QUALITY ASSURANCE PLAN

CCAL WATER ANALYSIS LABORATORY

Department of Forest Science
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Acronyms/Abbreviations

AAII	Technicon Auto-AnalyzerII
AD	analytical duplicate
ASTM	American Society for Testing and Materials
BB	bottle blank
CCAL	Cooperative Chemical Analytical Laboratory
CFR	Code of Federal Regulations
cm	centimeter
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
ML	Minimum Level of Quantification
ng	nanogram
NIST	National Institute of Standards and Technology
NPS	National Park Service
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
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
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 Science, specializes in analysis of nutrient research samples for lake, stream, precipitation and groundwater, with samples coming from all over 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 full time chemists and three student interns. Personnel and their primary responsibilities include:

Kathryn Motter, Chemist and Lab Manager: Dionex IC, Shimadzu TOC, Varian AA, Total Phosphorus, documentation, budget, database management, data quality reports, laboratory organization, web page management and quality assurance monitoring

Cameron Jones, Chemist and Lab Supervisor: Technicon AutoAnalyzerII, Alkalinity, pH, conductivity, sample preparation, sample tracking and data reporting, invoicing, customer service, quality assurance monitoring, delegation of responsibilities to students

Kaleb Jentzsch, intern: sample log-in, sample preparation, sample setup for Total Phosphorus and Total Nitrogen digestions, Ortho-phosphorus sample setup and analysis, chemist support

Jeremy Unrau, intern: Total Dissolved Solids, Total Suspended Solids, sample storage and organization, cleaning of glassware and bottles, general laboratory maintenance, chemist support

Adam Silbernagel, intern: cleaning of glassware and bottles, general laboratory maintenance, chemist support

Student intern responsibilities shift and overlap with time and employee turnover.

3.0 Quality Assurance Objectives

Quality Assurance (QA) Objectives for CCAL are outlined in Table 3.1. Project specific QA Objectives supersede those listed.

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 0.5 M 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 427 mL 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 twice with DIW. Rinse bottles with acid bath and follow with thorough DIW rinse. Wash bottles in dishwasher on the ultra wash, followed by a DIW rinse cycle. Remove bottles from the dishwasher and place upside down on rubber mat covered drying shelves to dry. 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 thoroughly with DIW and dried on the rubber mat covered 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 Total Phosphorus Flasks

Rinse capping beakers (50/30 ml) 4X with DIW, fill and let soak for at least an hour in the sink. Rinse 4X with DIW and place upside down on tray to dry.

Remove sample identification markings with acetone. Rinse flasks four times and place in racks. Place rack(s) of flasks in dishwasher, and run on rinse only setting. Soak flasks in acid bath overnight. Rinse 4X with DIW, and air dry flasks on wooden drying rack. When completely dry, cap flasks and store in the appropriate color coded drawer. Date that the clean procedure was completed is recorded; flasks with oldest clean date are used first. Flasks with wash date older than one month are rewashed before use.

4.4 Ortho Phosphorus Tubes

Empty tubes, remove labels and rinse tubes with DIW three times. Fill tubes with DIW and soak for at least an hour. Place tubes in acid bath and soak overnight. Tubes are then rinsed again three times with DIW, inverted in wooden racks on clean absorbent lab matting and stored. Date that the clean procedure was completed is recorded; tubes with oldest clean date are used first. Tubes with wash date older than one month are rewashed before use.

4.5 pH, Alkalinity, Titration and Conductivity Beakers

Empty, remove markings and flush thoroughly DIW. Rinse three 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.

4.6 Dissolved Solids Beakers

Remove markings, rinse, fill with DIW and soak overnight. Empty and scrub with brush to remove residue. Rinse 4X 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.

4.7 Filter Equipment

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

4.8 Atomic Absorption Sample Tubes

Rinse tubes 4X with DIW. Fill with DIW and soak for at least one hour. Soak overnight in acid bath. Rinse 4X with DIW and place tubes in styrofoam holders. Dry upside down on paper covered racks. Dry tubes are stored in a plastic basin.

Table 3.1 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 10A.0	Alkalinity; APHA 2320	Radiometer TIM840 Auto-Titrator	0.2 mg $CaCO_3$/L	NA
		pH; APHA 4500 H		NA	0 - 14 pH units
Determination of Specific Conductance	CCAL 11A.0	APHA 2510	YSI 3200 & YSI 3256 probe with temperature correction	0.4 µS/cm	0 µS - 3 S
Determination of Suspended Sediments	CCAL 12A.0	APHA 2540 B; EPA 160.2	Gravimetric	5 mg/L	NA
Determination of Total Dissolved Solids	CCAL 13A.0	APHA 2540 C; EPA 160.3	Gravimetric	5 mg/L	NA
Determination of True Color	CCAL 14A.0	APHA 2120 B	Visual Comparison	0 CU	0 - 70 CU
Analysis of Dissolved and Total Organic Carbon	CCAL 20A.0	APHA 5310 B; EPA 415.1	Shimadzu TOC-VCSH Combustion Analyzer	0.05 mg/L	0.05 – 5.0 mg/L
Analysis of Ammonia in Fresh Waters	CCAL 30A.0	APHA 4500-NH3 G; EPA 350.1	Technicon Auto-Analyzer II Phenate Method	0.01 mg N/L	0.01 - 2.0 mg N/L
Analysis of Nitrate/Nitrite in Fresh Waters	CCAL 31A.0	APHA 4500-NO3 F; EPA 353.3	Technicon Auto-Analyzer II Cadmium Reduction Method	0.001 mg N/L	0.001 - 10 mg N/L
Analysis of Silicate in Fresh Waters	CCAL 32A.0	APHA 4500-SiO2 E	Technicon Auto-Analyzer II Molybdate-Reactive Silica	0.20 mg SiO2/L	0.20 - 20 mg SiO2/L
Digestion and Analysis of Fresh Water Samples for Total Nitrogen and Total Dissolved Nitrogen	CCAL 33A.0	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 - 2.0 mg N/L

Analyte	CCAL Method Number[1]	Reference Method[2]	Instrument and Method Description	Detection Limit[3]	Working Range[4]
Analysis of Orthophosphrus in Fresh Waters	CCAL 40A.0	APHA 4500-P E; EPA 365.2	Milton-Roy 601 Spectrophotometer Ascorbic Acid Method	0.001 mg P/L	0.001 - 0.30 mg P/L
Digestion and Analysis of Fresh Water Samples for Total Phosphorus and Total Dissolved Phosphorus	CCAL 41A.0	APHA 4500-P B; APHA 4500-P E;EPA 365.2	Persulfate Digestion with subsequent analysis on Milton-Roy 601 SpectrophotometerAscorbic Acid Method	0.002 mg P/L	0.002 - 0.30 mg P/L
Analysis of Chloride, Bromide and Sulfate in Fresh Waters by Ion Chromatography	CCAL 50B.0	APHA 4110 B; EPA 9056A	Dionex 1500 Ion Chromoatograph with Chemical Suppression of Eluent Conductivity	Cl; 0.01 mg/L Br; 0.01 mg/L SO4; 0.02 mg/L	Cl; 0.01 - 5 mg/L Br; 0.01 – 5 mg/L SO4; 0.02 - 5 mg/L
Analysis of Cations in Fresh Waters by Atomic Absorption Spectrometry	CCAL 60A.0	APHA 3111; EPA 7000B	Varian SpectrAA 220 Flame Atomic Absorption Spectrometer	Ba: 0.2 mg/L 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 Sr: 0.02 mg/L	Ba: 0.2 – 15.0 Ca: 0.06 - 10.00 Fe: 0.06 - 2.00 K: 0.03 - 3.00 Mg: 0.02 - 1.50 Mn: 0.02 - 2.00 Na: 0.01 – 6.00 Sr: 0.02 - 0.15

[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, FAA 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.

4.9 Total Nitrogen 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. Date that the clean procedure was completed is recorded; tubes with oldest clean date are used first. Annually, put tubes in foil tray and bake in Muffle Furnace at 550ºC for at least three hours.

4.10 Technicon Autosampler Tubes

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

4.11 Suspended Sediment Watch Glasses

Remove sample numbers with acetone. Do not remove the dividing lines; remark dividing lines if necessary. Rinse thoroughly with DIW and dry in the black drying rack.

4.12 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 the hood.

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. Put tubes in foil tray and place in Muffle Furnace. Bake at 550ºC for at least three hours. Cool overnight in the furnace. Tubes and lids are stored in air-tight containers.

4.13 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. See Lab Aide Manual (appendix) for project specific protocols.

4.14 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.

5.1 Sample Custody

A sample log, 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 log sheet or communicated to CCAL staff, with order of priority, prior to sample delivery. Once at the laboratory, samples are entered into the tracking system. 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 and logged in to the electronic database, a project code is assigned (see Table 5.1) and samples are numbered consecutively within that code for each individual project. Labeled aliquots for various analyses are prepared and delivered to appropriate storage area for requested analysis.

Table 5.1 CCAL Water Analysis Laboratory: Projects in FY07 and FY08

Code	Investigator	Project Name	Location
ABOS	D. Shaw	ABO Rusty Blackbird	Alaska
CAKN	T. Simmons	Central Alaskan Network	Alaska
CALL	A. Larsen	Central Alaskan Lakes	Alaska
CTLK	Larson	Crater Lake	Crater Lake
DCMC	M. Chandler	Dunes City	South of Florence
DIAM	J. Eilers	Diamond Lake	Diamond Lake
DLKM	R. Miller	Diamond Lake	Diamond Lake
EILJ	J. Eilers	Eilers	Various
GREP	T. Pennington	Greenhouse Water	Portland State University
HBNJ	S. Johnson	Hydrologic Benchmark Netw	HJA
HINK	K. Cromack	Hinkle Creek	Hinkle Creek
HJAN	S. Johnson	H.J. Andrews	HJA

JDOM	J. Mills	John Day Otoliths	John Day Basin
LCNM	P. Moran	Lake Crescent Nutrient	Lake Crescent
LEAF	L. Ashkenas	Leaf	HJA
LINX	S Johnson, Ashkenas	Linx	HJA
LOWS	Salinas	Lake of the Woods	Lake of the Woods
MACH	R. Haggerty	Mack Creek	HJA
MILL	Salinas	Miller	Miller Lake
MTBE	J. Eilers	Mt. Baker	Mt. Baker - Snoqualmie
NOCA	R. Glesne	North Cascades	North Cascades
OLYF	S. Fradkin	Olympic	Olympic National Park
PMAS	A. Straub	Macroinvertebrate Study	Portland State University
RAIN	Samora	Mt. Ranier	Mt. Ranier
REDS	T. Suminski	Redoubt	Redoubt Lake
RIVH	A. Herlihy	RIVMAP	Various
SCWM	C. Myers	South Coast Watersheds	Oregon
SILV	S. Van De Wetering	Siletz Nutrient Monitoring	Siletz River
SPLK	C. Crisafulli, D.	Spirit Lake	Spirit Lake
TRAJ	S Johnson, Ashkenas	Trask	Trask River Basin
UCKC	M. Cover	UC Berkely	Klamath Basin
WALD	D. Bates, Johnson	Waldo	Waldo Lake
WILL	Flaherty	Willow	Willow
WINE	Salinas	Winema	Winema

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 bi-weekly. Historical records are kept on file at CCAL. Analyzed samples are held for three weeks after submission of final database, 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 5.2 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 5.2 Typical CCAL Hold Times

	Storage Temperature		
Analysis	**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
Chloride	-20ºC or 4ºC	-----	28 days unless frozen
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
Specific conductance	-----	4ºC	7 days
Sulfate	-20ºC or 4ºC	-----	28 days unless frozen

**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 tables 5.3 and 5.4.

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 3.1 and general laboratory procedures are documented here. Additional methods may be developed upon request, and as new instrumentation is obtained. See Table 5.3 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 2% 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. All records of certification and standard preparation are kept on file at CCAL.

Table 5.3 CCAL Water Analysis Laboratory: Master Tracking Sheet for FY2007

Project	Analysis/Determination																					
	Filter	pH	Alk	Cond	SSED	DSOL	DOC	TOC	NH3	NO3	SiO2	PO4	TDN	UTN	TDP	UTP	Cl	SO4	Ca	Na	K	Mg
ABOS		X	X	X									X		X				X			
CAKN		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
CTLK		X	X	X					X	X	X	X	X	X	X	X	X	X	X	X	X	X
DIAM	/	/	/	/			/	/	/	/	/	/		/		/						
DLKM		X	X	X		X	X		X	X	X	X		X		X						
EILJ		X	X	X			X	X	X	X	X	X		X		X						
HBNJ		X	X	X			X		X	X	X	X	X		X		X	X	X	X	X	X
HINK	X	X	X	X	X				X	X	X	X	X	X	X	X	X	X	X	X	X	X
HJAN	X	X	X	X	X		X		X	X	X	X	X	X	X	X	X	X	X	X	X	X
LCNM										X				X		X						
LEAF									X	X												
LINX							X		X	X		X			X							
LOWS		X	X	X		X			X	X	X	X		X		X	X	X	X	X	X	X
MACH							X		X	X			X									
MILL		X	X	X		X			X	X	X	X		X		X	X	X	X	X	X	X
MTBE		X	X	X					X	X	X	X		X		X						
NOCA									X	X		X	X		X		X	X	X	X	X	X
OLYF						X	X		X			X	X		X							
PMAS																						
RAIN					X	X			X	X		X	X		X				X	X	X	X
REDS		X	X	X					X	X	X	X	X	X	X	X			$X^{F/U}$	$X^{F/U}$		
RIVH										X				X		X	X	X				
SCWM														X								
TRAJ		X	X	X			X		X	X		X	X		X		X	X	X	X	X	X
X = requested analysis; / = optional procedure for the project																						

Table 5.4 CCAL Water Analysis Laboratory: Analysis Tracking Sheet

Project	Sample Series	Arrival Date	Login Date	Collect Date	Analysis/Determination															
					Filter	pH	Alk	Cond	SSED	TDS	DOC	NH3	NO3	SiO2	PO4	TDN	UTN	TDP	UTP	Anions	Cations

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 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 requested.

To estimate accuracy, CCAL participates in the United States Geological Survey (USGS) Standard Reference Surface Water test program for analysis of test samples for nutrient and chemical constituents of natural waters. See a summary 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 run semiannually to monitor laboratory wash procedures.

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.

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 Calculation of Data Quality Indicators

Measurement Data Quality Objectives presented in Table 8.1 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 8.1 Measurement Data Quality Objectives

Determination of DQOs still in progress; estimated values only

Analyte	Method Detection Limit	Precision and Accuracy	Transition Value*
Alkalinity	0.2 mg $CaCO_3$/L	± 0.02 mg/L or ± 2 %	1 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, Dissolved Organic	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 uS/cm or ± 2 %	50 uS/cm
Dissolved Solids	5 mg/L	± 5 mg/L or ± 10 %	50 mg/L
Iron	0.06 mg/L	± 0.03 mg/L or ± 5 %	0.6 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
Silicate	0.20 mg SiO2/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
Sulfate	0.02 mg SO4/L	± 0.02 mg/L or ± 5 %	0.4 mg/L

** The value above which precision and bias 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, analytical results, quality control results and calibration statistics are entered and tracked through a database in Microsoft Visual FoxPro. 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 and Notepad formats. 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 Forestry Sciences Laboratory. 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.

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 Reguations. Protection of Envornment. 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 12.1 CCAL Standard Operating Procedures Revision History

	Standard Operating Procedure	CCAL Method Number	Date of Version
Basic Determinations:			
Revision for new instrumention (see SOP):	Determination of pH and Alkalinity	CCAL 10B.0	May 2008
	Determination of Specific Conductance	CCAL 11A.0	March 2006
	Determination of Suspended Sediments	CCAL 12A.0	April 2006
	Determination of Total Dissolved Solids	CCAL 13A.0	March 2006
	Determination of True Color	CCAL 14A.0	April 2006
Carbon Analysis:			
	Analysis of Dissolved and Total Organic Carbon	CCAL 20A.0	June 2006

	Standard Operating Procedure	CCAL Method Number	Date of Version
Automated Analysis, Colorimetric:			
	Analysis of Ammonia in Fresh Waters	CCAL 30A.0	March 2006
	Analysis of Nitrate/Nitrite in Fresh Waters	CCAL 31A.0	March 2006
	Analysis of Silicate in Fresh Waters	CCAL 32A.0	March 2006
	Digestion and Analysis of Fresh Water Samples for Total Nitrogen and Total Dissolved Nitrogen	CCAL 33A.0	March 2006
Spectrophotometric Analysis:			
	Analysis of Orthophosphrus in Fresh Waters	CCAL 40A.0	April 2006
	Digestion and Analysis of Fresh Water Samples for Total Phosphorus and Total Dissolved Phosphorus	CCAL 41A.0	April 2006
Ion Chromatography:			
	Analysis of Chloride and Sulfate in Fresh Waters by Ion Chromatography	CCAL 50A.0	April 2006
revision to add bromide and other minor changes (see SOP):	Analysis of Chloride, Bromide and Sulfate in Fresh Waters by Ion Chromatography	CCAL 50B.0	May 2008
Atomic Absorption:			
	Analysis of Cations in Fresh Waters by Flame Atomic Absorption Spectrometry	CCAL 60A.0	May 2008

Appendix A: CCAL Recommended Sample Collection Protocol

Sample collection requirements vary greatly across programs. Specific project protocols will 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 will be 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 filtered or unfiltered sample in an acid washed polyethylene bottle; prerinse 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 Receipt Tracking

Project	Investigator	Lab # Range	Arrival Date	Initial	Sample Counts						Comments	Date Logged In	Co-Fro-Bo?
					Fresh		Frozen		Acidified				
					# filt	# unf	# filt	# unf	# filt	# unf			

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 758-8764
Cam Jones 750-7336

		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 #:	
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 08; M186			Fall 07; M184			Spring 07; M182		
Major	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	Alkalinity	48.74*	50.4	2	41.66 mg/L	43.5	2	34.14	34.9	4
	Bromide	1.39	1.42	4	0.93 mg/L	0.928	4	0.36	0.43	1
	Calcium	11.90	11.9	4	13.16 mg/L	13.3	4	11.09	11.9	1
	Chloride	23.40	23.6	4	7.63 mg/L	7.8	4	21.4	21.3	4
	Magnesium	7.05	6.27	0	4.76 mg/L	4.37	0	5.56	4.77	0
	pH	8.7	9.10	2	7.9	7.77	3	8.2	8.68	2
	Potassium	6.44	6.62	3	2.90 mg/L	2.84	4	4.75	4.7	4
	TDS (DSOL)	119	128	3	103 mg/L	100	4	96	96	4
	Silica	11.20	11.3	4	7.84 mg/L	7.6	3	7.83	7.49	3
	Sodium	17.83	17.8	4	12.17 mg/L	12.3	4	9.4	9.87	2
	Conductance	217.4	220	4	169.9 µS/cm	171	4	167.1	171	2
	Sulfate	14.62	14.6	4	19.83 mg/L	19.9	4	10.22	10.3	4
	TP as P	0.084	0.080	4	0.895 mg/L	0.91	4	0.051	0.048	4
				3.23	Average Rating		3.38			2.69
		Spring 08; P-50			Fall 07; P-49			Spring 07; P-48		
Precipitation	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	Calcium	0.91	0.898	4	1.99 mg/L	1.92	4	0.58	0.564	4
	Chloride	1.60	1.58	4	1.17 mg/L	1.12	3	2.65	2.56	3
	Magnesium	0.26	0.232	1	0.11 mg/L	0.1	2	0.07	0.05	1
	OP as P	0.048	0.048	4	0.034 mg/L	0.038	2	0.029	0.031	4
	pH	6.3	6.32	4	6.9	6.87	4	5.3	5.24	4
	Potassium	0.30	0.301	4	0.14 mg/L	0.13	3	0.36	0.34	3
	Sodium	0.60	0.610	4	0.55 mg/L	0.54	4	1	1.03	4
	Conductance	13.5	14.4	3	14.9 µS/cm	15.9	3	13.6	14.8	3
	Sulfate	0.84	0.844	4	0.60 mg/L	0.6	4	0.27	0.29	4
				3.56	Average Rating		3.22			3.33
		Spring 08; N-97			Fall 07; N-95			Spring 07; N-93		
Nutrient (low)	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	TKN	0.39	0.390	4	0.21 mg/L	0.2	4	0.17	0.18	4
	NH3-N	0.223	0.230	4	0.186 mg/L	0.175	3	0.136	0.132	4
	NO3-N+NO2-N	0.355	0.360	4	0.089 mg/L	0.09	4	0.132	0.136	3
	OP as P	0.213	0.225	2	0.220 mg/L	0.227	3	0.184	0.16	3
	TN as N	0.74	0.740	4	0.30 mg/L	0.302	4	0.3	0.309	4
	TP as P	0.409	0.410	4	0.228 mg/L	0.231	4	0.194	0.191	4
				3.67	Average Rating		3.67			3.67
		Spring 08; N-98			Fall 07; N-96			Spring 07; N-94		
Nutrient (high)	**Analyte**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**	**RV**	**MPV**	**Rating**
	TKN	0.80	0.675	3	0.87 mg/L	0.7	2	1.01	0.715	0
	NH3-N	0.650	0.645	4	0.689 mg/L	0.649	3	0.662	0.66	4
	NO3-N+NO2-N	1.065	1.04	3	1.101 mg/L	1.16	2	0.958	0.954	4
	OP as P	0.593	0.594	4	0.959 mg/L	0.96	4	0.75	0.74	4
	TN as N	1.86	1.71	2	1.97 mg/L	1.91	3	1.76	1.66	0
	TP as P	0.591	0.601	4	0.948 mg/L	0.975	3	0.759	0.75	4
				3.33	Average Rating		2.83			2.67
				3.45	Overall Average		3.28			3.09

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

RV = Reported Value; MPV = Most Probable Value

OP = Orthophosphorus; TDS (DSOL) = Total Dissolved Solids; TKN = Total Kjeldahl Nitrogen; TN = Total Nitrogen; TP = Total Phosphorus

RED = reporting error

*with new titrator

Appendix F: Lab Aide Manual

Responsibilities of Lab Aide

Daily Routine

- Check deionized water gauge in room 341.
- Survey all laboratories.
- 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 labs:
    - Put away clean, dry glassware.
    - Clean dirty glassware.
    - Housekeeping (paper towel and Kimwipe supplies, repaper trays, drawers and hoods, dusting).
- *Friday Only* Complete the task list for Friday stuff, which is found in the lab aid drawer in room 341.

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 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. It is a good habit.
- Do not use tap water on glassware.
- 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 Room/Dish Room (room 331)
   - Contains two dishwashers—both can be used for phosphorous flasks and plastic sample bottles.
   - Check all bottles and lids for dirt, water, or spots before capping.
   - Metal drying rack shelves—shelves, covered with rubber matting, used for drying all clean bottles and lids.

2. Atomic Absorption Lab (room 333)
   - Storing atomic absorption tubes.
   - Atomic absorption tubes cleaning and drying.
   - Carbon vials/caps cleaning and storage

3. Auto-Analyzer Lab (room 337)
   - Miscellaneous glassware.
   - Pipettes (cleaning, drying, storing).
     STORING—DO NOT TOUCH WITH FINGERS!

4. Water Processing Lab (room 341)
   - Check Deionized Water Gauge
   - Phosphorous beakers.
   - Phosphorous flasks.
   - TN Tubes.
   - TN caps
   - 150 ml pH beakers.
   - 100 ml pH beakers.
   - Dissolved solids beakers.
   - Filter watchglasses.
   - Miscellaneous glassware.
     - Filtering equipment.
     - Measuring equipment, i.e., pipettes, graduated cylinders etc.

    - Miscellaneous beakers, flasks, etc.
- HJA carboys cleaning.
- Washing and storing filters.
- Repaper drawers and trays.
- Clean microwave oven.
- Balance: weigh filter papers and dissolved solids beakers.
  DO NOT TOUCH BEAKERS WITH FINGERS

5. Oven Room (Growth Chamber)
   - Drying ovens (2).
   - Clean/loaded dissolved solids beakers.
   - Drying filters.

6. Acid Baths
   - HCl acid baths—change every 3 to 4 months or as needed.
   - Exception: Room 337 HCl acid bath. Change when supervisor specifies. IMPORTANT to fill this bath to the volume mark on the side of the tub.
   - Pipette acid bath (room 337)—change when supervisor specifies.

Acid Bath Preparation

- Equipment
  - Large 50 liter acid carboy.
  - Concentrated hydrochloric acid (HCl).
  - 500 ml graduated cylinder.
  - 4 liter beaker.
  - 10 liter carboy.

- Procedure
  - Mix 10 liters of acid bath at a time in the hood in room 337.
  - Place 8 liters of DIW into the 10 liter carboy.
  - USE GOGGLES, GLOVES, AND LAB COAT.
  - Measure 427 ml of concentrated HCl with graduated cylinder.
  - Add acid to the 8 liters of DIW in the 10 liter carboy.
  - Bring up to 10 liters with DIW.
  - Empty into 50 liter carboy in room 331.
  - Repeat as needed.

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7. Storage Room (room 381)
   - HJA carboy storage
   - Cooler storage room

8. General Duties
   - Replenishing supplies:

  - Paper towels—some are stockpiled in room 341. You must stock ALL labs.
  - Kimwipes and tape—kept in closet in room 337. Stock ALL labs.
  - Parafilm – Cut new parafilm squares from dispenser located on lab bench. Rooms 337 and 341.
  - 125 ml plastic sample bottles—room 337 and room 341.
  - Acid baths—all labs.
- Removing broken glass—all labs.
- Acid storage room (outside): storage and inventory of all acids. Remember to update the inventory sheet in room 337.
- Clean and recycle empty acid bottles.
- Reorganize/organize freezer and cold room.
- Clean coolers. Prepare coolers for shipping. Record shipping destination and contents on cooler tracking log sheet.
- Paper: drawers, trays, under hoods.
- Labels: Remove all ink labels from glassware with acetone. CAUTION: Do not remove 50 ml lines on phosphorous flasks or standard markings. Remove tape labels from glassware and bottles.
- Friday stuff; complete the task list for Friday stuff, which is found in the lab aid drawer in room 341.

Appendix G: Lab Aide Manual with Expanded Procedures

Laboratory Aide Expanded Procedures

1. Room 331: Bottle washing room

Bottle washing

Remove all tape labels. Empty the bottles down the drain, and put the lids into the 4L beaker in the sink. Rinse each bottle 2X with DIW, make sure to rinse the outside as well. Acid rinse the inside of each bottle by filling the bottle completely with acid bath, then transfer it to the next pre-rinsed bottle. **Transfer the acid up to 12 total bottles**. When the acid has been transferred to the final bottle, dump the acid into the beaker with the lids in it. After acid rinsing, rinse each bottle 4X with DIW then place the bottles in the dishwasher, mouth side down, and cover them with the metal racks. 125's should go on the top dishwasher shelf only, 250's may go on the bottom shelf, but only if they are supported by a shelf prong, and then held in place by a metal rack. It is important to use the metal racks to keep the bottles from being dislodged by the water pressure. 500's and 1L bottles should go on the bottom shelf. When the dishwasher is full, or you are out of bottles to put into it, close the dishwasher and press the "Rinse Only" button. The rinse cycle should last approximately 10 minutes. Once the rinse cycle is done, take the bottles out of the dishwasher and place them mouth side down on the rubber mat covered drying shelves.

Bottle Caps

While the dishwasher is running the rinse cycle, add fresh acid bath to the 4L beaker with the bottle caps until all of the caps are completely submerged. Thoroughly mix the caps around in the beaker using a gloved hand in order to dislodge any air pockets, and contact all of the surfaces of the caps with acid. Using the large plastic funnel, dump the acid down the sink while keeping the caps in the 4L beaker with the funnel. ***REMEMBER:*** *When dumping any acid in the sink, make sure to flush the sink with PLENTY of water from both the DIW tap and the regular tap, and pour the acid slowly so that it can dilute into the flush water. Allow the water to run for about a minute after the acid is gone to flush the acid through the drain, and then rinse down the entire interior of the sink with DIW to prevent corrosion*. Once the acid is dumped, fill the beaker with DIW, mix the caps around thoroughly, then dump the water using the funnel. Repeat the DIW rinse 4X, and shake out any extra water between rinses. After the final DIW rinse, place the caps interior surface side down on the rubber mat covered drying racks.

Finishing

When you are done with the dishwasher for the day, make sure all bottles have been removed from the dishwasher, close the door, and then press the button that says, "Drain Off". The bottles should take about 2 days to completely dry. Once the bottles and caps

are completely dry, check each bottle and cap for black specks, or drops of water. If there are black specks, the bottle must go through the wash procedure again. If there are water drops, then allow the bottle another day to dry. If the bottles are clean and dry, then cap the bottles and store them in their respective places.

New Bottles

New bottles need to be rinsed 4X with DIW, then acid soaked overnight. After the acid soaking, rinse the bottles 4X with DIW then soak them in DIW for two nights, changing the water after the first night. The bottles can then be treated as regular dirty sample bottles and put through the normal bottle washing procedure above.

2. Room 333: Atomic Absorption Lab

AA Tubes

Rinse tubes and styrofoam holder 4X with DIW, then soak tubes overnight in the acid bath to the right of the sink. Remove tubes from bath the following day and rinse 4X with DIW and place tubes in styrofoam holders. Dry upside down on paper covered racks. Dry tubes are stored in room 333 in the white plastic basin. Do not touch tubes on ends, only in the middle. Checked for cracked tubes, if any are found, dispose of them.

Carbon Vials

Day 1

Retrieve the two tubs with lids, and the plastic screen (it's the one held together with zip ties) from under the sink or in the fume hood in 333. Dump the contents of the carbon vials down the sink, put the lids in one tub, and arrange the vials vertically, with the mouths up, in the other (you can fit about 64 vials in a tub). Once the tub is full of vials, press the plastic screen over the top of the tub, and hold it in place while filling the tub with deionized water. This prevents the vials from floating up and falling over. When the vials are totally submerged in water, and all vials are full of water, place the screen over the top (one side fits better than the other, you should be able to push the "edges" of the screen over the edges of the tub so it snaps into place, however it is still necessary to hold the screen in place for dumping). Dump all the water out of the vials, and repeat the filling process until the vials have been rinsed four times. Submerge the vials overnight in the acid bath next to the sink in 333 and repeat the acid soaking procedure until all the dirty vials have been rinsed and immersed in acid bath. The caps are then rinsed thoroughly with DIW and immersed in the acid bath with the vials. Immerse all vials before immersing the caps. Stir the caps in the acid to ensure that all surfaces of every cap come into contact with the acid. *If there are more than 80 vials to wash at once, it is easier to fit them all in the acid bath if you arrange them all standing up, that way when the lower portion is filled with vials, you can place another layer on top of them.* Move the tubs and plastic screen to the sink, thoroughly rinse them with DIW and place them back under the sink or in the fume hood to dry.

Day 2
Take all caps out of the acid bath, and place them in one of the tubs used the previous day. Rinse the caps four times using the screen, then place them in the one-gallon jar located under the sink in 333 and fill it with DIW; leave the caps to soak overnight. Pull the vials out of the acid, line them up in the tubs and rinse with DIW four times using the screen as above. Fill the tub completely with DIW, with the vials still in it, and cover with the lid; let soak overnight. *If there are more than about 130 vials they will not all fit in the two tubs, sink the remaining vials in the one-gallon jar with the lids.* Thoroughly rinse the screen with DIW and put it away.

Day 3
Dump out the water in the tubs/vials, and rinse them four times with DIW using the screen, then place them in the drying rack in the fume hood in 333. Approximately 145 vials will fit in the drying rack. Transfer the caps from the jar into one of the empty tubs, rinse thoroughly with DIW using the screen, and shake the excess water from them. Transfer caps into the basket in the fume hood (the basket with holes in all four sides). Cover the top of the basket with plastic-wrap, DO NOT cover the holes in the sides, so the caps can air dry. Thoroughly rinse tubs, plastic screen and jar with DIW and put them back under the sink or in the fume hood to dry.

Day 4
If the vials are dry, place them in aluminum trays located in the wall cabinet in 333. Arrange vials so they are lying down, do not stack higher than two layers (about level with the edges of the tray). Take vials to the blast furnace in Sherri's Lab (room 353) and bake for at least three hours at 550ºC. **Make sure to turn the furnace off before leaving for the day.** Leave vials in the furnace overnight to cool.

Day 5
Pull vials from the furnace, transfer them to room 375 and place in the large plastic tubs located on the East wall metal racks (the tubs should be labeled "Washed and Blast Furnaced"). If the caps are dry, place them in the tub with the other clean caps. The cap tub is located on the same shelf as the clean vial tubs.

3. Room 337: Auto-Analyzer Lab

Miscellaneous glassware
Generally, all glassware should be rinsed 4X with DIW and placed upside down on a tray or drying rack to dry.

Pipettes
Rinse 4X with DIW and place in acid bath in plastic cylinder next to sink. After soaking over night, remove and rinse 4X with DIW. Do not touch pipettes by the delivery tip. Place in rack next to sink to dry. Store in appropriate drawer when completely dry.

4. Room 341: Water Processing Lab

Deionized water gauge
Check first thing upon arrival. Located on the wall above the sink in room 341. Turn the deionized water tap on. Tank "A" DIW quality should be greater than10.0MΩ. If not, inform supervisor and go through the procedure for changing the tanks in the core.

Ortho tubes
Rinse all tubes, clean or dirty, 4X with DIW and place in acid bath for 2 hours. Remove and rinse 4X with DIW. Replace paper towels under wooden racks. Place tubes upside down in wooden rack located on counter. Be certain to change hand towels in racks for each use. Put standard tubes in the first four left side spots in the following order: blank, 0.05, 0.10, 0.20. Store on top of refrigerator when dry. Rotate older clean tubes to the front. Record date washed on ortho tube Tracking Sheet.

Phosphorous flasks and capping beakers
Rinse capping beakers (50/30 ml) 4X with DIW and place upside down on tray to dry. Place rack(s) of flasks in dishwasher. Cover with metal racks. Use the rinse only cycle. Remove and place flasks in acid bath in room 341 overnight. Remove flasks the next day, and rinse 4X with DIW. Dry flasks upside down on wooden drying rack next to the sink. Cap dry flasks with dry capping beakers. Match flask and beaker colors. Store capped flasks in the appropriate color coded drawer. Record date washed on Phosphorous Flask Tracking Sheet.

TN Tubes
Check fume hood in room 341 for total nitrogen sets that are done with analysis and ready to be sank in acid bath (they will be on the left side of the hood under the green tape). Collect the 4 liter plastic beaker and the plastic funnel from the wooden drying rack, and take to the fume hood sink along with the TN sets. Empty the contents of the tubes down the fume hood sink with the water running, place the caps in the 4 liter beaker and the tubes back in the racks. Using the funnel to aid you, rinse the caps 4X with DIW at the sink in the counter, next to the acid baths, and place them inner-surface side down on a papered tray to dry. Rinse the funnel and plastic beaker and place back on wooden drying rack. Now rinse the TN tubes 4X each with DIW, and sink them overnight in the acid bath labeled **HCL ACID BATH FOR TOTAL NITROGEN**. The next day, pull tubes from acid bath and rinse 4X with DIW and place them in numerical order upside down in their racks. Allow them to dry on the counter top next to the door. Once dry, cap the tubes and place them on the top shelf in the cabinet next to the refrigerator.

pH beakers
These 150 ml beakers are marked with colored tape on the beaker bottom. Rinse 4X with DIW and dry upside down on a clean papered tray. When completely dry store in pH beaker drawer, making sure to rotate older beakers to the front. Once a year (usually during supervisor's vacation), remove the tape and acid soak the beakers overnight; remark with colored tape when dry. 100 ml beakers are also used for pH, alkalinity and

conductivity. These are not marked with tape. They are stored in two drawers in the middle bench in room 341. 100 ml beakers are washed the same as the 150 ml pH beakers.

Dissolved solids beakers

Soak used beakers with DIW overnight. After soaking, thoroughly scrub the inside of the beaker with a brush. Rinse 4X with DIW and put beakers upside down on a papered tray to drip dry, and transfer to the dissolved solids oven in the growth chamber. DO NOT TOUCH BEAKERS WITH BARE HANDS AFTER WASHING. Use a Kimwipe or gloves (or tongs for hot beakers) to move beakers. Store beakers upright in oven until supervisor advises that there are samples ready to be analyzed.

Filter watchglasses

Remove sample numbers with acetone, and rub the area thoroughly with a paper towel. Do not remove the dividing lines. Remark dividing lines if necessary. Rinse thoroughly with DIW and allow to dry in black drying rack. Store in drawer marked watchglasses.

Filtering equipment

Equipment consists of a filter funnel, filter stage, funnel barrel, and wire screen. Rinse all pieces thoroughly with DIW. Be cautious not to break the tall filter funnels. Acid wash if pieces look dirty when dry. Scrub carefully with a brush if necessary.

HJA plastic carboys/buckets

Remove tape from carboys and buckets. Remark all faded tare weights. Rinse 2X with DIW. Acid rinse carboy and cap interiors. Rinse 4X with DIW. Clean outside of carboys/buckets with water as needed. Put clean rinsed carboys/buckets on special wooden drying cart (located in room 381).

Glass carboys and plastic sample beakers

Rinse 2X with DIW. Acid rinse carboy interior. Rinse 4X with DIW. Place upside down on metal racks on far west counter in room 341 to dry. Use paper towels to absorb excess water under the racks. If metal racks are in use or unavailable for all carboys, place carboys upside down on a clean paper towel. Acid rinse plastic beakers and rinse 4X with DIW. Place upside down on wooden drying rack next to sink until carboys and beakers are dry. Cap carboys with beakers (match numbers) and store on far west counter in room 341.

Filter papers

Washing

Use tweezers to handle filter papers at all times. Use caution so as not to put holes in the filter with the tweezers. Place 5 GF/F (large size) or 6-10 GF/C (small size) unwashed filter papers grid-side down in a Buchner funnel used specifically to wash filter papers. Turn on vacuum and pour one liter DIW through filters. Disconnect vacuum and carefully place washed filters on foiled cardboard trays. To prevent filters sticking together, remove filters individually

from buchner and overlap slightly on foiled tray. All filters will need to have the wash date and preparer's initials recorded on the original filter box.

Drying
Place foiled tray with washed filters in the blue Matheson oven in basement for a minimum of 5 days. Watchglasses with loaded filters are also dried in this oven. Do not dry clean and loaded filters simultaneously.

Weighing
Some filters will need to be weighed prior to storage. Filters of this type must be placed directly into a desiccator from the oven. Allow to cool overnight. Filters are then assigned individual filter numbers. Filter numbers are sequential and will continue the numbering sequence from the filter paper tare log sheet. These numbers are marked softly in pencil directly on the edge of the top side of the filter. The top side of the filter is the non-grid side of the filter. Weigh each filter using the four place Mettler balance in room 341 and record its tare weight on the filter paper tare log sheet. Weighings must be QA checked by supervisor. After QA check, place weighed GF/F filters in sequential order into their original boxes, 25 per box. Record date weighed and weighers' initials on the filter box. Do the same for weighed GF/C filters, but place 100 filters per box. GF/F and GF/C weighed filters have separate filter paper tare log sheets.

Storing
Storage will depend on type of filter. GF/F filters for general usage will be stored in their original box. GF/C filters for general usage must be foil wrapped in packets of 25 – 30 filters. Record the number of filters per packet and the date filters were washed on the outside of the packet using a tape label. Store foiled packets in room 341.

Repaper drawers and trays
Kay-dry paper for covering carts, trays, and drawers is stored in the cupboard below the acid baths in room 341.

Cleaning microwave oven
Once a month, wipe down the interior of the microwave oven with a damp paper towel. Turn the power off to avoid electrical shock.

Using the Mettler Balance

Calibration
Dissolved solids breakers and filter papers are weighed on the Mettler balance in room 341. The Mettler balance is an extremely delicate instrument, and great care should be exercised at all times during its use. Remove the grey protective cover from the outside of the balance. Check to see that no weight was accidentally left on the balance. AT NO TIME SHOULD WEIGHT BE LEFT ON THE BALANCE IF NOT IN USE! Check the balance level by looking at the level bubble located on the top front of the balance. The level bubble should be

within the confines of the level circle. If it is not level, adjust by turning the level adjustment dials. There are three level adjustment dials located on the two front corners and at the rear middle of the balance. Open one of the sliding side doors and very carefully clean the balance pan with a soft brush (located in the beaker to the left of the balance). Be careful not to start the pan swaying as this will accelerate wear of the precision parts. Close the side door. Both weighing compartment doors must be closed to make accurate weighings. The on/off switch is located on the left side of the instrument. The switch has three settings: (1) When the knob is straight up and down, the balance is turned off. All additions of weight must be made with the balance switched off. (2) Turning the knob forward will activate the coarse weight mode—this is a less sensitive setting to check for the proper weight range. Use this mode when weighing something with an unknown weight. (3) Turning the knob to the rear will activate the fine weight mode—the mode that all final weighings must be made in. Do not use the fine weight mode without either knowing an approximate tare weight or ascertaining the correct weight range with the coarse mode. The light in the viewfinder will come on when the coarse or fine modes have been activated. Turn the balance to the fine mode to check the zero tare. Look through the viewfinder. When the main numbered scale stops moving, the fixed scale (numbered 0 – 10) "0" should line up with "00" from the main scale. If it does not line up, then the zero must be adjusted. The zero adjustment knob is located on the right side of the balance. Carefully move the zero knob in the appropriate direction (forwards or backwards) until the fixed "0" and the "00" from the main scale line up. Switch the balance off. Recheck the zero. If the balance is not zeroed, then repeat zeroing procedure. If the balance is still zeroed, proceed with weighings.

Weighings

Carefully place the object to be weighed on the balance pan. Never place chemicals directly on the balance plan, always use a weighing paper or weighing boat. Never place anything on the balance pan that will leave a residue. Only clean dry dissolved solids beakers or dry filter papers should be placed directly on the balance pan. In general, only clean dry surfaces should contact the balance pan. If the tare weight of the object to be weighed is known, then dial-in the appropriate weights using the weight dials on the front of the balance. Weight is added in 10 gram increments using the black dial, one gram increments using the blue dial, and 0.1 gram increments using the red dial. If the object weight is unknown, then the weight must be determined by successive approximation. Make a gross estimation of the object weight. A single filter paper will not weigh more than one gram while a dissolved solids beaker will weigh more than 50 grams. With the balance in the off position, add weight with the 10 gram increment dial. Using the coarse weight mode, check to see if the added weight is too much or too little. Look through the viewfinder after activating the coarse weight mode. If the field is dark, then there is too little weight. If the moving scale drops and does not bounce, then there is too much weight. Switch the balance off and adjust the weights accordingly. THE BALANCE MUST BE IN

THE OFF POSITION TO ADD OR REMOVE WEIGHT. Recheck the weight using the coarse mode after adjusting the weight. The ultimate goal is to determine the weight increments that bracket the true weight. Adding 10 grams should cause the viewfinder field to indicate too much weight, while removing the same 10 gram increment will cause the viewfinder field to indicate too little weight. Leaving the established 10 gram weight multiple in place, repeat the process for the one gram range and then the 0.1 gram range. Upon determining the proper weight range to 0.1 gram switch the balance to the fine weight mode. The movable scale should fall within the "00" to "100" range. To determine the final recorded weight, read from left to right. The weight of 76.8397 grams will be configured as follows: The 10 gram increment will be set to 70, the 1 gram increment will be set to 6, the 0.1 gram increment will be set to 0.8, and the "0" line of the fixed scale in the viewfinder will fall between the 39 and 40 on the movable scale. Read the final value (0.1 mg) by observing which line of the fixed scale lines up evenly with any of the lines on the movable scale. In this example, the 7 line of the fixed scale would line up with a line on the movable scale. The final weight should be stable with no trend downwards (losing weight) or upwards (gaining weight). Recheck the zero after every five weighings. If the zero has changed by more than 0.2 mg, then the balance should be rezeroed and the previously weighed items should be reweighed. RECHECK THE ZERO OFTEN, AND ALWAYS END A WEIGHING SESSION BY CHECKING THE ZERO. This checks the accuracy of the previous weighings and also assures that no weight will be left on the balance. Recover the balance when finished weighing. Be certain that the balance is switched off.

Weighing beakers

Clean dissolved solids beakers are placed in the dissolved solids oven in basement for a minimum of 5 days to drive-off all moisture. Loaded beakers, beakers with sample added, are dried in the same oven for a minimum of 5 days before reweighing. The day before beakers are to be weighed, remove the beakers from the oven and place in a desiccator. Two desiccators used to cool and stabilize objects for all precise weighings are located on a cart in room 341. Place ten beakers in each of the two desiccators. Use tongs to handle hot beakers. Never handle clean or loaded dissolved solids beakers with bare hands. Handling dissolved solids beakers with bare hands can add weight to the beakers in the form of natural skin oils. These oils can be driven-off during the sample evaporation phase of the analysis causing incorrect beaker weights. Allow the beakers to cool and stabilize to ambient conditions overnight. Zero the balance. Using a clean Kimwipe, carefully remove a dry beaker from the desiccator and place it upon the balance pan. Select the proper tare weight from the beaker tare weight sheet and dial in the weight. Check the tare weight by activating the coarse weight mode. If the tare weight is not correct, then recheck the tare weight on the tare weight sheet and adjust if necessary. Recheck the tare weight using the coarse weight mode. NEVER USE THE FINE WEIGHT MODE WITHOUT FIRST CHECKING THE BEAKER WEIGHT WITH THE COARSE WEIGHT MODE. If the tare weight still does not give an appropriate response, then

determine the tare weight by successive approximation (see section on using the Mettler balance). Beaker tare weights can change due to chipping or by deposition of hard to remove residue from the previous analyses. If the tare weight has changed, record the new tare weight on the beaker tare weight sheet. Loaded beakers will be heavier than the tare weights. If a loaded beaker is heavier than the weight range of its tare weight, add weight in 0.1 mg increments until the proper weight range is achieved. If a loaded beaker is significantly lighter than its tare weight, then something has occurred (chipping) to remove weight from the beaker. This will invalidate the analysis and the sample will have to be repeated. Check with the lab supervisor if a loaded beaker weight is lighter than its tare weight. When the proper tare weight/loaded weight range has been established, turn the balance to the fine weight mode. Allow the balance to stabilize. Record the weight to 0.1 milligram on the dissolved solids weighing sheet in the 1st weighing column. Recheck the recorded weight with the reading on the balance to be certain it is correct. Turn the balance off. Leave the beaker on the balance pan. After 3 minutes, reweigh the beaker and record the weight on the dissolved solids weighing sheet in the 2nd weighing column. If the successive weighings match to within 0.1 mg, then the last recorded weight is the correct weight. If the successive weighings do not match to within + / - 0.1 mg, continue to weigh the beaker until successive weighings match. Loaded beakers may start to gain weight. If they do, then the last weighing before starting to gain weight is the correct weight. Remove the beaker using a Kimwipe and return it to the desiccator. Repeat for all beakers. Rezero the balance after every other beaker. Beakers are usually weighed in sets of 20 (ten beakers in each desiccator), each set being ten beakers. Have the first set QA checked by the lab supervisor before beginning the second set. QA weights must agree to within + / - 0.5 mg. If QA weights do not agree, then beakers must be redried and reweighed. After each set has been weighed and then checked by the lab supervisor, transcribe the correct final weight to the appropriate column on the dissolved solids data sheet (tare or tare + sample). BE CERTAIN THAT THE CORRECT WEIGHT IS RECORDED FOR THE CORRECT BEAKER NUMBER. Keep the weighing sheet(s) together with the data sheet and turn in all sheets to the lab supervisor when complete. The lab supervisor will transcribe QA weights after all weighing and data sheets have been turned in.

<u>When weighing filters, don't touch with fingers</u>! Use tweezers.
<u>When weighing beakers, don't touch with fingers</u>! Use Kimwipes.

<u>Dessicant baking procedure</u>

The desiccant pans are located on the bottom shelf of the desiccator cart. Use one pan for the desiccant in each desiccator. Pour the desiccant into the pans and take to Sherri's lab, Room 353. Check the blast furnace log to make sure the oven is not in use, and log in the appropriate information. Set the furnace to 230ºC, and load the pans one on each shelf. While the desiccant is baking, wash out the dessicators, and remove the old high vacuum grease using acetone. When the desiccators are dry, (they may need to be dried with a paper towel) apply new high vacuum grease to the seal and make sure it seals completely

around the rim. **Use only enough grease to ensure a good seal. Excess grease will ooze down the inside of the desiccator and create a potential contamination problem.** The desiccant should bake for 2 hours, be sure to allow warm up and cool down time. When removing the pans they will be very malleable, use the small glove for dexterity and the large one as support for the bottom of the pan. The gloves are located on the bottom of the desiccator cart. Pour the desiccant back into the desiccators carefully, ensuring none of the desiccant touches the grease seal. Wipe off any powder stuck to the inside walls of the desiccators. Date and initial the tape on each desiccator.

5. Growth Chamber: Oven drying room

Cleaning drying ovens
Once a month, wipe down the interior of a drying oven with a damp paper towel. Turn the power off to avoid electrical shock. Be careful not to cause burns by touching the hot oven surfaces. Clean a different oven each month.

Dissolved solids beakers
See sections 5 for specific procedures.

Drying filter papers
See section 5 for specific procedures.

6. Acid baths (all labs)

Acid bath preparation
Prepare 10 liters of acid at a time in the hood in room 337. Retrieve the 10 liter carboy from room 331. Obtain the following equipment and supplies: concentrated hydrochloric acid (HCl) (room 333), 500 ml graduated cylinder (room 337), 4 liter plastic beaker (room 331). It is REQUIRED to wear safety goggles, gloves, and a lab coat when working with concentrated acid. Place 8 liters of DIW into the 10 liter carboy. Cautiously measure 427 ml of concentrated HCl acid with the graduated cylinder. Avoid spilling or splashing concentrated acid. Clean up any spills or splashes IMMEDIATELY by neutralizing with baking soda solution. Add the acid to the 8 liters of DIW in the 10 liter carboy. Bring up to 10 liters with DIW. Using a cart, transport prepared acid bath to room 331. Cautiously pour into the large 50 liter carboy. Repeat as needed. When preparing the last 10 liter batch of acid bath, cautiously rinse the graduated cylinder 4X with DIW adding each rinse to the carboy. DO THIS CAUTIOUSLY SINCE WATER IS BEING ADDED TO ACID. Keep track of how much water is added from the rinsing. Bring up to 10 liters with DIW. Rinse all mixing and measuring equipment thoroughly with DIW when preparation is complete.

7. Room 381: Storage Room

HJA carboy storage

All plastic HJA carboys should be placed upside down on wooden cart once washed, and placed in room 381. Carboys should be shaken everyday to remove excess water after washing in order to speed the drying process.

Cooler Storage
CCAL coolers should be washed out with DIW and placed in room 381 once dry.

8. General duties (all labs)

Paper towels
There are some paper towels stockpiled in room 341. If low on towels, leave a note on the door to room 341 asking the janitor to leave some.

Kimwipes and tape
Supplies of Kimwipes and tape are located in the supply closet in room 337. If low on supplies, inform supervisor to reorder. It is important not to run out of these supplies.

125 ml plastic sample bottles
See bottle washing section 1.

Acid baths
See acid bath section 7.

Removing broken glass
There is a container in room 333 to place broken glass. When this container is full, inform supervisor.

Acid storage and inventory
The acid storage room is located outside to the south of the loading dock. It is locked at all times. A key for the acid storage room can be obtained from the supervisor or the front desk. New shipments of acid must be inspected and logged into inventory before being stored. Inspect each bottle for breakage and/or unusual conditions. Assign inventory numbers to each bottle and record inventory numbers on the acid inventory sheet. Inventory numbers are sequential starting from the last bottle number. The acid inventory sheet is located in room 337 on the corkboard next the door. Mark inventory numbers directly on the bottle label with a coarse tipped Sharpie. Do not obscure label safety information. Return the inventoried acid bottles to their original packing box and transport to the acid storage room with a cart. Unload inventoried bottles onto storage shelves. Separate acid bottles by type of acid. Limit the amount of concentrated acid stored in the laboratories to one bottle of each acid. Hydrochloric acid and phosphoric acid are stored in room 333 under the hood. Sulfuric acid is stored in room 341 in the right-side hood. Transport individual bottles of acid using an acid carrier. Acid carriers are stored in room 337. DO NOT USE THE STAIRS WHEN TRANSPORTING ACID, USE THE ELEVATOR. WEAR LAB COAT AND SAFETY GOOGLES WHEN TRANSPORTING ACID. Remember to mark off any acid brought up on the inventory sheet in room 337.

Cleaning empty acid bottles

Empty acid bottles must be rinsed out 2X with DIW or cold tap water. Do this in the fume hood as fuming will occur. DO THIS CAUTIOUSLY SINCE WATER IS BEING ADDED TO ACID. Rinse bottles an additional 2X with DIW/tap water and then soak with DIW/tap water for at least 7 days. Clearly mark soaking bottles to indicate they are filled with water and not acid. Water soaking acid bottles should be stored in room 337 underneath the sink. After soaking, empty the bottles down the drain. Cleaned acid bottles are recycled in room 199.

Reorganize/organize freezer and cold room

Check the bottle dump sheet located in room 338 on a weekly basis. Periodically, the lab supervisor will give specific instructions for these tasks.

Clean coolers

All sample coolers must be cleaned as soon as possible. Remove old/worn mailing labels. Rinse out the coolers with DIW. Remove with a paper towel any dirt not removed by the rinsing procedure and re-rinse with DIW. Leave cooler open and inverted to speed the drying process. CCAL coolers are stored in room 381 when dry. Clean and dry coolers belonging to investigators but not slated for mailing are stored in room 381. CCAL coolers that are mailed must be tracked on the cooler tracking chart. Record cooler ID and destination. Record date they are returned. Clean coolers that belong to investigators should be returned to the investigator as quickly as possible. Sample bottles may need to be included. Generally, coolers that are mailed will need to be taped shut with glass fibre strapping tape. Coolers are to be shipped FedEx; obtain shipping address, phone number, and FedEx account number from supervisor. Take to reception on first floor and give them the package along with its corresponding information. Make sure you get the printed tracking receipt and place in log book (room 341). Check with lab supervisor for specific instructions for each cooler.

Labels

Labels are made using label making program. Label information must be correctly formatted and color coded. Lab supervisor will inform of pertinent label information. Remove all ink labels from glassware with acetone. CAUTION: do not remove 50 ml lines on phosphorous flasks or standard markings. Remove tape labels from glassware and bottles. Clean label glue from bottles/glassware with acetone. ALWAYS WEAR GLOVES AND GOOGLES WHEN WORKING WITH ACETONE.

Appendix H: Lab Aide Weekly Task Sheet

Lab Aid Task List: WEEKLY

Activity	Room #	Date										
Paper Towels	333											
	337											
	341											
	Bottle R.											
Kimwipe Supply	333											
	337											
	341											
Fume Hood	337											
Drains	341											
Eye Wash	337											
	341											
Floor Drains	337											
	341											
Clean/Flush	333											
Sink Drains	337											
	341											
	Bottle R.											
Acid Baths	333											
(as needed)	341											
	Bottle R.											
Acetone Wash	333											
Bottles	337											
	341											
	Bottle R.											
Acid Wash	333											
Bottles	337											
	341											
Ethanol Bottle	337											
Data Sheets	337											
	341											
Soda Wash	333											
Bottles	337											
	341											
	Bottle R.											
Parafilm	337											
	341											
K-Dry	341											
Sweep	333											
	337											
	341											
Recycleables	333											
	337											
	341											

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
Dust Labs	333												
	337												
	341												
	Bottle R.												
Check Acid Inventory	HCl												
	H2SO4												
	HNO3												
	H3PO4												
Wash Drying Racks/ Replace Paper Towels	333												
	337												
	341												
Wash Pegs & Pegboards	337												
	341												
Wash Microwave	341												
Clean Fume Hoods	333												
	337												
	341												
Fire Extinguishers	333												
	337												
	341												