS, AP and AAII, the working range has been established as that in which most sample concentrations occur. An alternative range may be used to meet specific sample requirements.

4.0 Sample Containers and Glassware Preparation

This section details the protocols for washing sample aliquot bottles, general laboratory glassware and analysis specific vials/tubes. Specific information on laboratory maintenance can be found in the Lab Aide Manual located in the Appendix.

4.1 10% v/v HCl Acid Bath Preparation

Always wear proper PPE when working with concentrated acids

Mix 10 Liters of acid bath at a time in the hood
Add 1-L of concentrated HCl to approximately 8-L of DIW in a 10 Liter carboy. Bring up to 10 Liters with DIW
Change or prepare as needed

4.2 Sample Bottles

Remove tape labels and rinse bottles four times with DIW. Rinse bottles with acid bath and follow with thorough DIW rinse. Wash bottles in dishwasher plumbed with DIW two times through the rinse cycle. Remove bottles from the dishwasher and place upside down on drying shelves. New bottles should be acid soaked with acid bath for at least 24 hours, soaked twice with DIW, rinsed thoroughly with DIW and washed in the dishwasher as above.

Caps are rinsed with DIW, followed with acid bath rinse and thorough DIW wash. Caps are dried on the drying rack shelves. When completely dry, bottles are capped and stored until use.

Semi-annually, the bottle wash procedure is verified with bottle blanks. Bottles for various analyses are filled with DIW, and allowed to sit for at least seven days at 4°C. Analytical results should be lower than one standard deviation over the detection limit.

4.3 pH, Alkalinity, Titration and Conductivity Beakers

Empty, remove markings and flush thoroughly DIW. Fill with DIW and soak for at least one hour. Rinse four times with DIW. Invert beakers on trays lined with clean absorbent lab matting. When dry, store in appropriate drawer. Beakers should be acid soaked at least once a year, or as needed.

4.4 Dissolved Solids Beakers

Fill beakers with DIW and soak overnight. Empty and scrub with brush to remove residue. Rinse 4X with DIW. Soak beakers overnight in Extran cleaning solution. Remove from Extran and scrub with brush. Rinse four times with DIW,

and put in the dissolved solids oven to dry. Do not touch beakers with bare hands after washing; use a Kim Wipe, gloves or tongs. Beakers are stored in the oven until use.

4.5 Filter Equipment

Rinse all parts thoroughly with DIW and dry on racks. Acid wash or scrub individual components as necessary to remove residue.

4.6 Atomic Absorption Sample Tubes

Rinse tubes 4X with DIW. Fill with DIW and soak for at least one hour. Empty tubes and submerge in acid bath; soak overnight. Rinse 4X with DIW and place tubes in racks. Dry upside down on drying shelves. Dry tubes are stored in a plastic basin with tight fitting lid.

4.7 Total Nitrogen and Total Phosphorus Tubes

Rinse caps four times with DIW over a large funnel. Shake to remove excess water. Place caps open side down on a tray lined with absorbent lab matting; allow caps to dry thoroughly.

Empty tubes and rinse tubes with DIW four times. Place tubes in acid bath and soak overnight. Tubes are then rinsed again four times with DIW, inverted in clean racks and allowed to air dry. When completely dry, tubes are capped and stored.

4.8 Technicon and Astoria Pacific Autosampler Tubes

Empty tubes and rinse with DIW 4X. Place tubes in acid bath and soak until needed. Tubes are prerinsed with DIW thoroughly before use.

4.9 Suspended Sediment Watch Glasses

Rinse thoroughly with DIW and dry in the black drying rack. Renumber with paint pens as needed.

4.10 Carbon Analyzer Tubes and Caps

Rinse septum caps four times with DIW and soak in acid bath overnight. Rinse four times with DIW, and soak in DIW overnight. Rinse caps with DIW, shake off excess water and dry thoroughly in clean environment.

Rinse tubes four times with DIW, and soak overnight in acid bath. Remove tubes from bath the following day, rinse four times with DIW and soak in DIW overnight. Rinse with DIW and place tubes upside down in rack to dry. Inorganic

carbon tubes are stored once dry. Bake organic carbon tubes in a Muffle Furnace at 550ºC for at least three hours; cool overnight in the furnace. Tubes and lids are stored in air-tight containers.

4.11 Miscellaneous Glassware, Sample Carboys and Plastic Beakers

Generally, all glassware should be rinsed 4X with DIW and placed upside down on a tray or drying rack to dry. Glassware is rinsed or soaked in acid bath as needed. See Lab Aide Manual (appendix) for project specific protocols.

4.12 Laboratory Maintenance

Strict laboratory hygiene is necessary for trace level analysis. See Lab Aide Manual (appendix) for a complete outline of regular laboratory cleaning and maintenance tasks.

5.0 Sample Custody, Preparation and Preservation

The accuracy of analytical data as a representation of true sample composition is dependent upon collection and treatment of samples before they arrive at the laboratory. Sampling techniques and procedures must be such that the sample does not deteriorate or become contaminated before it reaches the lab. Samples should be collected in clean, acid-washed bottles and filtered and frozen (when appropriate) unless sent to the laboratory within 24 hours. CCAL staff has recommended protocols for field collection personnel. See documentation in the appendix or on the website.

5.1 Sample Custody

A Sample Submission Form, and labeled sample aliquots, should be delivered to the lab as soon as possible following sample collection. Requested sample analyses should be stated on the submission form or communicated to CCAL staff, with order of priority, prior to sample delivery. Once at the laboratory, samples are entered into the database and sample log tracking system. A project code is assigned (see Table 2) and samples are numbered consecutively within that code for each individual project. Sample condition, number of samples and date of receipt are recorded (see Sample Receipt and Tracking Form in the appendix). Samples may be frozen at this time until time of analysis.

Prior to analysis, samples are thawed (if necessary) and labeled aliquots for various analyses are prepared and delivered to appropriate storage area for requested analysis.

Table 2: CCAL Water Analysis Laboratory: Projects in FY17 and FY18

Code	Investigator	Project Name	Location
ALBA	A. Argerich	HJA Nutrient Analyses	HJA
ANJO	J. O'Donnell	NPS Arctic Network	Alaska
APPL	T. Lundell	Applegate	Rogue Basin, Oregon
ATAL	A. Cardwell	Aquatic Toxicology, ALUM	Albany Lab
BRAJ	S. Johnson	Blue River Reservoir	Blue River, Oregon
BREF	K. Fesler	Barney Reservoir	Barney Reservoir
CAKN	T. Simmons	Central Alaskan Network	Alaska
CALL	A. Larsen	Central Alaskan Lakes	Alaska
CLAP	K. Page	Crater Lake	S. Oregon University
CTLK	S. Girdner	Crater Lake	Crater Lake
DAKS	S. Stehn	Denali Road Study	Alaska
DCMC	M. Chandler	Dunes City	South of Florence
DERM	R. Miller	Dexter Reservoir	Oregon
DLKM	R. Miller	Diamond Lake	Diamond Lake
DRIP	E. Amt	PGE, Deschutes River and Res.	Madras, Oregon
EAJO	G. Jones	Event A	Willamette/Deschutes
ESOW	A. Goddard	Oregon Coast	Oregon Wildlife Foundation
FRAD	R. Heindel	Front Range Dust Deposition	Univ. of Colorado Boulder
HAWA	A. Olson	Hatchery Water	Medvejie Hatchery, AK
HBNJ	S. Johnson	H.J. Andrews	HJA
HJAN	S. Johnson	H.J. Andrews	HJA
JAFS	D. D'Amore	Juneau Alaska	Alaska
LILA	R. Schweinfurth	Fire Effect	Columbia River
LOCR	T. Lundell	Lost Creek	Rogue Basin, Oregon
LVLD	E. Dinger	Klamath Network	California, Oregon, Nevada
MESO	S. Serchan	Stream C Team: Mesocosms	HJA
MOJN	M. Steiner	Mojave Network	Great Basin
NOCA	C. Welch	North Cascades	North Cascades
OCWA	K. Gerber	Oak Creek Greenhouse	Corvallis, Oregon
OLYF	S. Fradkin	Olympic	Olympic National Park
PPDO	J. Mitzel	P&P DOC	Corvallis, Oregon
RAIN	R. Lofgren	Mt. Rainier	Mt. Rainier
REDS	T. Suminski	Redoubt	Redoubt Lake
RIDG	M. Goni	River DOC	Umpqua & Eel River Basins
ROMN	W. Schweiger	Rocky Mountain Network	Montana, Wyoming, Colorado
RREM	C. Murphy	Reservoir Research	Oregon
SAMI	D. Noakes	Salmon Migration	Elk River, Oregon
SIEN	A. Heard	Sierra Nevada Network	Nevada, California
SLAK	C. Addis	Spearfish Lake	The Dalles, Oregon
SLTZ	A. Sweet	Lincoln Soil & Water Cons. Dist.	Siletz River
STAR	E. Hinckley	Various	Univ. of Colorado Boulder
TIHI	M. Harrison	Temporal Isotopic & Hydrochem. Inv.	Johnson Creek Watershed
UMTI	J. Istok	Bromide Tracer Study	Umatilla, Oregon
WICR	S. Burnett	Willow Creek	Univ. of Idaho
WILL	T. Lundell	Willow	Willow
WIRI	D. Griffith	Willamette River	Willamette Univ.
ZORT	C. Dieterle	Zooplanton Recovery	Oregon

5.2 Sample Storage

Samples are stored in the walk-in cold room (4ºC), or one of the laboratory freezers (-20ºC) or refrigerators (4ºC). Storage temperature is monitored using traceable memory monitoring thermometers (see data sheet in the appendix). High, low and current temperatures are logged weekly. Historical records are kept on file at CCAL. Analyzed samples are held for three weeks after submission of final database and then disposed of unless further analyses or reanalyses are requested or other arrangements are made.

5.3 Sample Processing and Preservation

Samples requiring filtered aliquots should be filtered as soon as possible after collection to minimize biological and algal activity. CCAL recommends filtering in the field if at all possible. Membrane filters (pore size approximately 0.5 um) and glass-fiber filters (Whatman GF/C-pore size 1.2 um or GF/F-pore size 0.7 um) are most commonly used. CCAL uses Whatman GF/F Glass Fiber Filters that are prewashed with DIW and oven dried at 80ºC, unless otherwise specified by project. After filtering, both filtered and unfiltered samples should be stored in the dark at 4°C until delivery at the lab.

In general, the most reliable analytical results are obtained when samples are analyzed immediately after collection. This is rarely possible. The most commonly used sample preservation methods consist of addition of chemical preservatives. CCAL does not recommend chemical preservation of samples as it increases the potential for contamination and interferes with some analyses. CCAL recommends freezing of a filtered sample aliquot for most analyses; other aliquots should be kept cold and in the dark. See Table 3 for various analysis-specific preservation and hold time procedures used by CCAL.

Regardless of the preservation method, complete stability for every constituent is unattainable. Strict rules for preservation of water samples do not exist and effectiveness of most methods are questionable for various analytes. Extensive studies have been published supporting preservation of water samples by freezing for many analytes. Whatever methods are used, they should be consistent across the life of the project and procedures should be well documented. The lab should be notified in advance of the preservation method used.

Table 3: Typical CCAL Hold Times

Analysis	Storage Temperature Filtered	Unfiltered	Hold Time*
Alkalinity	-----	4ºC	7 days
Ammonia-nitrogen*	-20ºC or 4ºC	-----	48 hours unless frozen
Bromide	-20ºC or 4ºC	-----	28 days unless frozen
Calcium	-20ºC or 4ºC	4ºC	30 days unless frozen
Carbon, dissolved or total organic	-20ºC or 4ºC	4ºC	14 days unless frozen
Carbon, inorganic	4ºC	4ºC	72 hours
Chloride	-20ºC or 4ºC	-----	28 days unless frozen
Dissolved Solids	4ºC	-----	7 days
Magnesium	-20ºC or 4ºC	4ºC	30 days unless frozen
Nitrate-nitrogen	-20ºC or 4ºC	-----	48 hours unless frozen
Nitrogen, total dissolved or total (Persulfate)	-20ºC or 4ºC	4ºC	28 days until digestion unless frozen
Phosphate, ortho	-20ºC or 4ºC	4ºC	48 hours unless frozen
Phophorous, total dissolved or total	-20ºC or 4ºC	4ºC	28 days until digestion unless frozen
pH	-----	4ºC	7 days
Potassium	-20ºC or 4ºC	4ºC	30 days unless frozen
Silica	4ºC	-----	28 days
Sodium	-20ºC or 4ºC	4ºC	30 days unless frozen
Solids, Suspended	-----	4ºC	Filtered within 7 days
Specific conductance	-----	4ºC	7 days
Sulfate	-20ºC or 4ºC	-----	28 days unless frozen
Suspended Sediment	-----	4ºC	7 days

**CCAL does not recommend freezing samples for more than 8 weeks whenever possible.*

5.4 Sample Tracking

Requested analyses are entered into the database at time of sample login. Sample analysis progress is tracked through data entry both electronically and in tables. See example Table 4 and Table 5.

Table 4: CCAL Water Analysis Laboratory: Master Tracking Sheet for FY2018

Analysis/Determination

Project	Ph	Alk	Cond	SSED	DSOL	DOC	TOC	DIC	NH3	NO3	PO4	SiO2	TDN	UTN	TDP	UTP	Cl	SO4	Br	Na	K	Ca	Mg	Fe	Mn	ANCA	Hard
ANJO*	X	X	X			X	X	X	X	X	X		X	X	X	X	X	X		X	X	X	X	X			
ATAL*		X				X								X		X											
BREF				X					X	X	X			X		X											
CAKN	X	X	X			X			X	X	X		X	X	X	X	X	X		X	X	X	X				
CALL*	X	X	X			X			X	X	X	X	X	X		X	X	X		X	X	X	X				
CTLK*	X	X	X				X		X	X	X	X	X	X	X	X	X	X		X	X	X	X				
DAKS																	X										
DCMC														X		X											
DLKM									X	X	X	X		X		X											
DRIP									X	X	X			X		X	X										
EAJO																											
FRAD											X																
HAWA*		X																				X	X	X			X
HBNJ						X			X	X	X		X														
HJAN*	X	X		X		X			X	X	X	X	X	X	X	X	X	X		X	X	X	X				
JAFS									X	X	X		X		X		X	X		X	X	X	X				
LILA						X																					
LVLD						X								X		X	X	X		X	X	X	X				
MESO						X		X																			
MOJN*	X	X	X							X			X	X	X	X	X	X		X	X	X	X				
NOCA					X	X			X	X	X		X		X	X	X	X		X	X	X	X				
OLYF					X				X	X	X		X		X		X	X		X	X	X	X				
PPDO						X																					
RAIN*					X				X	X	X		X		X		X	X		X	X	X	X				
REDS	X	X	X						X	X	X	X	X	X	X	X						X^F/U	X^F/U				
RIDG						X																					
ROMN		X		X		X			X	X	X		X	X	X	X	X	X		X							
SIEN	X		X							X			X		X		X	X		X	X	X	X				
SLAK									X	X	X			X		X								X^F/U	X^F/U		
SLTZ				X			X		X	X	X			X		X											
STAR*											X	X			X	X											
TIHI										X	X						X	X						X^F/U			
UMTI*																	X		X								
WICR									X	X	X			X		X								X^F/U	X^F/U		
WILL									X	X	X			X		X								X^F/U	X^F/U		
WIRI*						X	X		X	X	X						X	X	X								

*Requested analyses may vary

Table 5: CCAL Water Analysis Laboratory: Analysis Tracking Sheet

						Hold Times		7d	7d	7d	7d		14d	48h	48h	48h	28d	28d	28d	28d	28d	28d	30d	
							Analysis/Determination																	
Data Sent	Data Prep	Project	Sample Series	Arrival Date	Thaw Date	Due Date	Filter	pH	Alk	Cond	SSED	TDS	DOC	NH3	NO3	PO4	SiO2	TDN	UTN	TDP	UTP	Anions	Cations	Invoiced

6.0 Calibration and Analytical Procedures

Standard Operating Procedures (SOPs) are available as individual documents for each analysis used at CCAL Water Analysis Laboratory. A complete list of methods is found in Table 1, and general laboratory procedures are documented here. Additional methods may be developed upon request, and as new instrumentation is obtained. See Table 4 for a list of current projects and requested analyses.

Run logs are maintained for each instrument. They contain information such as analysis run details, samples analyzed, instrument maintenance, problematic symptoms, troubleshooting and response.

Descriptions of analytical procedures including instrument calibration are detailed in each analyte specific SOP. General laboratory procedures are outlined below.

6.1 Balance and Pipette Calibration

All laboratory balances are calibrated yearly by an external vendor. The vendor is called in for repairs and/or maintenance if any abnormalities are observed in the interim. Pipette calibration is checked before every use by weight to within 1% of theoretical weight of aliquot volume.

6.2 Calibration Standard Preparation

Standards are prepared by serial dilution (if necessary) of standards purchased from vendors that provide traceability to National Institute of Standards and Technology (NIST) standards. Preparation of stock and working standards is recorded on worksheets (see example in appendix) and documented by the weight of standard added to a given flask before dilution to volume with DI water. The weight of standard dispensed must be within 2% of the expected value. Balances are checked with certified weights before standard preparation. All records of certification and standard preparation are kept on file at CCAL.

6.3 General Calibration and Analysis Procedures

Generally, analytical instrumentation is calibrated at the beginning of each analysis set with three to six working standards. A second source check standard (SSCS) is analyzed after the calibration and after every 10 samples. For most analyses, the SSCS is followed by a blank. The SSCS is prepared from a source or lot different than that used for the calibration standards. Check standard recovery must be within 10 % of theoretical value, or within normal observed limits of variability, to accept the sample data preceding it. In addition to the SSCS, a detection limit standard and/or a bulk quality control check standard (QCCS) may be analyzed once each run. Approximately 10% of the samples analyzed are duplicated; duplicate values must be within 10% of the original value.

6.4 Method Detection Limits

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 and is determined from analysis of a sample in a given matrix containing the analyte (U.S. EPA, 40CFR136, App. B). The MDL is determined by repeated analysis of a standard solution approximately five times the concentration of the estimated detection limit. The standard sample used in determination of the MDL should complete all normal sample processing steps used in the analytical method. At least seven measurements are recommended for determining the MDL. The MDL is calculated as follow:

MDL = t*S

t = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

S = standard deviation of the replicate analyses

7.0 Internal Quality Control Checks

Analytical instrumentation is calibrated using standard solutions of the analyte of interest. CCAL uses prepared, NIST traceable standard. Calibration correlation should be greater than 0.995. For most analyses, drift is monitored with check standards throughout the analysis run. Check standards are from a source or lot other than that of the calibration standards. If drift outside 10% recovery is observed, the run is stopped and the instrument recalibrated, or the analysis is repeated. Samples beyond the last acceptable check standard are reanalyzed.

For most analyses, a bulk, surface water Quality Control Check Standard (QCCS) is analyzed once each analysis run. The QCCS results may be used to establish control charts. Response is required for results outside three standard deviations of control values, and may include recalibration and reanalysis, instrument maintenance and/or repair. Some analyte concentrations may change over time and this must be taken into account when determining appropriate response.

Sample duplicates are used to estimate precision. When sample volume allows, 10 % of the samples are duplicated for every analysis. Field duplicates may be included upon request.

To estimate accuracy, CCAL participates in the United States Geological Survey (USGS) Standard Reference Surface Water test program and the National Water Research Institute's (NWRI) Environment Canada Proficiency Testing (PT) Program for analysis

of test samples for nutrient and chemical constituents of natural waters. See summaries of recent results in the appendix.

Other quality checks performed during analysis may include blanks run throughout the analysis to monitor carry-over, detection limit standards run once each analysis, and filter and bottle blanks.

Temperatures of all sample storage areas are monitored using traceable memory monitoring thermometers and tracked on forms attached to each refrigerator and freezer (see data sheet in the appendix). High, low and current temperatures are logged bi-weekly. Historical records are kept on file at CCAL. If the temperature exceeds the acceptance limit, corrective action must be taken which may include moving the samples to another refrigerator or freezer until the problem is corrected.

Oven temperatures are monitored using traceable memory monitoring thermometers. Current temperatures are recorded weekly, upon use or as required to show consistency.

Semi-annually, the bottle wash procedure is verified with bottle blanks. Bottles for various analyses are filled with DIW, and allowed to sit for at least seven days at 4°C. Analytical results should be lower than one standard deviation over the detection limit.

8.0 Data Quality Objectives

Measurement Data Quality Objectives presented in Table 6 represent the 99 % confidence intervals about a single measurement. At lower concentrations, precision objectives are equivalent to the MDL, and based upon the standard deviation (*sd*) of a set of repeated measurements:

$$sd = \sqrt{\sum (x - \bar{x})^2 \Big/ (n-1)}$$

where x is an individual measurement and $\bar{x}$ is the mean of the measurement set. For higher concentrations, the precision objectives are based on the percent relative standard deviation (*%RSD*).

$$\%RSD = \frac{sd}{\bar{x}} * 100$$

This reduces the problems of unreasonable objectives for low or high analyte concentrations. Concentration ranges are specified to determine the concentration at which absolute or relative terms apply. The division between the ranges, the Transition Value (*tv*), is estimated by:

$$tv = \frac{\sqrt{\frac{sd}{2} * sd}}{RSD} - \frac{sd}{2}$$

where $RSD = \%RSD/100$.

Table 6: Measurement Data Quality Objectives

Analyte	Method Detection Limit	Precision and Accuracy	Transition Value*
Alkalinity	0.2 mg $CaCO_3$/L	± 0.2 mg/L or ± 5 %	4 mg/L
Ammonium	0.01 mg N/L	± 0.003 mg/L or ± 5 %	0.06 mg/L
Barium	0.2 mg/L	± 0.2 mg/L or ± 5 %	4 mg/L
Bromide	0.01 mg/L	± 0.01 mg/L or ± 5 %	0.2 mg/L
Calcium	0.06 mg/L	± 0.06 mg/L or ± 5 %	1.2 mg/L
Carbon, Organic	0.05 mg/L	± 0.05 mg/L or ± 5 %	1 mg/L
Carbon, Inorganic	0.05 mg/L	± 0.05 mg/L or ± 5 %	1 mg/L
Chloride	0.01 mg/L	± 0.01 mg/L or ± 5 %	0.2 mg/L
Conductivity	0.4 µS/cm	± 1 µS/cm or ± 2 %	50 µS/cm
Dissolved Solids	5 mg/L	± 5 mg/L or ± 10 %	50 mg/L
Iron	0.06 mg/L	± 0.06 mg/L or ± 5 %	1.2 mg/L
Magnesium	0.02 mg/L	± 0.02 mg/L or ± 5 %	0.4 mg/L
Manganese	0.02 mg/L	± 0.02 mg/L or ± 5 %	0.4 mg/L
Nitrate/Nitrite	0.001 mg N/L	± 0.001 mg/L or ± 5 %	0.02 mg/L
Nitrogen, Total	0.01 mg N/L	± 0.01 mg/L or ± 5 %	0.2 mg/L
Ortho-Phosphorus	0.001 mg P/L	± 0.001 mg/L or ± 5 %	0.02 mg/L
pH	NA	± 0.1 pH unit	NA
Phosphorus, Total	0.002 mg P/L	± 0.002 mg/L or ± 5 %	0.04 mg/L
Potassium	0.03 mg/L	± 0.03 mg/L or ± 5 %	0.6 mg/L
Silicon	0.20 mg Si/L	± 0.05 mg/L or ± 5 %	1 mg/L
Sodium	0.01 mg/L	± 0.01 mg/L or ± 5 %	0.2 mg/L
Strontium	0.02 mg/L	± 0.02 mg/L or ± 5 %	0.4 mg/L
Sulfur	0.01 mg S/L	± 0.01 mg/L or ± 5 %	0.2 mg/L
Suspended Sediment	2 mg/L	± 1 mg/L or ± 10 %	10 mg/L

** The value above which precision and accuracy are expressed in relative terms.*

To use difference instead of the standard deviation to evaluate precision, the difference between two measurements is used for the absolute term and the relative percent difference (*RPD*) is used for the relative term:

$$RPD = \frac{|x - x_2|}{\bar{x}} * 100$$

9.0 Data Reduction, Validation and Reporting

Analytical results are collected in various formats, dependent upon the instrumentation output. All sample information, project data, billing, customer and project information, analytical results, quality control results and calibration statistics are entered and tracked through a SQL database with Microsoft Access interface. All QA and QC indicators are reviewed at time of analysis, and the analytical results are validated and the QA/QC checked again before final submission of the database.

Analytical results, sample information and calibration summaries are sent electronically to the project PI in Excel format. Investigators have three weeks to review the results and request reanalyses.

Validation of analytical results may include the following calculations:

- For projects requesting a complete analytical suite of anions, cations, pH and alkalinity, Ion Balance may be run to check for completeness and identify any outlying values. The balance may be skewed if there is an abundance of an ion not analyzed, but the balance check works well for most waters.

$$\text{Ion balance} = \frac{\sum anions}{\sum cations}$$

  Where:
  $\sum anions$ = HCO_3 + SO_4-S + Cl + NO_3-N + PO_4-P
  $\sum cations$ = H + Ca + Mg + K + Na + NH_4
  All ion concentrations are in units of ueq/L

- Total nitrogen concentration should be greater than the sum of ammonia and nitrate/nitrite.

- Total phosphorus concentration should be greater than orthophosphorus.

- Total (unfiltered) results should be greater than dissolved (filtered) results.

Data Quality Analysis Reports may be requested for detailed analysis of all indicators used by CCAL (fees apply).

Electronic and hard copy reports of all laboratory records are stored at Oak Creek Building, College of Forestry, Oregon State University. Historical records are available upon request, with permission from the initiating PI (fees apply).

10.0 Performance and System Audits

CCAL has participated in the USGS inter-laboratory comparison study for laboratory quality assurance testing semiannually since 1981. The program provides Standard Reference Samples for Trace Elements, Major Ions, Precipitation and Nutrient samples. Accuracy of CCAL's analytical results are ascertained based on performance in the program. See the appendix for a summary of recent results.

In 2009 CCAL began participating annually in Environment Canada's National Water Research Institute Proficiency Testing Program. CCAL has ramped up participation and now participates semiannually in the Rain & Soft Waters, Major Ions and Total Phosphorus Studies. The advantage of this study is a much larger, more diverse, sample set. See the appendix for a summary of results.

11.0 References

11.1 Standard Methods for the Examination of Water and Wastewater, American Public Health Association. 21st Edition, 2005.

11.2 Code of Federal Regulations. Protection of Environment. Section 40, Appendix B to Part 136. Definition and procedure for the determination of the method detection limit. Revision 1.11. Revised July 1, 1990. Office if the Federal Register, National Archives and Records.

11.3 ASTM. American Society for Testing and Materials. Standard Specifications for Reagent Water. D1193-77 (reapproved 1983). Annual Book of ASTM Standards, Vol. 11.01. ASTM: Philadelphia, PA, 1991.

11.4 Water Chemistry Laboratory Manual, Wadeable Streams Assessment. U.S. Environmental Protection Agency, Office of Water, Washington DC; EPA841-B-04-008, 2004.

11.5 Recommended Guidelines for Sampling and Analyses in the Chesapeake Bay Monitoring Program, U.S. Environmental Protection Agency; EPA 903-R-96-006, 1996.

11.6 D.T.E. Hunt and A.L. Wilson, "The Chemical Analysis of Water: General Principles and Techniques". Royal Society of Chemistry; Burlington House, London; 1986

11.7 Chaloud, D. and Peck, D.V. (Eds) 1994. Environmental Monitoring and Assessment Program: Integrated Quality Assurance Project Plan for the Surface Waters Resources Group, 1994 Activities. EPA 600/X-91/080, Rev. 2.00. U.S. Environmental Protection Agency, Las Vegas, Nevada.

11.8 Patton, C.J. and Gilroy, E.J. 1999. U.S. Geological Survey; Nutrient Preservation Experiment – Experimental Design, Statistical Analysis, and Interpretation of Analytical Results; Water-Resources Investigations Report 98-4118; U.S. Geological Survey. Denver, Colorado.

11.9 U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1-A9, available online at http://pubs.water.usgs.gov/twri9A.

11.10 Environment Canada, Analytical Methods Manual; August 1979. Inland Waters Directorate; Water Quality Branch; Ottawa, Canada.

12.0 Document History Log

Table 7: CCAL Standard Operating Procedures Revision History

Standard Operating Procedure	CCAL Revision	Date of Initial Version	Date of Most Recent Revision
Basic Determinations:			
Determination of pH and Alkalinity	CCAL 10C.1	March 2006	February 2015
Determination of Specific Conductance	CCAL 11A.2	March 2006	February 2015
Determination of Suspended Sediments	CCAL 12A.3	April 2006	February 2015
Determination of Total and Total Dissolved Solids	CCAL 13A.1	March 2006	February 2010
Determination of Color by Visual Comparison	CCAL 14A.1	April 2006	February 2010

Determination of Color by Color Kit	CCAL 15A.1	January 2010	February 2015
Determination of Turbidity	CCAL 16A.1	February 2010	June 2013
Carbon Analysis:			
Analysis of Dissolved and Total Organic Carbon	CCAL 20A.3	June 2006	March 2015
Analysis of Dissolved and Total Inorganic Carbon	CCAL 21A.1	February 2010	March 2015
Automated Analysis, Colorimetric:			
Analysis of Ammonia in Fresh Waters	CCAL 30A.2	March 2006	April 2014
Analysis of Nitrate/Nitrite and Nitrite in Fresh Waters	CCAL 31B.1	March 2006	April 2014
Analysis of Silicon in Fresh Waters	CCAL 32B.1	March 2006	April 2018
Digestion and Analysis of Fresh Water Samples for Total Nitrogen and Total Dissolved Nitrogen	CCAL 33A.4	March 2006	April 2014
Analysis of Orthophosphrus in Fresh Waters	CCAL 34B.2	April 2006	April 2014
Digestion and Analysis of Fresh Water Samples for Total Phosphorus and Total Dissolved Phosphorus	CCAL 35B.2	April 2006	April 2014
Spectrophotometric Analysis:			
Digestion and Analysis of Fresh Water Samples for Total Phosphorus and Total Dissolved Phosphorus	CCAL 41A.1	April 2006	March 2010
Ion Chromatography:			
Analysis of Chloride and Sulfate in Fresh Waters by Ion Chromatography	CCAL 50B.2	April 2006	May 2015

Atomic Absorption:			
Analysis of Metal Cations in Fresh Waters by Flame Atomic Absorption	CCAL 60B.1	April 2008	May 2015

13.0 Document Revision History

Original Document: May 2008
Version: 0
Title: Quality Assurance Plan, CCAL Water Analysis Laboratory

Edit Date: April 2010
New Version: 1
Address Update
General editing and updating throughout document
Section 7.0: add Environment Canada Proficiency Testing Program participation

Edit Date: June 2012
New Version: 2
General editing and updating throughout document
Section 4.0: remove manual total phosphorus reference

Edit Date: June 2013
New Version: 3
Table 3.1: revise suspended sediment detection limit, change magnesium range, update SOP versions
Table 5.2: add suspended sediment hold time
Tables 5.1, 5.3, Appendix E, Appendix F: updates for current year
General editing throughout document for grammatical correctness and clarity
Table 8.1: revise suspended sediment detection limit, correct alkalinity transition value
Appendix G, Appendix H: update Lab Aide manuals to more accurately reflect current procedures used.
Table 12.1: Update SOP versions and dates

Edit Date: April 2018
New Version: 4
General editing and updating throughout document
Section 2.0: update personnel
Table 3.1: update CCAL Method numbers and SSED detection limit
Section 4.1: update acid bath concentration
Table 5.1: replace with current project list
Table 5.2: add gravimetric determinations
Table 5.3: replace with current project list
Table 5.4: update to include expanded information
Section 9.1: replace Microsoft Visual Fox Pro with SQL database
Table 12.1: update CCAL Method numbers and dates

Appendix A: CCAL Recommended Sample Collection Protocol

Sample collection requirements vary greatly across programs. Specific project protocols depend upon study objectives, program requirements and cross project comparability. Whatever protocol you decide to adopt, be consistent throughout the life of the program. We are happy to assist you in making a decision and provide clean, and/or baked bottles at your request (fees apply). Contact the lab for more information.

Generally, collect a bulk sample, filter a portion of the homogenized sample, and transfer filtered and unfiltered sample aliquots to well labelled, acid washed, high density polyethylene bottles; prerinse bottles with sample, fill to the brim (negative meniscus) and cap tightly. Store sample away from sunlight at 4ºC, and transport to the lab as soon as possible.

Conversely;

- USGS Field Manual (OWQ, 2002) states that bottles designated for analysis of organic compounds should not be prerinsed.
- Polyethylene containers are suggested for TOC sample collection in EPA's EMAP QAPP (EPA 600/X-91/080, 1994), Environment Canada's Analytical Methods Manual (1979) and EPA Test Method 9060A (EPA SW-846). Alternatively, Standard Methods (AWWA, 2005) and USGS Field Manual (OWQ, 2002) recommend use of only baked, glass bottles for collection of organic samples.

See documented references for complete sample collection protocols.

Appendix B: Sample Receipt and Tracking Form

CCAL Sample Tracking Log

Abbreviations Key:

S = small	B = blue	O/T/B = ours/theirs/both
M = medium	R = red	WaBo? wash bottles?
L = large	O = other	
	PM = packing material	

			Sample Counts						(not CCAL cooler)			Locate	Date					
			Fresh		Frozen		Acidified											
Project	Lab # Range	Initials	# filt	# unf	# filt	# unf	# filt	# unf	Cooler(s) # / N?		Cooler Desc.	Cold	Dump			Service/Supplies with Misc Charges	Displacement Date	Additional Comments / Tracking
Investigator	Arrival Date	Condition / Comments							WaBo?	O/T/B	PM	Fro	pull	wash	sent	What Shipped?	To Who?	
Project	Lab # Range	Init	# filt	# unf	# filt	# unf	# filt	# unf	Cooler?		CD	Cold	Dump			Service/Supplies to charge	Displacement Date	Additional Comments / Tracking
Investigator	Arrival Date	Condition / Comments							WaBo?	O/T/B	PM	Fro	pull	wash	sent	What Shipped?	To Who?	
Project	Lab # Range	Init	# filt	# unf	# filt	# unf	# filt	# unf	Cooler?		CD	Cold	Dump			Service/Supplies to charge	Displacement Date	Additional Comments / Tracking
Investigator	Arrival Date	Condition / Comments							WaBo?	O/T/B	PM	Fro	pull	wash	sent	What Shipped?	To Who?	
Project	Lab # Range	Init	# filt	# unf	# filt	# unf	# filt	# unf	Cooler?		CD	Cold	Dump			Service/Supplies to charge	Displacement Date	Additional Comments / Tracking
Investigator	Arrival Date	Condition / Comments							WaBo?	O/T/B	PM	Fro	pull	wash	sent	What Shipped?	To Who?	

Appendix C: Sample Storage Temperature Monitoring

CCAL Water Analysis Laboratory
Temperature Monitoring of Sample Storage Unit

Unit Description:

Target Temperature:

Acceptance Range:

Contacts: Kathy Motter 541-737-5120
Laura Hartley 541-737-0826

		Temperature, C		
Date Recorded	Current T	Low	High	Comments

Appendix D: Standard Preparation Worksheet

CCAL Standard Preparation Worksheet

Standard:

Starting Material

Chemical/Sample Name:
Manufacturer:
Chemical ID / Lot #:
Concentration:
Expiration (if applicable):

Stock Standard Preparation

Theoretical Volume or Weight of Standard Aliquot:	
Actual Weight of Standard Aliquot:	
Final Volume:	

Final Concentration:

Preparation Documentation

Balance Check

Balance	
Weight Set and Weight used	
Weight observed	

Pipette Performance Check

Pipette	
Volume of DI pipetted (mL)	
Weight of DI pipetted (g)	

Working Standards

Volume of Standard Pipetted (mL)	Weight of Standard Pipetted (g)	Final Standard Solution Volume (mL)	Final Concentration of Standard (mg/L) as ____

Comments:

Analyst / Date Prepared:

Appendix E: Summary of USGS Interlaboratory QA Study Results

USGS Office of Water Quality, Standard Reference Sample Project
Interlaboratory Standard Reference Sample Comparison Study Results Summary

		Spring 18; M-226			Fall 17; M-224			Spring 17; M-222		
Major	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	Alkalinity	53.61	49.8	0	23.11	23.3	4	58.48	57.4	3
	Bromide	0.09	0.055	2	0.26	0.265	4	0.09	0.101	4
	Calcium	13.48	13.4	4	6.96	6.79	3	10.73	10.8	4
	Chloride	25.62	26.2	3	9.85	10.2	2	7.75	7.57	3
	Fluoride	0.23	0.242	3	0.68	0.700	3	0.32	0.351	2
	Magnesium	9.08	8.84	3	1.43	1.44	4	8.44	8.56	4
	pH	9.9	9.81	4	7.5	7.39	4	9.2	9.15	4
	Potassium	2.26	2.27	4	1.11	1.07	3	1.16	1.17	4
	TDS (DSOL)	113	126	3	57	64.0	2	90	88	4
	Silica	10.52	10.5	4	9.57	9.56	4			
	Sodium	14.51	14.0	3	101.2	99.2	3	7.04	7.08	4
	Conductance	217.5	220	3	5.69	5.64	4	156.5	158	4
	Sulfate	14.14	14.2	4	0.028	0.033	3	10.01	9.77	3
	TP as P	0.050	0.053	4	11.47	11.0	3			
				3.14			3.29			3.58
		Spring 18; P-70			Fall 17; P-69			Spring 17; P-68		
Precipitation	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	Calcium	0.52	0.501	3	0.18	0.220	0	1.01	0.931	1
	Chloride	22.53	23.5	2	2.54	2.51	4	2.27	2.23	3
	Fluoride	0.05	0.060	3	0.05	0.050	4	0.03	0.03	4
	Magnesium	1.43	1.40	3	0.07	0.066	3	0.14	0.109	0
	OP as P	0.086	0.083	4	0.001*	0.0015	3	0.011	0.014	0
	pH	4.1	4.12	4	4.1	4.04	3	4.5	4.5	4
	Potassium	0.33	0.345	4	0.01	0.011	4	0.17	0.166	4
	Sodium	11.10	11.1	4	0.25	0.247	4	0.4	0.38	3
	Conductance	105.9	112	2	46.5	46.8	4	24.3	26.6	2
	Sulfate	0.51	0.520	4	0.24	0.287	4	1.32	0.508	0
				3.30			3.30			2.10
		Spring 18; N-137			Fall 17; N-135			Spring 17; N-133		
Nutrient (low)	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	NH3-N	0.102	0.110	3	0.154*	0.154	4	0.096	0.093	4
	NO3-N+NO2-N	0.324	0.326	4	0.447	0.436	3	0.091	0.09	4
	OP as P	0.111	0.112	4	0.153*	0.15	4	0.088	0.088	4
	TN as N	0.46	0.450	4	0.64	0.616	3			
	TP as P	0.133	0.135	4	0.167	0.163	3			
				3.80			3.40			4.00
		Spring 18; N-138			Fall 17; N-136			Spring 17; N-134		
Nutrient (high)	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	NH3-N	0.459	0.430	3	0.438*	0.370	0	0.116	0.115	4
	NO3-N+NO2-N	0.784	0.790	4	0.647	0.639	4	0.809	0.801	4
	OP as P	0.597	0.607	4	0.391*	0.388	4	0.269	0.263	4
	TN as N	1.34	1.27	3	1.07	1.06	4			
	TP as P	0.612	0.620	4	0.384	0.388	4			
				3.60			3.20			4.00
				3.46	Overall Average		3.30			3.42

M = Major; P = Precipitation; N = Nutrient (low and high); * new instrumentation

RV = Reported Value; MPV = Most Probable Value

Appendix F: Summary of NWRI Environment Canada Proficiency Test

National Water Research Institute, Environment Canada

Rain and Soft Waters

Analysis	Study #0111 December 2017	Study #0110 June 2017	Study #0106 June 2015
Alkalinity			
Ammonia	1 flag, high bias	3 flags, low bias	Ideal
Calcium	high bias	1 flag, high bias	Ideal
Chloride	Ideal	1 flag	Ideal
Conductivity	Ideal	Ideal	Ideal
DIC	Ideal	Ideal	Ideal
DOC	Ideal	Ideal	Ideal
Fluoride	Ideal	1 flag	Ideal
Magnesium	Ideal	4 flags, high bias	Ideal
Nitrate + Nitrite	Ideal	Ideal	2 flags, high bias
pH	Ideal	Ideal	Ideal
Potassium	Ideal	Ideal	Ideal
Reactive Silica	Ideal		low bias
Sodium	3 flags, low bias	2 flags	2 flags
Sulfate	Ideal	Ideal	Ideal
Total Hardness	Ideal	Ideal	Ideal
Total Nitrogen	Ideal	Ideal	Ideal
Overall Performance:	*Good*	*Fair*	*Good*

Major Ions

Analysis	Study #0111 December 2017	Study #0110 June 2017	Study #0106 June 2015
Ammonia	1 flag	Ideal	Ideal
Calcium	1 flag, high bias	Ideal	Ideal
Chloride	1 flag	Ideal	Ideal
Conductivity	Ideal	high bias	Ideal
DIC	Ideal	Ideal	Ideal
DOC	Ideal	Ideal	Ideal
Fluoride	1 flag	3 flags	Ideal
Magnesium	high bias	Ideal	Ideal
Nitrate + Nitrite	Ideal	1 flag	2 flags, high bias
pH	Ideal	high bias	Ideal
Potassium	Ideal	Ideal	Ideal
Reactive Silica	1 flag	low bias	low bias
Sodium	1 flag	Ideal	Ideal

Sulfate	Ideal	Ideal	Ideal
Total Alkalinity	high bias	Ideal	Ideal
Total Hardness	high bias	Ideal	Ideal
Total Nitrogen	1 flag	Ideal	Ideal
Overall Performance:	*Fair*	*Good*	*Good*

	Study #0111 December 2017	**Study #0110 June 2017**	**Study #0106 June 2015**
Total Phosphorus	3 flags, low bias	Ideal	Ideal
Overall Performance:	*Poor*	*Very Good*	*Very Good*

Five-Year Historical Performance:			
Rain and Soft Waters	*Good*	*Very Good*	*Very Good*
Major Ions	*Good*	*Good*	*Good*
Total Phosphorus	*Very Good*	*Very Good*	*Very Good*

Appendix G: Lab Aide Manual

Responsibilities of Lab Aide

Daily Routine

- Survey the laboratory.
- Check with supervisor for specific, urgent needs (i.e., acid baths, bottle shortages, replace broken glassware, specific glassware shortage, wash/weigh filter papers).
- After survey of lab:
    - Put away clean, dry glassware.
    - Clean dirty glassware.
    - Housekeeping (paper towel and Kimwipe supplies, repaper trays, drawers and hoods, dusting, garbage, recycling).

Things to Remember

- Never be lulled into a false sense of complacency. Remember that you are working with HAZARDOUS CHEMICALS.
- The lab aide has the most important job in the lab. Why? Because without clean glassware, our results are not dependable.
- Any glassware that should not be touched should be handled with a Kimwipe, gloves, or tongs.
- Do not touch any glassware, including sample bottles, in such a way as to cause contamination—avoid areas that will contact with the water sample.
- Dependability. The lab aide must be dependable in both coming to work and the work you do.

General Rules and Guidelines

- Always wear a lab coat.
- Always wear eye goggles/glasses and gloves when working with concentrated acid, chemicals or disposing of chemical solutions.
- When immersing/removing glassware from acid baths always wear goggles and gloves.
- Remove glassware from acid baths after 24 hours unless otherwise specified.
- Never place glassware with tape or ink labels into acid bath. Remove labels first. This avoids contaminating the acid baths.
- Always wear gloves and goggles when working with acetone.
- Rinse glassware with DIW before placing in acid bath. This avoids contaminating the acid baths.
- Replace acid bath lids tightly.
- Clean up acid and acid bath spills immediately.
- Routinely rinse everything 4X with DIW.
- Do not use tap water on any glassware/bottles.
- Cap and store clean bottles in the bottle room as soon as they are dry to minimize contamination.
- Never put your fingers inside clean glassware/bottles even if you are wearing gloves.
- Never use soap on glassware. Rinse your hands well with DIW after washing with soap.
- Dry glassware upside down whenever possible.

Outline of Duties

1. Bottle Station/Dish Station (OCB242)
   - Dishwater - used for plastic sample bottles
   - Bottle Cages - check all bottles and lids for dirt, water, or spots before capping. Follow with second person check before caging.
   - Metal drying racks - used for drying all clean bottles, glassware and lids
   - TN tubes & caps
   - DOC/DIC vials and caps
   - pH/alkalinity beakers
   - Suspended sediment watchglasses
   - Miscellaneous glassware
     - Filtering equipment
     - Measuring equipment, i.e., pipettes, graduated cylinders etc.
     - Miscellaneous beakers, flasks, etc.

2. Atomic Absorption Station (OCB248)
   - Storage tubs for sample tubes
   - Shelves for racks

3. Auto-Analyzer Station (OCB248)
   - Miscellaneous glassware
   - Pipettes
   - Acid bath for sample vials – replace upon request

4. Macro-Filter Station (OCB150)
   - HJA carboys cleaning
   - Filter carboy cleaning
   - Filter equipment
   - Carboy weigh
   - Filtration setup

5. Balance Station (OCB242A)
   - Balance: weigh filter papers and dissolved solids beakers
     DO NOT TOUCH BEAKERS WITH FINGERS
   - Desiccant baking & desiccator clean
   - Washing and storing filters

6. Ovens (OCB242)
   - Cleaning ovens
   - Track/monitor temperatures

7. Acid Baths (OCB242, OCB248, OCB150)
   - HCl acid baths - change as needed or upon request
   - Acid Bath Preparation

- Fill 10-L carboy with approximately 8-L DIW
- In the fume hood, slowly add 1-L concentrated HCl
- Fill carboy to the 10-L mark with DIW

8. Storage Rooms (OCB333, greenhouse foyer, cold room)
   - HJA carboy storage
   - Cooler storage room
   - Sample storage

9. General Duties
   - Replenish supplies:
     - Paper towels
     - Kimwipes and tape
     - Parafilm
     - 125 mL plastic sample bottles
   - Dispose of broken glass
   - Acid storage inventory
   - Garbage & recycling out
   - Freezer and cold room organization
   - Shipping: ship coolers, bottles or other upon request
   - K-dry: trays, drawers, work stations
   - Weeklies, Monthlies, Yearlies (see Tables H & J)
   - Sample login, labelling and preparation
   - Clean drying racks
   - Monitor and log refrigerator and freezer temperatures

Appendix H: Lab Aide Weekly Task Sheet

		- Date -						
Activity	**Room #**							
Paper towels	150							
	242							
	248							
	254							
Eye Wash	150							
	242							
	248							
	254							
Clean/Flush	150							
Sink Drains	242							
	248							
	254							
Sweep	150							
	242							
	248							
	254							
Waste Bins	150							
	242							
	248							
	254							
Recyclables	150							
	242							
	248							
	254							
K-Dry	242							
Acetone, Ethanol,	242							
Bicarb, DIW, Acid	248							
Shelving and								
Dump Dates								
Cold Room Drain								
Sweep Cold Room								

Appendix I: Lab Aide Monthly Task Sheet

Lab Aid Task List: MONTHLY													
Activity	**Room**	**Jan**	**Feb**	**Mar**	**Apr**	**May**	**Jun**	**Jul**	**Aug**	**Sep**	**Oct**	**Nov**	**Dec**
Check Acid	HCl												
Inventory	H2SO4												
	HNO3												
	H3PO4												
Wash Drying	48												
Racks	56												
Wash Pegs &	48												
Peg Boards	56												
Clean Fume	48												
Hoods	56												

Lab Aid Task List: BI-YEARLY													
Activity	**Room**	**Jan**	**Feb**	**Mar**	**Apr**	**May**	**Jun**	**Jul**	**Aug**	**Sep**	**Oct**	**Nov**	**Dec**
Showers	48												
	56												
Dust Labs	48												
	56												