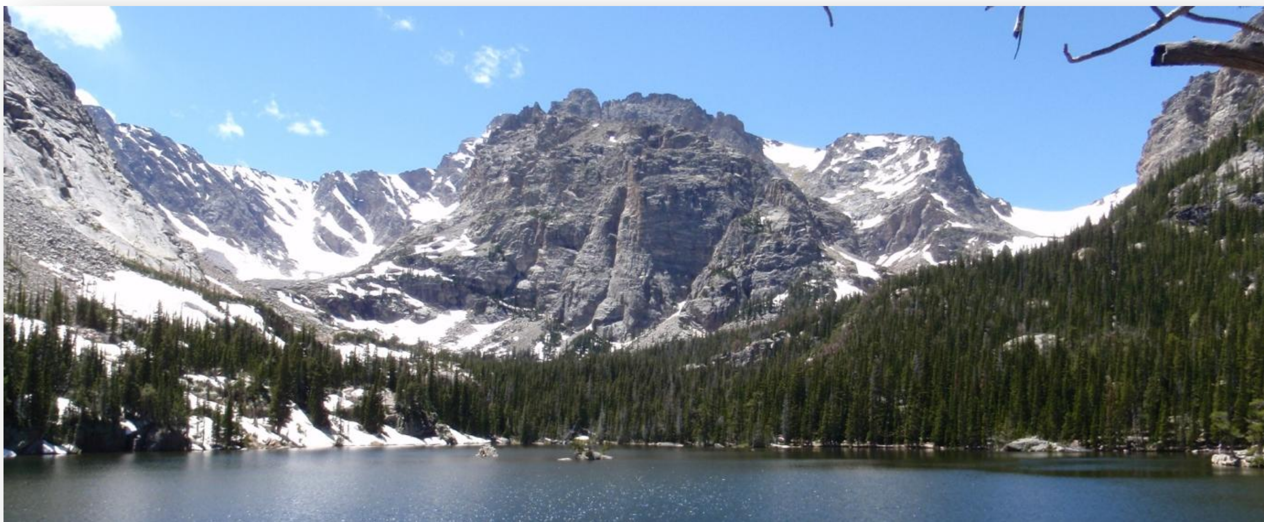
Loch Vale Watershed Long-term Research and Monitoring Program:

Methods Manual 2011



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Forward

This manual is intended to serve as a guide for technicians, students, and other participants in the Loch Vale Watershed (LVWS) long-term ecological research and monitoring program in Rocky Mountain National Park, and others like it. Procedures are described for sample collection, analyses, quality assurance, and database management.

This is a living document, so we welcome suggestions and questions. This document was most recently revised on July 28, 2011.

Please forward comments to Jill Baron (970-491-1968) or Eric Richer (970-491-2153).

Introduction

The National Park Service (NPS) initiated the LVWS monitoring program in 1983 with funding from the Aquatic Effects Research Program of the National Acid Precipitation Assessment Program. Initial research objectives were to understand natural variability and the processes that mitigate or accelerate the effects of atmospherically deposited pollution on soil and surface water chemistry, and to build a record in which long-term trends could be identified. The project is currently a cooperative effort between the NPS, U.S. Geological Survey (USGS), and Colorado State University (CSU).

Routine sampling of precipitation chemistry, surface water chemistry, hydrology, and meteorological conditions and began in 1983. Since then, all samples and data have been analyzed according to accepted and published methods (e.g., Clesceri et al. 1998; U.S. Environmental Protection Agency 1987).

Collection and analyses methods for all data have been rigorously tested. Quality assurance methods and results are described in reports produced by LVWS personnel (Denning 1988, Edwards 1991, Allstott 1995, Allstott et al. 1999, Botte and Baron 2004, and Richer and Baron 2011).

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FIELD PROCEDURES

General Introduction

Weekly field procedures in LVWS include: 1) collection of precipitation for chemical analysis, 2) measurement of precipitation amount, and 3) sampling of surface waters for chemical analysis.

The National Atmospheric Deposition Program (NADP) monitoring site, CO98, was established within the LVWS in 1983. NADP is a national network of precipitation monitoring sites. The network is a cooperative effort between many different groups, including the State Agricultural Experiment Stations, USGS, U.S. Department of Agriculture, and other governmental and private entities. The objective of the network is to provide data on the amounts, trends, and geographic distributions of acids, nutrients, and base cations in precipitation. Samples from each station are collected weekly using clean-handling procedures, and sent to the Central Analytical Laboratory (CAL) in Champaign, Illinois for chemical analysis.

Aerochem Metrics samplers collect precipitation samples for analysis of water chemistry. Concentrations of solutes in precipitation water can be multiplied with precipitation volume to estimate chemical deposition. Methods for operating precipitation collectors and raingages are found in the NADP National Trends Network Site Operation Manual (Dosset and Bowersox 1999). An Alter-shielded Belfort raingage measured precipitation volume for CO98 from August 1983 to August 2010. NADP updated CO98 with an electronic NOAH IV raingage in June 2007. An additional, or co-located, NADP site was installed in October 2010 under the name CO89. CO89 consists of an Aerochem Metrics precipitation collector and NOAH IV electronic raingage. After recording three years of co-located data with the CO98 NOAH IV, the CO98 Belfort was removed from the Loch Vale NADP site on August 9, 2010.

A Parshall Flume with stilling well was installed at the Loch Outlet in 1983. Prior to August 2006, stage was measured with a float within the stilling well. Stream levels were recorded with a Campbell Scientific CR500 stream recorder, and a Leupold and Stevens strip chart recorder served as a backup for the Campbell. Stage data were then converted to discharge values (m^3s^{-1}). Operation of the Loch Outlet stream gage was transferred to the USGS Water Resources Division (WRD) in August 2006. Stage is currently monitored with a depth transducer and data logger.

Surface water samples are analyzed for chemical concentrations. Five sites are currently sampled within the LVWS, as well as five others within Rocky Mountain National Park (RMNP). Sampling locations are shown in Appendix A. Sampling frequency ranges from weekly (at the Loch Outlet) to annually (for Emerald, Haiyaha, Louise, and Husted lakes). A detailed sampling schedule is listed in Appendix B. As stream and lake waters in the

LVWS are chemically dilute, sampling protocols need to be followed carefully to avoid contamination.

Weather conditions were measured at a Remote Area Weather Station (RAWS) near the Loch Vale NADP site from 1983 to 1998. The RAWS was shut down on November 24, 1998. Details of the decommissioning process can be found in the LVWS Quality Assurance Report for 1995-1998 (Allstott et al. 1999). The Main Loch Vale Weather station was established in 1991 approximately 30 meters from the location of the RAWS, and is operated and maintained by the USGS-WRD.

Field Safety

LVWS is a remote basin that can be demanding of advanced fitness and survival skills. Never consider a trip to LVWS to be a simple day hike. Field days can be very long and stormy, even in the summer. Be prepared with appropriate gear to keep warm and dry during inclement weather. Gear should include, but are not limited to, a good waterproof and windproof shell, warm hat, two pairs of warm gloves, comfortable waterproof boots (suitable for long hikes), warm socks (plus an extra pair), light to medium weight long underwear, warm pants and sweater (fleece works well), headlamp or flashlight, knife, compass, map, emergency blanket, well-stocked food bag (bring more than you think you'll eat), water (drink at least 2 liters per day), and a well-stocked first aid kit (with the knowledge of how to use it).

Summertime hazards include lightning, hail, snowstorms, and hypothermia. Winter hazards include avalanches, blizzards, hypothermia, and frostbite. There is danger of serious injury (broken bones, cuts, concussions, etc...) in all seasons.

Ground squirrels in LVWS can be carriers of bubonic plague, so do not approach or feed them. Mountain lions have also been sighted in the area. If confronted by a lion, raise your hands high above your head and slowly back away. Fight back if attacked.

Make sure all group leaders have a working two-way radio. All group leaders should also be certified in first aid and CPR.

It is best to start the day with a clear plan of action. Know all your sampling points, routes between them, and how long you expect the work to take at each point. Reevaluate the plan, and your progress within the plan, frequently throughout the day. It is best to hike out while there is still light in the sky. Plan accordingly! Never go to the field alone, and never go out in a group without someone in town knowing where you plan on going and when you are expected to return. Once back in town, make sure you contact the person(s) awaiting your return.

The bottom line is to never endanger yourself or anyone else for a sample. In the best conditions, a park-led rescue will take a minimum of 5 hours. If the weather is bad, a rescue can take over a day (even during summer!). Stay safe.

Snow Stability Evaluation

Avalanche danger is a serious concern in LVWS. A good rule of thumb is that if a slope can be skied, it can also produce an avalanche. Standard routes to the most frequently visited sampling sites in LVWS involve relatively little avalanche danger. In spite of this, the trip leader needs to be constantly aware of present and changing snow conditions. When traveling to a non-standard sampling point during the winter, snow stability is your primary concern. If, after careful evaluation of snow stability, someone in the group is in doubt of the party's safety, TURN AROUND! The loss of a sample is inconsequential compared to injury or death.

Be sure to call the Colorado Avalanche Information Center (CAIC) hotline (970-498-5311) before traveling to the mountains, or check the CAIC website at: http://avalanche.state.co.us/index.php. Know what to expect before leaving town. Weather, terrain, and snow conditions all influence avalanche danger. Be wary of slopes with angles greater than 25 degrees after new snowfall (especially when accompanied with winds greater than 15 mph). Potential avalanche terrain based on slope alone is shown in Figure 1.

South facing aspects are generally more stable during the colder winter months, while north facing slopes are generally more stable in the spring and summer. Dry slab avalanches are the primary danger during winter months. The recipe for a dry slab avalanche is a more consolidated layer of snow on top of a less consolidated layer, when combined with slopes over 25 degrees and a trigger (i.e., YOU!) these avalanches can be fatal. Be aware of runout angles when you are below steep terrain. Stay far enough away from steep terrain above you to keep the top of the potential avalanche path at less than 20° from you to it. When traveling in avalanche terrain, all parties must carry an avalanche beacon, probe, and shovel. Your partner or group is responsible for saving your life should you become buried in an avalanche, and vice-versa.

There is no substitute for proper avalanche awareness training. There are many avalanche awareness courses offered throughout the fall and winter. Awareness of all major contributing factors is the first, and possibly best, means of staying out of harm's way. The following tests can be used as tools in your evaluation of how safe a zone is for travel, but they are not your only tools.

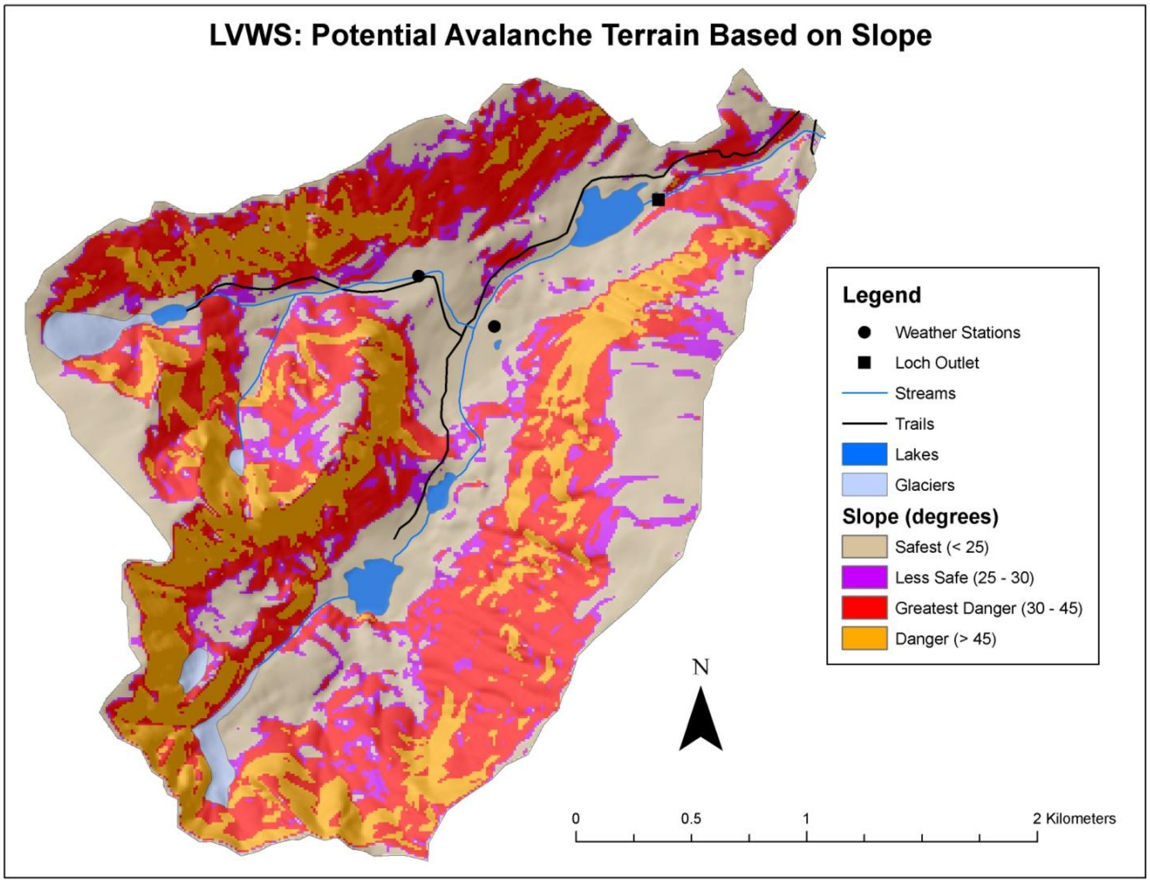

Figure 1. Potential avalanche terrain within the Loch Vale Watershed bases on slope alone. Note: Other factors besides slope contribute to avalanche risk!

The Hasty Pit:

The pit provides information to evaluate the questions: Are there slab layers? How large and extensive are the slabs? Approximately how well are the slabs bonded to other layers? What might it take to make a slab fail? In the pit, you will be able to identify snow layers, snow types, layer cohesiveness, and snow density. Pits can be used to identify buried surface hoar and faceted snow layers. Never dig a hasty pit where you might trigger a slide. The site should be safe and representative of the slopes you are really evaluating. Your pit site should have the same elevation, slope angle, and aspect as the slopes of interest. A hasty pit can be dug and analyzed in 15-20 minutes with practice. Make the pit about 3 feet wide. Dig to ground if practical but go at least 4 to 6 feet deep. The actual digging of your pit will give you information about the layering of the snow, so pay close attention. Make the uphill face vertical and smooth. Observe this face for layers by brushing the face with your glove. Check hardness of the layers by pressing into the snow. If you can easily push in your fist the snow is "Very soft"; four fingers is "Soft"; one finger is "Medium"; a pencil is “Hard”; a knife blade is "Very hard" snow. The hasty pit is not a complete stability evaluation. Informed decisions can only be reached by combining input from weather, terrain, snowpack, skier capabilities, and information provided by your hasty pits.

The Shovel Shear Test:

Isolate a column of snow about the width and depth of a shovel blade from your smooth face. Cut both sides and the back to isolate the column from the surrounding snowpack. This lets us evaluate the shear strength of the layers since that is all that is now holding them together. Insert the shovel 1-2 feet vertically behind the column. Do not lever the shovel! If the column fails without pressure this is a "very easy" shear. If failure occurs with light horizontal pressure, this is an "Easy" shear. These are indicators of unstable snow. The rating then goes to "Medium", "Hard", and "Very hard" as you exert progressively more force. The force you exert is relative to your own technique, therefore, so are these ratings. When a layer shears, examine the snow at the shear plane to identify the sliding layer and the failure source (e.g., buried surface hoar, ice lens, faceted snow). The shear test in a hasty pit will identify many weak layers. Examining crystals along these planes may help explain the source of instability.

The Compression Test:

- Dig a hasty pit
- Isolate a column of snow 30 cm wide and with a 30 cm upslope dimension that is deep enough to expose potential weak layers on the smooth walls of the column. A depth of 100-120 cm is usually sufficient as the compression test rarely produces fractures in the deeper weak layers.
- Completely isolate the column on all sides.
- If the snow surface slopes, remove a wedge of snow to level the top of the column.
- Place a shovel blade on top of the column.
    - Tap 10 times with fingertips, moving hand from wrist.
        - While tapping, if the upper part of the column slides off or no longer "evenly" supports further tapping on the column, remove the damage part of the column, level the new top of the column, and continue tapping.
    - Tap 10 times with fingertips or knuckles moving forearm from elbow.
    - Hit the blade 10 times with open hand or fist moving the arm from the shoulder.
- Record when the column fractures and note characteristics of snow layer below fracture.

Term	**Description**	**Reporting**	**Risk**
Very Easy	Fractures during cutting	CTV/Q	Red
Easy	Fractures within 10 light taps using finger tips only	CT1-10/Q	Red
Moderate	Fractures within 10 moderate taps from the elbow using fingertips	CT11-20/ Q	Yellow
Hard	Fractures within 10 firm taps from whole arm	CT21-30/	Green

	using palm or fist	Q	
No Fracture	Does not fracture	CTN	Green

Recording: "CT# Q# @↓##cm; test depth ###cm"
CT#: number of taps until column fractured
Q#: quality of shear
##: depth from the surface of the fracture
###: depth of the isolated column

Shear Quality:

Q1	Clean, planar, smooth, and fast shear surface. Slab typically slides easily into the snow pit; can break like its spring loaded.
Q2	Shear surface appears smooth, but slab does not slide as readily as Q1. Shear surface may have some irregularities, but not as irregular as Q3. The entire slab does not slide into the snow pit.
Q3	Shear surface is non-planar, uneven, irregular, and rough. Shear fracture typically does not occur through the whole slab/weak layer interface being tested. After the weak layer fractures, the slab moves little or may not move at all, even on slopes steeper than 35°.

Radio Protocol

Radios are your lifeline to the outside world in the event of an emergency. They also provide communication between separate groups in LVWS. In either case, you should use proper radio etiquette. It is very important to keep communications with ROMO dispatch brief and to the point, since the rest of ROMO staff also uses these frequencies.

Make sure radios have charged batteries and are in good physical condition before going to the field. Batteries need to be on the charger for at least four hours for a complete charge. An optimum charge can only be attained if the batteries are periodically drained of all charge. Do this by leaving power on overnight to the weather channel.

Before starting up the trail to LVWS, ensure your radios are functioning properly. To do this, turn on radio by turning volume knob clockwise. Then adjust squelch by turning squelch knob clockwise until static is heard. Turn squelch down (counterclockwise) until static is no longer audible. Turn channel selector knob to channel one. Listen to make sure there is no other radio traffic you would be interrupting. Once you are sure the channel is clear, key the transmit switch and say "ROMO, Researcher Baron - Radio check." Release key! They will answer "Researcher Baron - loud and clear." You then say, "Copy – Researcher Baron clear." If ROMO cannot hear you, there will obviously be no reply to your call. Repeat your initial call two more times (spaced by 30 seconds apiece). If no contact is made, say "No contact. VIP Researcher clear."

Channels one and two are the primary channels used by RMNP personnel within the park. Channel 11 is recommended for communication between LVWS staff while in the field. Keep your radio set on ROMO (i.e., channel one or two) while traveling as a group, that way you can assist if an incident occurs elsewhere. If you encounter an incident, do not hesitate to call for help. You should not have any trouble reaching ROMO from anywhere in the LVWS. You may have to move to higher ground to increase quality of transmission. If your group separates at some point, use channel 11 for communication.

RMNP will provide a volunteer to assist with winter-time sampling. This volunteer is generally responsible for all radio communication, but all LVWS staff traveling to the field should be familiar with radio operation and etiquette. When traveling with a Park Service volunteer, you must file a back-country travel plan at the Resource Management Office at park headquarters. When filing this plan, we must include call signs for all parties in the group. Park Service volunteers will use a call sign of "VIP" (e.g., VIP Messina). LVWS staff should use a call sign of "Researcher".

All frequencies programmed into radios (except the USGS line of sight channel) are not intended to be used unless necessary, and if they are used, proper radio etiquette must be followed. This consists of brief concise transmissions. A typical call would resemble the following:

Us:	"ROMO, Researcher Baron"
Them:	"Researcher Baron"
Us:	"I have a 28 year old male with possible femur fracture. Break."
Them:	"Go ahead"
Us:	"Patient is breathing and responsive, no external bleeding, vitals steady. Break."
Them:	They will lead conversation from this point.

First Aid Kit

Quantity	Item	Comments
2	Pairs of nitrile or latex gloves	Non latex gloves are best in order to prevent allergic reactions
4	Ziplock style freezer bags	Can be used for used dressings, bandages, miscellaneous garbage
4+	Safety pins	Keep several sizes
2	Topper sponges	Different sizes
6	Triangular bandages or Cravats	
2+	Surgical dressings (lg), sanitary napkins	Any dressing that can be used for absorbing large quantities of blood is acceptable.
8	4″x4″ sterile dressings (Telfa)	
Assorted	Band-Aids	Keep different styles on hand
	Moleskin	Useful for blisters
2	Roller bandages (3″ or 4″)	
1	Roller bandage (2″)	
1	Roll of adhesive tape	Athletic tape works well
2	SAM splits, wire ladder, or other splint	
2	Tongue depressors	
1	Accident release form	Used if aid is declined by patient
2	Butterfly closures	Useful for deep incisions
1-2	Packets of sugar, honey, or glucose	Used for diabetics
1	Pen light	
1	CPR mask with one way valve	
1	Suction device	e.g., a modified turkey baster
1	Traction application gear	Most people use a specially made tip and tail for a ski. If you don't have these, make sure you have something as effective (a nice new alternative is the Kendrick traction device)
1	Windlass for traction	6″ wood dowel
6′	Avalanche cord	For use in pulling traction
1	Trauma shears	
1 package	Antiseptic wipes	e.g., PAWS

Basic Equipment for your Backpack

Quantity	Item	Comments
1	Pliers	Leatherman type
1	Screwdriver	Combo Phillips & flat head
1	Knife	Pocket knife or Leatherman (which combines all 3 of the above)
1	Notepad & Pencil	
1	Plastic whistle	
1	Space blanket	
1	Razor knife	
2	40 gallon 2 ply trash bags	
	Glove liners	
	Sunscreen	
	Lip balm	
1	1/4x3″ bolt & nut	Ski repair equipment
1	Shovel	Suitable for digging through avalanche debris
1	Avalanche Beacon	Mandatory
1	Headlamp	

Precipitation Sampling

a. Introduction

The LVWS is part of the National Atmospheric Deposition Program (NADP) precipitation chemistry-monitoring network. Aerochem Metrics collectors capture precipitation samples that are collected weekly for chemical analysis at the Central Analytical Laboratory (CAL) in Champaign, IL. The Aerochem collector lid covers a clean plastic bucket when it is not raining. The lid opens when precipitation activates a sensor. The sensor is heated, so water evaporates when precipitation stops which closes the lid back over sample bucket. Sample buckets are collected for the LVWS co-located NADP sites (CO98 and CO89) every Tuesday morning. Detailed sampling and maintenance procedures are provided in the National Trends Network Site Operation Manual (Dosset and Bowersox 1999), located in NREL A223. You must be familiar with these procedures before operating a collector. Training in NADP procedures is offered yearly, and should be taken by all regular operators of the LVWS NADP site. The following instructions are given as a brief synopsis of appropriate methods.

b. Equipment and supplies to be taken to the field

- Two clean buckets and lids (leave in plastic bags)
- LVWS field book with pencil

c. Estimated time to complete procedure

Once on site, approximately 10 minutes are required to change the buckets and check the sensors. If there are problems that require trouble-shooting or repair, or if the weather is bad, the time spent at the NADP site can increase substantially.

d. Preparation

Buckets and lids for the Aerochem are shipped to and from the CAL in “6-packs”. Within each 6-pack, there should be six buckets, lids, 1-L sample bottles, and boxes for shipping sample bottles to CAL. Each 6-pack should also include a supply of Field Observer Report Forms (FORFs). Two to three 6-packs are stored in NREL A223. When six used buckets and lids have been accumulated, a used 6-pack of buckets and lids should be assembled and shipped to CAL. CAL will automatically ship a clean replacement 6-pack when they receive a used 6-pack of buckets and lids. When picking up mailers from the mailroom at NREL, be careful not to confuse LVWS boxes with those used at other sites.

e. Procedures

Carry two new buckets and lids (still in their plastic bags) to the LVWS NADP site each Tuesday. Once on site, check the foam lid seal to ensure good contact with the sample buckets. There should be no gaps or openings. Check the temperature of sensors by touching a finger to the sensor plate (not on the ribs suspended above the plate). It should be cool, but above freezing. Sprinkle a few drops of water, or snow, onto the ribs above sensor plate to open the wet-side bucket. If the lid does not open, consult the NADP Site Operation Manual (Dosset and Bowersox 1999) for troubleshooting procedures.

Peer into the wet-side bucket from the downwind side (i.e., northeast). Note any contamination in the field book. Using the plastic bag as a glove, seal the clean lid onto the sample bucket. Ensure the lid is firmly sealed on the bucket. Check the sensor heater again, it should now be warm. Remove the sealed wet-side sample bucket from the Aerochem. Install the clean bucket using the bag as a glove. Blow any remaining water off the sensor and away from wet-side bucket, or wait for the heater to evaporate remaining water on the sensor. Observe the lid seal as it covers the newly installed wet-side bucket. If the seal is not complete, airborne particulates could contaminate the sample. Notify the NADP Site Liaison (217-244-0372), if lid does not seal completely.

Place the sample bucket into the plastic bag from the clean lid or clean bucket. Label the plastic bag with the collector ID, either CO98 or CO89. DO NOT WRITE ON THE BUCKETS! Record the bucket change time and date in the field book, and note the bucket weight written on the side of each bucket by CAL. CAL bucket weights can be used to identify each bucket back in the lab and ensure precipitation samples for CO98 and CO89 do not get confused. Record any other relevant information (i.e., contamination, malfunction, etc...) in the field notebook. Load sample buckets into the bottom or onto the outside of your pack. It is best to have two people carry one bucket each.

f. Be Aware

The Aerochem collectors can malfunction in many ways, but the most consistent problem is power loss during prolonged snow storms. Each Loch Vale NADP site is independently powered by solar panels. Be sure to remove snow from the solar panels and trample down snow that accumulates in front of the solar panels. Wiring for the solar power system is detailed in Figure 2. The solar power systems are designed for three days of autonomy. Power to the collectors and raingages will automatically disconnect when battery banks drop below 40-50% capacity. In the summer, the solar panels, batteries, and wiring must be checked and repaired if needed. Also, check connections to the Aerochem motor box and tighten any loose screws and nuts, especially those related to the movement of the roof and lid seal.

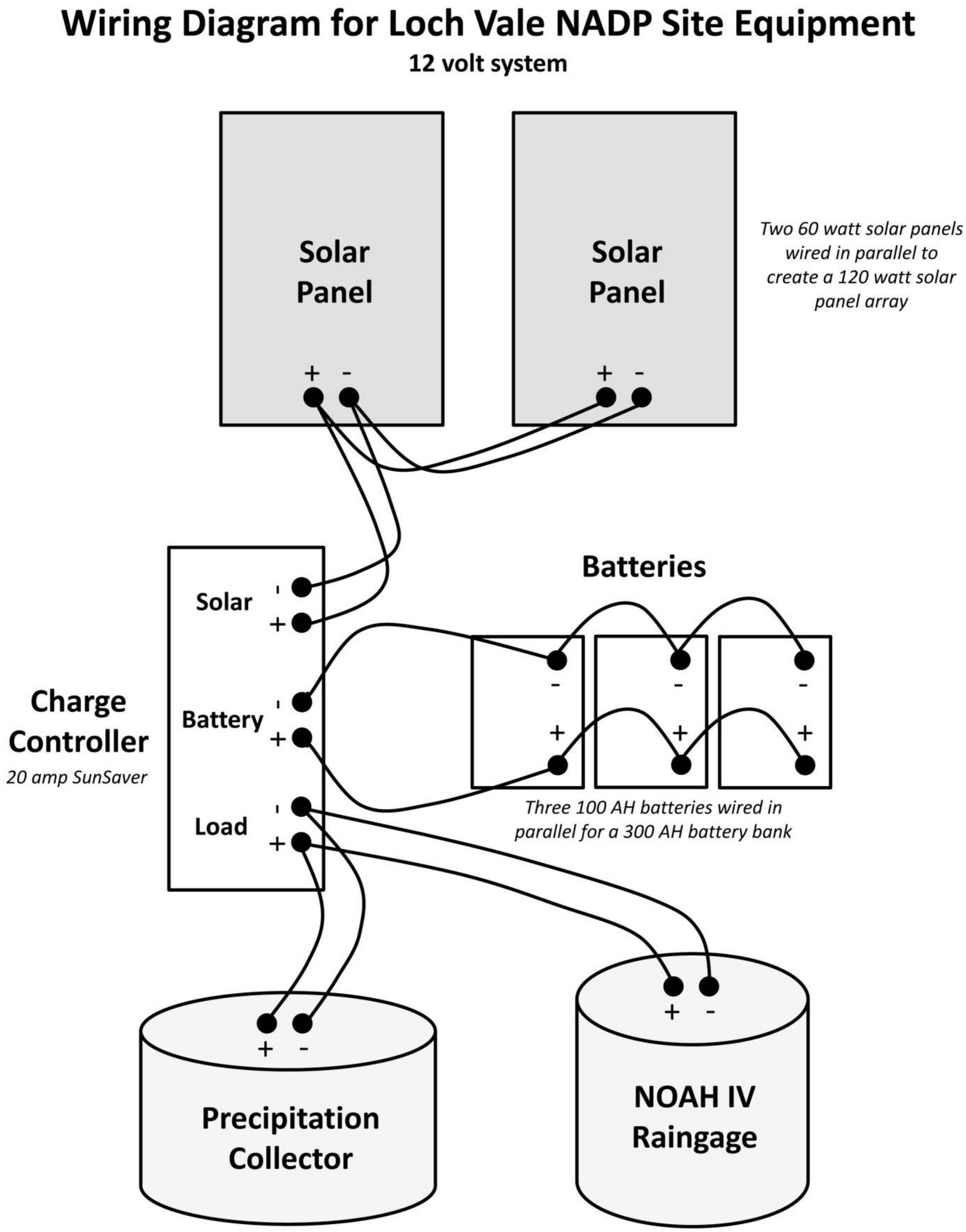

Figure 2. Wiring diagram for the Loch Vale NADP sites, CO98 and CO89, each site is wired independently.

The dry-side bucket is changed monthly and the foam lid seal is changed twice a year. DI rinsed dry-side buckets should be carried to the NADP the first Tuesday of every month. Replace the dry-side buckets before replacing wet-side buckets. The CAL will send new lid seals necessary. Follow installation instructions that come with the lid seal.

If you have concerns or problems with any NADP equipment, contact the NADP Site Liaison, Matt Layden, immediately.

The CAL contact for all questions and problems is:

Matt Layden, NADP Site Liaison
Telephone: (217) 244-0372
Email: ntn@sws.uiuc.edu

NOAH IV Raingage

a. Introduction

An Alter-shielded NOAH IV electronic raingage (egage) was installed at the CO98 NADP site in June 2007. The ETI NOAH IV Total Precipitation Gage provides accurate and unattended measurements of rain and snow precipitation over a full range of temperatures and environmental conditions. An additional egage was installed at the LVWS NADP site in October 2009 when the co-located CO89 NADP site was established. Site operators are required to download precipitation data from both NOAH IVs every Tuesday. Raingages are kept in a winterized state all year, requiring the catch buckets to be emptied periodically.

Detailed maintenance and repair procedures can be found in the NOAH IV Total Precipitation Gage Technical Manual. The manual is located in NREL A223.

b. Equipment and Supplies

- Personal Digital Assistant (PDA) and downloading instructions are kept in NREL A223 (be sure to fully charge the PDA prior to leaving for the field).
- Antifreeze and funnel are kept in the storage box at the NADP site.

c. Estimated time to complete procedure

It takes around five minutes to download data from each NOAH IV, an additional 20-30 minutes if either catch bucket is full. To prevent overflow, consider a bucket full when bucket depth reaches 10 inches.

d. Preparation

Charge the PDA the day before going in the field. Be sure there is an adequate supply of antifreeze at the NADP should you need to empty and winterize one of the egages. Make sure you have enough empty antifreeze or Nalgene containers to accommodate the volume of precipitation waste from the raingage.

e. Procedures

Download data from CO98 NOAH IV Precipitation Gage (a.k.a. NTN(1)):

1) Stand next to the CO98 NOAH IV raingage and turn on PDA
2) Tap the “Bluetooth” key to open the Bluetooth Manager
3) Double tap the CO98 icon to establish the connection
4) Close the Bluetooth Manager (tap on the “X” in the upper right corner)

5) Tap the “NADP Rain” key to start the NADP Rain program
6) Tap on the “Retrieve Data from Raingage” button
7) Tap on the “Connect” button to connect to CO98 raingage
8) Once “Connected” is displayed on the screen check the gage information
9) Tap on “Download” to move to Download page
10) Select start date and time (i.e., bucket on from previous week), tap on “Download”
11) Tap on “Data” to move to Data page
12) Select Sample Start (i.e., bucket on from previous week) and End dates (i.e., bucket off for current week), then set Network to NTN(1) and tap “View” to view daily precipitation totals. **RECORD THE DAILY PRECIPITATION TOTALS IN THE FIELD BOOK**
13) Tap “X” to close window
14) Navigate to Raingage page by tapping “Time” and then “Raingage”
15) At Raingage page, Tap “Refresh”. Note voltage and bucket depth.
16) Tap “Collectors” then “Refresh” and note status of both CO98 (NTN(1)) and CO89 (MDN(2)) collectors
17) Tap “Disconnect” to move to Disconnect page, then tap “Disconnect” and “Exit” and “Exit” again

Download data from CO89 NOAH IV Precipitation Gage (a.k.a. MDN(2) and NPX3):

Repeat steps 1-17 from above, but use CO89, MDN(2) and NPX3 for gage and collector references. Turn off PDA.

Transfer data files to NADP Archive folder and email to NADP:
1) Back in the office, connect the PDA to the computer
2) Navigate to the folder where the data files are stored on the PDA.
3) Copy new files to: \data\baron\NADP_Archive\year\site
   There should be two files for each NOAH IV.
   Example file names for CO98 (site name CO98):
   20091027T1543CO98.txt
   CO9820091027T1543.xml
   Example file names for CO89 (site name NPX3):
   20091027T1548NPX3.txt
   NPX320091027T1548.xml
   200991027T1548 = Year (2009) Date (1027) Time (T1548)
4) Send all four files to: nadp-precip@isws.illinois.edu
   Subject: CO98/CO89 NOAH IV Files
   Include local download time plus start (bucket on) and stop (bucket off) dates

f. Be Aware

The catch buckets in NOAH IV raingages can hold a maximum of 14 inches. Buckets should be emptied and winterized when bucket depth reaches 10 inches. You can check the current bucket depth with the PDA by navigating to the Raingage page within the NADP Rain program using procedures detailed above. Once on the Raingage page, click "Refresh" to view the current bucket depth.

To empty the bucket, you will first need to place the egage in Service Mode. Once you navigate to the Raingage page on the PDA, you must click "Service Mode". Service Mode will time-out after 300 seconds. As it will likely take longer than five minutes to empty and winterize the gage, you will need to click "Refresh" to extend Service Mode prior to automatic time-out. While in Service Mode, you will need to remove the collection chamber. First, remove the inlet orifice and set aside, be sure to clean any debris off the O-ring before re-installing the office. Gently remove the collection chamber by lifting the bucket straight up. ***Make sure the collection chamber is not dropped onto the platform. This could cause irreversible damage to the load cell!***

To empty the collection chamber, funnel waste into empty antifreeze or Nalgene containers. Containers of used antifreeze should be brought down from the station immediately (in a plastic bag to protect against leakage). Add two inches of fresh antifreeze to the empty chamber. Return the collection chamber to the inside of gage housing and onto the platform. ***Again, be careful not to drop it onto the platform!*** Make sure the alignment guideposts are inserted into the alignment ring on the bottom of the chamber. Align the black mark on the collection chamber with the black alignment mark between the two LED emitters. The emitters must have an unobstructed view of the detectors through the opening in the chamber. Finally, place the black inlet orifice over the housing, and press down on the inlet orifice until it sits securely on the housing.

The antifreeze mixture brought down from the weather station should be carried in antifreeze jugs or Nalgene containers. Back at the lab, funnel waste antifreeze into the five-gallon metal jugs labeled "Un-Regulated, Contents: 20% Ethylene Glycol, 80% Water." Include start and stop dates on the label for each jug. These jugs are found in NESB A101A. Dispose of waste through the CSU Hazardous Waste Office (x1-6745).

Refer to the NOAH IV Total Precipitation Gage Technical Manual for detailed maintenance procedures. Service Mode should be enabled prior to and during all maintenance procedures.

Contact the NADP Site Liaison, Matt Layden, (phone: 217-244-0372; email: ntn@sws.uiuc.edu) immediately if there are any problems with the egages.

Ambient Ammonia Sampling

a. Introduction

LVWS (CO98) is part of the NADP Ammonia Monitoring Network (AMoN). Passive Diffuse Ammonia (NH_3) Samplers are exposed for a 14-day sampling period. Exposed samples are removed and new samples installed on Tuesday mornings in accordance with the AMoN sampling schedule. NH_3 monitoring entails the use of two samples: (1) a travel blank and (2) sampler for field deployment. In most cases, a single sampler is deployed. Periodically, triplicate samplers will be deployed as part of network quality assurance. The procedure for deploying the samplers is the same regardless of whether there is a single sampler, or triplicate samplers. Additional information and detailed procedures are provided at the NADP AMoN website. You must be familiar with AMoN procedures before handling a NH_3 sampler. The following instructions provide a brief synopsis of appropriate methods.

b. Equipment and supplies to be taken to the field

- To-be-retrieved NH_3 sampler box containing:
  - travel blank
  - Prepaid return shipping tag
  - Vinyl gloves
  - Sample field form
- To-be-deployed NH_3 sampler box containing:
  - NH_3 sampler(s) and travel blank
  - Prepaid return shipping tag
  - Vinyl gloves
  - Sample field form
- LVWS field book with pencil

c. Estimated time to complete procedure

Once on site, approximately 10 minutes are required to change the NH_3 sampler(s).

d. Preparation

A set of new, unexposed samplers will be shipped to you in advance of each change date. Upon arrival, open the outer box to verify that the sampler(s) and QA travel blank arrived in good condition. The samplers should be sealed in two glass jars. Notify CAL of any problems with the shipment, and also note any issues with the shipment in the REMARKS section of the field form. Once you verify the sampler(s) and travel blank are okay, close the box and store it in the NREL freezer. Sampler(s) and travel blanks should be stored in the NREL freezer at all times to minimize NH_3 contamination, excluding transportation to and deployment in the field.

e. Procedures

1) Once on site, inspect the sampler(s) and shelter for any contamination (e.g., bird droppings or dirt) or other conditions that might affect sample integrity. If necessary, clean the sample shelter with DI water.
2) Retrieve the exposed sampler(s) – put on clean vinyl gloves provided by NADP. Detach the sampler(s) from the holder one at a time. Handle the samplers by the flexible tubing only. Place the sampler(s) with the attached flexible mounting tube into the glass jar that they were shipped in. Seal the jar, and wrap the jar with bubble wrap to protect it during transportation and shipment. Place the wrapped, sealed, sampler jar and its corresponding wrapped, sealed, travel blank into the shipping bag for return shipment to CAL. Leave the citric acid impregnated filter paper in the shipping bags. The citric acid filter paper acts as an ammonia scavenger. Record the Sample Off date and time.
3) Install the new sampler(s) – remove and save the bubble wrap from the new sampler jar. Remove a single new sampler from the jar. Handle the sampler buy its flexible tubing only. DO NOT TOUCH THE BLUE TUBE (i.e., radiello diffusive sampler body). Attach the sampler onto the mounting bolt inside the shelter. Ensure that the sampler is secure. Reseal the now empty glass jar and wrap it in bubble wrap. Place the resealed empty jar in the bag containing travel blank and citric acid filter paper. Record the sample start date/time. Repeat installation procedure for any remaining samplers.
4) Samper Storage – bring the two sampler boxes (i.e., for the retrieved sampler and the newly installed sampler) back to NREL. Store the box for the newly installed sampler with its travel blank in the NREL freezer. Complete the field form for the retrieved sampler and ship the complete box to CAL.
5) Completing the field form – record the NADP site ID (i.e., CO98), your name/initials, and the sample start and end dates/times. Report site conditions applying to the sampling period. Record the field blank storage location. Note any unusual site conditions in the REMARKS section. Each sampler is labeled with a unique two letter code on the bottom of the sampler.
6) Packing and shipping exposed sampler(s) – ensure the shipping bag with the citric acid filter paper is sealed, and place it in the shipping box. Include the top (white) copy of the field form. Seal the box with tape, affix the prepaid shipping form and send to:

   Phyllis Ballard
   Central Analytical Laboratory
   Illinois State Water Survey
   2204 Griffith Dr. Dock B
   Champaign, IL 61820-7463

f. Be Aware

Beware of ammonia contamination! Be sure to store all samplers in the NREL freezer while they await deployment to the field or shipment to CAL. This will minimize sampler contamination. Be sure to wear the vinyl gloves provided by NADP whenever handling a NH_3 sampler.

If the sampler falls onto the ground or another surface, but remains intact, simply pick it up, ensure the flexible tubing is firmly attached to the cartridge, and deploy or pack the sampler as you would normally. Make a note on the field form. If in doubt, contact the CAL for guidance:

AMoN Site Liaison
Telephone: (800) 952-7353 (toll-free)
Email: amon@isws.illinois.edu

Measuring Conductivity

a. Introduction

The conductivity of a solution is its ability to conduct an electrical current, and is the reciprocal of its electrical resistance. It is an indicator of the ionic strength of a solution. As with all other analyses we perform, conductivity measurements are susceptible to contamination. Conductivity of tap water can be 50 times greater than that of surface waters in LVWS. Make sure that everything that may contact sample waters has been thoroughly rinsed with DI before attempting any measurement.

The conductivity and temperature of all stream and lake samples are measured in the field prior to sample collection. Previously, precipitation samples were analyzed for conductivity at NREL. NADP no longer requires that conductivity be measured prior to shipment of precipitation samples. Therefore, laboratory conductivity measurements for precipitation samples are no longer collected as of 2010.

b. Equipment and Supplies (found in NREL A235)

- Field notebook
- Orion Conductivity meter Model 105A+
- Deionized (DI) water
- VWR Traceable Conductivity Standard 10 $\mu S\ cm^{-1}$ (Catalog #23226-567)
- VWR Traceable Conductivity Standard 100 $\mu S\ cm^{-1}$ (Catalog #23226-589)
- $75 \pm 1\ \mu S\ cm^{-1}$ Specific Conductance Standard (SCS) solution from NADP
- $14 \pm 2\ \mu S\ cm^{-1}$ Quality Control (QC) check solution from NADP
- Kimwipes

c. Estimated time to complete procedures

Allow 5 minutes for measurement of conductivity and temperature in the field.

d. Preparation

Be sure the conductivity meter has sufficient battery power and is calibrated prior to taking it into the field. The conductivity meter should be checked against standard solutions weekly, and calibrated as necessary. The meter must also be calibrated every time the battery is replaced. Calibration procedures are detailed in the Thermo Orion Instruction Manual, in the LVWS lab drawer. The nominal cell constant for the conductivity probe is 0.475 cm^{-1}. Check the amount and date of the standard and QC solutions prior to calibration. Recipes for the NADP QC solution (pH = 4.9 ± 0.15; conductivity = $14 \pm 2\mu S$

cm^{-1}) and SCS (pH = 4.9 ± 0.15; conductivity = 14 ± 2μS cm^{-1}) can be found in Appendix E. Standard conductivity solutions can also be ordered from NADP and VWR.

e. Procedures

To check the meter for accuracy:

1) Turn meter on.

2) Rinse conductivity cell and all vials to be used with DI water under faucet. Fill a vial with 10 ml of DI, dip probe, and record value in lab notebook.

3) Rinse conductivity cell and all vials to be used with DI water under faucet. Shake vials dry and rinse first one 3X with 14 ± 2 μS cm^{-1} QC conductivity solution. Fill a vial with 10 ml QC conductivity solution, dip probe, and record value in lab notebook.

4) Rinse another vial 3X with 75 ± 1 μS cm^{-1} SCS conductivity solution. Fill a vial with 10 ml SCS conductivity solution, dip probe, and record value in lab notebook.

5) If the conductivity values are outside of the acceptable range, calibrate the meter and repeat steps 3-4 until both readings are accurate.

6) Turn meter off, rinse probe with DI, and discard vials.

7) Put meter away in drawer.

To measure conductivity and temperature of surface waters in the field:

1) At each site, conductivity and temperature are measured prior to collecting water samples.

2) Turn on the meter, and submerge the probe in the stream/lake. Agitate the probe until "Ready" is displayed.

3) Record the time, conductivity, and temperature in the field book.

4) Turn the meter off.

Surface Water Sampling

a. Introduction

Surface water samples are collected from flowing streams, lake inlets, and lake outlets throughout the year (see sampling schedule in Appendix B). Stream samples are analyzed for pH, specific conductance (i.e., conductivity), major ions, silica, DOC, DON, DOP, and phytoplankton. Samples from the Loch Outlet are used in conjunction with precipitation chemistry to calculate chemical budgets and for long-term trend analysis. Samples taken elsewhere in the watershed are useful for analyzing processes and trends.

Stream and lake waters in LVWS are chemically dilute. Therefore, it is crucial that sampling protocols are carefully followed to avoid contamination.

Three types of samples are collected from the Loch Outlet: normal, duplicates, and blanks. Quality control samples must be collected on a regular schedule (Appendix B). Ten percent of all LVWS samples must be either duplicate (DUPE) or blank (BLANK) samples. Repeatability and accuracy of analysis is tested with duplicate sample results. Bias is assessed by analyzing blank samples. Instructions for conducting QA/QC sampling are in Appendix C.

b. Equipment and Supplies

- Field notebook and pencil
- Orion conductivity/temperature meter
- Butyl or neoprene sampling gloves
- 20 ml plastic vial of Lugol's solution with 2 ml disposable pipette (for monthly phytoplankton sample - see recipe for Lugol's solution in Appendix E)

Bottle set (this set is repeated for all locations unless otherwise noted)

- (1) 500 ml translucent HDPE acid-washed bottle (for filtered analysis)
- (1) 250 ml brown HDPE acid-washed bottle (for raw unfiltered sample)
- (1) 60 ml translucent HDPE acid-washed bottle (for SiO_2)
- (1) 480 ml baked borosilicate bottle (for DOC, DON, and DOP)
- (1) 125 or 250 ml baked borosilicate bottle – collected monthly at Loch Outlet only (for phytoplankton sample)

c. Estimated time to complete procedure

Once on site, it will take ~15 minutes to collect all samples.

d. Preparation

All supplies needed for surface water sampling are located in the cabinet below the lab bench, in NREL A223, or in the boxes underneath the lab bench. Instructions for ordering new supplies are located in Appendix G. Orders are frequently filled within a few days, but can also take weeks, so plan ahead! Check with a supervisor prior to making a purchase to ensure you have the correct account number.

Assemble a bottle set for each site. Fill appropriate number of gallon-sized Ziploc bags with contents listed in "Bottle set" section above. Label each bag with the site name, sample type (i.e., NORM, DUPE, BLANK), and collection date (YYMMDD). Do not forget to pack the conductivity meter and field notebook! Be sure the conductivity meter has been calibrated recently and that the battery has adequate charge.

All HDPE bottles are acid-washed and stored filled with DI to minimize sample bottle contamination. Prior to inclusion in a bottle set, the conductivity of the water inside each HDPE bottle must be tested. If the conductivity is <2 µS/cm, the bottle may be included in a sample set. If the conductivity is >2 µS/cm, the bottle must be removed from rotation and acid-washed again prior to use.

e. Procedures

1) Begin sampling at a given site by recording site name, weather conditions, time of sampling, and names of technicians in the field notebook.

2) For the Loch Outlet, be sure to record a stage reading from the staff gage inside the flume at the time of sampling.

3) Turn on conductivity/temperature meter. Immerse probe in stream until reading stabilizes, "Ready" should be displayed once the values stabilize. For all sites, record sampling time, conductivity, and temperature in the field notebook.

4) Wear rubber or neoprene gloves to prevent cold hands and minimize sample contamination. Rinse all bottles three times with sample before filling. To rinse, dip bottle to depth of 0.2-0.5 m with bottle mouth upstream, fill to one third of capacity, replace cap, shake vigorously, uncap, and pour water from bottle over threads of cap, and from cap over threads of bottle. Make sure you pour rinse water downstream, or away from eventual sample point.

   When finished rinsing, fill bottles to shoulder and replace cap. Follow the same procedure for all bottles, except for the phytoplankton bottle (which is rinsed 3X and filled to shoulder before adding 1-2 ml of Lugol's solution).

f. Be Aware

Make sure no person or animal is disturbing the stream immediately upstream of the sampling site. You do not want any sediment in the sample. If necessary, wait until water has cleared before sampling.

Streams flow at very low levels underneath the snow in the wintertime. If you have to dig away snow or chop out ice in order to sample, choose a spot with sufficient flow. Wait for water to clear of snow, ice, and sediment before sampling. An igloo or other shelter built just upstream from the throat of the flume at the Loch Outlet works well to minimize shoveling snow during winter.

Collection Permits and Fee Waivers

Our presence in RMNP is a privilege granted yearly by park staff, and as such, we apply yearly for collecting permits. We must also file an Investigator's Annual Report after each year of research, or the privilege to work in the park will be revoked.

We renew our Scientific Research and Collecting Permits every 1-2 years, depending on the expiration date. The project title for our primary research permit is "Long-term Ecological Research and Monitoring in the Loch Vale Watershed". Research permits are renewed through the NPS Research Permit and Report System. Application and renewal instructions are found at: https://science.nature.nps.gov/research/ac/ResearchIndex. Existing LVWS permits can be viewed and renewed by searching for last name "Baron" with the Investigator Lookup during the application process.

Every person must be pre-approved by RMNP Natural Resources staff to collect samples of any kind in the park. Each December, submit the names of all those projected to be working in LVWS the following summer to Judy Visty (judy_visty@nps.gov). Make sure that fee waiver permits are requested at the same time as collection permits. Fee waivers can be used for free entry at all RMNP entrance stations. Keep the list to a minimum number of people. Be sure to bring the LVWS Scientific Research and Collecting Permit to the field with you every week for entry to the park.

LABORATORY PROCEDURES

General Introduction

The NREL laboratories are shared among a number of different projects. It is important to follow general laboratory practices of safety and cleanliness. All lab users must complete the lab safety training with a lab manager. If you have any questions or concerns, do not hesitate to ask the lab manager. Make sure all LVWS personnel (including volunteers) are properly trained for the tasks they are asked to perform.

The following procedures are used both in preparation for field collection and for the processing of samples after returning from the field. Refrigerate all water samples upon return from field (in walk-in cooler, not the freezer!). All samples should be processed by noon the day following collection. Surface water samples were previously analyzed for pH and specific conductivity at NREL. Currently, specific conductivity is measured in the field at each sampling location for all stream and lake water samples. Specific conductivity and pH measurements in the NREL laboratory were discontinued in 2010, due to redundancy with conductivity and pH measurements that are obtained during water chemistry analyses at the Rocky Mountain Research Station (RMRS) and NADP Central Analytical laboratories. Samples are still filtered, preserved (as directed), and refrigerated at NREL before delivery to the appropriate analytical laboratory.

A note on cleanliness… Since LVWS waters are extremely dilute, you must assume any surface you touch is sufficiently contaminated to affect the outcome of analysis. Therefore, be very careful of what you, sample bottles, and lids come in contact with during lab work. Wash hands frequently with DI water. Latex gloves are available in the lab (however, the gloves themselves are not completely clean, and they become very slippery when wet). To minimize the potential for sample contamination, the LVWS lab bench at NREL is used exclusively for processing LVWS water samples. Clean the bench top before starting lab work, and continually sponge away any water that accumulates during lab work. Leave the area clean for the next user.

Procedures for processing and storage of surface water samples are summarized in Tables 1 and 2.

Table 1. Summary of laboratory procedures for processing LVWS weekly and synoptic surface water samples.

Bottle Set for Surface Water	Treatment	Label	Analysis	Destination
500 ml HDPE acid-washed bottle	Filtered with peristaltic pump into 60 ml translucent HDPE acid-washed bottle	F.U. (i.e., Filtered Untreated)	Ion concentrations	RMRS lab
250 ml brown HDPE acid-washed bottle	None	R.U. (i.e., Raw Untreated)	ANC, pH, and specific conductivity	RMRS lab
60 ml translucent HDPE acid-washed bottle	None	SiO_2	SiO_2 Concentration	NREL refrigerator
Pre-baked 480 ml amber borosilicate bottle	1) Filtered with baked glass filter tower into pre-baked 120 ml amber borosilicate bottle **AND** acidified with 2 drops of 37% HCl	DOC/DON/DOP	DOC/TDN and PO_4^{3-}	NREL refrigerator
	2) **NOT** filtered, but transferred into pre-baked 120 ml amber borosilicate bottle **AND** acidified with 2 drops of 37% HCl	TOTAL P	Total P	NREL refrigerator

Table 2. Summary of laboratory procedures for processing LVWS monthly surface water samples.

Bottle Set for Surface Water	Treatment	Label	Analysis	Destination
500 ml HDPE acid-washed bottle	Filtered with peristaltic pump into 250 ml brown HDPE acid-washed bottle	ARCHIVE - F.U.	As needed	NREL freezer; FORT freezer
Pre-baked 250 ml amber borosilicate bottle	Stained with 1-2 ml of Lugol's solution in the field	PHYTO	Species richness and abundance	NREL refrigerator then algal taxonomist

Filtration for Major Ions

a. Introduction

Solutes present in surface waters are primarily derived from precipitation inputs and geochemical weathering. Because samples must be delivered to laboratories for chemical analysis, it is important that the original chemical composition remains unchanged before and during transport. Particulates in a sample can affect chemical results as well as clog lines of sensitive instrumentation. Biological activity in the sample can alter chemical composition. Therefore, we filter to remove particulate materials, spores, and bacteria from sample water to minimize their effect on chemical analysis. Samples that have been filtered with the peristaltic pump are labeled F.U. (Filtered Untreated), and are used to determine the concentration of major ions.

An ARCHIVE sample is collected from the Loch Outlet once a month. This sample is filtered with the peristaltic pump into a brown 250 ml HDPE bottles, labeled as "ARCHIVE - F.U.", and stored in the NREL freezer. Archive samples are transferred from the NREL freezer to the LVWS chest freezer in the USGS Fort Collins Science Center (FORT) laboratory once a year.

b. Equipment and Supplies

Equipment and supplies are located in the NREL A235.

- Bottle(s) with sample
- Peristaltic pump with Antlia filter holders
- Millipore Isopore 0.4 µm membrane filters (Catalog# HTTP04700)
- Clean forceps
- Deionized (DI) water
- Waste beaker
- Clean 60 ml HDPE bottle labeled with site name, date, sample type, & F.U.
- Clean 250 ml brown HDPE bottle labeled with site name, date, sample type, & ARCHIVE - F.U. (collected once a month from the Loch Outlet)

c. Estimated time to complete procedure

Allow approximately 15 minutes per sample, although filtering times may vary greatly depending on the amount of particulate matter in the sample.

d. Preparation

All pump apparatus should be acid-washed once a month. This can be done on the lab bench. To clean, fill a one liter bottle with 10% HCl from acid bath, run acid through pump as you would a regular sample (only without the filter membrane), allow acid to sit in tubing for ~4 hours, then copiously rinse with DI water (at least 10 liters). Conductivity of the rinse water should be <2 μS cm^{-1}, even after sitting in the pump for several hours. pH paper can be used to check that the pH of the DI rinse water did not change as it passed through the peristaltic pump (i.e., that all acid has been removed from the pump).

Make sure there are enough filters on hand to process all samples. Filters may take several weeks to be delivered from scientific supply houses, so plan ahead!

Plastic bottles must be acid-washed before use (see acid washing section). Bottles are stored filled with DI water. Check the conductivity before using any bottle. A bottle with a conductivity >2 μS cm^{-1} should be taken out of rotation, and re-cleaned.

e. Procedures for filtering sample

Rinsing pump

1) Get a piece of clean tubing from the Tupperware box in the LVWS clean-drawer. Handle tubing on the end marked with ink. Rinse inside and out with DI water.

2) Place non-inked end of tube in 1-liter bottle of DI water.

3) Attach inked end of tube to the stopcock on peristaltic pump.

4) Place waste beaker under filter holder.

5) Open stopcock. Turn pump on forward to a setting of 3. Rinse pump tubing with 1-liter of DI before working with sample.

6) Turn pump off.

Loading filter

1) Being careful not to touch any of its inner surfaces, remove bottom half of filter holder. Place on clean surface (e.g., a Kimwipe or inside of filter box lid).

2) Flame forceps with Bunsen burner and douse with DI water.

3) Using forceps, remove rubber ring from filter holder and set it on a clean surface (e.g., a Kimwipe or inside of filter box lid).

4) Use forceps to remove paper over filter and carefully remove one filter (Isopore 0.4 μm) from box. Filters rest in box upside down. Turn filter over from its position in box and

place directly into of filter holder. If filter is not centered, carefully adjust its position by pulling on edge with forceps. Be careful handling filters, as any contact between the filter and forceps in any area except its extreme outer edge will result in contamination or damage to the filter. Do not use any filter you think may be punctured or contaminated.

5) Replace the rubber ring on top of filter and carefully secure in filter-head by pushing into outer groove of filter holder with forceps.

6) Screw filter holder back into the filter-head and rinse the assembly with more DI water (10 seconds is sufficient).

Filtering sample

1) Take tube out of DI water bottle, walk away from bench, and spin tube to remove all DI water.

2) Place tube into sample bottle and attach to stopcock. Turn pump on to a forward setting of 3 (to purge all DI water and allow tubing and filter to be rinsed with sample). Watch bubbles from the sample as they move through the pump tubing, be sure you are collecting filtered sample and not DI rinse water. Samples that have been refrigerated overnight will be colder than the DI rise water. When you feel colder water coming through the filter, you know that sample water is passing through the pump. Filtering pressure must be less than 15 lbs in^{-2} so that the filter does not rupture. This corresponds to a setting of 3 on pump motor control.

3) Rinse bottles three times with filtrate before filling (a few ml per rinse is sufficient). If sample contains enough matter to clog the filter before the F.U. bottle is filled, it may be necessary to stop, change the filter, and resume filtering. Remember to rinse the new filter with sample before beginning to fill bottle again. <u>Be sure to collect an Archive sample from the Loch Outlet once a month</u>!

4) Change filters between samples. Follow filter rinsing procedure as previously directed. Also, rinse and spin inked tube with DI water between samples.

5) After all samples have been filtered, give the unit a final rinse with DI water (at least 1 liter) to purge all sample water. Be sure there is no water left in the filter tubing.

6) Rinse inked tube with DI water and place back into box in clean drawer.

7) Store the 60 ml F.U. sample bottles in the walk-in cooler before delivery to analytical laboratory. Store the monthly Archive sample from the Loch Outlet in the NREL freezer.

a. Be Aware

It is a good idea to have a number of pre-cut, acid-washed pieces of Antlia silicone replacement tubing ready. Disassemble the pump tubing head to replace the old piece of tubing with a new piece of tubing. Wrap the outlet side of silicone tubing with strapping tape to prevent ballooning under filtration pressure. Pump assembly is easier if the pump rotor is rotating slowly (do this by hand with screwdriver) while the tubing is held in place and the plastic pump head halves are gradually pushed together. New tubing for pump is located in drawers under lab bench. Any other pump related supplies can be bought from VWR.

Filtration for DOC, DON, and DOP

a. Introduction

Dissolved organic carbon (DOC) is created in natural systems by leaching of soil organic matter, or from decomposition of algal biomass. DOC is operationally defined as organic carbon that passes through a 0.45 µm filter. Therefore, we filter water samples prior to analyzing DOC. Proper washing/baking is essential to remove all surface contaminants (most importantly, carbon!). Proper washing and baking will also prolong the useful life of the filtration apparatus. Methods for analyzing DOC are detailed in the Schimadzu section of the LVWS Methods Manual.

Dissolved organic nitrogen (DON) is determined by calculating the difference between Total Dissolved Nitrogen (TDN) and Total Inorganic Nitrogen (TIN). TIN is calculated by converting nitrate (NO_3^-) to nitrate expressed as nitrogen ($NO_3{-}N$) and ammonium (NH_4^+) to ammonium expressed at nitrogen (NH_4^+-N). Once nitrate and ammonium concentrations are expressed as nitrogen they can be summed to get TIN. Methods for analyzing TDN are detailed in the Schimadzu section of the LVWS Methods Manual.

Dissolved organic Phosphorus (DOP) is estimated by calculating the difference between Orthophosphate (PO_4^{3-}) and Total Phosphorus (TP). Methods for analyzing PO_4^{3-} and TP are detailed in the AlpKem Section of the LVWS Methods Manual. PO_4^{3-} samples are filtered but TP samples are not.

b. Equipment and Supplies

Equipment and supplies are located in drawers below the LVWS lab bench in NREL A235.

- 480 ml amber borosilicate bottle filled with sample (from cooler)
- Whatman GF/F filters (pre-baked at 500 °C for 5 hours - see instructions below)
- Forceps
- Pre-baked filtration apparatus (ground fitted glass 1000 ml flask, ground glass 47 mm fritted glass filter support, ground glass 47 mm funnel, & clamp)
- Vacuum line (< 15" Hg vacuum)
- (2) Pre-baked 120 ml amber borosilicate bottles. Once bottle should be labeled "DOC/DON/DOP" and the other bottle should be labeled "Total P". Both bottles labels should include the site name, sample type, and date.
- 37% concentrated hydrochloric acid (HCl)

c. Estimated time to complete procedure

Allow ~10 min per sample, although filtering times may vary greatly depending on the amount of particulate matter in the sample.

d. Preparation

All glassware and glass fiber filters (GF/F) must be baked in the muffle furnace (in NREL A121) before use. Rinse equipment in DI water and cover all orifices with aluminum foil. Place in muffle furnace for five hours at 500 °C (one hour to reach temperature, and four hours at full temperature). Temperature in the furnace must not exceed 500 °C. If it does, the glass will deform. Allow oven to cool overnight (to < 150 °C) before opening furnace and removing glassware, otherwise it may crack from sudden cooling.

e. Laboratory Procedures

Total Phosphorus

1) Rinse one 120 borosilicate amber bottle 3X with sample, and then fill to shoulder. Label the bottle "Total P", be sure to include site name, sample type, and date on the label.

2) Add two drops of 37% HCl to the Total P bottle, invert, and shake vigorously.

3) Repeat for each sample site.

4) Store sample bottles in walk-in cooler in appropriate location (i.e., with other Total P bottles)

DOC/DON/DOP - Rinsing filter tower

1) Assemble filtration apparatus by placing filter support on flask; center a single filter over fritted glass on filter support using forceps, center funnel on filter support, and clamp support/funnel together with clamp. Do not connect to vacuum line yet.

2) Add ~20 ml of sample to funnel. Swirl to rinse sides of funnel, and then connect vacuum line to port on flask. Disconnect vacuum line just as rinse water completely leaves funnel (before significant amount of air passes through the filter - otherwise, trapped particles may fracture and be washed into the sample).

3) Shake and swirl filtrate around in flask, and then pour into waste vessel.

4) Rinse two more times.

DOC/DON/DOP - Filtering sample

1) Pour remaining sample into funnel, attach vacuum line, and pull sample into flask. If there is substantial amount of suspended material in the sample, filtering will be very slow. If the sample appears to contain substantial suspended material, only pour half of the sample into the filter tower so that the filter can be changed midway through sample.

2) Once the sample has been filtered, rinse a 120 borosilicate amber bottle 3X with sample, and then fill to shoulder. Label the bottle "DOC/DON/DOP", be sure to include the site name, sample type, and date on the label.

3) Add two drops of 37% HCl to the DOC/DON/DOP bottle, invert, and shake vigorously.

4) Repeat all steps for each sample. Be sure to rinse the filter tower with DI between samples, and use a new filter for each sample.

5) Store sample bottles in the walk-in cooler in appropriate location.

6) After processing samples, rinse all glassware thoroughly with DI water. Cover all orifices with aluminum foil and bake as previously described. Bake glassware on either Friday or Monday so it is ready for use by Wednesday morning.

Measuring Silica

a. Introduction

Read Silica, High Range Method (adapted by Hach from Clesceri et al. 1998). If necessary, make new standards and keep in refrigerator. Use Hach's 10 mg/L SiO_2 standard to make standards (range of 0.0, 0.1, 0.5, 1.0, 2.0, 5.0 mg/L) using formula $C_1V_1=C_2V_2$, where C_1 is the initial concentration, V_1 is the initial volume, C_2 is the desired concentration, and V_2 is the desired volume. Refer to the Spectronic 20$^+$ Series Spectrophotometer User's Guide (Thermo 2003) for trouble shooting and additional operating procedures.

b. Equipment and Supplies (found in walk-in refrigerator and NREL A256)

- Thermo Spectronic 20D$^+$ spectrophotometer (in NREL A256)
- Vortex Genie 2 stirrer (in NREL A258)
- Timer or clock
- Wide mouth/flat bottom optically matched cuvettes
- 10 ml automatic pipette and clean pipette tips
- Hach High Range Silica Test Kit
- 10 mg/L SiO_2 solution for standards
- Acid Reagent Powder Pillows
- Molybdate Reagent Powder Pillows
- Citrate Acid Pillows
- Samples for analysis

c. Preparation

Zero the absorbance

1) Turn on the spectrophotometer turning the Power Switch (knob on the left side of the instrument) clockwise. Allow the spectrophotometer to warm up for at least 15 minutes.

2) After the warm up period, set the analysis wavelength to **420 nm** by rotating the Wavelength Control Knob.

3) Set the **filter lever** to 340-599 nm.

4) Set the display mode to TRANSMITTANCE by pressing the MODE key until the appropriate LED is lit.

5) Adjust the display to 0% Transmittance using the knob on the front left side of the instrument. Make sure that the sample compartment is empty and the cover is closed.

6) Set the display mode to ABSORBANCE by pressing the MODE key until the appropriate LED is lit.

7) Fill a clean cuvette with DI and wipe the cuvette with a Kimwipe to remove liquid droplets, dust and fingerprints.

8) Place the DI filled cuvette in the sample compartment and align the guide mark on the cell with the guide mark at the front of the sample compartment. Press the cell firmly into the sample compartment and close the lid.

9) Adjust the display to 0.0 Absorbance (0.0 Absorbance = 100% Transmittance) with the Transmittance/Absorbance Control (knob on the right side of the instrument).

10) Remove the cuvette from the sample compartment.

Matching cuvettes

1) Allow samples and standards to reach room temperature.

2) Fill each cuvette with ~10 ml of DI water.

3) Wipe each cuvette with a Kimwipe, as any scratches or marks will skew results. After wiping a cuvette, insert it into the sample compartment and note the absorbance in the lab notebook. All matching cuvettes should have absorbance values ≤ 0.002.

4) Set cuvettes that do not match aside. Keep all that do match.

d. Procedures

Standard curves

1) Begin by running a standard curve to get the respective absorbencies.

2) Perform a linear regression to find the equation that describes the relationship between the standard concentrations and their absorbencies. A new standard curve is necessary for every 2-3 runs, and for each new day's run(s).

3) If the R^2 is ≥ 0.99, proceed to the next step. If not, check date on standards and either rerun the standards or make new ones.

Running samples

1) Check that the pipette is dispensing the correct volume of sample (i.e., 10.00 ml).

2) Once pipette is calibrated, begin pipetting standards and samples into 12 cuvettes. Keep a written log of what sample or standard goes into each cuvette in the silica lab notebook.

3) Each group of 12 cuvettes should contain a standard, blank, and duplicate.

4) Rinse cuvettes with a few ml of sample or standard three times before dispensing 10 ml of analyte.

5) After analytes are pipetted into cuvettes, add 1 "Acid Reagent Powder Pillow" to each cuvette. Use the Vortex Genie to stir the reagent in with the analyte. This speeds dissolution greatly.

6) Add 1 "Molybdate Reagent Powder Pillow" to each cuvette and use Vortex Genie to dissolve reagent, wait 10 minutes before next step.

7) Add 1 "Citrate Acid Pillow" to each cuvette and use Vortex Genie to dissolve reagent.

8) Wait two minutes before placing cuvette into meter. Wipe the outside of each cuvette with a Kimwipe before placing into meter.

9) Record the absorbance reading from spectrophotometer in the silica lab notebook.

10) Run 2 to 3 sets of 12 samples before running a new standard curve.

11) Use the standard curve equation to convert absorbance to concentration. Enter the absorbance as the "x" variable.

AlpKem Procedures

The Alpkem is an instrument that colorimetrically analyzes aqueous solutions for concentrations of different chemicals. NREL's AlpKem can be configured to run ammonium (NH_4^+), nitrate (NO_3^-), and orthophosphate (PO_4^{3-}). Technicians must have proper training from a laboratory manager before attempting to use this instrument.

a. Introduction

Please refer to the "Flow Solution Manual IV" by OI Analytical 2001 for NH_4^+, NO_3^-, PO_4^{3-}, and total phosphorus (TP; persulfate digest) methods. This manual is located in NREL A256. Modifications to the TP method are supplied by the Hedin lab and are described below.

b. Equipment and Supplies (located in Rm. A256)

– OI Analytical AlpKem Spectrophotometer
– Samples for NH_4^+/NO_3^-/PO_4^{3-}/Total Phosphorus (KCL/DI matrix)
– Standards (KCl/DI matrix)
– 2-ml analyzer cups and racks
– KCl/DI squeeze bottles
– 8-micron membrane filters
– Reagents (recipes are described in Appendix E)
– Test tubes (TP)
– Linerless caps (TP)
– Oxidizing solution (Potassium Persulfate for TP)
– Autoclave (TP)
– 5 ml pipette and tips (for samples, standards, washes) (TP)
– 1 ml pipette and tips (for oxidizing solution) (TP)

AlpKem Reagents

NH_4^+	**NO_3^-**	**PO_4^{3-}/TP**
KCl/DI Carrier	DI Carrier	PO_4^{3-} Color Reagent
NH_4^+ Working Buffer	NO_3^- Color Reagent	Dowfax
Sodium Salicylate/Sodium Nitroferricyanide	NO_3^- Buffer (NH_4Cl) (check filter for clogs)	DI Carrier
Hypochlorite (use fresh bleach)	Cadmium Reduction Column	Potassium Persulfate

c. Preparation

1) Allow samples and standards to reach room temperature by setting on counter while setting instrument up.

2) Make sure all of the reagents are full, and your standards, reagents, and DI are fresh.

3) Turn on the computer from the power strip and follow the set-up procedures detailed below.

4) The water and NH_4^+/NO_3^- waste must be collected and disposed of as hazardous waste. Consult the lab manager if you questions regarding hazardous waste disposal. DO NOT ALLOW THE PINK AND GREEN WASTES TO MIX UNDER ANY CIRCUMSTANCES! Mixing these substances will produce noxious gasses.

5) Be sure the correct channels are set up for your particular needs.

d. Procedures

Ammonium/Nitrate Directions

1) Check that the instrument is configured for NH_4^+/NO_3^-: The line from the sampler should be connected to the "T" where the NH_4^+ sample line and the NO_3^- pull through line meet. The debubbler on the channel 3 detector (NH_4^+/ PO_4^{3-}) should be connected to one line labeled "NH_4 from debubbler" and another line labeled "NH_4 to spec". If not, see the section: Switching the AlpKem from PO_4^{3-} to NH_4^+/NO_3^-.

2) Remove NO_3^- color reagent and NH_4^+ salicylate-nitroferricyanide bottles from the fridge. Place the NO_3^- color reagent in a warm water bath to warm up to room temperature.

3) Check that the sampler wash waste is flowing into the sink.

4) Turn on compressed air at round black knob on bottom of regulator.

5) Turn on main power switch and make sure NH_4^+ heater is on. Let the sampler initialize.

6) Open Winflow 4.0 software. If software is already open, exit and reopen. This causes the sample probe to go into the wash reservoir.

7) Empty and refill the large DI water bottle.

8) Lock down the pump platens and pull the engaging levers straight up for all the pump tubes labeled NH_4^+ and NO_3^- plus "to sampler wash", "from sampler wash", and "sample pull through".

9) Press the "run stop" button on the pump, the display should read –50.0.

10) Connect the sampler wash and NO_3^- carrier lines to the large DI wash bottle (be sure to connect to the ports as labeled: the sampler wash will not flow properly if connected to the port labeled NO_3^- carrier).

11) Connect the NH_4^+ lines: buffer, hypochlorite, and salycilate, to the NH_4^+ manifold startup solution.

12) Connect the NO_3^- lines: color reagent and buffer, to the NO_3^- rinse bottle.

13) Let pump for 10 minutes and check for leaks.

14) Discard old hypochlorite solution and make fresh (12 ml bleach and 178 ml DI water).

15) Move the NO_3^- buffer line to the buffer bottle.

Condition the cadmium column

1) Place the column, syringe with tube to fit column, open specimen cup of NO_3^- buffer, and open cup of 0.2% $CuSO_4$ on the bench within easy reach.

2) Draw about 1 ml of buffer into the syringe and remove all air bubbles from the tube. Break the tubing connection on the column and elevate the connector end slightly until a drop of liquid appears at the tube end.

3) Hold the syringe plunger end up and depress the plunger until a drop of buffer appears at the connector end.

4) Connect the syringe to the column without introducing any air.

5) Place the free end of the column into the buffer cup and slowly depress the plunger to expel the air from the free end of the column.

6) Transfer the free end to the cup of $CuSO_4$ and draw up about 2-4 ml.

7) Depress the plunger slightly to release any tension on the liquid in the column and transfer the end to the cup of buffer.

8) Draw up 3-5 ml of buffer, and then depress the plunger slightly to release any tension on the liquid in the column.

9) Remove the end from the buffer, place the column, tubing and syringe flat on the bench, then disconnect the syringe and connect the two ends of the column together.

10) Take the column and syringe to the sink and rinse with DI water. Put the buffer and $CuSO_4$ cup away and wipe the bench with a wet sponge. The NO_3^- buffer contains NH_4^+, which can contaminate your samples!

Connect the cadmium column to the instrument

1) Make sure there are no bubbles in the nitrate cartridge (about 10 minutes after moving the NO_3^- buffer line to the buffer bottle).

2) Press the “run stop” button on the pump, press the “mode” button 4 times, the display should read “2.0”, press the “run stop” button again, this puts the pump speed to minimum.

3) Break the connection in the line between the “to column” and “from column” ports on the NO_3^- cartridge.

4) Break the tubing connection on the column and note which end will connect to the line from the “to column” port on the cartridge. Elevate the other end of the tubing until a drop of liquid forms at the end. Wait until a drop forms on the end of the “to column” line and connect it to the column without introducing any air.

5) Connect the other end of the column to the “from column” line on the cartridge.

6) Press the “run stop” button on the pump, press the “mode” button 3 times, the display should read “-50.0”, press the “run stop” button again, this puts the pump speed back to normal.

7) Move the NO_3^- color reagent line to the color reagent bottle.

8) Move the NH_4^+ reagent lines to their respective reagent bottles.

9) Make sure the line from the sampler is completely clear of all other lines and the reagent bottles so the sample probe can move freely.

10) Let the system pump at least 30 minutes before beginning a sample run.

Setting up a sample run

Press the sample table button on the main tool bar in Winflow4.

Type in “cup #”, “name” and “type” for each sample and standard to be analyzed. Refer to the typical sample table on the page 7 and the notes below.

Cup Numbers

Positions 1-20 are the large tubes at the back of the sampler and are used for standards, checks and baselines (washes). The instrument can sample from these cups up to 6 times each.

There are 3 racks for the 2 ml sample cups. The one on the left (closest to the sampler wash cup) holds cup numbers 101-190. The middle rack holds cup numbers 201-290 and the rack on the right holds number 301-390. These cups should only be sampled one time each.

Sample Name

Sample names can be anything that fits in the field. However, the standards used in the calibration curve (anything labeled “C” in the “type” column) must be named exactly the same as they are named in the calibrant table in the method file.

To view the calibrant table, click the “edit method” button on the main toolbar, click file, open, and select the correct method file. Consult the lab manager for the correct method file. The calibrant table is the last page of the method file.

The format for naming calibration standards is: X/Y where X is the concentration of NH_4^+ and Y is the concentration of NO_3^-. No leading zeros on values less than 1 and no decimal points except for fractional concentrations.

Type

SYNC - This is always the first cup in a run, it is a high standard (usually top standard) which will yield a large peak to let the software know the sample peaks have started.

C - indicates a calibration standard to be included in the calibration curve. Do not use this for internal standards or checks unless you want them included in the calibration curve.

RB - indicates a baseline (wash). The instrument will use these peaks to correct for baseline drift so they should always be a 0 ppm standard.

U - unknown. Should be used for all samples, checks, and internal standards.

After the calibration curve, the basic pattern should be 10 samples, 1 standard, 10 samples, and 1 baseline. Repeat to a maximum of about 80 samples. Larger sets should be broken into multiple runs. Sample runs should always end with a baseline.

Other columns in the sample table

R = Number of replicate samples to be taken from the cup. Should be set to 1.
Dil = dilution factor of the sample, usually 1.
Wt = weight of the sample, usually 1.
After the sample table is prepared, be sure to save it.

Starting a sample run

1) Click the collect data button on the main toolbar. The software will ask for an operator name and ID, type in your initials for both. The software will then ask for a sample table name and a method name. Consult the lab manager for the correct method. Select the correct file names and press OK. The software will ask for a filename for the results with the default being the same as the sample table with an .rst extension. Select or type in a file name and press OK. The software will not allow you to overwrite or append and existing file.

2) The data collection window will appear on the screen. Press the "play" button to start the data collection. At this point, the software will monitor the baselines for 60 minutes and then start sampling.

3) Debubble both flow cells by pinching and releasing the outflow tubing. Repeat until no bubbles appear.

4) Pour standards and samples into the cups and place in the sampler. Get at least 20 cups poured before starting the sampler.

5) Check baselines for stability and drift, should be less than 500 micro-absorbance units.

6) If there are any peaks or jumps in the baseline traces, Press the "stop" button. The software will ask if you want to stop, press yes. Press the "rewind" button and then the "play" button.

7) Press the "fast forward" button to begin sampling.

8) The first NO_3^- peak will appear about 75 seconds after the first sample is drawn. The first NH_4^+ peak will appear after about 250 seconds.

Instrument shutdown

1) Press the “run stop” button on the pump to stop the reagent flow.

2) Disconnect the cadmium column from the NO_3^- cartridge and connect the two ends together. Be careful to minimize the amount of air let into the column.

3) Connect the “to column” and “from column” lines together.

4) Press the “run stop” button on the pump to restart the flow.

5) Move the NO_3^- lines to the NO_3^- rinse bottle and the NH_4^+ lines to the NH_4^+ manifold startup solution bottle. Let pump about 10 minutes.

6) Disconnect all the lines and let the system pump until all the liquid is out of the instrument, about 15 minutes.

7) Press the “run stop” button to stop the pump and turn off the main power switch to the instrument.

8) Push the engaging levers on all the pump platens to horizontal and disconnect one end of each platen from the pump.

9) Empty the NH_4^+ waste into the proper hazardous waste container.

10) Empty the NO_3^- waste into the proper hazardous waste container.

11) Empty and discard the sample cups and tubes. Rinse the sampler racks with DI water and set on the bench to dry.

12) Return the NO_3^- color reagent and NH_4^+ salycilate-nitroferricyanide bottles to the fridge.

13) Refrigerate the standards if the instrument will be used the next day or freeze them if it will be longer.

14) Wipe down the instrument and benches with a moist sponge.

15) Turn off the ball valve on the compressed air.

Switching the AlpKem from PO_4^{3-} to NH_4^+/NO_3^-

1) Make sure the PO_4^{3-} waste container is empty. The waste goes in the bottle on the bottom shelf of the acid cabinet in room A258.

2) Disconnect the "PO_4 waste from spec" line from the outflow tube of the channel 3 (PO_4^{3-}/NH_4^+) detector and connect the "NH_4 waste from spec" line in its place.

3) Disconnect the "PO_4 to spec" line from the bottom connection on the debubbler on the channel 3 detector and replace it with the "NH_4 to spec" line.

4) Disconnect the "PO_4 from debubbler" line from the top connection on the debubbler and replace it with the "NH_4 from debubbler" line.

5) Turn the PO_4^{3-} heater off and the NH_4^+ heater on.

6) Disconnect the sampler line from pump tube labeled "PO_4 sample" and connect it to the "T" where the "NH_4 sample" line and the "NO3 sample pull thru" line meet.

Orthophosphate/Total Phosphorus Directions

Orthophosphate

1) All glassware and bottles used should be acid washed before beginning.

2) Check that the instrument is configured to run PO_4^{3-}. The line from the sampler should be connected to the pump tube labeled "PO_4 sample" and lines labeled "PO_4 to spec" and "PO_4 from debubbler" should be connected to the channel 3 debubbler. If not, see the section: "Switching the AlpKem from NH_4^+/NO_3^- to PO_4^{3-}".

3) Empty the large DI water bottle and fill with 1.2 M HCl from the acid bath. Let it sit for at least 15 minutes. Carefully pour the acid back into the acid bath, rinse 6 times with DI, and fill with DI.

4) Dissolve 1.8 grams ascorbic acid in about 70 ml DI water in a 100 ml volumetric flask. Dilute to 100 ml with DI water.

5) Check sampler wash waste reservoir under bench to right of instrument. Empty into sink if more than half full.

6) Turn on main power switch and make sure PO_4^{3-} heater is on.

7) Open Winflow 4.0 software. If software is already open, exit and reopen. This causes the sample probe to go into the wash reservoir.

8) Lock down the pump platens and pull the engaging levers straight up for all the pump tubes labeled PO_4^{3-} plus “to sampler wash” and “from sampler wash”.

9) Press the “run stop” button on the pump, the display should read –50.0.

10) Connect the sampler wash line to the port labeled sampler wash on the large DI water bottle. Connect the PO_4^{3-} color reagent line to the port labeled NO_3^- carrier on the large DI water bottle (be sure to connect to the ports as listed: the sampler wash will not flow properly if connected to the port labeled NO_3^- carrier).

11) Connect the “PO_4 Dowfax” line to the dowfax bottle.

12) Let the system pump for 15 minutes and check for leaks.

13) Prepare the color reagent by adding the following stock solutions to the color reagent bottle IN ORDER and mixing after each addition:

    150 ml 5N sulfuric acid
    15 ml antimony potassium tartrate solution
    45 ml ammonium molybdate solution
    90 ml ascorbic acid solution

14) If there are no leaks, move the PO_4^{3-} color reagent line to the color reagent bottle.

15) Let the system pump 15 minutes before running samples.

Setting up a sample run

Press the sample table button on the main tool bar in Winflow4.

Type in “cup #”, “name” and “type” for each sample and standard to be analyzed. Refer to the typical sample table on the page 5 and the notes below.

Cup Numbers

Positions 1-20 are the large tubes at the back of the sampler and are used for standards, checks and baselines (washes). The instrument can sample from these cups up to 16 times each.

There are 3 racks for the 2 ml sample cups. The one on the left (closest to the sampler wash cup) holds cup numbers 101-190. The middle rack holds cup numbers 201-290 and the rack on the right holds number 301-390. These cups can be sampled up to 4 times each.

Sample Name

Sample names can be anything that fits in the field. However, the standards used in the calibration curve (anything labeled “C” in the “type” column) must be named exactly the same as they are named in the calibrant table in the method file.

To view the calibrant table, click the “edit method” button on the main toolbar, click file, open, and select the appropriate method. Consult the lab manager for the correct method. The calibrant table is the last page of the method file.

Type

SYNC - This is always the first cup in a run, it is a high standard (usually top standard) which will yield a large peak to let the software know the sample peaks have started.

C - indicates a calibration standard to be included in the calibration curve. Do not use this for internal standards or checks unless you want them included in the calibration curve.

RB - indicates a baseline (wash). The instrument will use these peaks to correct for baseline drift so they should always be a 0 ppm standard.

U - unknown. Should be used for all samples, checks, and internal standards.

After the calibration curve, the basic pattern should be 10 samples, 1 standard, 10 samples, and 1 baseline. Repeat to a maximum of about 80 samples. Larger sets should be broken into multiple runs. Sample runs should always end with a baseline.

Other columns in the sample table

R = Number of replicate samples to be taken from the cup. Should be set to 1.
Dil = dilution factor of the sample, usually 1.
Wt = weight of the sample, usually 1.
After the sample table is prepared, be sure to save it.

Starting a sample run

1) Click the collect data button on the main toolbar. The software will ask for an operator name and ID, type in your initials for both. The software will then ask for a sample table name and a method name. Consult the lab manager for the correct method. Select the

correct file names and press OK. The software will ask for a filename for the results with the default being the same as the sample table with an .rst extension. Select or type in a file name and press OK. The software will not allow you to overwrite or append and existing file.

2) The data collection window will appear on the screen. Press the "play" button to start the data collection. At this point, the software will monitor the baselines for 60 minutes and then start sampling.

3) Debubble the flow cell by pinching and releasing the outflow tubing. Repeat until no bubbles appear.

4) Pour standards and samples into the cups and place in the sampler. Get at least 20 cups poured before starting the sampler.

5) Check baselines for stability and drift, should be less than 500 micro-absorbance units.

6) If there are any peaks or jumps in the baseline traces, Press the "stop" button. The software will ask if you want to stop, press yes. Press the "rewind" button and then the "play" button.

7) Press the "fast forward" button to begin sampling.

Instrument shutdown

1) Move the "PO_4 color reagent" line to the "NO_3 carrier" port on the large DI water bottle. Let the system pump for about 10 minutes.

2) Disconnect all the lines and let the system pump air for about 15 minutes or until all the liquid is out of the instrument.

3) Press the "run stop" button to stop the pump and turn off the main power switch to the instrument.

4) Push the engaging levers on all the pump platens to horizontal and disconnect one end of each platen from the pump.

5) Pour the unused color reagent into the PO_4^{3-} waste container.

6) Empty the PO_4^{3-} waste into the container in the acid cabinet in A258.

7) Empty and discard the sample cups and tubes. Rinse the sampler racks with DI water and set on the bench to dry.

8) Refrigerate the standards if the instrument will be used the next day or freeze them if it will be longer.

9) Wipe down the instrument and benches with a moist sponge.

Switching the AlpKem from NH_4^+/NO_3^- to PO_4^{3-}

1) Make sure the NH_4^+ waste container is empty. The waste goes in the bottle on the floor by the sink.

2) Disconnect the "NH_4 waste from spec" line from the outflow tube of the channel 3 (PO_4^{3-}/NH_4^+) detector and connect the "PO_4 waste from spec" line in its place.

3) Disconnect the "NH_4 to spec" line from the bottom connection on the debubbler on the channel 3 detector and replace it with the "PO_4 to spec" line.

4) Disconnect the "NH_4 from debubbler" line from the top connection on the debubbler and replace it with the "PO_4 from debubbler" line.

5) Turn the NH_4^+ heater off and the PO_4^{3-} heater on.

6) Disconnect the sampler line from the "T" where the "NH_4 sample" line and the "NO_3 sample pull thru" line meet and connect it to the pump tube labeled "PO_4 sample".

Total Phosphorus

Methods for total phosphorus are taken from the Hedin lab at Cornell. This is a modification of the total nitrogen method with a few slight differences. By analyzing samples for orthophosphate (PO_4^{3-}) and total phosphorus (TP), dissolved organic phosphorus (DOP) can be calculated by simple subtraction.

Be sure the test tubes and threads are free of chips and cracks since contents may be lost during autoclaving. Set autoclave for liquid cycle, 250 F, 50 min. using only the cycle 4 button. If system is set for a different cycle, please see Dan Reuss (lab manager).

1) Pipette 5 ml of standards, samples, washes, and duplicates into acid washed and oven dried borosilicate glass test tubes.

2) Add 1 ml of potassium persulfate oxidizing solution to each tube (12.5 g potassium persulfate to volume in a 250 ml volumetric, adjust recipe depending on sample size).

3) Tightly cap the tubes with acid washed linerless caps and arrange in autoclavable racks.

4) With remaining persulfate, make up a 1:5 mixture of potassium persulfate to di in 1000 ml flasks (80 ml persulfate to 400 ml di). Two flasks should be enough for 1 run of 90 samples or 2 smaller runs. This is to be used in place of di in the sampler wash line. Be sure to leave plenty of head space (1/2 of flask) so oxidant does not overflow in autoclave. Use foil and autoclave tape to seal the flasks.

5) Place racks with tubes and sealed flasks in metal pans in autoclave.

6) Fill the pans with a di water bath to the depth of the solution in the flasks and tubes. This will minimize leaks due to rapid changes in temperature and pressure.

7) Turn on the power to the autoclave and allow to warm up for ~10 min.

8) Press the 4 button twice to start the autoclave cycle.

9) Once cycle is finished, crack door 1 inch and allow slow cool for 10 min.

10) Use caution when taking samples and flasks from autoclave as solutions are extremely hot. Allow samples to cool to room temperature for immediate run or place in cooler and run within 24 hours.

11) Follow same procedures to run samples on AlpKem as for Orthophosphate (only difference is instead of di for sampler wash use 1:5 persulfate solution from autoclave.)

Schimadzu Procedures

For the analysis of non-purgeable organic carbon (NPOC) and total nitrogen (TN) in stream, lake and precipitation waters.

a. Introduction

The Schimadzu TOC-V instrument measures the amount of total carbon (TC), inorganic carbon (IC) and total organic carbon (TOC) in water. Non-purgeable organic carbon (NPOC) can also be measured when the POC accessory is installed. "Oxidative combustion-infrared analysis" is a widely used TOC measurement method that has been adopted by the JIS and other international standards. By installing the optional TN unit, total water-borne nitrogen (TN) can be measured, using the principles of "oxidative combustion-chemiluminescence." As LVWS water samples are filtered prior to analysis, the resultant concentrations are operationally defined as dissolved organic carbon (DOC) and total dissolved nitrogen (TDN).

Methods can be found in: JIS K-0102 "Industrial Waste Water Testing", JIS K-0551: "Total organic carbon (TOC) testing methods for ultra-pure water", U.S. Pharmacopoeia 23, EPA 415.1, EPA 9060A, ASTM D2575, Standard Methods for Examination of Water and Waste Water 5310B. Refer to the Schimadzu user manual in A256.

b. Equipment and Supplies (located in room A256)

- Schimadzu TC TN Auto-Analyzer
- Filtered/acidified DOC/DON/DOP samples
- Calibration standard (6 ppm C / 2 ppm N)
- Check sample (3 ppm C / 1 ppm N)
- 2 M HCl
- 40 ml clear sampler vials
- Parafilm

c. Preparation

1) Allow samples and C and N stocks to reach room temperature by setting on counter while setting instrument up.

2) Open the two valves on the air tank (main valve to tank and the on/off valve connected to the blue air line which goes to instrument.)

3) Turn on the power to the instrument (bottom right corner).

4) Load the correct template file (consult the lab manager for the correct template file) and save as a new file with today's date (e.g., lvws_2010Mar18). Connect the instrument to the PC and allow to warm-up by clicking on the lightning bolt button.

5) Make up fresh calibration standard, check sample and carbonate removal check sample:

Calibration Standard (6 ppm C/2 ppm N)	**Check Sample (3 ppm C/1 ppm N)**
In 500 ml volumetric flask add: 3 ml organic C 1 ml N 20 ml 2M HCl And fill to meniscus with DI	In 1000 ml volumetric flask add: 3 ml organic C 1 ml N 40 ml 2M HCl And fill to meniscus with DI

d. Procedures

Use the following procedures when working from the template sample table:

1) Each row will be assigned a specific sample and must be named according to the sample site and date. For example "LO_NORM_031215" refers to a Loch.O normal sample from Dec. 15, 2003. Be sure to name all samples, washes and checks in the empty "sample name" column. The "vial" number is the sample table must correspond with the location of the sample in the sample tray.

2) Prepare the first twelve sample vials in the sample tray according the following table. These vials will be used to wash the machine and determine calibration curves.

Vial	Contents
1	DI
2	Calibration Std.
3	Calibration Std.
4	DI
5	Calibration Std.
6	Calibration Std.
7	Check Std.
8	DI
9	Check Std.
10	DI
11	Check Std.
12	DI

3) Fill the 40 ml sampler vials with at least 15 ml of analyte and note the position of each sample in the tray. Cover all sampler vials with parafilm to prevent evaporation and contamination. Along with field blanks, field dupes, and an occasional lab dupe (every

20 samples), be sure to insert a DI (wash) and check (3C/2N) after every 10 samples. DI wash and check samples should be included in the template file. End the run with a wash and check.

4) Fill a brown 480 ml acid washed borosilicate bottle with check solution and insert the small tube protruding from the front left corner of the Schimadzu into the bottle. This is vial number 0.

5) Once the sample table has been set-up and everything looks in place, start the machine by hitting the “start” button, which looks like a traffic light signal. Click “shut down instrument”, “standby”, “OK”, “external acid addition OFF”, and “start”.

Use the following procedures if you need to set up a sample table from scratch:

Setting up calibration curves

1) Set-up the calibration curves. “File”, “new”, “calibration curve” (for NPOC).

2) Choose “edit calibration points manually” and “dilution from a standard solution”.

3) Change “analysis” to NPOC, select “zero shift” off, “multiple injections” on, and name the curve according to the run date and particular curve. For example “031215NPOC”.

4) Choose NPOC “acid addition” to be 2.0%. There is no acid addition for the TN method.

5) Create NPOC calibration curve using “Edit Calibration Point Parameters” page. The “injection volume” should be changed to 150 µl. From top to bottom, standards should read 6, 3, 1.5, 0.75, 0. This is accomplished by clicking “add”, typing “0” in the “Standard Solution Concentration” box, “1” in the “Auto Dilution” box, and “OK”. Next, type “6” in the “Standard Solution Concentration” box, “1” in the “Auto Dilution” box, and “OK”. Continue this process using 6 as the “Standard Solution Concentration” and by increasing the “Auto Dilution” box number to dilute the four top standards to the ones listed above.

6) Select to use default settings and **do not** enable history log.

7) Repeat the above steps for the TN curve. The “injection volume” should read 150 µl. From top to bottom, TN standards should read 0, 0.25, 0.5, 1, 2. This is accomplished by clicking “add”, typing “1” in the “Standard Solution Concentration” box, “1” in the “Auto Dilution” box, and “OK”. Next, type “2” in the “Standard Solution Concentration” box, “2” in the “Auto Dilution” box, and “OK”. Continue this process using 2 as the “Standard Solution Concentration” and by increasing the “Auto Dilution” box number to dilute the 1 top standard to the concentrations listed above. This set-up allows the instrument to pull 6 and 3 for NPOC from vial #2, 1.5 and 0.75

for NPOC from vial #3, 0 and 0 for NPOC and TN from vial #4, 0.25 and 0.5 for TN from vial #5, and 1 and 2 for TN from vial #6.

Setting up method

1) Select “file”, “new”, “method”.

2) For “analysis” choose “NPOC/TN” and name the method. For example “LVS031215” for stream samples or “LVPPT031215” for precipitation samples.

3) Browse for the NPOC calibration curve you just created and open.

4) You can go through the “injection parameters wizard” or choose to skip this option. **Do not** check “USEPA” or “History Log”.

5) Repeat these steps to locate and select the TN calibration curve.

Setting up sample table

1) From blank sample table with cursor on row 1, select “insert”, “auto generate”, choose method you just created, select 3 samples for the “number of samples” box, make sure “start vial” is 1, change sample name to “DI”, and click “next”.

2) Click “next” on the “Perform Calibrations” page and “Insert Control Samples” page without changing any options.

3) From the “Sparging / Acid Addition” page, make sure to change the “vial” column related to the 3 DI samples to read “1”. This will tell the instrument to pull 3 samples of DI from vial #1. Click “OK”.

4) From the next blank row, choose “insert”, “calibration curve”, choose NPOC curve you recently set-up for the run, click “open” and change the vial numbers column to “2,2,3,3,4”. Click “OK”.

5) From the next blank row, choose “insert”, “calibration curve”, choose TN curve you recently set-up for the run, click “open” and change the vial numbers column to “4,5,5,6,6”. Click “OK”.

6) From the next blank row, choose “insert”, “auto generate”, select the method you just created and add the number of samples you will run and the “start vial” number. Clear the “sample name” field and click “OK”.

7) Each row will be assigned a specific sample and must be named according to the sample site and date. For example “LO031215” refers to Loch.O from Dec. 15, 2003. Be sure to name all samples, washes and checks in the empty “sample name” column.

8) Along with field blanks, field dupes, and an occasional lab dupe (every 20 samples), be sure to insert a di (wash) and check (3 ppm C/1 ppm N) after every 10 samples. Also be sure to end the run with a wash and check.

9) Once the sample table has been set-up and everything looks in place, hit the “start” button, which looks like a traffic light signal. Be sure to check the “external acid addition” **off**.

LECO CN Analyzer

a. Introduction

The LECO TruSpec CN carbon and nitrogen analyzer is a non-dispersive, infrared, microcomputer based instrument, designed to measure the carbon and nitrogen content in a wide variety of organic compounds. For LVWS purposes, the LECO is used for the analysis of percent (%) carbon (C) and nitrogen (N) on finely ground soil and plant samples.

b. Equipment and Supplies (located in Rm. A253).

- LECO TruSpec CN analyzer
- plant/soil samples (ground and stored in well-labeled scintillation vials)
- ball mill soil/tissue grinder (Room A127A inside Room A121)
- microbalance (to weigh samples)
- foil for analyzer cups
- blanks and standards (i.e., Mixed Meadow Grass (plant) or Sidhigh (soil))

c. Preparation

1) Separate plant or soil samples by site and date.

2) Grind oven dried samples to a fine powder using the ball mill grinder for soil or Wiley mill for plants in downstairs lab.

3) Place each ground sample in a well-labeled scintillation vial and store according to date, site, and treatment.

e. Procedures

1) Check gases:
Compressed Air should read 40 psi.
Helium and Oxygen should read 35 psi on the left gage. Consult a Lab Manager if the pressure on the Oxygen or Helium tanks is less than 300-400 psi (regulator closest to the tank).

2) **Check furnace temperature:** the temperature should be close to 950°C.

3) **Check to see if crucible is in the furnace** (same procedure used to change crucible)
   - Carefully lift off sampler carousel and gently set aside
   - Loosen the 3 main screws on the loading head – *very carefully* remove the loading head and set it on its side on top of the machine

- Screw the lance extractor tool into the lance tube, *carefully* remove the tube and place in the tray next to the machine. THE LACE TUBE IS EXTREMELY HOT!!!
- Replace crucible with tongs if necessary – don't forget to reset the counter if you replace the crucible
- Replace the lance tube, use a Kimwipe to clean any grit from the O-ring
- Replace the loading head by *very carefully* lining up the connecting pins and stems. NOTE: you should hear compressed gas is seated properly, this is normal and will stop once the loading head is tightened
- Carefully push down on the loading head, and then tighten the bottom right hand screw first until you feel resistance. Tighten the other two screws until you feel resistance, and then fully tighten each screw by rotating among the screws.

4) **Balance:**
Turn on the balance. Use the $\frac{42\ g}{205\ g}$ button to switch display to 0.0000 g. Press the **CAL** button to calibrate the balance.

5) **Counters:**
- Select **Configuration** from the menu bar to access the counter window, then click on **Counters.** The *Warn* column indicates the maximum number of samples that should be run before that item needs replacement or maintenance. The *Count* column will increase by 1 each time you run a blank, standard, or sample. Make sure that the *Count* column will not exceed the *Warn* value during your run.
- NOTE: *It is especially important that the **crucible** Count is not exceeded. Overfilling the crucible can result in expensive damage to the machine and considerable downtime. Please consult the Lab Manager if you are uncertain if the crucible needs to be replaced.*
- **Resetting the Counters (if necessary) –** After changing the crucible or performing other maintenance, please reset the *Count* to 0. To access this window, select **Maintenance**, then click **Login**, select the item that maintenance has been performed on, click **OK.** Click **Yes** if maintenance was performed. Click **No** if maintenance was not performed. Return to the Counter window to ensure the proper item was reset.

6) **Leak Checks:**
- Before running any samples it's in your best interest to check for leaks. Select **Diagnostics** from the main menu, then click **Leak Check.**
- Select **Whole O2**, click *Start*, wait until *Passed* or *Failed* appears in the Results column. If *Passed* appears, be sure the value is less than 2.0 mbars.
- Select **Whole He**, click *Start*, wait until *Passed* or *Failed* appears in the Results column. If *Passed* appears, be sure the value is less than 2.0 mbars.
- Contact a Lab Manager if either of the leak checks fails.

7) **Calibration:**

– **Initial Blanks –** First, make sure the next available (i.e., empty) cell on the spreadsheet has been selected. Select **Samples** from the main menu, then click **Login.** From the *Sample* window, use the drop down arrow next to *Sample Name* to select **Blank**. The *Mass* must be 1.0000 (which is the default), select **4** repetitions. Using the drop down arrow at the end of the *Method* line, select the appropriate ***Method.***

Material	Method	Standard/ Check	Weight Range (g)	Carbon Range (%)	Nitrogen Range (%)
Plant	PLANT 10ML LOOP	Mixed Grass	0.1000 - 0.1100	10.8 – 100	0.62 – 8.5
Soil	SOIL 10ML LOOP	Sidhigh	0.2000 - 0.2100	0.62 – 5.3	0.065 – 0.55

Note: The organic soils collected for the ongoing fertilization experiment in Loch Vale are high in both Carbon and Nitrogen, and are therefore analyzed with the PLANT 10ML LOOP method.

Enter your name or supervisor's name on the Operator line. The other *Attributes* should be left blank in most cases. Click **OK** to add the Blanks to the sample list. Click **Cancel** if necessary to close the *Sample* window. Four blanks should have been added to the sample table. To help keep track of your samples, change the numbers in the *Location* column to match the numbers on the sample carousel.

– **Initial Standards** – Select **Samples** from the main menu, and click **Login Drift Samples.** Select the appropriate method using the drop down arrow, and the associated calibration standard will appear in the *Drift Standards* box, along with the weight range (your standards must be within this range, see table above). Select **4** repetitions. The standards appear after the blanks on the spreadsheet. *Note: The weights in the Mass column for the standards will appear with a ~ symbol, this is just to remind you of the approximate weight you should use. The actual weights will replace the reminder weights as you enter them using the* **Print** *button on the balance.*

– To start the analysis, click the **Analyze** button of press **F5** to start analyzing the blanks and standards. To halt the machine after the blanks (in order to run the blank calibration), first left click on the row containing the first standard, then right-click, select **Pause** from the **Samples** menu, choose **Manually** from the drop down list. A red stop sign should appear next to the first standard. This will stop the analysis temporarily after the last blank sample.

– **Blank Calibration –** If the carbon values vary by less than 0.04 and the nitrogen values vary by less than 0.02, you may proceed with the calibration. If not, continue running blanks until the values stabilize. After the blanks have finished running (and the machine is in pause mode), choose the blanks you wish to use for the blank calibration by highlighting the appropriate group of blanks (left click and drag, or left-click and use control to select individual blanks). Select **Configuration** from the main menu, click **Blank.** From the *General Blank* window, *Include* both Nitrogen and

Carbon if necessary, using the *Include/Exclude* button. Click **Ok.** After the blank calibration, restart the analyses by clicking the **Analyze** button or **F5.**

- **Standard Calibration –** Select the appropriate standards you want to use in calibration. Select **Configuration** from the main menu, click **Drift,** from the next menu click **Drift** again. *Include* both Nitrogen and Carbon if necessary, using the *Include/Exclude* button. Click **OK.**

8) **Running Samples:**
   - Run one blank and one standard immediately after calibration to ensure the values are correct. If the values are out of range, run a few more blanks and/or standards, checking to see if they stabilize in the correct range. Contact the Lab Manager if values are still questionable, and enter a **Pause** at the beginning and end of the calibration check.

Standard	**Carbon (± 5%)**	**Nitrogen (± 5%)**
Mixed Grass	43.78 (41.59 – 45.97)	2.53 (2.40 – 2.65)
Sidhigh	2.589 (2.460 – 2.718)	0.269 (0.255 – 0.282)

   - Enter the sample name in the **Name** column. Weigh the appropriate amount of sample for the method and press the **Print** button on the balance, the sample weight should appear next to the name you just entered.
   - Run a blank and/or standard every 10 samples to ensure the machine is running properly.
   - At the end of your run, **DO NOT** shut off the machine and **DO NOT** close the software. Change the crucible if necessary, select and download your data, and fill out the log book.

9) **Exporting Data:**
   Select the sample you want to download by clicking and dragging in the row column. Go to **Samples** on the main menu bar and click on **Text Export Data.** Enter a filename and the appropriate drive, click **Save.**

10) **Printing Data:**
   Select the rows containing the samples you want to print. Select **Samples** on the main menu bar, click **Print,** click **OK.**

Acid-washing Procedures

a. Introduction

All plastic bottles and tubing used for collection, processing, and storage of water samples must be cleaned in 10% HCl acid prior to use. This cleaning removes any contaminants adhered to the walls of bottles that can affect sample quality.

b. Equipment and Supplies

Acid baths are located in NREL A254, and the plastic bottles and caps are in NREL A235

- Plastic bottles and caps, various sizes from 2 L to 60 ml
- 10% (by volume) HCl acid bath (see recipe in Appendix E)
- DI water
- Plastic dishpans
- Lab coat, goggles, rubber gloves, and closed toe shoes
- Sodium bicarbonate to neutralize acid spills

c. Estimated time to complete procedure

Allow one-half hour for the first 12 bottles, and 15 minutes for each additional set of 12 bottles

d. Preparation

1) Make sure you are aware of safety procedures for dealing with strong acids. The NREL lab manager must certify you for safe laboratory practices.

2) Bottles to be washed should have no visible dirt or oil. If necessary, wash them in hot water and Alconox detergent before acid cleaning; making sure all detergent has been thoroughly rinsed off with tap water. The bottles and caps should have a final DI rinse before entering acid bath.

3) Remove any tape or labels on bottles before placing in the acid bath. The acid will alter the labels, causing them to fragment and dirty the acid bath, as well as make them much harder to remove from the bottle.

4) The acid baths are good for about six months depending on how heavily they are used. Replace the acid baths if there is substantial particulate material in it or if the acidity has decreased. Acid must be neutralized before disposal. Consult a lab manager for assistance neutralizing the acid, after which is can be poured down the drain with copious amounts of tap water.

e. Procedures

1) Submerge bottles and caps (except for the green Teflon lined caps used for amber borosilicate bottles - these caps get soaked and rinsed with DI water only) in acid bath. Be sure bottles have been DI rinsed before entering bath.

2) Bottles must be completely immersed in the acid bath for at least three hours prior to removal and DI rinses. Wear all protective gear available when submerging bottles, being careful not to splash or drip acid outside the acid bath. Sodium bicarbonate and water can be used to neutralize and clean up any acid spills.

3) After gently pouring acid back into bath, place bottles and caps into a plastic dishpan. Again, be careful not to spill or drip acid. Rinse all outside surfaces briefly with DI water.

4) Place the caps into a large mouthed jar or beaker and fill, shake, and dump DI seven times to rinse caps. Set aside in jar filled with DI until needed.

5) Fill each bottle of 1/3 full with DI water, shake, and empty. Repeat 6X to remove any residual acid.

6) After seventh rinse, fill bottle with DI water and screw on rinsed cap of appropriate size.

7) Store washed bottles under the lab bench in NREL A235.

8) Check conductivity of water in the bottles before use (see page 34 for instructions on how to measure conductivity). Rewash any bottle with a conductivity value >2 $\mu S\ cm^{-1}$.

f. Be Aware

Protective clothing must be worn when working with strong acid solutions.

Bottles may soak longer than three hours, but should not remain in the acid bath for longer than three days. It is easiest to fill acid bath with fresh bottles after rinsing a batch, then you are ready to rinse another set when needed.

Dirty bottles are returned to us from the analytical laboratory, they are stored in NREL A254 until needed.

SHIPPING SAMPLES

Surface Water Samples

a. Introduction

Deliver surface water samples weekly to the USFS Rocky Mountain Research Station water chemistry analytical laboratory (417-2394; piersond@usgs.gov) for chemical analysis within 24 hours of collection. When Wednesday's lab work is complete, simply walk the R.U. and FU samples over to the USFS building and place them in the appropriate refrigerator.

DOC samples were previously delivered to George Aiken (303-541-3057) at the USGS-WRD in Boulder every other week (3215 Marine Street, Suite E-127). DOC is now analyzed at NREL, so DOC samples are no longer shipped.

b. Equipment and Supplies

Equipment and supplies are located in NREL A223 and NREL A235. The following supplies are used in the shipment of surface water samples, as necessary.

- Small plastic cooler
- Ice pack
- Packing material

c. Estimated time to complete procedure

Allow 15-30 minutes for delivering samples to the RMRS laboratory.

d. Preparation

Samples must be filtered and stored in the walk-in cooler until ready for transport.

e. Procedures

1) Place one or two "blue ice" packets in the cooler.

2) Make sure all sample bottles are sealed tightly.

3) Pack samples into cooler.

4) Deliver samples to the USFS RMRS laboratory and place them in the "Sample Drop Off" location in the walk-in-cooler.

Precipitation Samples

a. Introduction

A 1-liter sample bottle is sent to the Central Analytical Laboratory (CAL) of the National Atmospheric Deposition Program (NADP) for chemical analysis of the precipitation sample. Following is a brief synopsis of procedures. Detailed instructions are given in the NADP/NTN Site Operation Manual (Dosset and Bowersox 1999). The person responsible for shipping the precipitation sample should be certified as an NADP site operator.

Precipitation samples are weighed in the bucket. Subtract lid and bucket weight (written on each bucket and lid) from the total weight of lid, bucket, and sample to find the sample volume. Decant the sample from the bucket into a clean NADP 1-liter HDPE Nalgene bottle and send to the NADP Central Analytical Laboratory (CAL) in Champaign, Illinois for complete chemical analysis.

b. Equipment and Supplies (NREL A223 and NREL A235)

- 1 liter bottle for precipitation sample
- Precipitation sample in white plastic bucket with snap on lid
- Card board box for 1 liter sample bottle
- Field Observer Report Form (FORF)

c. Estimated time to complete the procedure

Allow 30 minutes to process sample, fill out FORF, and ship the bottle.

d. Preparation

The sample, bucket, and lid must be weighed on the balance in NREL A231. All precipitation samples of any volume must be transferred to the 1 liter sample bottle. See the NADP field procedures for further details.

e. Procedures

1) Enter all necessary information in the Field Observer Report Form. Remove the pink copy, which is added to the NADP Field Forms notebook in NREL A235.

2) Place used bucket back into plastic bag, label as "Used NTN Sampling Material", and place into used 6-pack (i.e., large boxes located in A223). Once six used buckets and lids have accumulated, the "used" 6-pack should be shipped to CAL.

3) Place the white and yellow copies of the FORF in the sample box with the 1 liter sample bottle. Seal the box with packing tape, which can be ordered from NADP by requesting additional supplies on the FORF, and write you name above the return address. You must also include the appropriate CSU account number on the box in the upper right hand corner. Consult the Program Manager or Principal Investigator if you do not know the correct account number.

4) For shipping used 6-pack back to CAL, after checking that there are six used buckets and lids in the large card board box, seal the box and place a NADP shipping label on the box lid. Write your name and appropriate CSU account number on the shipping label.

5) All boxes to be shipped should be placed in or near the out-going mail box near the NREL elevator.

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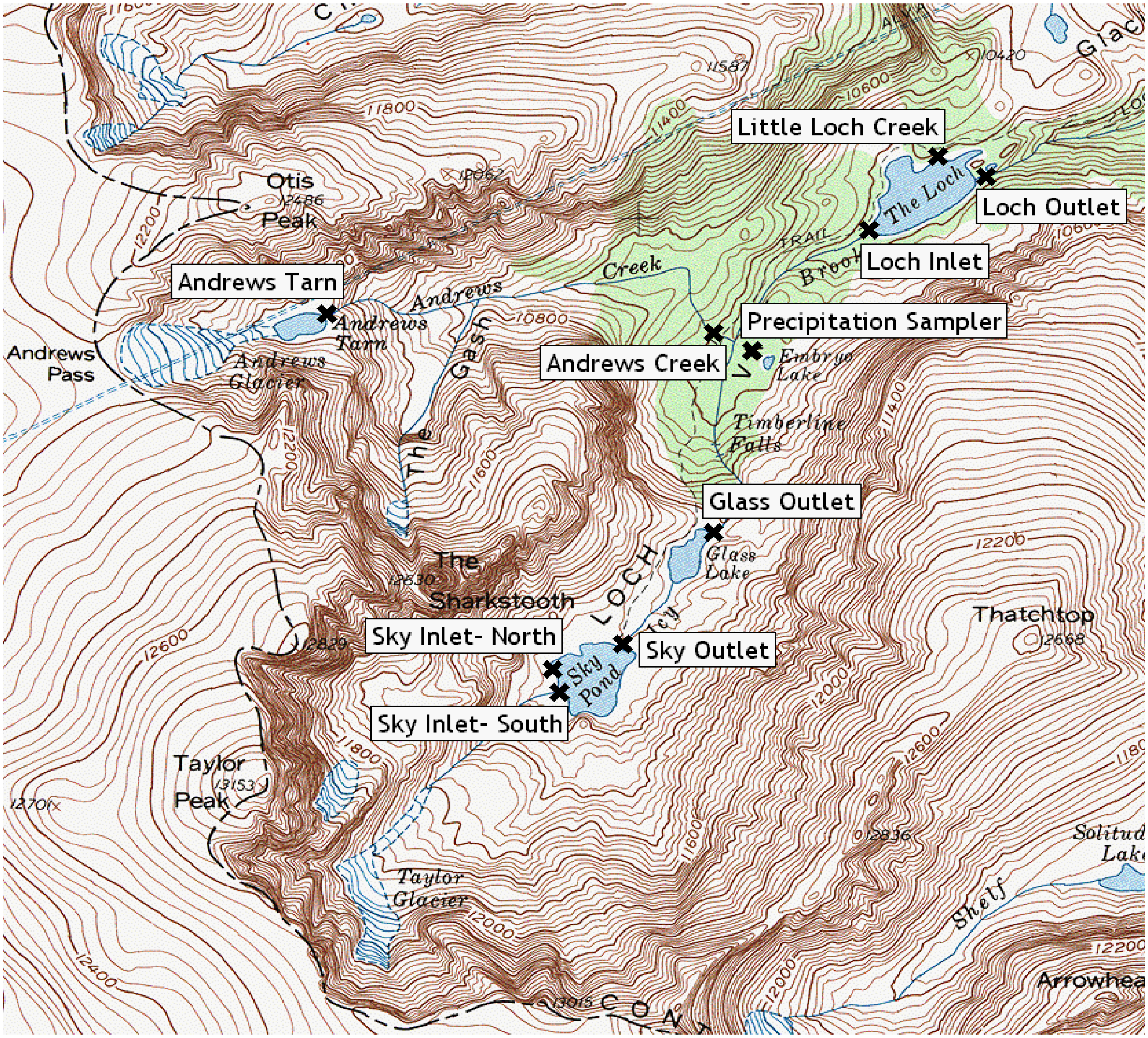
APPENDICES

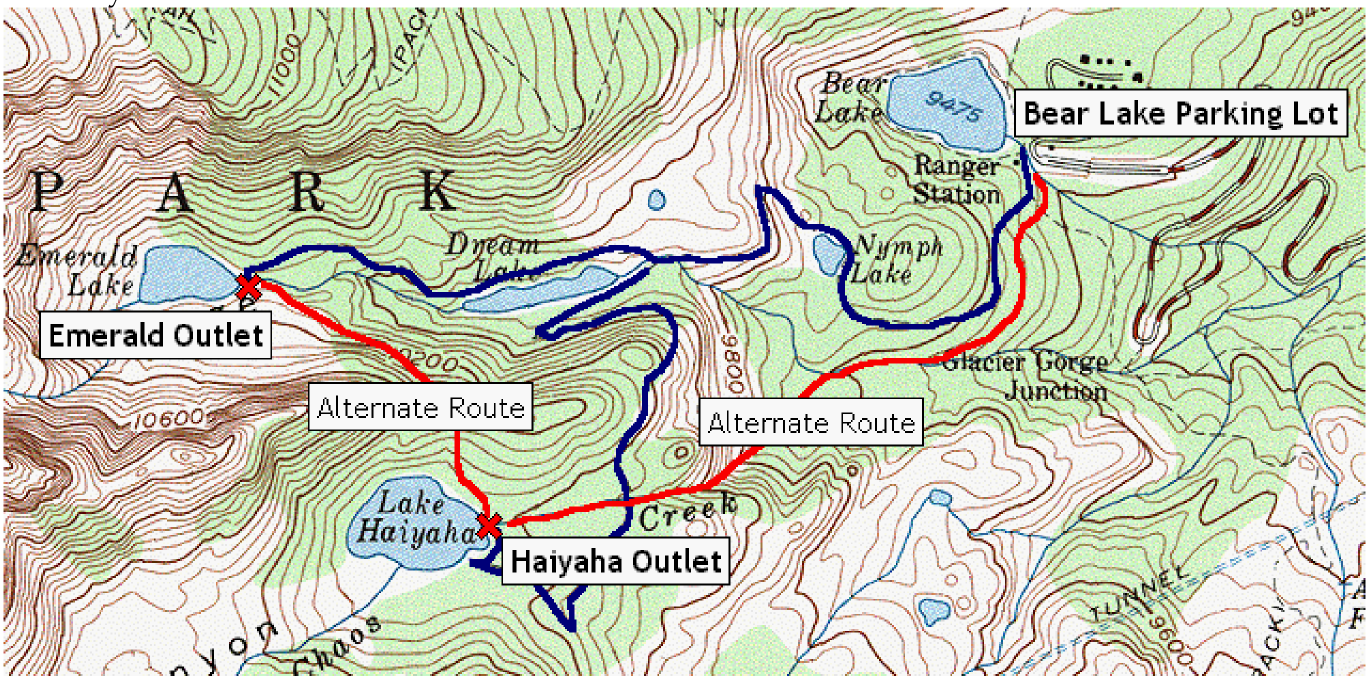
Appendix A: Sampling Locations and Trails

For LVWS:



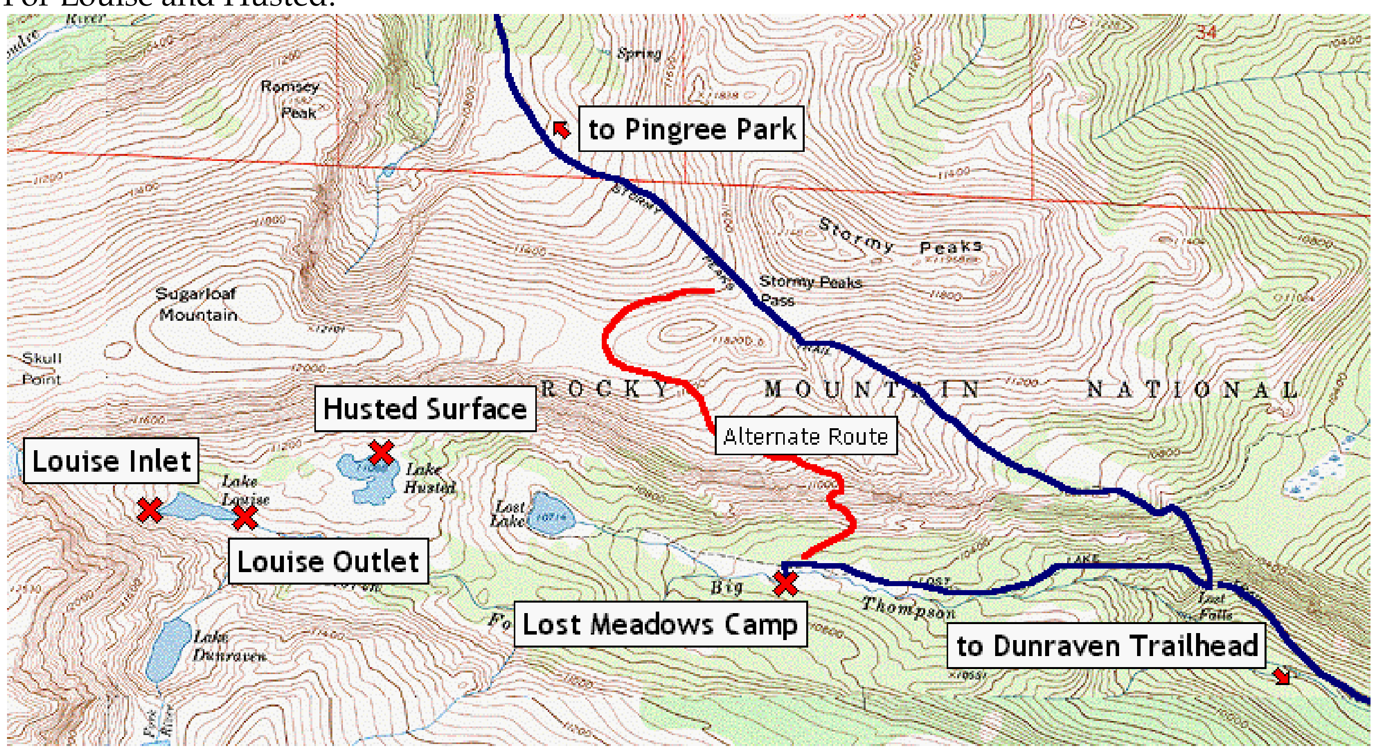
Nearly all sample points are on or near the trail. Consult with supervisor for exact locations.

For Haiyaha and Emerald:

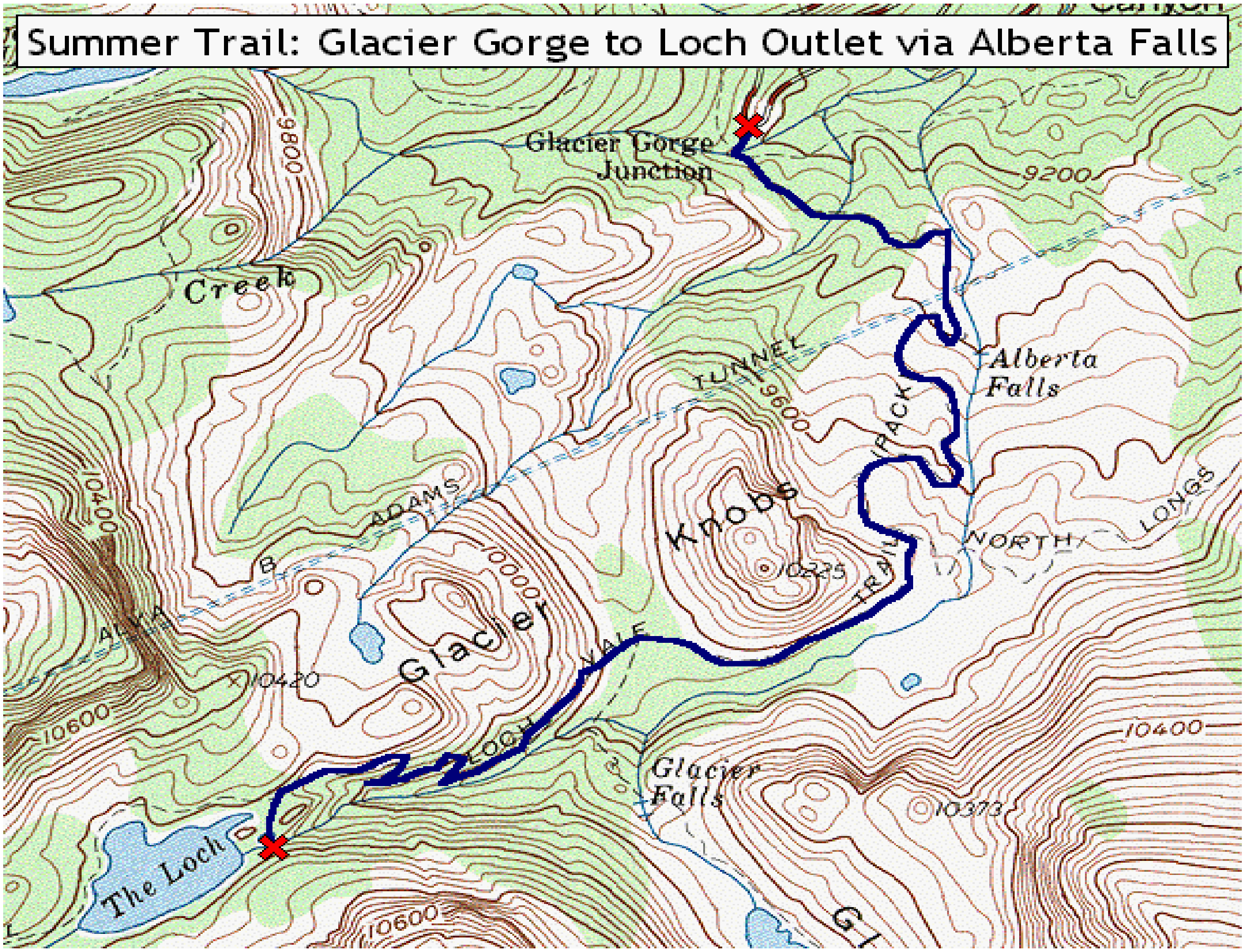


Alternate routes are off trail, and are very rugged. Travel with caution.

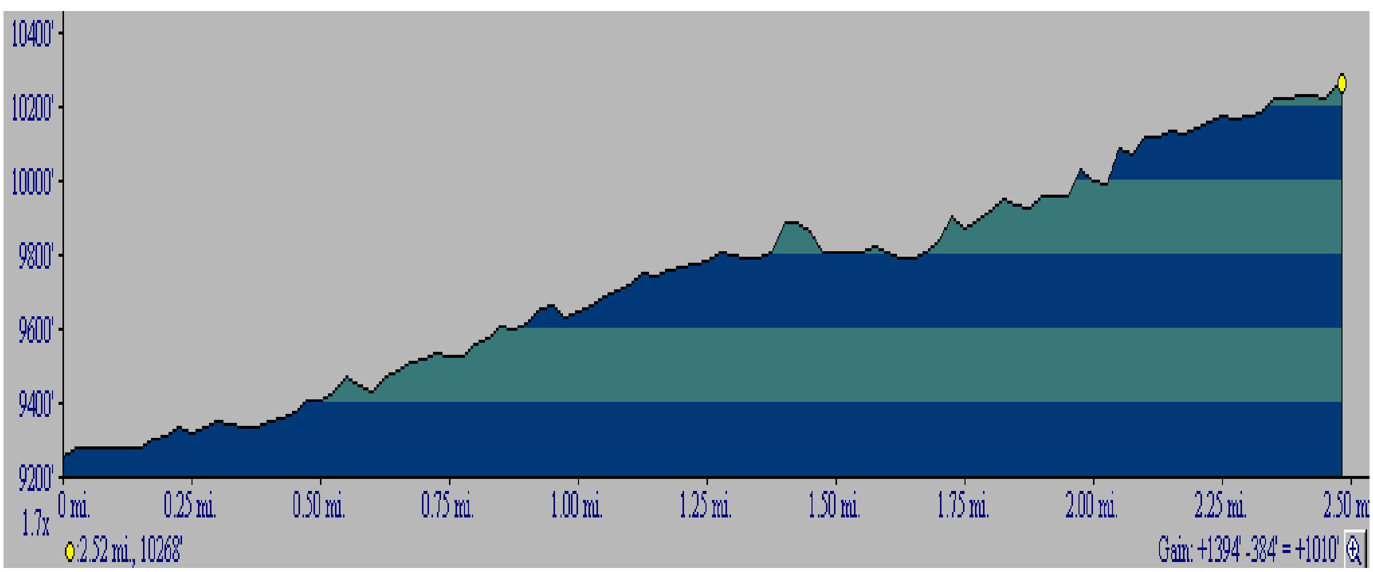
For Louise and Husted:



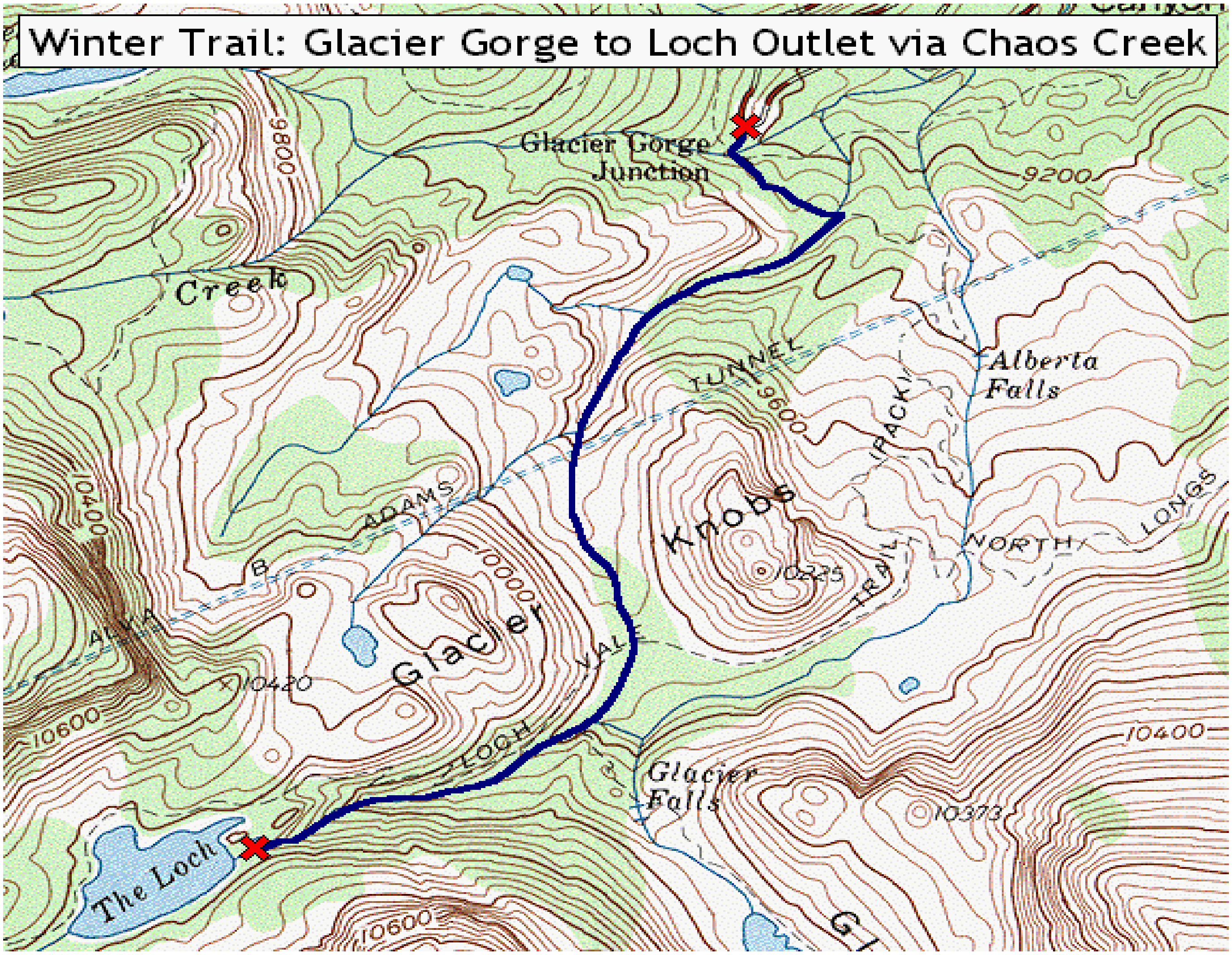
Alternate route involves a very steep descent into the Lost Meadow Basin. Travel with caution.

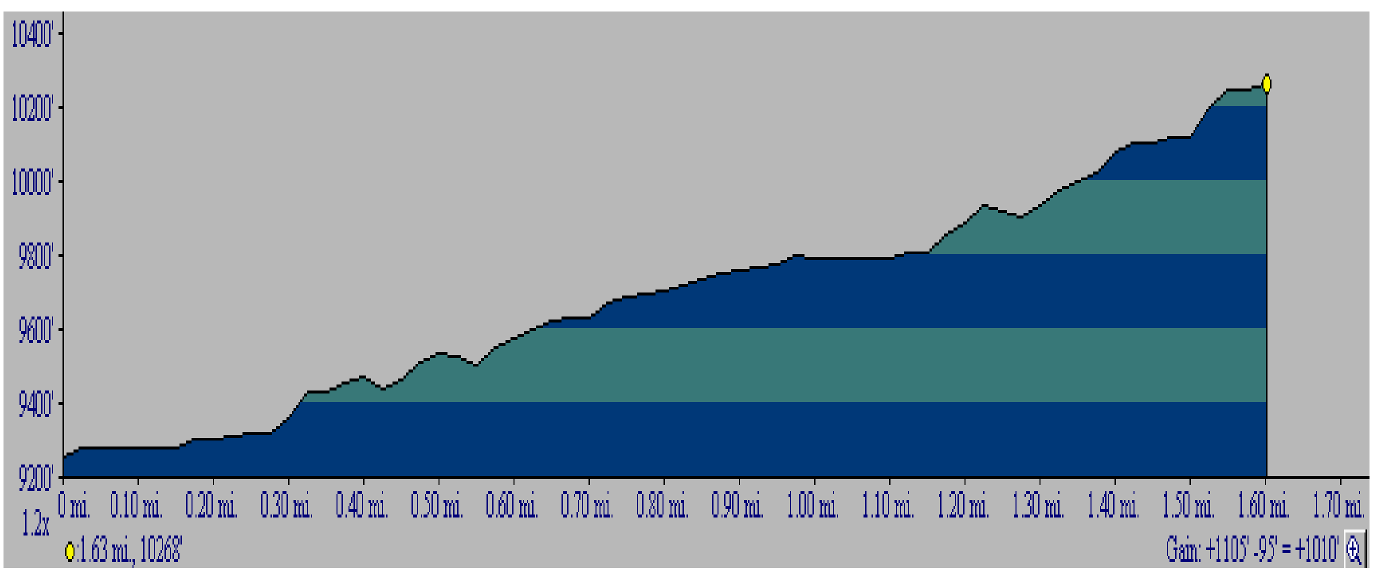


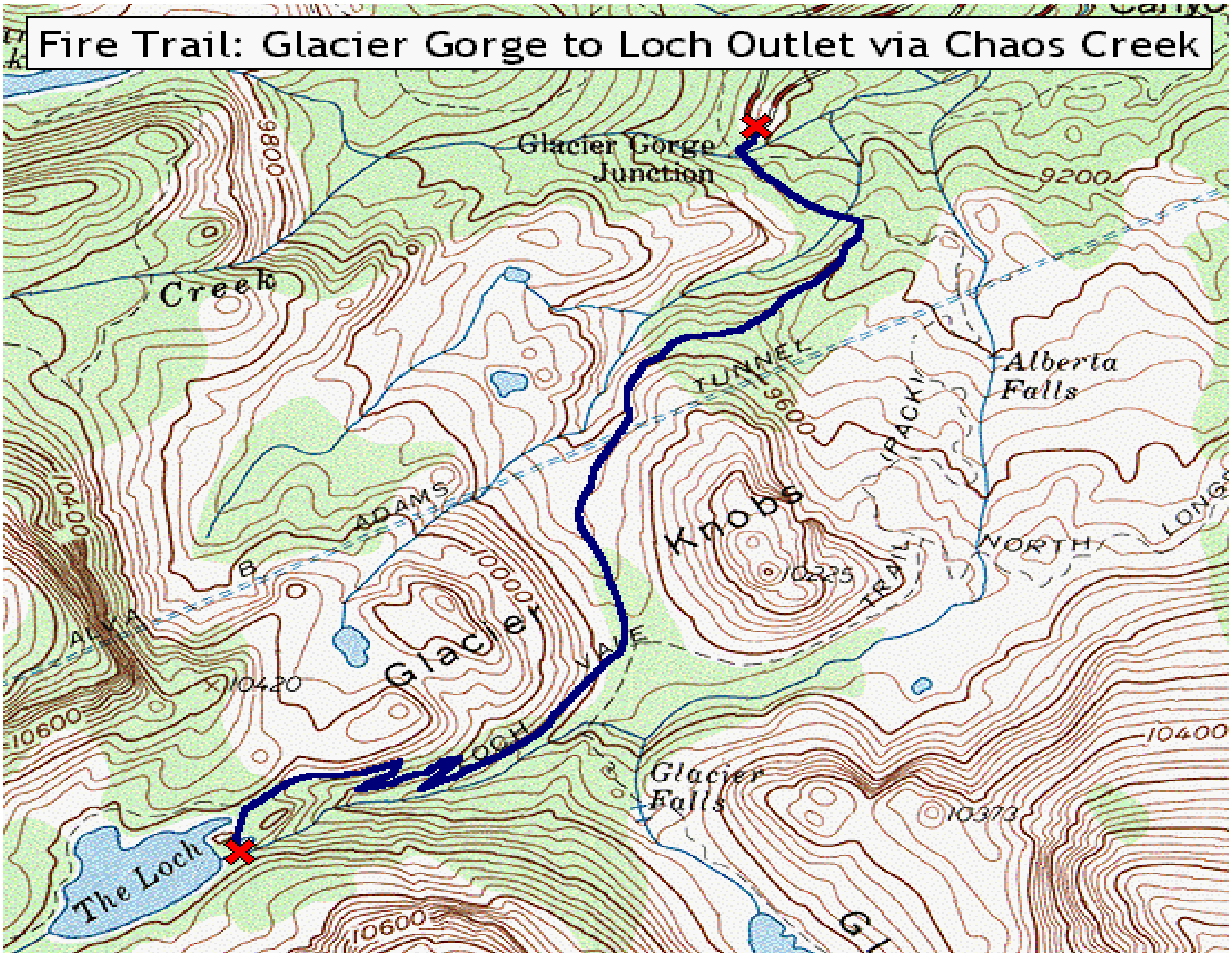
Summer Trail: Glacier Gorge to Loch Outlet via Alberta Falls
Glacier Gorge Junction
9200
Creek
TUNNEL
9600
Alberta Falls
PACK
ADAMS
Knobs
10225
NORTH
LONGS
TRAIL
10420
Glacier
VALE
10600
LOCH
10400
Glacier Falls
10373
The Loch
10600

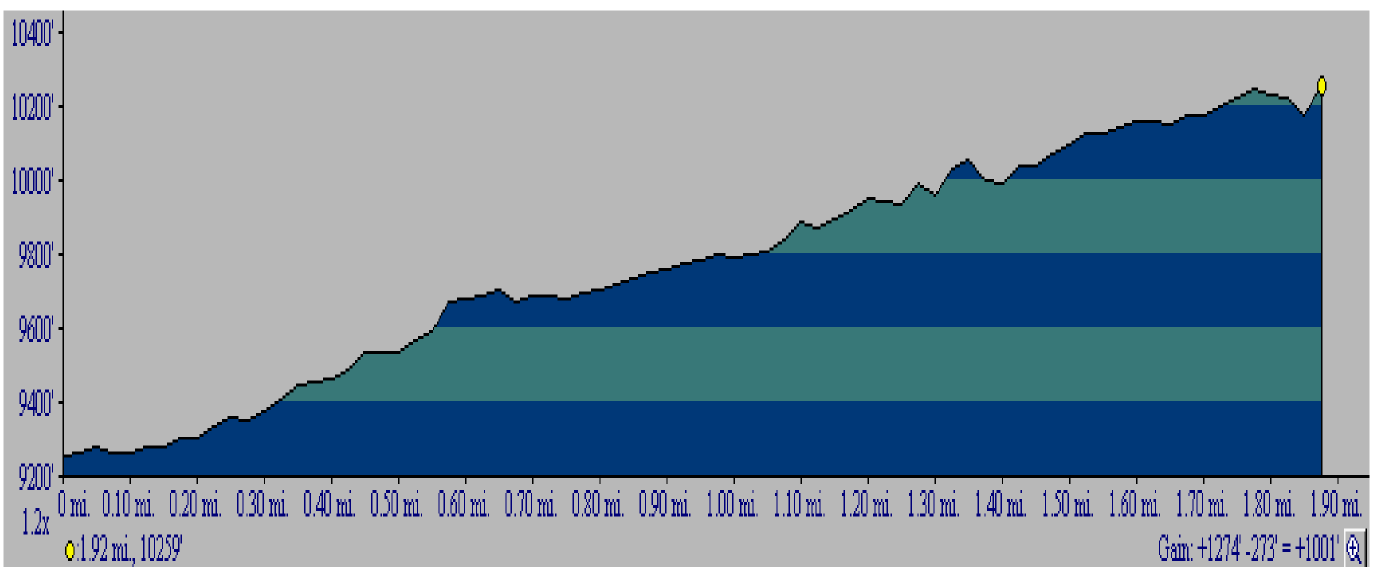


10400'
10200'
10000'
9800'
9600'
9400'
9200'
1.7x
0 mi.
0.25 mi.
0.50 mi.
0.75 mi.
1.00 mi.
1.25 mi.
1.50 mi.
1.75 mi.
2.00 mi.
2.25 mi.
2.52 mi., 10268'
Gain: +1394' -384' = +1010'


Winter Trail: Glacier Gorge to Loch Outlet via Chaos Creek
Glacier Gorge Junction
Creek
9200
9800
9600
TUNNEL
Alberta Falls
Knobs
10225
TRAIL
NORTH
LONGS
ADAMS
Glacier
VALE
LOCH
ALVA
10420
10600
10400
Glacier Falls
10373
The Loch


10400'
10200'
10000'
9800'
9600'
9400'
9200'
1.2x
0 mi. 0.10 mi. 0.20 mi. 0.30 mi. 0.40 mi. 0.50 mi. 0.60 mi. 0.70 mi. 0.80 mi. 0.90 mi. 1.00 mi. 1.10 mi. 1.20 mi. 1.30 mi. 1.40 mi. 1.50 mi. 1.60 mi. 1.70 mi.
1.63 mi., 10268'
Gain: +1105' -95' = +1010'


Fire Trail: Glacier Gorge to Loch Outlet via Chaos Creek
Glacier Gorge
Junction
9200
9800
Creek
TUNNEL
9600
(PACK)
Alberta
Falls
ADAMS
Knobs
10225
TRAIL
NORTH
LONG
10400
10000
B
Glacier
ALVA
VALE
10420
10600
LOCH
Glacier
Falls
10400
10373
The Loch
10600


10400'
10200'
10000'
9800'
9600'
9400'
9200'
1.2x
0 mi. 0.10 mi. 0.20 mi. 0.30 mi. 0.40 mi. 0.50 mi. 0.60 mi. 0.70 mi. 0.80 mi. 0.90 mi. 1.00 mi. 1.10 mi. 1.20 mi. 1.30 mi. 1.40 mi. 1.50 mi. 1.60 mi. 1.70 mi. 1.80 mi. 1.90 mi.
1.92 mi., 10259'
Gain: +1274' -273' = +1001'

Appendix B: Sampling Schedule

Table B1. LVWS surface water sampling schedule for active sites.

Name	Sampling Frequency	Analyses
Andrews Creek	1/month, summer	RU, FU, DOC, DOP, DON
Emerald Outlet	1/year, fall	RU, FU, DOC, DOP, DON
Haiyaha Outlet	1/year, fall	RU, FU, DOC, DOP, DON
Husted Surface	1/year, fall	RU, FU, DOC, DOP, DON
Loch Outlet	1/week, year round	RU, FU, DOC, DOP, DON, Phyto*, Archive♣
Loch Inlet	1/month, summer	RU, FU, DOC, DOP, DON
Louise Inlet	1/year, fall	RU, FU, DOC, DOP, DON
Louise Outlet	1/year, fall	RU, FU, DOC, DOP, DON
Sky Outlet	1/month, summer	RU, FU, DOC, DOP, DON
Sky Inlet South	1/month, summer	RU, FU, DOC, DOP, DON

*Phytoplankton samples are collected once a month at the Loch Outlet.

♣Archive samples are collected once a month from the Loch Outlet. Archive samples are filtered with the peristaltic pump into brown 250 ml HDPE bottles and stored in the NREL freezer. The frozen archive samples are transferred from the NREL freezer to the LVWS chest freezer at the USGS Fort Collins Science Center (FORT) laboratory once a year.

Synoptic sampling occurs once a month during the summer, starting in June and ending in September. Synoptic sampling entails collecting surface water sample at Sky Inlet South, Sky Outlet, Andrews Creek, and Loch Inlet, in addition to the regular weekly sampling at the Loch Outlet.

A sampling schedule should be prepared by the Program Manager prior to the beginning of a new calendar year. Ten percent of the total number of samples collected must be quality assurance samples (i.e., DI field Blanks or field Duplicate samples). Around 80 samples (Normal, Blanks, and Duplicates) are collected yearly. To obtain 10% quality assurance, a Blank or Duplicate sample should be collected from the Loch Outlet every 6-7 weeks. QA sampling at the Loch Outlet should alternate between field Blanks and field Duplicates. Details of taking/processing these samples are found in Appendix C.

Table B2. Historical LVWS sampling sites that are no longer actively sampled.

Name	Sampling Frequency	Analyses
Andrews Tarn	1/year, summer	RU, FU, DOC
Andrews Tarn Inlet	1/year, summer	RU, FU, DOC
Emerald Inlet	Variable, summer	RU, FU, DOC
Emerald Hypolimnion	Variable, summer	RU, FU, DOC
Emerald Metalimnion	Variable, summer	RU, FU, DOC
Emerald Surface	Variable, summer	RU, FU, DOC
Glass Inlet	Variable, summer	RU, FU, DOC
Glass Hypolimnion	Variable, summer	RU, FU, DOC
Glass Metalimnion	Variable, summer	RU, FU, DOC
Glass Surface	Variable, summer	RU, FU, DOC
Glass Outlet	1/month, summer	RU, FU, DOC, DOP, DON
Haiyaha Hypolimnion	Variable, summer	RU, FU, DOC
Haiyaha Metalimnion	Variable, summer	RU, FU, DOC
Haiyaha Surface	Variable, summer	RU, FU, DOC
Husted Inlet	Variable, summer	RU, FU, DOC
Husted Hypolimnion	Variable, summer	RU, FU, DOC
Husted Metalimnion	Variable, summer	RU, FU, DOC
Husted Lake Surface	Variable, summer	RU, FU, DOC
Husted Outlet	Variable, summer	RU, FU, DOC
Little Loch Creek	1/month, summer	RU, FU, DOC, DOP, DON
Loch Hypolimnion	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto
Loch Metalimnion	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto
Loch Surface	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto
Louise Hypolimnion	Variable, summer	RU, FU, DOC
Louise Metalimnion	Variable, summer	RU, FU, DOC
Louise Surface	Variable, summer	RU, FU, DOC
Sky Inlet North	1/month, summer	RU, FU, DOC, DOP, DON
Sky Hypolimnion	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto
Sky Metalimnion	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto
Sky Surface	Bi-monthly, winter	RU, FU, DOC, DOP, DON, Phyto

Appendix C: Quality Assurance/Quality Control Samples

Blank samples are taken in the following manner:

1) When taking a blank sample, a normal Loch Outlet sample is taken at the same time. Prepare two normal bottle sets for the Loch Outlet in the lab.

2) On the day of sampling, fill a 1 liter acid-washed/rinsed plastic bottle with conductivity tested DI water from the lab. Conductivity of this water must be <2 $\mu S\ cm^{-1}$. Notify lab manager if it is not, and postpone the blank sample.

3) After taking normal Loch Outlet sample, rinse and fill each bottle in the blank sample set from the 1 liter DI water bottle using the same methods as described in the Surface Water Sampling section of the LVWS Methods Manual (only pouring DI into sample bottles instead of dipping into stream).

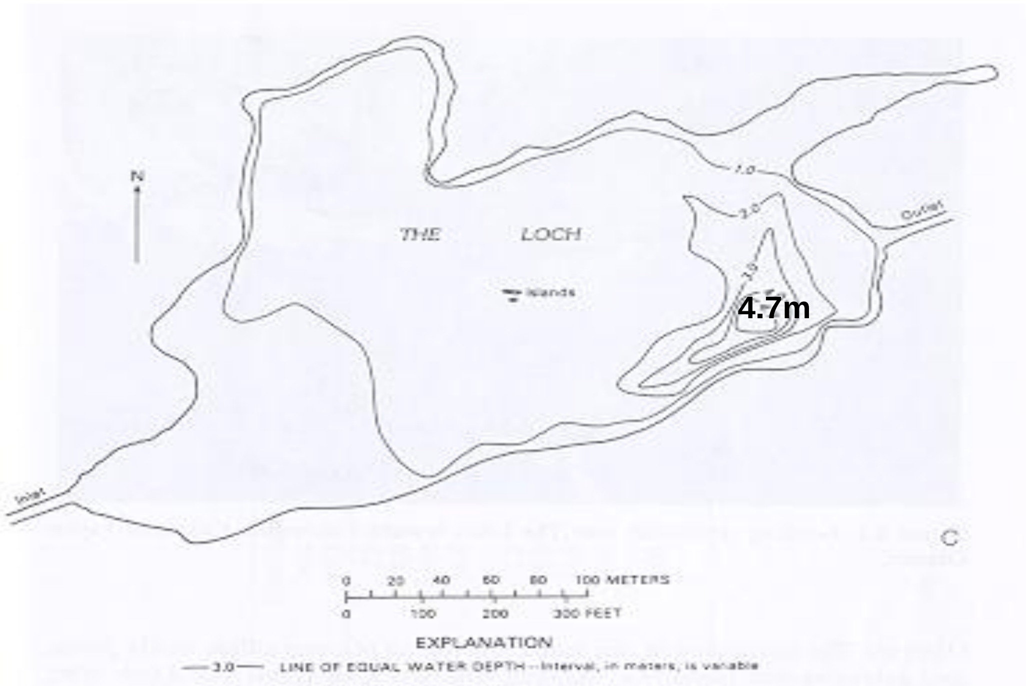
4) Process the sample just as you would a normal sample. Label the blank samples bottles with sample type "BLANK" instead of "NORM".

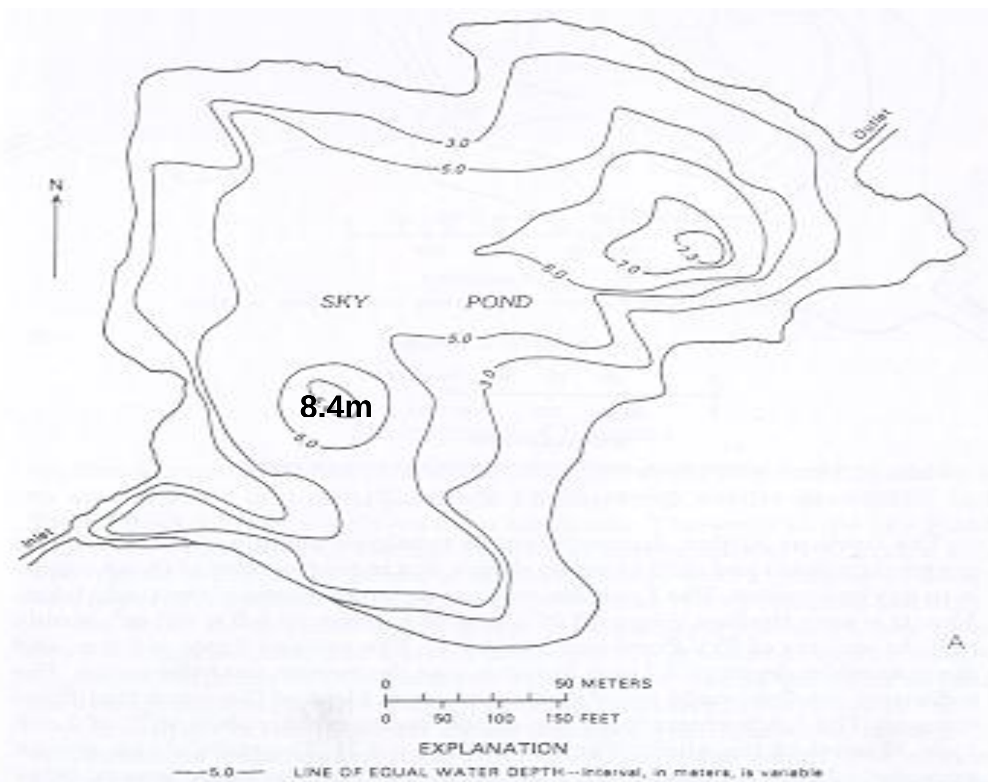
Duplicate samples are taken in the following manner:

1) A duplicate sample is simply that - a duplicate of a normal sample from the Loch Outlet. Take two identical sets of samples according to the methods detailed in the Surface Water Sampling section of the LVWS Methods Manual. Label one bottle set "NORM", and the other "DUPE".

5) Process the sample just as you would a normal sample. Label the bottles with sample type "DUPE" instead of "NORM".

Appendix D: Lake Bathymetry





Appendix E: Recipes

***Lugol's Fixative Solution*:** Dissolve 10 g I_2 (pure iodine; *caution*: toxic) and 20 g KI in 200 ml distilled water and 20 ml concentrated glacial acetic acid. Store in amber borosilicate bottle. Transport to field in 20 ml plastic vial.

Acid Baths: To make new acid baths, add one part concentrated HCl to nine parts water under a fume hood. Make sure you use safe procedures for working with strong acids (add acid to water, **NOT** water to acid!)

NADP Quality Control Check Sample (QC): pH = 4.9 ± 0.15; conductivity = 14 ± 2 µS cm^{-1}. To make this quality control check sample, get 0.5 liters of DI water stirring in a 1 liter volumetric flask on a stir plate. Slowly add 4.00 g NaCl and 0.78 ml HNO_3. Bring volume up to 1 liter. Dilute 1 ml of this solution to 1 liter. Freeze remainder of original liter for later use. This standard does not need to be refrigerated, but it should be stored in a dark cupboard and shaken before use.

NADP Specific Conductance Standard (SCS): pH = 5.62 ± 0.20; conductivity = 74 ± 1 µS cm^{-1}. To make this conductivity standard, first dry ~100 g of KCl crystals at 105°C for >5 hours. Then dissolve 93.2 g of the dried KCl in a 1 L volumetric flask to create a 1.25 M KCl stock solution. Dilute 0.40 ml of this stock solution to 1 L to make the 5.0×10^{-4} M KCL SCS.

***Alpkem Reagents*:**
Prepare all recipes in their written order.
When filtering is necessary, use 8.0 µm membrane filters located in NREL A256.

For NH_4 Channel

Manifold Startup Solution
Add 0.5 ml BRIJ or other surfactant to 1L DI

NH_4 Buffer
12 g NaOH
29 g Na Citrate
50 g disodium EDTA
Bring up to 1000 ml with DI water
Filter through 8.0 µm polycarbonate membrane
Add ~6 drops Dowfax to actual dispensing container

Salicylate
0.6 g Na Nitroferricyanide
300 g Na Salicylate
Bring up to 1000 ml with DI water

Filter through 8.0 µm polycarbonate membrane
Store in refrigerator in dark glass bottle

Hypochlorite
24 ml bleach
Bring up to 400 ml with DI water
Be sure to use fresh bleach (opened within the week)

For NO_3 Channel

NO_3 Color Reagent
100 ml H_3PO_4
40 g sulfanilamide
2 g N-1-Naphthylethylenediamine
Add to 800 ml DI water
Bring up to 1000 ml with DI water
Filter through 8.0 µm polycarbonate membrane
Store in refrigerator in dark glass bottle

NO_3 Buffer
638 g NH_4Cl
0.75 g disodium EDTA
Bring up to 7.5 liters with DI water
Filtered inline from dispensing bottle

NH4/NO3 Standards
Use the formula $C_1V_1=C_2V_2$ to make ammonium/nitrate working standards from a 1000 mg/L stock solution

For Orthophosphate Analysis

Stock Sulfuric Acid 5N
140 ml concentrated H_2SO_4
Slowly added to ~800 ml of DI water in a cool water bath
Bring up to 1000 ml with DI water

Stock Antimony Potassium Tartrate Solution
3 g Antimony Potassium Tartrate
Dissolve into ~500 ml DI water
Bring up to 1000 ml with DI water
Store at 4°C in a dark glass bottle

Stock Ammonium Molybdate Solution
40g Ammonium Molybdate
Dissolve in ~800 ml DI water
Bring up to 1000 ml with DI water
Store at 4°C in polyethylene bottle

Stock Ascorbic Acid Solution
1.8 g Ascorbic Acid
Dissolve into ~70 ml DI water
Bring up to 100 ml with DI water
Stable for 1 week at 4°C

PO_4 Color Reagent
100 ml Stock Sulfuric Acid (5N)
10 ml Stock Antimony Potassium Tartrate Solution
30 ml Stock Ammonium Molybdate Solution
60 ml Stock Ascorbic Acid Solution
Add reagents in order
Filter through 8.0 µm polycarbonate membrane
Prepare daily

Stock 1000 mg/L P (1L)
4.393 g Potassium DI-hydrogen Phosphate (KH_2PO_4)
Dissolve into ~900 ml DI water
Bring up to 1000 ml with DI water

Intermediate Calibrant 100 mg/L P (100 ml)
Using a volumetric pipette, add 10 ml Stock to ~80 ml DI
Bring up to 100 ml with DI water

Working Calibrants (100 ml)
Use $C_1V_1=C_2V_2$ to measure out desired range of standards
Bring up to 100 ml with DI water

For TP analysis:

Potassium Persulfate (Oxidizing Solution).
Dissolve 5g $K_2S_2O_8$ into ~90 ml DI water using stir bar and low heat
Bring up to 100 ml with DI water

Stock Sulfuric Acid 5N
140 ml concentrated H_2SO_4
Slowly added to ~800 ml of DI water in a cool water bath

Bring up to 1000 ml with DI water

<u>Stock Antimony Potassium Tartrate Solution</u>
3 g Antimony Potassium Tartrate
Dissolve into ~500 ml DI water
Bring up to 1000 ml with DI water
Store at 4°C in a dark glass bottle

<u>Stock Ammonium Molybdate Solution</u>
40 g Ammonium Molybdate
Dissolve in ~800 ml DI water
Bring up to 1000 ml with DI water
Store at 4°C in polyethylene bottle

<u>Stock Ascorbic Acid Solution</u>
1.8 g Ascorbic Acid
Dissolve into ~70 ml DI water
Bring up to 100 ml with DI water
Stable for 1 week at 4°C

<u>PO_4 Color Reagent</u>
100 ml Stock Sulfuric Acid (5N)
10 ml Stock Antimony Potassium Tartrate Solution
30 ml Stock Ammonium Molybdate Solution
60 ml Stock Ascorbic Acid Solution
Add reagents in order
Filter through 8.0 µm polycarbonate membrane
Prepare daily

<u>Stock 1000 mg/L P (1L)</u>
4.393 g Potassium DI-hydrogen Phosphate (KH_2PO_4)
Dissolve into ~900 ml DI water
Bring up to 1000 ml with DI water

<u>Intermediate Calibrant 100 mg/L P (100 ml)</u>
Using a volumetric pipette, add 10 ml Stock to ~80 ml DI
Bring up to 100 ml with DI water

<u>Working Calibrants (100-ml)</u>
Use $C_1V_1=C_2V_2$ to measure out desired range of standards
Bring up to 100-ml with DI water

Appendix F: Supplies

Order new supplies (glassware, bottles, instruments, etc…) from the VWR catalog via the Kuali Financial System website. CSU gets a contract-pricing rate (-40% of list) from VWR, so it is much less expensive to get new things from them versus other lab supply houses. Check with NREL's purchasing administrator about what to do if VWR does not stock an item you need. Purchases through Kuali do not require a Purchase Request Form. For all other purchases, you must fill out a Purchase Request Form on the NREL website.

Supplies can also be obtained from the CSU Chemistry Stockroom in the Chemistry building on campus. Purchases from the Chemistry Stockroom do not require a Purchase Request Form, but do require a CSU Chemistry Department Stockroom Card. The LVWS Program Manager should have a Stockroom Card, and be able to make purchase for other LVWS personnel. Contact the NREL admin office to request a Stockroom user card.

To make a purchase via Kuali, login to the Campus Administrative Portal (CAP) via the CSU website. Once logged into CAP, click on Kuali Financial System (KFS). One in Kuali, click on Shop Catalogs and browse to the company and supplies you need. Add the desired supplies to your basket, click Review, check that the shipping address is correct, and fill out the form as appropriate. For the Object, type "6201" for supplies. Consult Karen Adleman (kadleman@nrel.colostate.edu) or someone else in the NREL admin office (NESB A202) if you need assistance navigating Kuali.

To fill out a Purchase Request Form, visit the NREL website. On the website, click on Internal Resources and login. Navigate to a blank Purchase Request Form by clicking on Procedures and Guidelines. Consult the NREL reallocater in the admin office or Program Manager if you are unfamiliar with Purchase Request Forms.

New field supplies can be purchased from either Ben Meadows or Forestry Supply after filling out a Purchase Request Form. Any purchase over $500 requires approval from the Principal Investigator. All purchases require a justification statement. Some frequently ordered supplies are listed below:

NADP (National Atmospheric Deposition Program)

- 75 ± 1 μS cm^{-1} Specific Conductance Standard (SCS) solution from NADP
- 14 ± 2 μS cm^{-1} Quality Control (QC) check solution from NADP
- Field Observer Report Forms
- Packing tape

VWR

- Plastic bottles (60 ml, 250 ml square brown, 500 ml, & 1000 ml HDPE Nalgene)
- Glass amber borosilicate bottles with green Teflon caps (120 ml, 240 ml, & 480 ml)
- Millipore Isopore 0.4 µm membrane filters (Catalog# HTTP04700)
- Whatman GF/F glass fiber filters (47 mm, 0.7 µm)
- 47 mm all glass filter tower (all glass funnel and support) 1000 ml flask (Kontes)
- pH buffers (4.00 and 7.00)
- Specimen cups
- Scintillation vials
- VWR Traceable Conductivity Standard 10 µS cm^{-1} (Catalog #23226-567)
- VWR Traceable Conductivity Standard 100 µS cm^{-1} (Catalog #23226-589)

Hach

- 10 mg/L SiO_2 standard solution
- Silica method 8185, Silicomolybdate method (high range: 0-100.0 mg/L SiO_2)
    - Acid Reagent Powder Pillows
    - Molybdate Reagent Powder Pillows
    - Citrate Acid Pillows

Appendix G: Radio Channels in Rocky Mountain National Park

List of channels available on LVWS Bendix/King Radios.

Channel	Area	Action	Use
E1 Dirct	East side	Radio to radio direct	1-5 miles
E2 Twin	East side	Radio to Twin Sisters repeater	East side long distance communications
E3 Tundr	East & West	Radio to AVC repeater	Trail Ridge above treeline
W4 Dirct	West side	Radio to radio direct	1-5 miles
W5 Red	West side	Radio to Red Mtn repeater	West side long distance communications
W6 Shdo	West side	Radio to Shadow Mtn repeater	West side long distance communications
M7 Dirct	East & West	Radio to radio direct	Maintenance division
M8 Prosp	East side	Radio to Prospect Mtn repeater	Maintenance division long distance communications
F9 Dirct	East & West	Radio to radio direct	Fire and Resource radio to radio direct
F10 Twin	East side	Radio to Twin Sisters Fire repeater	Fire and Resource long distance communications
Ch11 Work	East & West	Radio to radio direct	ROMO work channel
L12law	East & West	Radio to radio direct, **LE Rangers Only**	NPS Law work channel
E13 Meadow	Southeast	Radio to Meadow Mtn repeater	Long distance communications in Wild Basin area
E14 Hagues	East & Northwest	Radio t Hagues Peak repeater	Long distance communications in North Fork and Hagues Creek areas

Transmit only as necessary on all channels except for #11 (Work). Channel 11 is open to work related conversation. Although you may talk freely on this channel, please keep conversation brief and concise.

Emergency radio transmissions should go out to ROMO-E1 first. Protocols are described on in the Radio Protocol section. If ROMO-E1 cannot be reached, try channel 2, and then either channel 9 or 10.

Appendix H: Database Management

1) All LVWS databases should be updated annually. Ensure that all new data have passed QA/QC procedures prior to adding data to the LVWS master databases. See LVWS Quality Assurance reports for QA/QC methodology (Denning 1988, Edwards 1991, Allstott 1995, Allstott et al. 1999, Botte and Baron 2004, and Richer and Baron 2011).

2) For new surface-water chemistry data: arrange quality assured data from the RMRS laboratory so that fields (i.e., columns) are consistent with the LWWS surface-water chemistry master database Ensure that all data field are in the correct order and units, and that all site names are consistent with those in the master database. Copy and paste the new data into the master database. Be sure all values that are below published detection limits are stored appropriately for each variable (i.e., if the detection limit is = 0.02 mg/L, then all values less than 0.02 should be stored as "<0.02"). Values of "0" should not be entered into the database. All missing data should be reported at "-999". The path to the LVWS surface-water chemistry master database is: \\data.nrel.colostate.edu\baron\LVWS_master\data\LVWS_masterdata_surfacewaterchemsitry.xlsx.

3) New precipitation data should be obtained from the National Atmospheric Deposition Program website, and added to the LVWS database as outlined in step 2 above. The path to the LVWS master precipitation database is: \\data.nrel.colostate.edu\baron\LVWS_master\data\.

4) New climate data should be obtained from the Water, Energy, and Biogeochemical Budgets program, and added to the LVWS database as outlined in step 2 above. The path to the LVWS master climate database is: \\data.nrel.colostate.edu\baron\LVWS_master\data\.

5) Be sure to plot all new data to ensure units and formatting are correct, and note all data that are preliminary and subject to change.

Appendix I: Historical Field and Laboratory Procedures

These procedures are no longer used to collect and analyze samples from the LVWS, but are provided as a reference for past methods.

Belfort Raingage *(Discontinued in August 2010)*

a. Introduction

An Alter-shielded Belfort raingage was used in LVWS to measure precipitation amount from August 1983 to August 2010. The Belfort recorded cumulative precipitation depth, as well as frequency and length of precipitation events. The Belfort was initially installed in 1983, and removed on August 16, 2010. The CO98 Belfort and CO98 NOAH IV provide a continuous precipitation record for the Loch Vale NADP site.

Detailed sampling and maintenance procedures for the gages are provided in the NADP/NTN Site Operation Manual (Dosset and Bowersox 1999) and in the Belfort Equipment Manual. Both manuals can be found in NREL A223.

b. Equipment and Supplies

- Raingage charts, cylinders, cylinder clips, rubber bands, dashpot oil, D-cell batteries, and pencils are kept in the NREL lab and/or inside shell of Belfort.
- Antifreeze and transmission fluid are kept in the storage box at the NADP site. Raingage ink is kept inside the Belfort.

c. Estimated time to complete procedures

It takes five to ten minutes to change charts, an additional 20-30 minutes if the Belfort catch bucket is full. To prevent overflow, consider bucket full when the chart reads 7 inches.

d. Preparation

Include a new chart with Tuesday's sample set. Make sure all required supplies are present in the gage shell each Tuesday. If some are missing, make sure they are replaced by the following Tuesday.

e. Procedures

To change charts, open the sliding access door. Move the pen up and down to mark the end of the week's record. Move pen away from chart with the pen shift bar. Remove drum and chart by lifting drum up off the clock gears. Write the time/date off in appropriate places on chart. Install new chart on drum. Fold edge at end of week and slip it under the beginning portion of the chart. Mark the station ID (CO98), date, and time on chart. Return drum and new chart to clock, making sure gears are properly meshed. Rotate the drum so pen rests on the current day and time. Refill the pen nib with ink if needed, usually a small drop is sufficient. The pen can usually go 2-4 weeks between refills. Do not over fill the pen. ONLY FILL THE PRECIPITATION PEN, WHICH IS THE BOTTM PEN. The top two pens are no longer used after installation of the NOAH IV electronic raingages. Mark the beginning of the week's record by moving pen up and down against the chart paper.

f. Be Aware

The catch bucket in the Belfort should be emptied when chart reads 7 inches; otherwise, the bucket will likely overflow during a major storm event. The gage works best in the range of 2-6 inches. When emptying bucket, funnel waste into empty antifreeze containers (most other containers will leak). Containers of used antifreeze should be brought down from the station immediately (in a plastic bag to protect pack against leakage). Add two inches of fresh antifreeze to the empty bucket and just enough transmission fluid to cover surface area of antifreeze (~0.5 cups). Return bucket to scale.

The mechanical linkages of the Belfort are sensitive to water, oil, dirt, dust, and abuse of any kind. Be careful to keep the internal area clean. Do not to allow the catch bucket to overflow with precipitation (see section on changing antifreeze above).

Calibration of the Alter-shielded Belfort must be checked once a year. Complete procedures are given in the Belfort manual. Advanced Technology Systems, Inc. provided a better set of instructions in 1999. These procedures are included as loose-leafs in the Belfort manual. Procedures are quite tricky, so if adjustment is required the process should be thoroughly understood before attempting.

The antifreeze mixture brought down from the weather station should be carried in antifreeze jugs and Nalgene containers, not milk jugs. Milk jugs are not durable enough to withstand the abuse they will receive on the trip down the trail. Back at NREL, funnel waste antifreeze into the five-gallon metal jugs labeled "Un-Regulated, Contents: 18% Ethylene Glycol, 2% Transmission Fluid, 80% Water." Include start and stop dates on the label for each jug. These jugs are found in NESB A101A, ask the Laboratory Manager for keys to A101A. Dispose of waste through the CSU Hazardous Waste Office (x1-6745).

Replace D-cell batteries in the clock at least twice a year (mandatory each November and March).

Measurement of Hydrologic Discharge ***(Transferred to USGS-WRD in 2006)***

a. Introduction

Operation of the Loch Outlet stream gage was transferred to the USGS-WRD in August 2006. LVWS personnel are no longer responsible for measurement of hydrologic discharge within the LVWS.

Icy Brook carries most surface water out of LVWS. A Parshall Flume with a four-foot throat was installed in Icy Brook approximately 100 meters downstream from the Loch during the fall of 1983. The flume is rated so that a given water level in the flume can be directly related to a discharge volume/rate. A Campbell Scientific CR500 stream recorder recorded stage height in a stilling well, and a Leupold and Stevens chart recorder served as a backup for the Campbell. Water level data from both gages were converted to instantaneous flow and cumulative flow (m^3s^{-1}). When the USGS-WRD assumed responsibility for operation of the flume at the Loch Outlet, a depth transducer was installed to measure water levels and a stage-discharge was developed for the flume.

The stilling wells can only function when sufficient flow is present. Generally, this is from late April to late October. Winter flow is a small proportion of the annual total, and was estimated prior to installation of the depth transducer in 2006. Although stage has been recorded during the winter since 2006, these measurements may be affected by ice formation within the flume.

b. Equipment and Supplies

- Flume field notebook and pencil
- Campbell Scientific CR500 stream recorder
- Eight D-cell batteries for CR500
- EnviroSystems encoder w/pulley wheel, pulley tape, float, and counter weight
- Laptop computer (with PC208 program), RS232 cable, and a SC32A converter
- A 9 to 25 pin serial cable
- Data storage module (SM192)
- One D cell battery for Stevens gage
- Stevens gage w/pulley line, float, and counter weight
- Strip chart paper, Stevens gage pen, and cylinder (kept in lab over winter)
- Cylinder paper spring and rubber bands (kept in lab over winter)

c. Estimated time to complete procedures

Initial set-up of the gages requires about an hour at the site, not including time to dig snow from around the flume. For weekly service, allow 20-30 minutes to download data, change chart, record the current stage height, and make minor adjustments. Closing down the flume for the winter requires only slightly more time than the weekly service.

d. Preparation

For initial set-up, make sure the CR500 is in operating order (do test runs in office). Also ensure the Stevens gage is in good shape with no visible corrosion. WD-40 and graphite dust work well to care for and prevent corrosion.

The area around the flume throat and stilling well must be dug out of the snowdrift. This is usually accomplished over several weeks beginning in early April. There should be no obstructions (i.e., snow) in the flume.

e. Procedures

Initial Set-up for CR500

1) The CR500 should be set up in mid to late April. Open the CR500 stilling well lid (it's the big PVC tube). Remove ice from the stilling well (by gently chopping with an ice axe or spud bar and by melting with water heated with a backpacking stove) when the outlet stage height is just greater than 0.1 foot. Be VERY careful not to crack or move the PVC barrel. The link from it to the flume is a point of instability.

2) Fasten the EnviroSystems shaft encoder to platform inside stilling well. Place float and weight in the stilling well, with the metal pulley tape passing over the external pulley of the shaft encoder. The float goes on the left and weight on right as you look towards the creek. Make sure the spikes on pulley mate with holes in pulley tape.

3) Attach screw cap connection to shaft encoder. Connect wires to right-hand white panel on CR500 (red=12V, white=C2/P3, black=G, and clear=G).

4) Boot computer, and start PC208. Connect battery pack to CR500. Attach CR500 to the computer using serial cable, SC32A, and an RS232 cable (blue). It goes from the CS I/O port on the CR500 to the data logger port on the SC32A. The terminal/printer port on the SC32A is linked to the printer port on the laptop with the serial cable.

5) Click the Connect button on the PC208 menu bar. Go to tools. Make sure the associated program is LOCHO.DLD. Click connect button on tools window. When connected, send the program to the data logger.

6) Go to the numeric display window. Enter the observed level reading from staff gage (meters) in the offset window. Monitor display to make sure readings are updated on the frequency set by program.

7) Disconnect the link to laptop. Close and lock shelter lid.

Weekly service for CR500

1) Attach SM192 to CS I/O on CR500 with blue RS232 cable.

2) Wait ten minutes, and then detach cable and close all enclosures.

Annual Shut Down for CR500

1) Temperatures will become increasingly colder as fall progresses. After a point, water in the stilling well will freeze. A drop in stage height below 0.1 feet usually accompanies this. In either case, it is time to shut down the gage for the year. First, perform the weekly maintenance as above.

2) Disconnect all equipment listed in section b. “Equipment and Supplies”. Load everything carefully into pack and return to lab.

Initial Set-up for Stevens gage

1) The Stevens gage should be set up in mid to late April. Open Stevens gage stilling well cover (combo: 16-18-24). Remove ice from stilling well when Outlet stage height is just greater than 0.1 foot. This is achieved by carefully chopping with an ice axe or spud bar and by melting with approximately one gallon of boiling water (heated with a backpacking stove).

2) Place float and weight in the stilling well, with the cable passing over the external pulley on the Stevens Recorder. The float goes on the left as you are looking towards the creek. Make sure the little balls on the cable fit into the holes in the pulley.

3) Place the "D" battery in the blue clock. Movement of the balance wheel should be visible through the inspection window.

4) Place clean chart paper on the drum in the recorder. Position the drum and the pen such that the pen writes the stage height on the horizontal axis and the day/time on the vertical axis. Make sure you write the date, time, and stage height reading at the beginning of the chart, as well as in the flume notebook. If flow is on the rising limb of the hydrograph (early season), position the drum so the whole chart is available for recording. This reduces the possibility that the pen will have to skip over the edges of the chart paper. After flow becomes less flashy, you should start the pen in the middle of the chart.

5) Replace the recorder cover. Close and lock the cover to the stilling well.

Weekly maintenance for Stevens gage

1) Visually read the water level along the staff gage (ft) in the flume, averaging the high and low water surges. Record the value and any adjustments or repairs made to the recorder or flume in the flume notebook.

2) Remove the Stevens recorder cover. Tilt pen back and remove chart drum. Replace the chart paper on the drum, and reposition the drum in the recorder. Slide pen carriage back so that the pen will begin recording at the correct time. Each line is equal to two hours. Position the cylinder so the pen will be unlikely to be hung up passing over chart ends, and lock in place. Write the date, time, and stage height on the end of the old chart and the beginning of the new chart. Record the date, time, and stage height in the flume notebook.

Annual Shut Down for Stevens gage

1) Temperatures will become increasingly colder later in October. After a point, water in the stilling well will freeze. A drop in stage height below 0.1 feet usually accompanies this. In either case, it is time to shut down the gage for the year. First, perform the weekly maintenance as above, except do not replace the chart paper.

2) Remove the battery from the clock. Bring all supplies back to the lab except for the gage, float, weight, and cable.

3) Remove the float, cable, and weight from the stilling well and place next to the recorder. Close and lock the stilling well cover.

f. Be Aware

The opening and throat of the flume must be clear for the stage height/discharge relationship to be correct. Check frequently (especially during high flows) and remove any obstructions.

The pen carriage rails in the Stevens Recorder may need to be cleaned so the pen can move smoothly along the time axis. Sometimes problems that appear as clock malfunction can be attributed to dirty rails. Lubricate with dry graphite or WD-40.

Usually the Stevens gage pen will last 2-3 seasons. Make sure there are spares in the supply bag located in the Stevens stilling well.

Winter Lake Sampling ***(Discontinued in 2002)***

a. Introduction

The lakes in the LVWS freeze over sometime in October. In the field book, note the date and percentage of ice cover of the Loch until frozen completely. Also, note the percentage of ice cover and date each week in the spring until the Loch is ice-free. Due to minimal flow after freezing, lakes become nearly closed basins until the following spring. When ice covered, lake water thermally stratifies, and in-lake biological processes are more likely to influence water chemistry than under summer flow conditions. Take water samples from upper (surface) and lower (hypolimnion) water layers of Sky Pond and the Loch (after boring through ice) bi-monthly during ice covered period.

b. Equipment and Supplies

- Hand-crank peristaltic pump
- 10 meters of acid-washed/ DI rinsed tubing
- Sink weight and duct tape
- Orion conductivity/temperature meter
- Butyl gloves, ice axe, ice auger (with spare blades), snow shovel
- 20 ml plastic vial of Lugol's solution (see recipe for Lugol's solution in Appendix E)
- One 2 ml disposable pipette

Sampling set (for each sampling point):
- (1) 500 ml translucent HDPE acid-washed (for chemical analysis)
- (1) 60 ml translucent HDPE acid-washed (for SiO_2)
- (1) 250 ml brown HDPE acid-washed (for raw, unfiltered sample)
- (1) 480 ml baked borosilicate bottle (for DOC, DON, and DOP)
- (1) 125 ml baked borosilicate bottle (for monthly Loch outlet phytoplankton sample)
- (1) 30 ml syringe for pH (for closed cell pH)

c. Estimated time to complete procedures

For one site, allow 15 minutes to drill hole in ice, and 30 additional minutes to collect samples and conductivity/temperature data.

d. Preparation

Acid-wash the pump tubing before each use (instructions for acid-washing on page 50). Make sure tubing is thoroughly rinsed in DI water. To rinse tube, run DI (at full blast) through it for at least 5 minutes. Drain and spin as much water out of the tubing as possible so it will not freeze during the trip to sampling site.

e. Procedures

1) Go to the deepest spot of lake. See bathymetric maps of lakes in Appendix D for locations. Start a hole in the ice with an ice axe. Finish the hole with ice auger. The ice is usually about a meter thick - so do not give up! Sharp blades are necessary. Either have them sharpened, or have a new set on hand before sample day.

2) Clear any loose ice out of open water in the hole with shovel and/or rubber gloves.

3) Measure the depth of lake and ice with tube to make sure you are over the deepest point of lake. Be careful not to disturb sediments. If you choose a site that is less than half as deep as the deepest point of the lake, abandon it and drill another hole.

4) Measure temperature profile of the lake by taking readings every meter. Also, take a reading just above and below the ice layer. Record all data in the field notebook.

5) Holding on to one end, drop sample tube into the water through hole. Weight the end of pump tubing so it falls in a straight line (a small bottle filled with sand duct taped to bottom of tube is easy to carry and works very well).

6) Collect two water samples from the lake. The first should be labeled "surface" and is collected from just below the bottom of the ice. The second should be labeled "hypolimnion" and is collected 0.25-0.5 m above lake bottom. Before drawing sample, flush pump with at least 45 revolutions of pump handle. Rinse all bottles and syringe 3X before filling (see rinsing procedures in Surface Water Sampling section). To collect the phytoplankton sample, rinse the bottle 3X, fill, dispense 1 ml of Lugol's solution with a disposable 2 ml pipette, and then cap. While one person operates the pump, the other person fills sample bottles. The person holding tube and bottles should wear insulated rubber gloves to keep hands warm and to prevent contamination of sample. Keep bottles insulated if they are in danger of freezing. Record the sample depths, sample times, and names of field technicians in the field notebook.

f. Be Aware

Water inside the pump will freeze if not constantly flowing. Once started pumping, do not stop! Difficulty of turning handle and diminished flow is evidence that freezing has begun. You may be able to thaw it by pumping more vigorously from the lake bottom. If this does not work, drain tube, put in plastic bag, and thaw inside your parka.

Prolonged exposure to cold and wind can lead to frostbite or hypothermia. Come prepared with adequate warm clothing, wind protection, hand warmers, and food. Do not ever endanger yourself or the party for a sample. If anyone is in doubt of the party's safety, pack it up and go.

Measuring pH *(Discontinued in 2010)*

a. Introduction

LVWS staff discontinued pH measurement for precipitation and surface water samples at NREL in 2010. The CAL and USFS laboratories measure pH for precipitation and surface water samples, respectively. Therefore, pH measurements at NREL were considered redundant and discontinued.

An important characteristic of water is its acidity. pH is defined as the negative log of the hydrogen ion activity. The standard method for pH measurement uses an H+ ion specific electrode that generates an electrical potential across a glass membrane. The pH meter is simply a voltmeter that measures this potential. A stable pH reading is operationally defined as one that does not change by more than 0.01 units in 30 seconds. There are extensive notes on pH measurement and specific hints on the Broadley-James electrode contained in the Instruction Manual for NADP/NTN Site Operation.

b. Equipment and Supplies (located in the NREL A235)

- Lab notebook
- pH meter (TTT85)
- Broadley-James pH electrode
- Calibration buffers: pH 4.00 and pH 7.00
- Quality Control (QC) Check Solution, pH 4.90 ± 0.15
- Fresh deionized (DI) water in squeeze bottle
- 2 ml plastic scintillation vials
- Kimwipes
- Waste beaker

c. Estimated time to complete procedure

Allow 30 minutes for calibration and 1 or 2 samples. Add 5-15 minutes for each additional sample, whether open cup or closed chamber.

d. Preparation

1) All solutions to be measured (including standards and check samples) should be at room temperature (within a few degrees).

2) Check amount and expiration dates of the standard buffers and QC solution. If needed, order more from the NADP Central Analytical Laboratory by contacting the NADP Site Liaison. It may take several weeks for them to arrive.

e. Procedures

Calibration

1) Unplug the LVWS Broadley-James pH electrode from the meter on the LVWS bench. Rinse the probe copiously with DI water.

2) Swap probes and turn off the TTT85. Be sure to hit the reset button on the circuit breaker to restore power.

3) Rinse the stir bar with DI water and blot dry on a Kimwipe. Add the stir bar to the scintillation vial containing the pH 7.00 buffer. Set the stir plate to the lowest setting and place the vial on the stir plate. Insert the pH probe into the vial so that the red "eye" is completely submerged.

4) Press the pH key on the TTT85.

5) Press the CAL key - 1. CAL. TEMP. XX.X C appears on the display.

6) Press the CAL key again – pH X.XXX BUF1 Y.YYY appears on the display. If necessary, change the pH value of buffer 1 (Y.YYY) (i.e., SHIFT, 7, . , 0, 0, 0, and STORE).

7) Wait for a stable measurement. As we record pH to two decimal places (e.g., 4.52), a measurement is considered stable when the hundredths place does not fluctuate. This could take some time (>15 minutes). Don't worry if the meter says that the pH is not 7.00. The calibration procedure will reset the meter.

   Once a stable measurement is obtained, press the CAL key. ZERO pH X.XXX CAL1 appears on the display for a short period, then 2.CAL TEMP. X.XX C will be displayed.

8) Rinse the pH probe and stir bar with DI water. Blot dry with Kimwipe.

9) Place the stir bar in the pH 4.00 buffer vial. Put the vial on the stir plate and insert the pH probe into the buffer solution.

10) Press the CAL key again – pH X.XXX BUF2 Y.YYY appears on the display. If necessary, change the pH value of buffer 2 (Y.YYY) (i.e., SHIFT, 4, . , 0, 0, 0, and STORE).

11) Wait for a stable measurement (X.XXX) and then press the CAL key. SENS XX.X & CAL2 appears on the display briefly, after 3. CAL TEMP. XX.X C is displayed. The sensitivity should be between 97.5 and 102.5. If not, re-calibrate starting with the pH 7.00 buffer.

12) Press the pH key to return to a measuring state. Be sure to rinse the probe and stir bar between samples.

Check solution and sample measurement

1) Rinse a vial with DI, then rinse the vial with QC check solution before filling with QC check solution.

2) Be sure to rinse the probe and stir bar with DI and then blot dry.

3) Immerse the pH probe in the QC check solution and wait for stable measurement.

4) If stable pH is 4.90 ± 0.15, record it. If pH is outside of this range, rinse probe with DI water and check QC check solution expiration. If the QC check solution is good, repeat calibration. Measured pH of check solution must be 4.90 ± 0.15 before any samples are measured. If you cannot get a good reading on check solution, the QC check solution likely needs to be replaced. Cleaning and troubleshooting procedures are described in NADP Site Operation Manual. If a probe is not able to read the check solution properly, consult the Lab Manager.

5) Remove probe and stir bar from check solution vial and thoroughly rinse with DI water. Blot excess water from probe tip and stir bar with Kimwipe.

6) Rinse and fill a vial with the sample to be measured.

7) Immerse probe in the sample vial and wait for stable measurement. Once the pH has stabilized to the hundredths place, record measurement on lab sheet/notebook.

8) Remove probe from the sample vial, and thoroughly rinse with DI water. Blot excess water from probe tip with Kimwipe. Rinse the stir bar with DI, blot dry, and transfer to next sample vial.

9) Repeat steps 6 through 8 for each additional sample. If measuring more than five samples, re-measure QC sample after every fifth cup samples (to check for drift). If QC sample is not 4.90 ± 0.15, re-measure last five samples if possible.

f. Be Aware

The plastic body of the pH electrode is fragile! Be careful not to crack or scratch it. Be especially careful not to allow anything solid to touch the spherical glass membrane at the tip of the probe. It is easily scratched, which ruins the electrode.

The electrical potential being measured is in the millivolt range; therefore the equipment is extremely sensitive to static charge. Stand back and keep motions to a minimum when attempting a measurement.

Appendix J: LVWS Watershed Staff

The LVWS Watershed Project staff is made up of a Principal Investigator, co-Principal Investigator(s), Program Manager, technicians, programmers, graduate students, undergraduate students, and collaborators.

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