

**QUALITY ASSURANCE REPORT**  
**Loch Vale Watershed Study, 1991-94**

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## **List of Acronyms**

AAS - Atomic Absorption Spectrophotometry  
ANC - Acid Neutralizing Capacity, equivalent to alkalinity  
CAL - Central Analytical Laboratory at the Illinois State Water Survey  
CSU - Colorado State University  
DAMS - Data Acquisition and Monitoring Subsystem  
DCP - Data Collection Platform  
DI - Deionized Water  
DOC - Dissolved Organic Carbon  
DQO - Data Quality Objectives  
EPA - Environmental Protection Agency  
GOES - Geostationary Operational Environmental Satellite  
IC - Ion Chromatography  
ICP - Inductively Coupled Plasma Spectrophotometry  
IPD - Ion Percent Difference  
LIS - Low Ionic Strength  
NADP - National Atmospheric Deposition Program  
NBS - National Biological Service  
NOAA - National Oceanic and Atmospheric Administration  
NPS - National Park Service  
NREL - Natural Resource Ecology Laboratory  
QA - Quality Assurance  
QC - Quality Control  
RSD - Relative Standard Deviation  
USFS - United States Forest Service  
USGS - United States Geological Service

## **Introduction**

The Loch Vale Watershed (LVWS) Project was initiated in 1980 by the National Park Service (NPS) under funding for the Aquatic Effects Research Program of the National Acid Precipitation Assessment Program. Major goals of the project originally were: 1. to provide a long term ecological/biogeochemical record of LVWS processes and, 2. to observe and differentiate natural biogeochemical and biological variability from human-caused disturbance in alpine and subalpine ecosystems through long-term study. Since the beginning of this project our goals have expanded to include the former as well as: 1. to understand and better quantify the role of climatic variability, particularly changes in precipitation and temperature, on alpine and subalpine processes, 2. To apply new and better methods to the addressing of the role of climate on winter snow accumulation, redistribution, and melt processes and, 3. to estimate potential ecosystem damage induced by changes in climate due to land use change and/or globally-increasing greenhouse gases.

Meteorology, hydrology, precipitation chemistry, and surface water chemistry are the four main parameters that have been collected regularly since 1982. Data are reviewed and stored on the UNIX computer network at the Natural Resource Ecology Laboratory at Colorado State University. This quality assurance report addresses data collected since January 1, 1991. Previous data quality reports include 1983-87 (Denning, 1987), 1988 (Baron, 1988), and 1989-90 (Edwards, 1991).

## **Meteorological Data**

Loch Vale Watershed meteorological data are collected by a solar powered remote area weather station (RAWS) located between Embryo Pond and the Icy Brook/Andrews Creek confluence. Weather parameters measured by this station are wind speed, wind direction, relative humidity, ambient air temperature, solar radiation, barometric pressure, and precipitation amount. Parameters are measured every 15 minutes by a Handar 524 data collection platform (DCP), and are relayed hourly to the National Oceanic Atmospheric Administration (NOAA) receive station at Wallops Island, Virginia via the Geostationary Operational Environmental Satellite West (GOES) satellite network. LVWS personnel download these data into the LVWS master database weekly.

Data received from the DOMSAT relay are quality checked and edited by LVWS personnel before loading into the master database for two reasons: 1. the program that loads the data into the database can process the data in only one particular format; and 2. so that incomplete or erroneous transmissions will not be included in the database.

The Data Acquisition and Monitoring Systems (DAMS) at the Wallops Island downlink monitors each hourly transmission for signal strength (EIRP), frequency offset, modulation index and received data quality (Integral Systems, Inc., 1990). As per previous LVWS QA/QC reports, fifty randomly selected hourly transmissions were analyzed for these parameters (Table 1).

**Table 1: Meteorological data signal quality parameters**

	Signal Strength(EIRP)	Frequency Offset (Hz)	Modulation Index	Data Quality
mean	45.520	0.694	always	always
SD	3.767	0.895	Normal	Normal
normal range	42 to 52	-0 to +3		
#outside norm	5 low	0		

All parameters except EIRP were within the normal range. One out of ten transmissions examined had a signal strength less than 42, however, these low EIRPs did not compromise the quality of data collected. Quality of data collected at the RAWS is not compromised by low EIRP unless it dips lower than 35.

Transmission accuracy was also tested by comparing the number of successful transfers to the total number of possible transfers (Table 2).

**Table 2: Total number of possible vs. received satellite transmissions**

period	possible	received	accuracy (%)
1/1/91 - 12/31/94	34,920	33,885	97%

There were 34,920 possible transmissions from January 1, 1991 till December 31, 1994. There are 33,885 transmissions which have been included in the database from this period. This marks a 97% transmission accuracy.

No existing documentation details all calibrations and servicing of weather station components since the last QA/QC report by R. Edwards (1991). Work on the weather station has been minimal due to the general lack of problems during the period covered by this report. The anemometer bearings were replaced in May of 1994. A complete service check of the weather station and calibration of all sensors was performed in mid-September by Dean Newkirk, a private consultant who is based in Denver. All checks and calibrations were performed to maintain the equipment within published limits. Newkirk's analysis was that the station is in good condition.

## Hydrological Data

The Loch outlet is located at the northeastern edge of The Loch, and has been gaged with a Parshall flume and stilling well since 1983. Stage height data are collected both mechanically (with a Leupold and Stevens chart recorder) and electronically (with a Omnidata DP115 datalogger) when measurable flow through the flume is present, typically from May through October.

Weekly checks of actual stage height are made by observing a staff gage located just inside the flume during low-snow or snow free months. This stage height is recorded into a field notebook and compared to the mechanically and electronically collected values. Any changes made to the data are noted and stored along with the data files at NREL.

Data are downloaded from the data storage modules at the end of the water year when measurable flow ceases. Any corrections needed due to equipment or operator error are performed at this time and noted in the database. The data are then run through a program that calculates the discharge ( $\text{m}^3 \text{sec}^{-1}$ ) using a formula provided by the Thompson Pipe and Steel Co.

The accuracy of Parshall Flumes is  $\pm 5\%$  (Winter, 1981) Before 1994, we estimated that an additional 5% uncertainty was introduced by water flowing around or under the frame structure. In June and October wing-walls were expanded, rebuilt, and re-packed with bentonite filled fine-meshed burlap bags, so that there is now no observable flow outside the flume.



## Precipitation Data

The National Atmospheric Deposition Program is responsible for the chemical analysis of Loch Vale precipitation and the quality assurance of those results. NADP has a quality assurance plan that details all phases of quality control procedure. As a minimum, the analytical laboratory achieves at least the detection limits listed in Table 3, with a maximum allowable bias of  $\pm 100\%$  at the detection limit,  $\pm 20\%$  at 10 times the detection limit, and  $\pm 10\%$  at 100 times the detection limit. (Aubertin, et al, 1990). Actual detection limits and bias are reported annually in Central Analytical Laboratory (CAL) quality assurance reports.

**Table 3: Data Quality Goals for Precipitation Chemistry** (Aubertin, et al, 1990).

Analyte	Units	Bias	Detection Limit
Ca	mg/l	100% at Detection Limit  20% at 10 times Detection Limit  10% at 100 times Detection Limit	.009
Mg	mg/l		.003
Na	mg/l		.003
K	mg/l		.003
NH <sub>4</sub>	mg/l		.02
SO <sub>4</sub>	mg/l		.03
NO <sub>3</sub>	mg/l		.03
PO <sub>4</sub>	mg/l		.02
Cl	mg/l		.03
pH < 5.0			$\pm 0.1 / \pm 0.03$
pH > 5.0		$\pm 0.3 / \pm 0.1$	
Specific Conductance			
10 - 100	$\mu\text{S/cm}$	$\pm 10\% / \pm 3\%$	
> 100	$\mu\text{S/cm}$	$\pm 6\% / \pm 2\%$	

A summary of sample validity showed that 198 LVWS precipitation samples were submitted to the Central Analytical Laboratory for analysis between January 1, 1991 and October 10, 1994 (NADP, 1995). Of these 198 samples, 184 (94.8%) were considered valid. Of the 14 samples rejected, eight were for being exposed to dry deposition for more than six hours, four for contamination, and two for mistakes in field protocol.

When samples do not meet certain goals they are reanalyzed. NADP sample reanalysis criteria are outlined in Table 4 and the following paragraph.

**Table 4: Sample Reanalysis Criteria**

Ion Percent Difference

If:	Reanalyze when:
(anions+cations) ( $\mu\text{eq/L}$ )	$[(\text{Anions}-\text{Cations}/(\text{Anions}+\text{Cations})\times 100]$
<50	> $\pm$ 60%
$\geq 50$ but <100	> $\pm$ 30%
$\geq 100$	> $\pm$ 15%

The Conductance Percent Difference (CPD) is calculated by subtracting the measured conductance from the calculated conductance, multiplying the difference by 100, and dividing the product by the measured conductance. Samples are reanalyzed if the CPD is outside of the range from -40% to +10%

LVWS field personnel participate in biannual intersite comparison quality assurance studies organized by the United States Geological Survey (USGS). In these studies a blind sample is sent to each NADP site operator. Conductivity and pH measurements are performed on this blind sample under standard protocols. Results are to be within the network wide median value for that sample. If they are not, the problem area within the analysis is identified and the necessary adjustments in procedure, equipment, or personnel are made. Results reported for pH and conductivity by LVWS personnel have always met NADP/NTN goals (Table 5).

**Table 5: USGS Intersite Laboratory Comparison Results**

Date	Expected pH	Measured pH	Expected Conductance	Measured Conductance
4/91	4.24	4.30	26.3	27.8
11/91	4.73	4.74	8.6	8.9
4/92	4.49	4.49	15.0	14.8
9/92	5.05	5.02	19.1	15.9
6/93	4.87	4.90	6.0	6.2
10/93	4.67	4.65	9.7	9.7
6/94	4.79	4.82	7.40	6.7
11/94	4.53	4.53	20.7	22.0

LVWS field personnel are responsible for measuring pH and conductivity of weekly precipitation samples for our own records and as a check on NADP analyses. Each analysis is accompanied by measurement of a quality control check sample. If pH is not within 0.1 unit and conductivity is not within 4 $\mu$ s/cm of the reported check sample value, the measuring instrument is re-calibrated or other necessary adjustments are made.

Although most of the precipitation samples taken in LVWS meet NADP sample validity criteria there is a problem with the catch efficiency of the standard Aerochemetrics collector. Alter and Nipher shielded raingages are much more effective devices for catching precipitation in windy areas than is the sample bucket. Weekly winter precipitation amounts shown by the Alter and Nipher shielded gages are usually one and a half to two times that of the amount shown in the NADP Aerochemetrics sample bucket. Summer precipitation amounts from gage to sample bucket typically match each other more closely, but rarely fall within the 5% desired.

LVWS procedures regarding NADP rigors are detailed in the LVWS Methods Manual and in the NADP Instruction Manual (Bigelow, 1988 rev. 1994).

Results of analyses performed at the CAL on LVWS precipitation samples are available from the NADP/National Trends Network (NTN) Annual Data Summary reports, or from the LVWS project.

## Surface Water Chemistry Data

### Introduction

Weekly surface water sampling has routinely taken place in LVWS since 1982. Year-round samples are drawn from The Loch outlet as long as sufficient flow is present. Samples are taken from other sites in the basin on a monthly basis during late-spring, summer and fall. Lake hypolimnion and surface water samples are drawn from Sky Pond and The Loch on an alternating monthly schedule during the months when the lakes are frozen.

Analytes measured year-round are ammonium, calcium, chloride, potassium, magnesium, nitrate, sodium, silica, and sulfate. LVWS waters are also examined for specific conductance, pH, dissolved organic carbon (DOC), and alkalinity. LVWS methods follow the established protocol for low ionic strength waters, (Lockheed, 1989), and each analytical laboratory the Loch Vale Watershed Project has participated with since 1991 has conducted its own internal quality assurance exercises according to standards for the analysis of low ionic strength natural waters (Friedman, 1982). Specific information regarding quality control procedures for the collection and handling of samples before chemical analysis are detailed in the LVWS Methods Manual (Newkirk, et al 1995).

Field collection/handling and laboratory treatment procedures are assessed through the use of replicate and blank samples. Ten percent of the total number of samples analyzed are quality checks. Bias and contamination due to field procedures are estimated through the use of blank samples. Analysis of precision is estimated by using duplicate and duplicate-split samples. Differences in the results between these four

samples illuminate deficiencies in the analytical process. Details of blank and duplicate sampling protocol are presented in the LVWS Methods Manual.

Currently, LVWS water samples are sent to the USFS Forest Service Rocky Mountain Station Biogeochemistry Laboratory. This Forest Service lab participates in the USGS's inter-lab analytical program in which standard reference samples are sent to more than 100 different labs and analyses compared. USFS values for this analysis fall within the country-wide mean for all relevant analytes (USGS, 1994).

#### Detection Limits

Detection limit is a numerical boundary below which the level of accuracy in the measurement of a particular analyte declines severely. This limit is defined as three times the standard deviation of signals produced by replicated blank analyses. The detection limits used by all labs the LVWS project has interacted with since 1991 are those set forth in the U.S. Environmental Protection Agency's 1987 Handbook of Methods for Acid Deposition.

#### QA Tests

Results of all tests performed on LVWS surface water samples are screened by USFS laboratory personnel to detect problems with individual samples. Analyses are repeated (up to three times) if the USFS quality check flags a particular sample. If a sample gets flagged three times the determination is made that any contamination present must have been introduced in the field, and gets marked as such. Criteria for a sample to be flagged are outlined in Table 6.

**Table 6: Chemical reanalysis criteria used by the Ft. Collins USFS water lab**

A. Anion/Cation Balance

	Maximum
Total Ion Strength ( $\mu\text{eq/L}$ )	% Ion Balance Difference <sup>a</sup>
<50	60
$\geq 50 < 100$	30
$\geq 100$	15

B. Conductance

	Maximum
Measured Conductance ( $\mu\text{S/cm}$ )	% Conductance Difference <sup>a</sup>
<5	50
$\geq 5 < 30$	30
$\geq 30$	20

<sup>a</sup>If the absolute value of the percent difference exceeds these values, the sample is reanalyzed. (O'Deen, et al. 1994)

USFS quality check procedures are double checked by LVWS personnel with an ion percent difference program in which the difference between the sum of the cations and the sum of the anions is divided by the total ion concentration. Results of this IPD program and the one used by the USFS water laboratory consistently are alike.

## Laboratory Changes

Analyses of LVWS surface waters took place at the USGS Water Quality Laboratory in Arvada, Colorado until December 12, 1992. Between December 12, 1992 and August 31, 1993 analyses were performed at the USGS Water Quality Laboratory in Reston, Virginia. Analyses of LVWS surface water have been performed at the USFS Water Quality Laboratory in Fort Collins, Colorado since August 31, 1993.

Dissolved organic carbon (DOC) was analyzed at Arvada and Reston with the other analyses until we transferred to the Fort Collins USFS Laboratory, at which time the site of analysis for DOC was changed to a USGS water laboratory in Boulder, Colorado.

Duplicate samples were sent to both labs during each period of change-over.

T-tests were performed on the data from these periods of overlap (Table 7). Results from the Arvada/Reston change show that, excepting pH,  $\text{NH}_4$ , and Cl, analytical results from these labs were not significantly different from each other. Differences in pH are uncertain in origin, while the differences found in  $\text{NH}_4$  and Cl could be attributed to a smaller number of samples available for inclusion into the T-test and/or lower detection limits. Results from the Reston/Ft. Collins change (Table 8) show that, excepting pH, analytical results from each lab were not significantly different from each other. Like the Arvada/Reston change, the differences found in pH for the Reston/Ft. Collins are of uncertain origin.



**Table 7: Number of samples included in each assay**

Analyte	USGS	Reston	USFS
alkalinity	328	42	59
ammonium	341	13	NA
calcium	355	42	59
chloride	356	42	59
magnesium	354	42	59
nitrate	351	42	59
potassium	355	42	59
pH	42	59	NA
silica	42	3	NA
sodium	354	41	73
sulfate	354	42	79

**Table 7: SAS T-test results: USGS- Arvada, Colorado vs. USGS- Reston, Virginia**  
 (means of all analytes except pH presented as mg/l)

analyte	mean (Arvada/Reston)	SD (Arvada/Reston)	P
alkalinity	97.27/107.70	12.64/13.99	0.1690
ammonium	0.02/0.07	0.01/0.02	0.0195
calcium	2.34/2.19	0.19/0.21	0.1689
chloride	0.18/0.37	0.01/0.02	0.0000
magnesium	0.35/0.34	0.03/0.03	0.4650
nitrate	1.54/1.45	0.20/0.23	0.4523
potassium	0.23/0.23	0.02/0.02	0.4569
pH	6.86/6.63	0.12/0.05	0.0005
silica	2.99/2.93	0.33/0.31	0.7564
sodium	1.00/0.96	0.13/0.14	0.5922
sulfate	2.94/2.83	0.39/0.30	0.5685

**Table 8: SAS T-test results: USGS- Reston, Virginia vs. USFS- Ft. Collins, Colorado**  
 (means of all analytes except pH presented as mg/l)

analyte	mean(Reston/FtCollins)	SD (Reston/Ft Collins)	P
alkalinity	43.70/42.23	4.33/5.01	0.6715
calcium	1.23/1.54	0.22/0.25	0.0725
chloride	0.14/0.13	0.03/0.02	0.6771
magnesium	0.20/0.22	0.04/0.05	0.4511
nitrate	0.94/1.12	0.23/0.21	0.2105
potassium	0.14/0.15	0.01/0.02	0.4031
pH	6.45/6.67	0.06/0.05	0.0004
sodium	0.50/0.51	0.06/0.06	0.7073
sulfate	1.58/1.66	0.25/0.31	0.6654

## pH

The mean of all reported field pH values (sample size of 472) was 6.40, and the mean for all pH values reported by the analytical laboratories (sample size of 423) was 6.55. These means were compared using a SAS T-test. Results of this test show the means to have a P of 0.0000. As noted in Aubertin et al. (1990), there is a systematic bias between field and lab pH for western precipitation samples. These authors attribute the bias to neutralization of salts during transit between field and laboratory. Another possible cause for this difference is the de-gassing of CO<sub>2</sub> from the vial during analysis which would slightly raise the pH. This statistical difference between means is also possibly due to the low variance found within each mean. Differences found in pH between reported field and lab values have minimal impact on interpretation of LVWS data because field pH values, not lab pH values, are those published.

## DOC

Dissolved organic carbon (DOC) analysis took place in Arvada, CO until December 12, 1992 then changed to Reston along with the rest of the analyses. On August 31, 1993 the site of LVWS DOC analyses was changed to a USGS water lab in Boulder, CO. Results of DOC analyses gained from Reston were inconsistently lower than those gained when the Arvada or Boulder labs were handling the analyses. Comparisons of means were performed in an effort to determine if there was an adjustment factor we could put to the Reston data (Tables 9 and 10)

**Table 9: Mean DOC values from each lab**

laboratory	sample size	mean (mg/l)
Arvada	191	1.85
Reston	43	0.94
Boulder	29	1.70
Arvada + Boulder	220	1.83

**Table 10: Comparison of DOC means of Arvada or Boulder vs. Reston**

laboratories	difference of means (mg/l)	P
Arvada vs. Boulder	0.15	0.54
Arvada vs. Reston	0.91	0.00
Boulder vs. Reston	0.76	0.01
Arvada & Boulder vs. Reston	0.89	0.00

There are general patterns of DOC efflux in LVWS that repeat each year, but due to annual differences in the timing of snowpack accumulation and release peak efflux of DOC varies from mid-May to late-June. No satisfactory adjustment factor could be calculated due to the seasonal inconsistency of variance of Reston values from the overall means (Table 11).

**Table 11: Seasonal Comparison of DOC means**

period	mean of Arvada & Boulder (mg/l)	mean of Reston (mg/l)	P of Arvada & Boulder vs. Reston
12/1 - 3/16	1.03	0.05	0.00
3/17 - seasonal peak	2.38	1.67	0.13
seasonal peak - 7/20	2.48	1.35	0.01
7/21 - 9/15	1.01	0.39	0.00
9/15 - 9/28	1.03	0.72	0.17

All DOC data gained from the Reston Lab has been discarded from the master LVWS database. A portion of the discarded data spanning from June 15, 1993 till September 28, 1993 will be replaced by data collected in LVWS by Diane McKnight of the USGS. McKnight's data is illustrated in Figure 1. Data from the Reston period outside the time span of McKnight's study (December 1, 1992 till June 14, 1993) will be noted in the database and future reports as missing.

Conclusions regarding laboratory changes

Numerous meetings with laboratory personnel from all of the involved labs in which the results of all these tests have been discussed have been held. Through these meetings we have decided that due to differing equipment, personnel, internal protocols, calibrations, detection limits, sensitivities, etc., analytical results will sometimes vary from laboratory to laboratory. Excepting DOC, analyses from Reston, results from each laboratory that the LVWS project has participated with from 1991-1994 should be considered valid and useful.



## **Conclusion**

For this project to continue its service to the public, the scientific community, and our land policy managers it is of utmost importance that the stringent QA/QC procedures followed in the past be continued and improved when possible. This and all previous QA/QC reports produced by the LVWS project show that we take great care in the collection and analysis of Loch Vale waters. Care in these areas of research provide reliable data for the synthesis stage of research. It is there, in the synthesis stage, that the work we do becomes of service to those mentioned previously. As we work to achieve our project objectives we must always remember that it all starts in the field and in the lab. Quality staff, methods, and equipment are the precursors to a reliable dataset which will grow stronger in value and be of great use for many years to come.

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