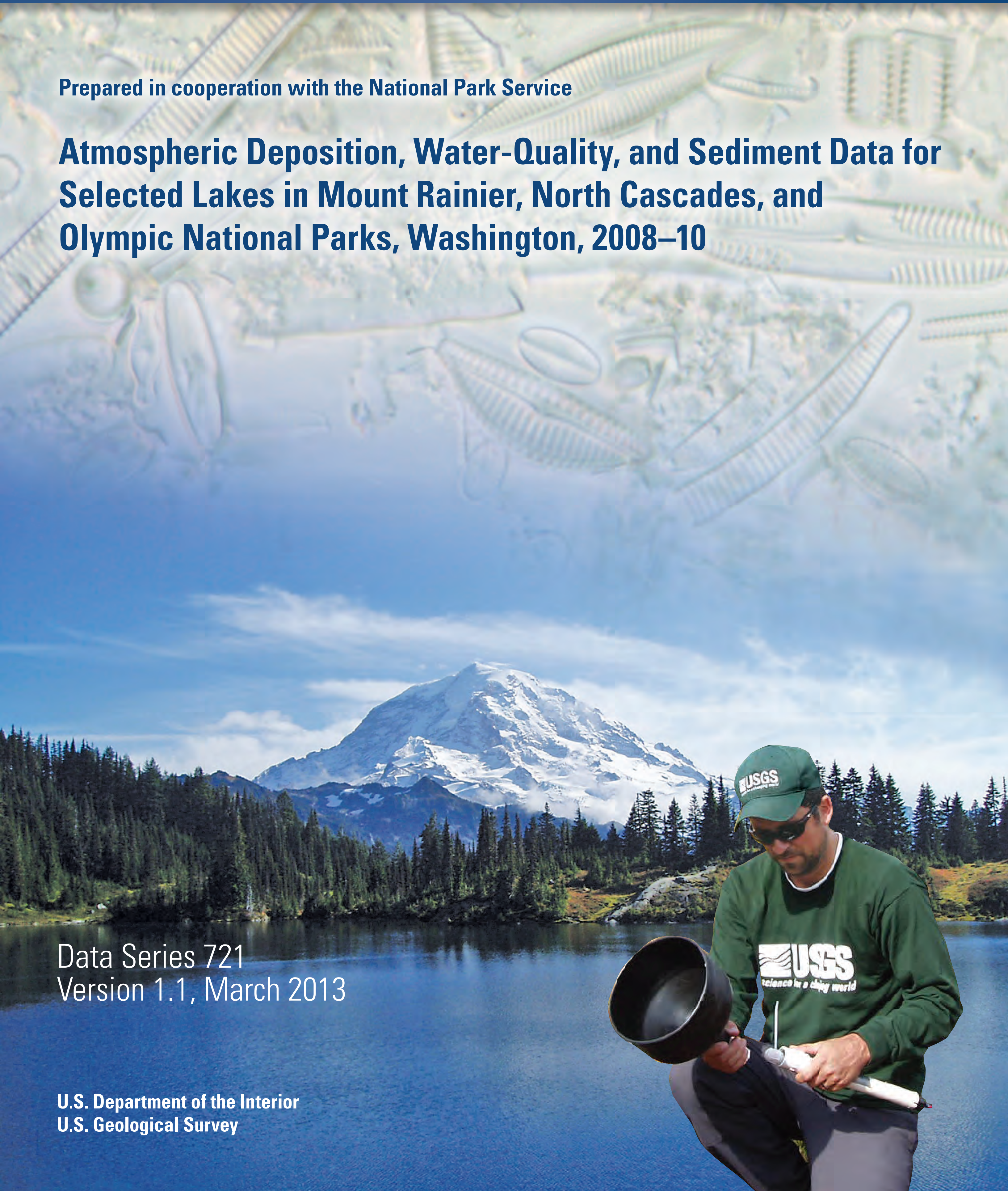

Prepared in cooperation with the National Park Service

Atmospheric Deposition, Water-Quality, and Sediment Data for Selected Lakes in Mount Rainier, North Cascades, and Olympic National Parks, Washington, 2008–10

Data Series 721
Version 1.1, March 2013

U.S. Department of the Interior
U.S. Geological Survey

Front cover:

Top: Electron microscope image of the first field view of diatom slide from Stiletto Lake, North Cascades National Park, at sediment depth of 30–31 centimeters. (Photograph taken by Mihaela Enache, Academy of Natural Sciences, Philadelphia, Pennsylvania, 2009.)

Bottom: U.S. Geological Survey scientist assembling passive atmospheric deposition collector near Snow Lake, Mount Rainier National Park, Washington. (Photograph taken by Rich Sheibley, U.S. Geological Survey, July 2008.)

Background: Eunice Lake, Mount Rainier National Park, Washington. (Photograph taken by Jonathan Richards, U.S. Geological Survey, 2008.)

Back cover:

Helicopter dropping sediment coring equipment at Hidden Lake, North Cascades National Park, Washington. (Photograph taken by Rich Sheibley, U.S. Geological Survey, September 2009.)

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U.S. Department of the Interior
KEN SALAZAR, Secretary

U.S. Geological Survey
Marcia K. McNutt, Director

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Tables—Continued

Conversion Factors, Datums, and Abbreviations and Acronyms

Conversion Factors

Inch/Pound to SI

Multiply	By	To obtain
	Length	
foot (ft)	0.3048	meter (m)
	Area	
square inch (in^2)	6.452	square centimeter (cm^2)

SI to Inch/Pound

Multiply	By	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
meter (m)	3.281	foot (ft)
meter (m)	1.094	yard (yd)
	Area	
hectare (ha)	2.471	acre
square centimeter (cm^2)	0.001076	square foot (ft^2)
square centimeter (cm^2)	0.1550	square inch (in^2)
hectare (ha)	0.003861	square mile (mi^2)
	Volume	
liter (L)	61.02	cubic inch (in^3)
liter (L)	1,233	cubic meter (m^3)
cubic centimeter (cm^3)	0.001233	cubic hectometer (hm^3)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)
	Density	
gram per cubic centimeter (g/cm^3)	62.4220	pound per cubic foot (lb/ft^3)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$°F=(1.8\times°C)+32.$$

Specific conductance is given in microsiemens per centimeter at 25 degrees Celsius (µS/cm at 25 °C).

Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter (µg/L).

Datums

Vertical coordinate information is referenced to the North American Vertical Datum of 1988 (NAVD 88).

Horizontal coordinate information is referenced to the North American Datum of 1983 (NAD 83).

Altitude, as used in this report, refers to distance above the vertical datum.

Conversion Factors, Datums, and Abbreviations and Acronyms—Continued

Abbreviations and Acronyms

Abbreviation or acronym	Definition
ANC	acid neutralizing capacity
ANSP	Academy of Natural Sciences, Philadelphia
HPGe	high purity germanium
IER	ion exchange resin
$CaCO_3$	calcium carbonate
CCAL	Cooperative Chemical Analytical Laboratory (Oregon State University, Corvallis)
LRL	laboratory reporting limit
LTEM	Long Term Ecological Monitoring
MORA	Mount Rainier National Park
MDL	method detection limit
MRL	method reporting limit
OLYM	Olympic National Park
NADED	North American Diatom Ecological Database
NAWQA	National Water Quality Assessment
NCCN	North Coast and Cascades Network
NOCA	North Cascades National Park
NPS	National Park Service
NWIS	National Water Information System
NWQL	National Water Quality Laboratory (U.S. Geological Survey)
PSRS	Pacific Southwest Research Station
ROMO	Rocky Mountain National Park
SRS	standard reference sample
STORET	STOrage and RETrieval
USFS	U.S. Forest Service
USGS	U.S. Geological Survey

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Atmospheric Deposition, Water-Quality, and Sediment Data for Selected Lakes in Mount Rainier, North Cascades, and Olympic National Parks, Washington, 2008–10

By Rich W. Sheibley, James R. Foreman, Patrick W. Moran, and Peter W. Swarzenski

Abstract

To evaluate the potential effect from atmospheric deposition of nitrogen to high-elevation lakes, the U.S. Geological Survey partnered with the National Park Service to develop a “critical load” of nitrogen for sediment diatoms. A critical load is defined as the level of a given pollutant (in this case, nitrogen) at which detrimental effects to a target endpoint (sediment diatoms) result. Because sediment diatoms are considered one of the “first responders” to ecosystem changes from nitrogen, they are a sensitive indicator for nitrogen deposition changes in natural areas. This report presents atmospheric deposition, water quality, sediment geochronology, and sediment diatom data collected from July 2008 through August 2010 in support of this effort.

Introduction

As a result of fossil fuel combustion, increased fertilizer production, and livestock waste generation, the amount of fixed nitrogen that can move from the atmosphere to terrestrial and aquatic ecosystems has doubled worldwide (Vitousek and others, 1997). This atmospheric nitrogen deposition can have many negative effects on the environment, such as nutrient imbalances that lead to eutrophication or algal blooms in many ecosystems (Swackhamer and others, 2004). Many lakes in national parks are oligotrophic, and these low-nutrient systems are especially sensitive to nitrogen deposition because of their inability to use atmospheric inputs for plant growth (Fenn and others, 1998).

The 1977 Clean Air Act Amendments (Public Law 95-95) designated 48 units of the National Park System as Class I air quality areas, affording these areas the highest degree of air quality protection. The National Park Service (NPS) conducts and supports monitoring and research that help identify air-pollution sensitive resources and effects thresholds in Class I areas. Three of the seven units in the NPS North Coast and Cascades Network (NCCN) are Class I areas—Mount Rainier, North Cascades, and Olympic National Parks.

Long-term research at Rocky Mountain National Park (ROMO) showed that increased nitrogen deposition altered the water quality of lakes and shifted their trophic status (Baron, 2006). In that study, analyses of historical sediment diatom community shifts in nitrophilic species allowed for the identification of a “critical load” for wet nitrogen deposition of 1.5 kilograms of nitrogen per hectare per year [(kg N/ha)/yr] for high alpine lakes at ROMO. The critical load for nitrogen is defined as the level of nitrogen deposition at which shifts in diatom communities occur that could potentially alter the ecosystem functioning at these lakes. From the work of Baron (2006), interest increased in applying the critical-load concept to the three Class I parks in the NCCN (fig. 1). In 2006, a multiagency group of researchers led by the NPS, held the “Pacific Northwest Nitrogen and Sulfur Deposition Critical Load Workshop” to identify research needs. During this workshop, the approach of examining diatom changes to identify a critical load at ROMO was indicated as a high research priority for NCCN lakes (Waddell and Greenwood, 2006). As a result, the U.S. Geological Survey (USGS) conducted a 3-year intensive study to determine critical loads of nitrogen deposition in NCCN lakes (the NCCN critical loads project) through the USGS-NPS Water Quality Partnership Program.

Purpose and Scope

This report presents atmospheric deposition, water quality, sediment chemistry, and sediment diatom data collected at 12 lakes in Mount Rainier, North Cascades, and Olympic National Parks. Detailed descriptions of the methods used to collect these data, as well as results from various quality-assurance samples, also are presented. These data were collected to support the development of a critical load for nitrogen deposition at the study lakes for a collaborative USGS-NPS project.

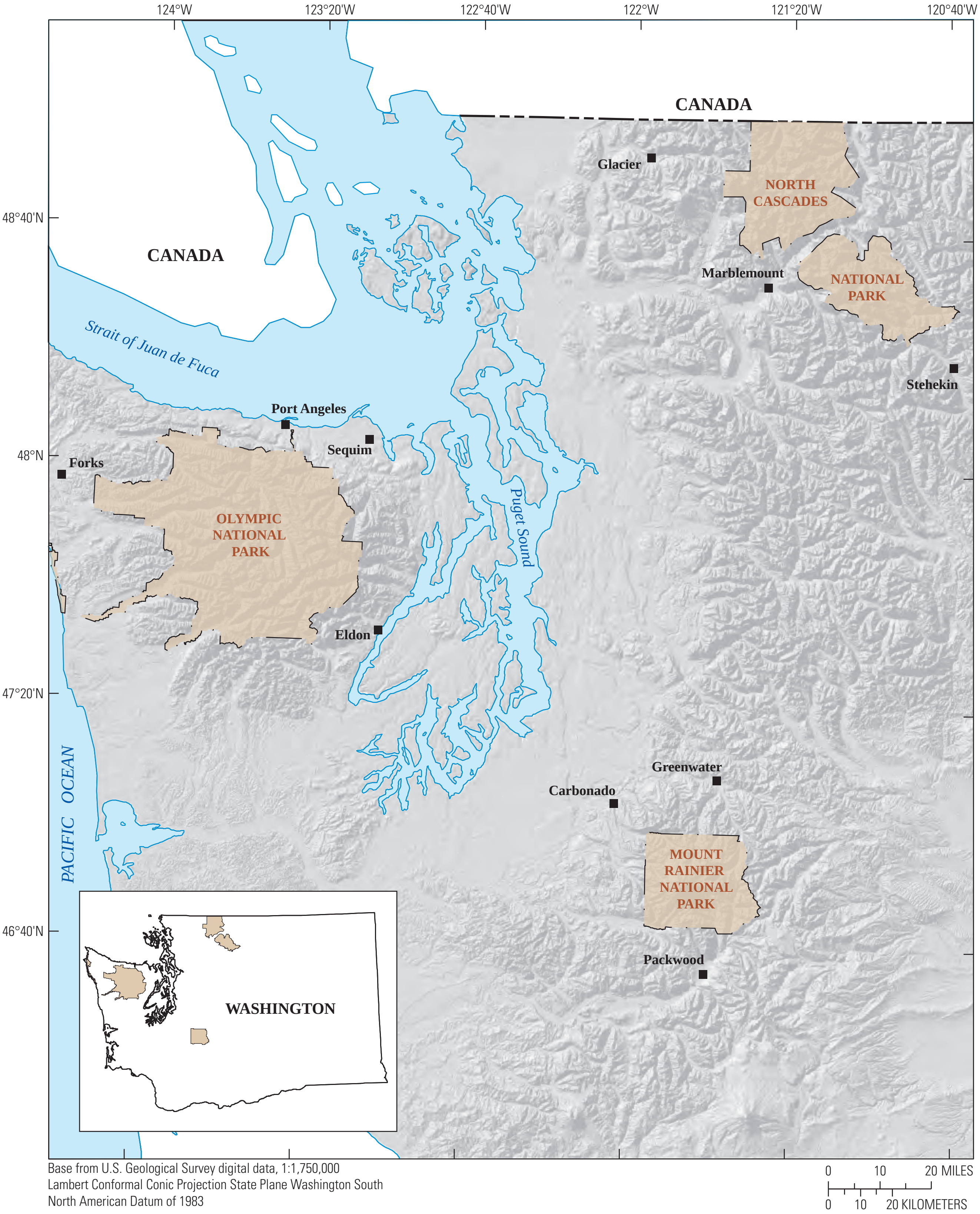


Figure 1. Locations of Mount Rainier, North Cascades, and Olympic National Parks, Washington. These parks make up the three Class I air quality areas of the North Coast and Cascades Network (NCCN) of National Parks.

Study Areas

Lakes in Mount Rainier (MORA), North Cascades (NOCA), and Olympic (OLYM) National Parks, Washington, were selected for this study. The goal of this study was to determine the potential effects of atmospheric nitrogen deposition on high-elevation lakes in these national parks. A methodical approach was used to select 4 lakes in each park, to include 12 lakes total in this study.

The initial phase of lake selection involved the compilation of historical water-quality data collected in each park to identify lakes that were susceptible to nitrogen deposition. More than 5,300 individual samples were collected across the three national parks from 1970 to 2008; the data resides in the USGS National Water Information System (NWIS) and the U.S. Environmental Protection Agency water-quality database (STOrage and RETrieval or "STORET"). Once assembled, the data were evaluated for completeness using the same methods described by Clow and Campbell (2008). Lakes from this dataset that were sampled for nutrient concentrations and acid neutralizing capacity (ANC) within the last 10 years (1998-2008) were considered for inclusion in this study. Other site selection criteria included:

Geographic spread within each park: The precipitation gradients for MORA and OLYM are east–west with the "drier" side of the park to the east. The precipitation gradient for North Cascades National Park (NOCA) is generally from north to south; the northern unit of the park receives more rain and snow. For this study, two "dry" and two "wet" lakes were selected in each park to capture the spatial variability in precipitation, which translates to differences in mass loading from atmospheric deposition. Furthermore, lakes that were considered high-visitor-use areas were avoided to minimize disruptions to the watershed and the field equipment.

High-elevation lakes: The average tree-line altitude (approximately 4,000 feet) was used in each park. Selection of lakes at or above this altitude was presumed to reduce the data variability associated with nitrogen fixation and geochemical processing by subalpine plant species, namely the red alder (*Alnus* sp.) (Rojas and others, 2001). Lakes at higher altitudes have been determined to have lower nutrient concentrations, making them more susceptible to trophic change through atmospheric inputs because of the lower amounts of terrestrial vegetation, higher percentage of bedrock within the watershed, and poorly drained soils (Fenn and others, 1998; Larson and others, 1999).

Avoid lakes with extensive fish populations: The stocking of alpine lakes with fish—particularly rainbow, cutthroat, and brook trout (*Oncorhynchus mykiss*, *Oncorhynchus clarki*, and *Salvelinus frontalis*, respectively)—has occurred in all three parks, most notably in MORA (May, 1966). Although this is no longer the practice, some of the lakes above 4,000 ft still contain fish and some populations have been maintained through illegal species reintroduction, presumably for sport-fishing purposes. Lakes with well-documented fish populations were excluded to further reduce data variability related to sediment-diatom communities and nitrogen content of the sediment, and to reduce bioturbation of the sediment as much as possible.

Minimum size and depth: During initial site-selection meetings with NPS scientists and managers, a decision was made to target lakes of a certain size and depth for this study. A goal was to study lakes that were not so shallow that other outside factors (such as wildlife disturbance) would influence the data collected, or so large that coring and sampling would be too difficult. As a result, an arbitrary lake size of less than 25 ha and a minimum water depth of 10 ft were selected for this study.

Trophic status: Because one of the goals of the project was to assess impacts from atmospheric nitrogen deposition, lakes that currently and historically were low in nutrients (oligotrophic) were desired. Nutrients are defined here as nitrogen and phosphorus ions important to biological growth and include ammonia, nitrate, and orthophosphate. These oligotrophic lakes should show a larger response to increases in nitrogen from deposition compared to a lake that has high nutrient levels.

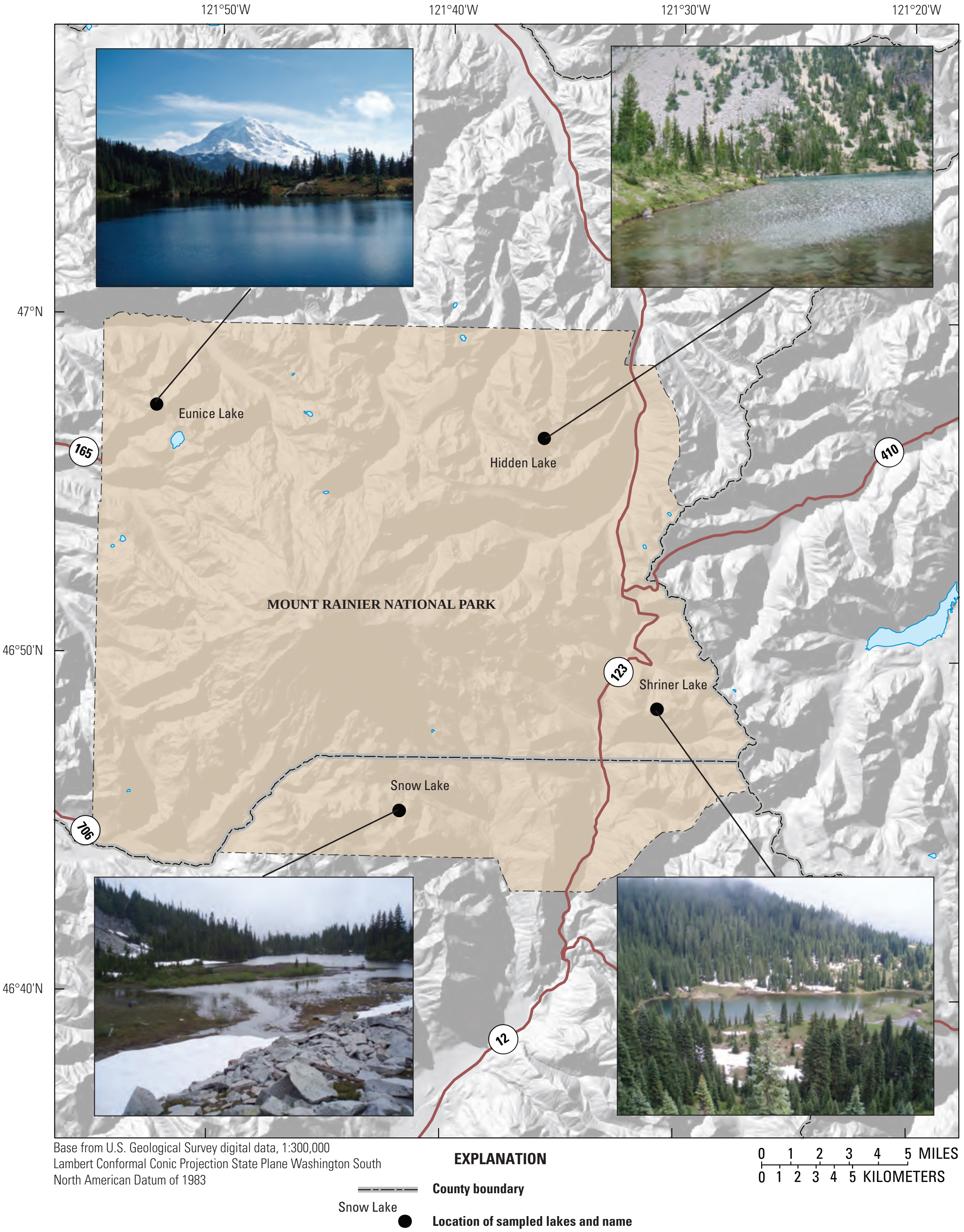
Build on existing data: Each of the NCCN parks maintains Long Term Ecological Monitoring (LTEM) programs at a subset of lakes within their jurisdictions. When existing LTEM sites fit within the other critical loads project site selection criteria, a LTEM site was selected to augment what was already known about these sites through the collection of new and complimentary data.

Based on these site selection criteria, staff from each park provided the USGS with a list of potential lake sites for comparison to the information gathered during the data-mining phase of this project. Lakes in the LTEM program that met the criteria for historical sampling, spatial distribution, and depth, size, and altitude limits were selected. At MORA and NOCA, two of the four lakes were part of the LTEM program, and at OLYM, all four lakes were part of the LTEM program. Properties of the study lakes selected for the project are listed in table 1, and shown in figures 2–4.

Table 1. Physical properties of selected study lakes used for the Pacific Northwest Critical Loads Project, Mount Rainier, North Cascades, and Olympic National Parks, Washington.

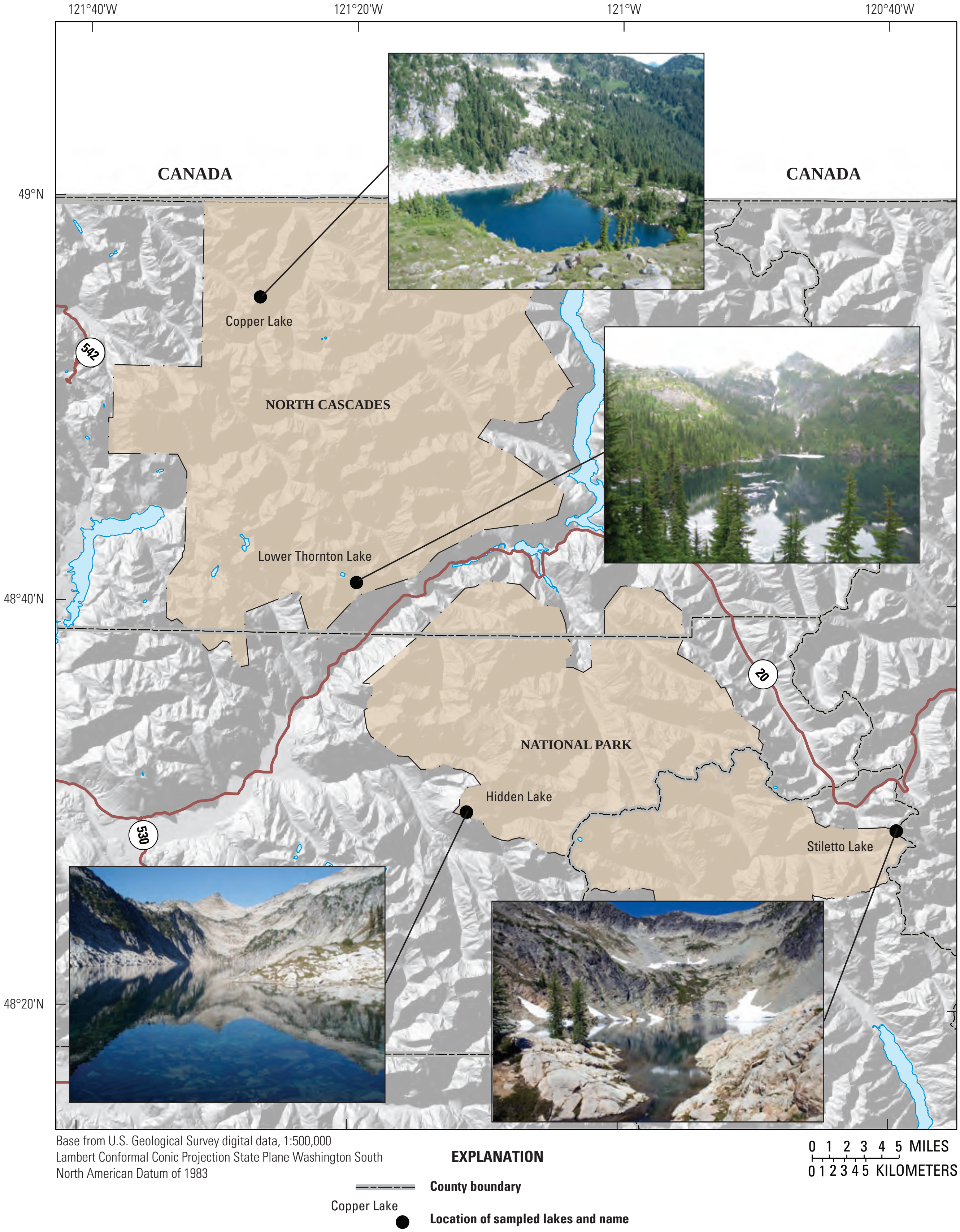
[Locations of study sites are shown in figures 2–4. **USGS Site No.**: Unique number for each site based on location. **Latitude and longitude**: At site, in decimal degrees, referenced to North American Datum of 1927 and National Geodetic Vertical Datum of 1929 (NGVD 29). **Lake elevation:** Referenced to NGVD 29. **Abbreviations**: ft, foot; ha, hectare; USGS, U.S. Geological Survey]

Site identifier	USGS site name	USGS site No.	Latitude	Longitude	Lake elevation (ft)	Lake area (ha)	Maximum depth (ft)
Mount Rainier National Park							
Eunice	Eunice Lake near Carbonado, WA	12091956	46.95563	121.87722	5,360	5.3	65
Hidden MORA	Hidden Lake near Greenwater, WA	12096700	46.94181	121.59812	5,930	2.1	23
Shriner	Shriner Lake near Packwood, WA	14223825	46.80900	121.51453	4,889	1.7	12
Snow	Snow Lake near Packwood, WA	14224590	46.75759	121.69825	4,680	2.4	32
North Cascades National Park							
Copper	Copper Lake near Glacier, WA	12215650	48.91818	121.45149	5,263	5.2	67
Hidden NOCA	Hidden Lake near Marblemount, WA	12181450	48.49583	121.18885	5,733	25	260
Lower Thornton	Lower Thornton Lake near Marblemount, WA	12178700	48.68420	121.32806	4,486	22	110
Stiletto	Stiletto Lake near Stehekin, WA	12450880	48.48197	120.65632	6,795	4.0	85
Olympic National Park							
Heather	Heather Lake near Sequim, WA	12047660	47.78508	123.17788	5,215	0.40	23
Hoh	Hoh Lake near Forks, WA	12040680	47.89872	123.78588	4,539	7.4	49
Milk	Milk Lake near Eldon, WA	12053810	47.62525	123.20604	4,708	1.1	46
PJ	PJ Lake near Port Angeles, WA	12047150	47.96069	123.42837	4,540	0.80	21



Base from U.S. Geological Survey digital data, 1:300,000
Lambert Conformal Conic Projection State Plane Washington South
North American Datum of 1983

Figure 2. Locations and views of study lakes in Mount Rainier National Park, Washington. (Photographs of Hidden, Shriner, and Snow Lakes taken by Rich W. Sheibley, U.S. Geological Survey; photograph of Eunice Lake taken by Jonathan Richards, U.S. Geological Survey, 2008.)



Base from U.S. Geological Survey digital data, 1:500,000
Lambert Conformal Conic Projection State Plane Washington South
North American Datum of 1983

Figure 3. Locations and views of study lakes in North Cascades National Park, Washington. (Photographs taken by Rich W. Sheibley, U.S. Geological Survey, 2008.)

Base from U.S. Geological Survey digital data, 1:550,000
Lambert Conformal Conic Projection State Plane Washington South
North American Datum of 1983

EXPLANATION

— — — **County boundary**

Milk Lake ● **Location of sampled lakes and name**

Figure 4. Locations and views of study lakes in Olympic National Park, Washington. (Photographs of Milk and PJ Lakes taken by Wendy Welch, U.S. Geological Survey, 2008; photographs of Heather and Hoh lakes taken by James R. Foreman, U.S. Geological Survey, 2008.)

Methodology

Many types of data were collected for this project, including estimates of atmospheric deposition of nitrogen and sulfur, lake surface-water quality, and sediment samples for various sediment chemical and physical properties and diatom identifications.

Atmospheric Deposition

Atmospheric deposition of nitrogen and sulfur was estimated at each of the 12 study lakes using a low-maintenance, passive deposition collector developed by Fenn and others (2002). This simple collector consists of a funnel attached to a small-diameter column that contains an ion exchange resin (IER; fig. 5). As rain water fell into the funnel, it passed through the IER column where anions and cations were sorbed onto the resin. After sufficient time at the field site, the columns were retrieved, capped, and sent to the laboratory for analysis. Columns were sent to the U.S. Forest Service (USFS) Pacific Southwest Research Station (PSRS) laboratory in Riverside, California, where this technique was developed for analysis of sorbed ions. In the laboratory, each column was extracted using a 2 normal potassium iodide solution, and the extract was analyzed for nitrate, ammonia, orthophosphate, and sulfate. Nitrite was not analyzed in any samples, as it is not commonly detected in atmospheric deposition. After the extraction process, ammonia was analyzed using a continuous flow analyzer, and nitrate, sulfate, and orthophosphate were analyzed using ion chromatography. Details of the laboratory analysis methods and results of field tests using this method are available in Fenn and others (2002, 2009) and Simkin and others (2004).

A set of five IER columns was deployed at each lake site during summer and collected in early autumn 2008. Five replicate columns were spread out around each lake basin, close to the shoreline, to account for site variability. Additionally, a control column that consisted of an IER column capped at both ends was left in the field at each site during the same period as the active columns. The control column from each site was used to account for the potential release of ammonia from the IER from natural diurnal heating and cooling as described by Fenn and others (2002). Other quality-control samples included IER columns spiked with low and high levels of nitrogen (N) and sulphur (S), which were sent to the laboratory as blind samples. All quality-control data for the IER columns are provided in appendix A.

The atmospheric deposition data are reported as the average total deposited mass of ammonia-nitrogen or nitrate-nitrogen and sulfur as sulfate from each site, after correction for background (control columns) and percent recovery of the extraction process using the following equation:

$$M_t = \left(M_e - M_c\right) / \left(percent\ recovery\right) \qquad (1)$$

where

M_t is the total mass of N or S,
M_e is the mass of N or S determined from the laboratory extraction,
M_c is the mass of N or S determined from the laboratory extracion of the field blank (control) columns, and
percent recovery is the percent recovery for N or S based on laboratory extractions of columns with a known mass of N or S.

Water Quality

Samples of surface water were collected at each of the study lakes to provide a snapshot of lake-water quality. These samples were collected prior to sediment coring, from the water surface (within upper 1 m of depth), and above the deepest part of the lake. Parameters analyzed followed the analytical suite recommended by the Water Resources Division of the NPS in their LTEM program, and included nutrients (nitrate plus nitrite, ammonia, orthophosphate, and unfiltered total nitrogen and total phosphorus), chlorophyll-*a*, pheophytin-*a*, acid neutralizing capacity (ANC), and major ions. Samples collected for total nitrogen, total phosphorus, chlorophyll-*a*, and pheophytin-*a* were unfiltered. Samples for major ions (two bottles, one acidified to a pH of less than 2 with nitric acid), nitrate plus nitrite, ammonia, and orthophosphate were filtered in the field using a 60-cm^3 syringe and a 0.45- or 0.22-µm membrane filter. Samples were kept cool during transport from each field site and nutrient samples were frozen immediately upon return to laboratory or office. All samples were collected and processed according to methods outlined in the USGS National Field Manual (Wilde and others, 2004; U.S. Geological Survey, 2006), with the exception of nutrient samples. USGS methods do not require freezing of nutrient samples; however, the nutrient samples for this project were analyzed by the Cooperative Chemical Analytical Laboratory (CCAL) at Oregon State University, Corvallis, whose procedures include freezing of nutrient samples to preserve them before analysis. The ANC of each lake was determined in the laboratory by titration and recorded as milligrams per liter of calcium carbonate (CaCO3) using the inflection point method (Rounds, 2006). Samples collected for chlorophyll-a and pheophytin-a were filtered onto a 0.45-µm glass-fiber filter disk, folded, wrapped in aluminum foil, and frozen until analysis.

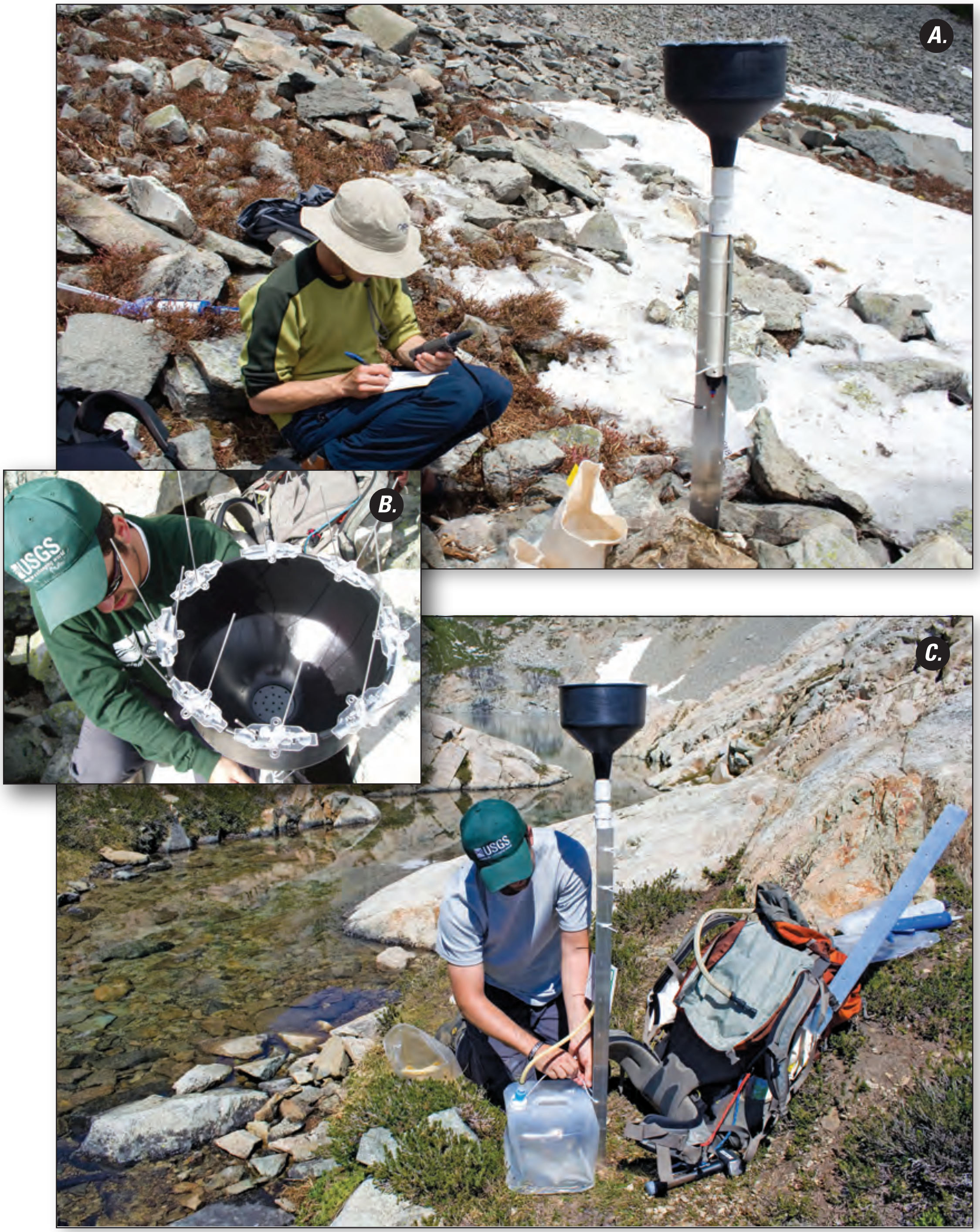


Figure 5. Passive deposition collectors placed at selected lakes, Mount Rainier, North Cascades, and Olympic National Parks, Washington. (*A*) U.S. Geological Survey hydrologist recording coordinates of a passive deposition collector at Eunice Lake in Mount Rainier National Park. The funnel (black) directs rainwater through the ion exchange resin (white); (*B*) small plastic and metal bird deterrents are attached to the top edge of the funnel to eliminate contamination from birds at Snow Lake in Mount Rainier National Park); and (*C*) two of the five collectors at each site have a collapsible plastic bag attached to the outlet of the resin column to estimate precipitation volumes at the sites (Stiletto Lake, North Cascades National Park). (Photographs *A* and *C* taken by Jonathan Richards, 2008; photograph *B* taken by Rich W. Sheibley, U.S. Geological Survey, 2008.)

Major anions, cations, chlorophyll-*a*, and pheophytin-*a* were analyzed by the USGS National Water Quality Laboratory (NWQL) in Lakewood, Colorado, using established methods (Fishman and Friedman, 1989; Fishman, 1993; Arar and Collins, 1997; American Public Health Association, 2005). Nutrient samples were analyzed by CCAL using a continuous-flow autoanalyzer following standard methods (Ameel and others, 1993; American Public Health Association, 2005). The decision to send samples to CCAL for nutrient analysis for this project was based on their ability to determine low-level nutrient concentrations and to maintain data consistency because the NCCN parks use CCAL for their LTEM nutrient analyses. All water-quality data for this project were censored at either the laboratory reporting limit or method reporting limit (Childress and others, 1999) for samples analyzed by the NWQL (major ions, chlorophyll-*a*, and pheophytin-*a*), or at the method detection limit for nutrients as defined by CCAL (Cooperative Chemical Analytical Laboratory, 2012).

Sediment Cores

Accurate paleolimnological reconstructions can be derived from lake sediments, specifically samples collected from lakes in wilderness areas (Last and Smol, 2001; Šporka and others, 2002). For this study, lake sediment cores were collected using a gravity corer (UWITEC, Inc.) and a pontoon boat that was assembled on site (fig. 6). All coring equipment was transported on foot or by helicopter to the site. Cores were collected from the deepest part of each lake. Depths were determined using a combination of lake bathymetry maps and echo-sounding with a commercially available depth finder. After the deepest part of the lake was identified, the gravity corer was lowered from the pontoon boat to 15 m above the lake bottom and allowed to fall freely. The corer was then reeled onto the boat by hand and the sample was inspected. Cores with a disrupted sediment-water interface, a steeply angled sediment surface, or with total core lengths of less than 20 cm were not used. Cores of sufficient length (target 30 cm) with a clear and level sediment-water interface were stored upright on the boat until a total of four cores were collected.

Once on shore, the cores were covered in an insulating sleeve and kept from direct sunlight prior to processing. A 2-cm surface sample was collected from one of the cores for analysis of sediment carbon (C) and nitrogen (for calculation of C:N ratios) and the Nitrogen-15 to Nitrogen-14 ratio ($^{15}N/^{14}N$). The remainder of the core was discarded. Two of the remaining cores were selected for diatom analysis and dating, based on visual similarities and overall length. Cores were sectioned in 0.5-cm increments for the first 10 cm, and then every 1 cm thereafter until expended. Subsamples were collected using a calibrated, screw-type, stainless steel extruder with the same equipment and field procedures used by Landers and others (2008) and collected in clean, resealable plastic bags. Sectioned samples were kept shaded and cool to prevent further chemical or metabolic processing.

When weather and time permitted, lake cores were sectioned at the field site. Samples collected from Heather and Milk lakes in OLYM, and Hidden Lake in NOCA were transported intact to a processing location by helicopter. The core from Hoh Lake in OLYM was transported out on foot, secured to an internal-frame backpack, after the first 10 cm of the core was sectioned and stored. This helped to stabilize the core for transport over land since the overlying water and upper flocculent sediments were removed. Of the 12 study lakes, cores were collected from 10 lakes: 3 in MORA, 3 in OLYM, and 4 in NOCA. Cores were not collected from two lakes because of sediment disturbances: PJ Lake in OLYM (avalanche disturbance) and Shriner Lake in MORA (high elk disturbance of lake sediments; lake was relatively shallow).

Sediment Chemistry

One surface sample (upper 2-cm section) from each lake was processed in the USGS Washington Water Science Center laboratory for determination of sediment carbon-to-nitrogen ratios as well as sediment $\delta^{15}N$, defined by (Révész and others, 2011):

$$\delta^{15}N = \delta\left(\frac{^{15}N}{^{14}N}\right) = \frac{R\left(\frac{^{15}N}{^{14}N}\right)sed - R\left(\frac{^{15}N}{^{14}N}\right)std}{R\left(\frac{^{15}N}{^{14}N}\right)std} \tag{2}$$

where

R is the isotopic ratio of ^{15}N to ^{14}N in the sediment sample (sed) or standard (std).

Values of $\delta^{15}N$ are reported as per mil (‰).

Figure 6. Collection of sediment cores at study lakes, Mount Rainier, North Cascades, and Olympic National Parks, Washington. (*A*) U.S. Geological Survey staff preparing the pontoon boat and gravity corer at Stiletto Lake, North Cascades National Park. Cores collected from the deep part of the lake were (*B*) reeled in by hand, (*C*) visually inspected, and (*D*) secured in an upright core holder for transport to shore. On land, (*E*) cores were extruded using a calibrated screw-type device, then (*F*) sediment sections were scraped into clean resealable plastic bags at 0.5 centimeter intervals for the first 10 centimeters, and then every 1 centimeter for the rest of the core. (Photographs (*A*), (*B*), and (*D*) by Rich W. Sheibley, 2009; photographs (*C*), and (*E*) by J. Foreman, 2009; and photograph (*F*) by S. Henneford, 2009.)

Surface sediment samples were stored in a resealable plastic bag and dried at 60°C for at least 24 hours until the sample was a constant weight. After drying, this sample was mixed using a mortar and pestle to grind the sample into a fine powder. Ground surface samples were then divided into two subsamples and placed in clean scintillation vials. One subsample was analyzed for total carbon and total nitrogen, and the other subsample was analyzed for sediment $\delta^{15}N$.

The total carbon and total nitrogen values were used to calculate sediment carbon-to-nitrogen ratios (C:N). Sediment carbon and nitrogen were determined at the University of California, Davis, Analytical Laboratory, and $\delta^{15}N$ was analyzed at the USGS Reston Stable Isotope Laboratory using accepted methods (Association of Official Agricultural Chemists International, 1997; Révész and others, 2011).

For each sediment slice, the wet weight was recorded before and after the diatom subsample was removed and each sediment section was dried (60°C for at least 24 hours) in its corresponding resealable plastic bag. Dried sediment samples were weighed and, given the mass of sediment removed from the diatom subsample, the original dry weight was calculated. This original dry weight was divided by the known volume of each sediment slice (by multiplying the diameter of the core by its section interval, 0.5 or 1.0 cm) to get the dry bulk density (mass divided by volume). Dry bulk densities were used to estimate the sedimentation rate for each lake and to determine the age of each sediment section (data not shown).

Sediment Geochronologies

The development of a coherent sediment geochronology is the foundation for understanding how a lake has evolved over recent time. A geochronology can help discern how input functions or perturbations within a watershed may have changed historically and consequently altered the ecological setting. The application of natural Uranium/Thorium-series and artificial (man-made, derived from past nuclear weapons testing) radionuclides as reliable geochronometers is well demonstrated (Ivanovich and Harmon, 1992; Swarzenski and others 2003; Swarzenski, 2007). Of these radionuclides, lead-210 (^{210}Pb) and cesium-137 (^{137}Cs) have been used extensively to reconstruct a record of sedimentation for relatively young (less than 100 years) sediment deposits (Appleby and Oldfield, 1978; Appleby, 2008). Lead-210 ($t_{1/2}$ = 22.3 yr) is a member of the uranium-238 (^{238}U) decay series and is delivered primarily to the surface of the Earth by natural atmospheric fallout. Because ^{210}Pb has a high affinity to adsorb onto particle surfaces, it is rapidly removed from a water column to sediment. This phase exchange occurs on the order of days in particle-laden water. The ^{210}Pb removed from the water column by particles will have a higher ^{210}Pb activity than the activity of its indirect radiogenic parent, radium-226 (^{226}Ra). As a result, there are two pools of ^{210}Pb—one is the parent-supported pool whose activity is considered close to that of ^{226}Ra, and the other is the unsupported (termed “excess”) ^{210}Pb pool, or “xs” ^{210}Pb. Sediment accumulation rates are derived from these xs^{210}Pb activities, and these sediment accumulation rates are used to calculate an approximate age of the sediment using various sedimentation models (see Appleby, 2008).

Dry sediment samples were ground to a fine powder at the USGS Washington Water Science Center laboratory using a mortar and pestle, and known amounts of the powdered samples were sealed in vials to allow for the ingrowth of daughter products of radon-222 (^{222}Rn). Samples were sent to the USGS Pacific Coastal and Marine Geology Science Center, Santa Cruz, Calif., where activities of ^{210}Pb, ^{137}Cs, and ^{226}Ra were determined using a high purity germanium (HPGe) well detector (Princeton Gamma Tech Instruments, Inc.) using established methods (Swarzenski and others, 2006; Swarzenski and others, 2008). Total ^{210}Pb activity was determined by measuring the 46.5 kilo electron volt (keV) gamma peak. Parent-supported levels of ^{210}Pb were determined by measuring the activity of ^{214}Pb (352 keV) and bismuth (^{214}Bi) (609 keV). Cs-137 activities were determined by measuring the 661.6-keV peak. Typically, the samples were counted for 24 hours. Self-absorption corrections were not applied because the standards used to calibrate the counting system and the environmental samples had similar densities and detector geometry. Efficiency curves for the HPGe well detectors were obtained using a suite of International Atomic Energy Agency gamma ray spectrometry reference materials (RGU-1, RGTh-1, and IAEA-300) using the same volume of standard as the sediment samples.

Diatom Analysis

After sectioning the lake core, a subsample from each resealable plastic bag was removed for diatom analysis. Diatom subsamples were collected in the USGS Washington Water Science Center laboratory in Tacoma, Washington, by gently stirring the sediment sample with a metal spatula, and scooping out a small amount of wet sediment for placement in a small scintillation vial. A total of 1–3 mL of wet material was used for classifying diatom communities.

Diatom samples were refrigerated until they were shipped to the phycology laboratory at the Patrick Center for Environmental Research at the Academy of Natural Sciences in Philadelphia, Pennsylvania (ANSP). Upon receipt at ASNP, sediment samples were digested with nitric acid in a microwave apparatus and mounted with Naphrax™ on glass slides. All samples were processed and analyzed following the ANSP Phycology Section standard operating procedures that are contained in Charles and others (2002). Two permanent diatom slides were made from each sediment sample. A suspension of cleaned diatom frustules was diluted repeatedly to remove residual acid and a subsample was spread evenly onto glass coverslips and allowed to dry. The glass coverslips were then mounted on glass slides using Naphrax™ mounting medium.

A minimum of 500 diatom valves were identified for each sample at a magnification of at least 1,000 times using a Leica™ DM LB2 microscope. The diatom taxonomy primarily was based on the references of Krammer and Lange-Bertalot (1986, 1988, 1991a, 1991b), Camburn and Cumming and others (1995), Charles (2000), Tanaka (2007), and taxonomic harmonization within the National Water Quality Assessment Program (NAWQA) reports (Academy of Natural Sciences, 2012). Each diatom species identified was assigned a unique North American Diatom Ecological Database (NADED) identifier in order to track and to adjust for any future changes in diatom taxonomy that might occur.

Quality of Data

Various quality-control data (field replicates, laboratory replicates, and field blanks) were collected for lake surface-water, sediments, and atmospheric deposition data. All quality-control data are provided in appendix A. In general, replicate and blank data for lake surface-water were within the project data-quality objectives. For field and laboratory replicates, the relative difference was less than 10 percent for duplicates with concentrations of greater than 0.5 mg/L and less than 20 percent for duplicates with concentrations less than 0.5 mg/L. For low concentrations (less than 0.1mg/L), the absolute difference was less than 0.050. For field blanks, all reported concentrations were less than the reporting limit for each analyzed parameter. For a few (non-nutrient) parameters, data did not meet these criteria and are outlined in appendix A. However, the lake-surface water was acceptable and of good quality overall.

Field and laboratory replicate data for surface sediment conformed to the project data-quality objectives for carbon and nitrogen content, which was a relative percent difference limit of 15 percent or less. For sediment $\delta^{15}N$ ratios, an absolute difference of 1.0 per mil was the targeted acceptance level.

Field blanks and laboratory spiked IER columns were used to assess data quality for determination of atmospheric deposition data. Average field blank values (M_c) for ammonia, nitrate, and sulfur were 0.02 mg of nitrogen, 0.03 mg of nitrogen, and 0.41 mg sulfate, respectively. Percent recovery values used in equation 1 for ammonia, nitrate, and sulfate are 96, 90, and 90 percent, respectively. Details on the determination of field blank and percent recovery data for the extraction process are presented in appendix A.

Atmospheric Deposition Data

Atmospheric deposition data were collected from the 12 study lakes for a minimum of 2 months (from 59 to 86 days) during summer 2008 (table 2). The data in table 2 are reported as the average total deposited mass of ammonia-nitrogen or nitrate-nitrogen and sulfur as sulfate from each site, after correction for background (control columns) and percent recovery of the extraction process using equation 1. The average values are from the 5 passive collectors that were deployed at each lake site. At Shriner Lake, four of the five columns were damaged as a result of large wildlife (elk) presence. At Hidden NOCA and Stiletto Lakes, only four of the five columns were located because snowfall the day before recovery made it difficult to locate the equipment.

Generally, mass deposition of sulfur was greater than either ammonia or nitrate across all sites, followed by nitrate, then ammonia. However, the mass deposition of ammonia was greater than nitrate at Eunice, Hidden MORA, and Shriner in MORA, and Hoh in OLYM. Total inorganic nitrogen deposition (the sum of ammonia and nitrate) was comparable to or greater than sulfur deposition across all sites. Depositional mass data in table 2 can be converted to a time- and area-weighted value by dividing the mass (in milligrams) by the number of days the columns were deployed, and the cross-sectional area of the funnels (54.5 in^2) to facilitate cross-site comparisons.

Table 2. Atmospheric deposition data at selected lakes in Mount Rainier, North Cascades, and Olympic National Parks, Washington, summer 2008.

[Locations of study sites are shown in figures 2–4. Values represent average depositional mass from passive collectors at each site after correction for field blanks and percent recovery (see text and appendix A for details). Values in parentheses are the standard deviation of the data. **Inorganic nitrogen**: Sum of ammonia nitrogen and nitrate nitrogen. mg, milligram]

Site identifier	Deployment date	Retrieval date	Number of days deployed	Number of columns	Average atmospheric deposition (mg)			
					Ammonia-nitrogen	Nitrate nitrogen	Inorganic nitrogen	Sulfate sulfur
Mount Rainier National Park								
Eunice	07-31-08	10-15-08	76	5	0.779	0.661	1.440	1.390
					(0.071)	(0.089)	(0.137)	(0.318)
Hidden MORA	07-23-08	10-16-08	85	5	0.476	0.401	0.878	0.965
					(0.143)	(0.144)	(0.280)	(0.313)
Shriner	07-23-08	10-17-08	86	1	0.781	0.467	1.250	0.868
					[1]	[1]	[1]	[1]
Snow	07-22-08	10-01-08	71	5	0.345	0.397	0.742	0.987
					(0.095)	(0.085)	(0.149)	(0.302)
North Cascades National Park								
Copper	08-12-08	10-10-08	59	5	0.604	0.683	1.290	1.290
					(0.121)	(0.063)	(0.182)	(0.227)
Hidden NOCA	07-30-08	10-10-08	72	4	0.697	0.703	1.400	1.180
					(0.048)	(0.153)	(0.129)	(0.401)
Lower Thornton	07-30-08	10-09-08	71	5	0.748	0.902	1.650	1.430
					(0.143)	(0.133)	(0.184)	(0.477)
Stiletto	08-05-08	10-10-08	66	4	0.293	0.462	0.756	0.712
					(0.262)	(0.151)	(0.254)	(0.172)
Olympic National Park								
Heather	08-18-08	10-22-08	65	5	0.155	0.283	0.437	0.535
					(0.094)	(0.067)	(0.060)	(0.304)
Hoh	08-23-08	10-22-08	60	5	0.284	0.276	0.560	1.350
					(0.145)	(0.087)	(0.173)	(0.396)
Milk	08-12-08	10-19-08	68	5	0.319	0.360	0.679	0.855
					(0.047)	(0.076)	(0.085)	(0.207)
PJ	08-05-08	10-24-08	80	5	0.188	0.263	0.452	0.963
					(0.086)	(0.033)	(0.055)	(0.129)

[1] No standard deviation because only 1 column was retrieved.

Water-Quality Data

Data for all water-quality environmental samples are presented in tables 3 and 4. Concentrations of nutrients at all sites were very low (table 3), and, for filtered samples, often equal to or less than the laboratory minimum detection limit. Concentrations of total nitrogen and phosphorus from unfiltered samples were also low. Generally, total nitrogen was always greater than total phosphorus. Phytoplankton metrics, chlorophyll-*a* and pheophytin-*a* also were low, and often equal to or less than the NWQL method reporting limits (Childress and others, 1999).

Specific conductance in water samples from all lakes was low (less than 100 µS/cm), and less than 20 µS/cm in all lakes in MORA and NOCA (table 4). Similarly, ANC was low (less than 5 mg/L [as $CaCO_3$]) in water samples collected from lakes in MORA and NOCA and although higher, still less than 20 mg/L (as $CaCO_3$) in water samples collected from lakes in OLYM. Major anions and cations across all sites typically were low (table 4) and, when combined with information on specific conductance and ANC, indicate that most high-elevation lake waters are dilute. No lake surface-water quality samples were collected for Shriner or PJ Lakes because these sites were too disturbed to warrant lake coring and subsequent diatom analysis.

Table 3. Field parameters, nutrients, and phytoplankton data from selected lakes, Mount Rainier, North Cascades, and Olympic National Parks, Washington, August 2009–August 2010.

[Locations of study sites are shown in figures 2–4. All samples collected from upper 1 meter of the water column over the deepest part of the lake. Constituents were analyzed from the same bottle collected in the field. Number in parentheses in column headings represent the U.S. Geological Survey National Water Information System parameter codes. **Site Identifier**: Hidden MORA, Hidden Lake in Mount Rainier National Park; Hidden NOCA, Hidden Lake in North Cascades National Park. **Abbreviations**: °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 degrees Celsius; N, nitrogen; P, phosphorus; mg/L, milligram per liter; µg/L, microgram per liter; E, estimated; NA, not analyzed; <, less than; –, no sample]

			Field parameters			Nutrients in filtered lake water			Nutrients in unfiltered lake water		Phytoplankton	
Site identifier	Sample date	Time	Temperature (°C) (00010)	Specific conductance (µS/cm) (00095)	pH (00400)	Ammonia (mg/L as N) (00608)	Nitrate plus nitrite (mg/L as N) (00631)	Orthophosphate (mg/L as P) (00671)	Total nitrogen (mg/L as N) (62855)	Total phosphorous (mg/L as P) (00665)	Chlorophyll *a* (µg/L) (70953)	Pheophytin *a* (µg/L) (62360)
Mount Rainier National Park												
Eunice	11-04-09	1215	5.50	7.5	6.5	<0.01	0.002	<0.001	0.25	0.01	0.40E	0.20E
Hidden MORA	08-19-10	1230	10.5	16	6.7	<0.01	0.001	0.001	0.060	0.004	0.70E	0.21E
Shriner	–	–	–	–	–	–	–	–	–	–	–	–
Snow	10-07-09	1230	12.3	13	5.4	<0.01	<0.001	<0.001	0.090	0.006	1.5E	0.42E
North Cascades National Park												
Copper	09-22-09	1130	6.60	5.6	6.1	<0.01	<0.001	<0.001	0.070	0.004	0.54	0.24
Hidden NOCA	09-24-09	1120	6.60	5.6	6.7	<0.01	0.001	<0.001	0.050	0.002	0.23	<0.10
Lower Thornton	09-23-09	1130	17.4	10	6.9	<0.01	0.004	<0.001	0.10	0.003	0.80	0.50
Stiletto	08-26-09	1255	10.8	9.4	6.3	<0.01	0.006	0.003	0.27	0.007	1.03	0.40
Olympic National Park												
Heather	09-10-09	1000	18.3	71	7.2	<0.01	0.007	0.001	0.060	0.005	0.62	0.14
Hoh	09-08-09	1410	5.60	62	7.5	<0.01	0.001	<0.001	0.16	0.007	0.63	0.28
Milk	09-11-09	1230	7.50	37	7.2	<0.01	<0.001	<0.001	0.040	0.004	0.28	<0.10
PJ	–	–	–	–	–	–	–	–	–	–	–	–

Table 4. Field parameters, acid neutralizing capacity, and major anion and cation data in water column from selected lakes, Mount Rainier, North Cascades, and Olympic National Parks, Washington, August 2009–August 2010.

[Locations of study sites are shown in figures 2–4. All samples collected from the upper 1 meter of the water column over the deepest part of the lake. Constituents were analyzed from the same bottle collected in the field. Number in parentheses in column headings represent the U.S. Geological Survey National Water Information System parameter codes. **Abbreviations**: °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 degrees Celsius; $CaCO_3$, calcium carbonate; mg/L, milligram per liter; µg/L, microgram per liter; E, estimated; NA, not analyzed; <, less than; –, no sample]

Site identifier	Sample date	Time	Field parameters			Acid neutralizing capacity (mg/L as $CaCO_3$) (00419)	Major anions and cations in filtered lake water									
			Temperature (°C) (00010)	Specific conductance (µS/cm) (00095)	pH (00400)		Chloride (mg/L) (00940)	Sulfate (mg/L) (00945)	Silica (mg/L) (00955)	Fluoride (mg/L) (00950)	Calcium (mg/L) (00915)	Magnesium (mg/L) (00925)	Sodium (mg/L) (00930)	Potassium (mg/L) (00935)	Iron (µg/L) (01046)	Manganese (µg/L) (01056)
Mount Rainier National Park																
Eunice	11-04-09	1215	5.50	7.5	6.5	1.50	0.412	0.154E	1.95	<0.08	0.694	0.073	0.538	0.39E	<6.0	0.390
Hidden MORA	08-19-10	1230	10.5	16	6.7	2.30	0.243	0.331	5.94	<0.08	2.068	0.20	0.717	0.24	3.2E	0.782
Shriner	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
Snow	10-07-09	1230	12.3	13	5.4	0.50	0.280	0.470	4.36	<0.08	1.390	0.12	0.740	0.11	10	1.40
North Cascades National Park																
Copper	09-22-09	1130	6.60	5.6	6.1	1.30	0.116E	0.370	1.63	<0.08	0.523	0.070	0.230	0.13	<4.0	0.825
Hidden NOCA	09-24-09	1120	6.60	5.6	6.7	1.50	0.146	0.215	0.956	<0.08	0.705	0.031	0.148	0.17	<4.0	0.252
Lower Thornton	09-23-09	1130	17.4	10	6.9	2.50	0.191	0.436	1.27	<0.08	0.533	0.073	0.203	0.16	3.4E	1.57
Stiletto	08-26-09	1255	10.8	9.4	6.3	4.50	<0.120	0.638	1.26	<0.08	1.24	0.066	0.186	0.15	<4.0	1.33
Olympic National Park																
Heather	09-10-09	1000	18.3	71	7.2	18.7	0.190	7.94	3.022	<0.08	10.6	0.90	1.092	0.18	2.4E	1.26
Hoh	09-08-09	1410	5.60	62	7.5	19.7	0.704	3.70	2.16	<0.08	9.18	0.59	0.969	0.085	2.2E	0.739
Milk	09-11-09	1230	7.50	37	7.2	14.4	0.214	0.61	1.23	<0.08	6.05	0.30	0.275	0.097	5.4	0.400
PJ	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–

Sediment Chemistry Data

Surface sediments (upper 2 cm in depth) were analyzed for percent carbon, percent nitrogen, and $\delta^{15}N$ (table 5). Nitrogen ranged from 0.17 to 2.0 percent, and carbon ranged from 1.96 to 22.4 percent across all lakes. Carbon and nitrogen percentages were used to determine the molar carbon-to-nitrogen ratio (C:N) for surface sediments, and ranged from 11.2 to 18.3. The $\delta^{15}N$ of the surface sediments ranged from values of -1.2 to 2.55 ‰ across all samples.

Table 5. Carbon, nitrogen, and selected ratios from lake sediment cores, Mount Rainier, North Cascades, and Olympic National Parks, Washington, August 2009–August 2010.

[Locations of study sites are shown in figures 2–4. **Abbreviations**: g, gram; ^{15}N, nitrogen isotope 15; N, nitrogen; ‰, per mil; –, not analyzed]

Site identifier	Sample date	Time	Sample net weight (g)	Total nitrogen (percent)	Total carbon (percent)	Molar carbon to nitrogen ratio	$\delta^{15}N$ (‰)
Mount Rainier National Park							
Eunice	11-04-09	1215	1.0028	1.2	12.3	12.4	0.900
Hidden MORA	08-19-10	1230	–	–	–	–	-0.0300
Snow	10-07-09	1230	1.0019	0.39	5.47	16.3	-1.20
North Cascades National Park							
Copper	09-22-09	1130	1.0040	0.83	10.3	14.6	0.660
Hidden NOCA	09-24-09	1120	1.0029	0.63	7.54	13.9	2.55
Lower Thornton	09-23-09	1130	1.0099	0.84	13.1	18.3	1.060
Stiletto	08-26-09	1255	0.9985	2.0	22.4	13.3	1.15
Olympic National Park							
Heather	09-10-09	1000	0.9928	0.33	3.62	13.0	0.280
Hoh	09-08-09	1410	1.0098	0.84	8.060	11.2	0.820
Milk	09-11-09	1230	1.0008	0.17	1.96	13.7	0.150

Sediment Geochronology Data

Four of the 10 lakes cored were analyzed for natural and artificial radionuclides for determination of sedimentation rates, from which the age of each sediment slice within a sediment core could be calculated. Excess ^{210}Pb, ^{226}Ra, and ^{137}Cs were measured for Snow Lake at MORA, Copper Lake at NOCA, and Hoh and Milk Lakes at OLYM (tables 6–9). Generally, these data successfully described the geochronology in each core as evidenced by a decrease in excess ^{210}Pb to near background (0.0 disintegrations per minute per gram [dpm/g]), and peaks in the ^{137}Cs. The first presence of ^{137}Cs greater than background and the peak of ^{137}Cs provide two time markers, 1953 and 1964, respectively, based on fallout from atomic testing (Appleby, 2008). The raw data in tables 6 through 9 can be used to calculate approximate age of each sediment slice.

Table 6. Bulk density and select radionuclide data from sediment core collected at Snow Lake, Mount Rainier National Park, near Packwood, Washington, October 7, 2009.

[Depths are in centimeters. **Abbreviations**: g/cm^3, gram per cubic centimeter; g/cm^2, gram per square centimeter; dpm/g, disintegrations per minute per gram; ±, plus or minus; –, not analyzed]

Depth	Mid-depth	Dry bulk density (g/cm^3)	Cumulative mass (g/cm^2)	Total lead-210 (dpm/g)	Radium-226 (dpm/g)	Excess lead-210 (dpm/g)	Cesium-137 (dpm/g)
0–0.5	0.25	0.23	0.11	32.0 ± 0.8	3.0 ± 0.2	29.0	5.2
0.5–1.0	0.75	0.11	0.17	–	–	–	–
1.0–1.5	1.25	0.29	0.31	30.6 ± 0.9	3.3 ± 0.1	27.3	6.2
1.5–2.0	1.75	0.30	0.46	–	–	–	–
2.0–2.5	2.25	0.32	0.63	27.5 ± 0.8	2.7 ± 0.2	24.9	5.8
2.5–3.0	2.75	0.36	0.81	26.4 ± 1.3	3.2 ± 0.2	23.3	6.4
3.0–3.5	3.25	0.39	1.00	16.0 ± 0.9	2.9 ± 0.1	13.1	6.8
3.5–4.0	3.75	0.37	1.19	–	–	–	–
4.0–4.5	4.25	0.35	1.36	12.3 ± 1.1	2.8 ± 0.1	9.5	5.9
4.5–5.0	4.75	0.31	1.52	–	–	–	–
5.0–5.5	5.25	0.30	1.67	7.8 ± 0.7	3.1 ± 0.1	4.6	1.6
5.5–6.0	5.75	0.31	1.82	–	–	–	–
6.0–6.5	6.25	0.29	1.96	5.1 ± 0.6	2.7 ± 0.1	2.4	1.0
6.5–7.0	6.75	0.28	2.10	–	–	–	–
7.0–7.5	7.25	0.27	2.24	2.4 ± 1.4	2.7 ± 0.1	-0.3	0.8
7.5–8.0	7.75	0.30	2.39	–	–	–	–
8.0–8.5	8.25	0.32	2.55	–	–	–	–
8.5–9.0	8.75	0.46	2.78	–	–	–	–
9.0–9.5	9.25	0.44	3.00	–	–	–	–
9.5–10.0	9.75	0.35	3.17	–	–	–	–
10.0–11.0	10.5	0.31	3.48	–	–	–	–
11.0–12.0	11.5	0.29	3.77	–	–	–	–
12.0–13.0	12.5	0.25	4.02	–	–	–	–
13.0–14.0	13.5	0.28	4.30	–	–	–	–
14.0–15.0	14.5	0.30	4.60	–	–	–	–
15.0–16.0	15.5	0.44	5.03	1.0 ± 0.5	3.2 ± 0.1	-2.2	0.0

Table 7. Bulk density and select radionuclide data from sediment core collected at Copper Lake, North Cascades National Park, near Glacier, Washington, September 22, 2009.

[Depths are in centimeters. **Abbreviations**: g/cm³, gram per cubic centimeter; g/cm², gram per square centimeter; dpm/g, disintegrations per minute per gram; ±, plus or minus; –, not analyzed]

Depth	Mid-depth	Dry bulk density (g/cm³)	Cumulative mass (g/cm²)	Total lead-210 (dpm/g)	Radium-226 (dpm/g)	Excess lead-210 (dpm/g)	Cesium-137 (dpm/g)
0–0.5	0.25	0.069	0.034	63.4 ± 1.5	0.6 ± 0.2	62.8	17.3
0.5–1.0	0.75	0.100	0.084	–	–	–	–
1.0–1.5	1.25	0.101	0.135	55.9 ± 1.4	0.7 ± 0.2	55.2	21.8
1.5–2.0	1.75	0.109	0.190	–	–	–	–
2.0–2.5	2.25	0.113	0.246	53.3 ± 1.4	1.0 ± 0.2	52.3	21.4
2.5–3.0	2.75	0.114	0.303	–	–	–	–
3.0–3.5	3.25	0.110	0.358	51.0 ± 1.2	1.0 ± 0.2	50.0	22.7
3.5–4.0	3.75	0.099	0.407	–	–	–	–
4.0–4.5	4.25	0.116	0.465	38.3 ± 1.0	0.7 ± 0.2	37.6	18.0
4.5–5.0	4.75	0.125	0.527	–	–	–	–
5.0–5.5	5.25	0.133	0.594	20.4 ± 0.9	0.9 ± 0.1	19.5	11.6
5.5–6.0	5.75	0.130	0.659	–	–	–	–
6.0–6.5	6.25	0.131	0.724	12.2 ± 0.7	0.7 ± 0.1	11.5	7.5
6.5–7.0	6.75	0.131	0.790	–	–	–	–
7.0–7.5	7.25	0.133	0.857	13.5 ± 1.1	1.5 ± 0.2	11.9	6.4
7.5–8.0	7.75	0.139	0.926	–	–	–	–
8.0–8.5	8.25	0.146	0.999	8.5 ± 0.8	0.9 ± 0.1	7.6	3.3
8.5–9.0	8.75	0.128	1.064	–	–	–	–
9.0–9.5	9.25	0.121	1.124	6.2 ± 0.9	1.3 ± 0.2	4.9	2.8
9.5–10.0	9.75	0.538	1.393	–	–	–	–
10.0–11.0	10.5	0.141	1.464	5.4 ± 0.9	1.0 ± 0.2	4.4	2.2
11.0–12.0	11.5	0.190	1.654	–	–	–	–
12.0–13.0	12.5	0.148	1.803	–	–	–	–
13.0–14.0	13.5	0.158	1.961	–	–	–	–
14.0–15.0	14.5	0.109	2.070	3.3 ± 0.6	1.0 ± 0.1	2.3	1.0
15.0–16.0	15.5	0.121	2.191	–	–	–	–
16.0–17.0	16.5	0.138	2.329	–	–	–	–
17.0–18.0	17.5	0.138	2.467	–	–	–	–
18.0–19.0	18.5	0.154	2.621	–	–	–	–
19.0–20.0	19.5	0.122	2.744	–	–	–	–
20.0–21.0	20.5	0.084	2.828	4.4 ± 0.6	1.2 ± 0.2	3.3	1.3

Table 8. Bulk density and select radionuclide data from sediment core collected at Hoh Lake, Olympic National Park, near Forks, Washington, September 8, 2009.

[Depths are in centimeters. **Abbreviations**: g/cm^3, gram per cubic centimeter; g/cm^2, gram per square centimeter; dpm/g, disintegrations per minute per gram; ±, plus or minus; –, not analyzed]

Depth	Mid-depth	Dry bulk density (g/cm^3)	Cumulative mass (g/cm^2)	Total lead-210 (dpm/g)	Radium-226 (dpm/g)	Excess lead-210 (dpm/g)	Cesium-137 (dpm/g)
0–0.5	0.25	0.015	0.007	150.7 ± 3.9	2.0 ± 0.5	148.7	10.3
0.5–1.0	0.75	0.015	0.015	–	–	–	–
1.0–1.5	1.25	0.062	0.046	126.4 ± 2.8	0.8 ± 0.7	125.6	12.5
1.5–2.0	1.75	0.053	0.072	–	–	–	–
2.0–2.5	2.25	0.004	0.075	119.9 ± 2.8	2.7 ± 0.4	117.2	13.5
2.5–3.0	2.75	0.048	0.098	–	–	–	–
3.0–3.5	3.25	0.045	0.121	70.7 ± 2.0	1.7 ± 0.4	69.1	21.4
3.5–4.0	3.75	0.053	0.147	–	–	–	–
4.0–4.5	4.25	0.084	0.189	37.7 ± 1.1	1.6 ± 0.2	36.1	23.4
4.5–5.0	4.75	0.078	0.228	–	–	–	–
5.0–5.5	5.25	0.108	0.283	35.9 ± 1.1	1.0 ± 0.2	34.8	23.1
5.5–6.0	5.75	0.101	0.333	–	–	–	–
6.0–6.5	6.25	0.092	0.379	62.6 ± 1.6	1.3 ± 0.2	61.3	28.3
6.5–7.0	6.75	0.047	0.402	–	–	–	–
7.0–7.5	7.25	0.063	0.434	75.7 ± 1.7	1.7 ± 0.3	74.0	31.3
7.5–8.0	7.75	0.051	0.459	–	–	–	–
8.0–8.5	8.25	0.054	0.486	55.6 ± 1.8	1.3 ± 0.3	54.3	55.6
8.5–9.0	8.75	0.074	0.523	–	–	–	–
9.0–9.5	9.25	0.077	0.562	33.2 ± 1.5	1.9 ± 0.3	31.2	45.5
9.5–10.0	9.75	0.081	0.602	–	–	–	–
10.0–11.0	10.5	0.292	0.748	16.7 ± 1.0	2.0 ± 0.2	14.7	5.6
11.0–12.0	11.5	0.319	1.068	–	–	–	–
12.0–13.0	12.5	0.246	1.313	8.6 ± 0.8	2.1 ± 0.1	6.6	2.6
13.0–14.0	13.5	0.263	1.576	–	–	–	–
14.0–15.0	14.5	0.238	1.815	8.4 ± 0.7	1.9 ± 0.2	6.5	1.6
15.0–16.0	15.5	0.279	2.093	–	–	–	–
16.0–17.0	16.5	0.248	2.342	7.4 ± 0.6	2.2 ± 0.1	5.2	1.1
17.0–18.0	17.5	0.313	2.654	–	–	–	–
18.0–19.0	18.5	0.299	2.954	5.0 ± 0.6	1.8 ± 0.1	3.2	0.4
19.0–20.0	19.5	0.288	3.242	–	–	–	–
20.0–21.0	20.5	0.325	3.566	4.2 ± 0.5	2.2 ± 0.2	2.0	0.2
21.0–22.0	21.5	0.313	3.879	–	–	–	–
22.0–23.0	22.5	0.310	4.189	–	–	–	–
23.0–24.0	23.5	0.282	4.472	–	–	–	–
24.0–25.0	24.5	0.296	4.767	–	–	–	–
25.0–26.0	25.5	0.303	5.071	2.6 ± 0.6	1.6 ± 0.1	0.9	0.1

Table 9. Bulk density and select radionuclide data from sediment core collected at Milk Lake, Olympic National Park, near Eldon, Washington, September 11, 2009.

[Depths are in centimeters. **Abbreviations**: g/cm^3, gram per cubic centimeter; g/cm^2, gram per square centimeter; dpm/g, disintegrations per minute per gram; ±, plus or minus; –, not analyzed]

Depth	Mid–depth	Dry bulk density (g/cm^3)	Cumulative mass (g/cm^2)	Total lead-210 (dpm/g)	Radium-226 (dpm/g)	Excess lead-210 (dpm/g)	Cesium-137 (dpm/g)
0–0.5	0.25	0.285	0.142	28.9 ± 0.8	2.9 ± 0.1	26.0	4.6
0.5–1.0	0.75	0.358	0.322	29.0 ± 0.9	1.4 ± 0.1	27.6	7.7
1.0–1.5	1.25	0.393	0.518	30.3 ± 0.8	1.5 ± 0.1	28.7	12.0
1.5–2.0	1.75	0.389	0.713	29.3 ± 1.0	2.1 ± 0.1	27.2	24.4
2.0–2.5	2.25	0.386	0.906	14.0 ± 0.4	1.3 ± 0.1	12.6	3.0
2.5–3.0	2.75	0.455	1.133	10.7 ± 0.7	1.9 ± 0.1	8.9	13.2
3.0–3.5	3.25	0.623	1.445	–	–	–	–
3.5–4.0	3.75	0.562	1.726	7.6 ± 0.5	1.8 ± 0.1	5.8	0.3
4.0–4.5	4.25	0.378	1.915	–	–	–	–
4.5–5.0	4.75	0.322	2.075	5.2 ± 0.6	2.4 ± 0.1	2.8	0.2
5.0–5.5	5.25	0.254	2.203	–	–	–	–
5.5–6.0	5.75	0.204	2.304	3.1 ± 0.6	2.2 ± 0.2	0.9	0.1
6.0–6.5	6.25	0.153	2.381	–	–	–	–
6.5–7.0	6.75	0.135	2.449	–	–	–	–
7.0–7.5	7.25	0.127	2.512	–	–	–	–
7.5–8.0	7.75	0.174	2.599	–	–	–	–
8.0–8.5	8.25	0.163	2.680	–	–	–	–
8.5–9.0	8.75	0.138	2.749	–	–	–	–
9.0–9.5	9.25	0.129	2.814	–	–	–	–
9.5–10.0	9.75	0.123	2.876	–	–	–	–
10.0–11.0	10.5	0.128	3.004	0.4 ± 0.8	1.3 ± 0.1	-0.8	0.1

Sediment Diatom Data

Sediment diatom assemblages were determined on 66 samples at 10 of the study lakes. The top and bottom slices of a sediment core from each lake were analyzed to screen for certain indicator diatom species. Four lakes (Snow Lake in MORA, Copper Lake in NOCA, and Hoh and Milk Lakes in OLYM) were identified for more detailed down-column analysis of diatom communities and sediment dating. These four sites were selected based on the presence of nitrophilic diatoms in upper core slices or because they were at locations with high nitrogen mass deposition data (table 2). All diatom counts are provided in tables 10 through 19, which are available as a Microsoft® Excel workbook. These tables include the full diatom taxon name, the short taxon name, the North American Diatom Ecological Database (NADED) identifier of each species identified, and the corresponding number of diatom valves counted in the sample for a given diatom species. A diatom valve is the unique siliceous covering on the top or bottom of a diatom cell, largely used to identify a given diatom species. The NADED identifier is a unique number used to track and adjust for any future changes in diatom taxonomy that might occur. Diatom counts were conducted until 500 valves were identified. One unexpected result of the diatom analysis during this study was the discovery of three previously unidentified diatom species (M. Enache, Academy of Natural Sciences Philadelphia, written commun., 2012). These species were *Psammothidium lacustre* sp. nov. Enache et Potapova, *Psammothidium alpinum* sp. nov. Potapova et Enache, and *Psammothidium nivale* sp. nov. Potapova et Enache, and were found in very low numbers in Hidden MORA, Snow, Copper, Hidden NOCA, Lower Thornton, and Milk Lakes. These newly identified species were not present at consistent depths.

Diatom Tables

All diatom counts are provided in tables 10 through 19**,** which are available as a Microsoft® Excel workbook. These tables include the full diatom taxon name, the short taxon name, the North American Diatom Ecological Database (NADED) identifier of each species identified, and the corresponding number of diatom valves counted in the sample for a given diatom species. These tables are available for download at http://pubs.usgs.gov/ds/721/.

Table 10. Diatom counts from sediment core collected at Eunice Lake (USGS lake station 12091956), Mount Rainier National Park, near Carbonado, Washington, November 4, 2009.

Table 11. Diatom counts from sediment core collected at Hidden Lake (USGS lake station 12096700), Mount Rainier National Park, near Greenwater, Washington, August 19, 2010.

Table 12. Diatom counts from sediment core collected at Snow Lake (USGS lake station 14224590), Mount Rainier National Park near Packwood, Washington, October 7, 2009.

Table 13. Diatom counts from sediment core collected at Copper Lake (USGS lake station 12215650), North Cascades National Park, near Glacier, Washington, September 22, 2009.

Table 14. Diatom counts from sediment core collected at Hidden Lake (USGS lake station 12181450), North Cascades National Park, near Marblemount, Washington, September 24, 2009.

Table 15. Diatom counts from sediment core collected at Lower Thornton Lake (USGS lake station 12178700), North Cascades National Park, near Marblemount, Washington, September 23, 2009.

Table 16. Diatom counts from sediment core collected at Stiletto Lake (USGS lake station 12450880), North Cascades National Park, near Stehekin, Washington, August 29, 2009.

Table 17. Diatom counts from sediment core collected at Heather Lake (USGS lake station 12947660) Olympic National Park, near Sequim, Washington, September 10, 2009.

Table 18. Diatom counts from sediment core collected at Hoh Lake (USGS lake station 12040680), Olympic National Park, near Forks, Washington, September 8, 2009.

Table 19. Diatom counts from sediment core collected at Milk Lake (USGS lake station 12053810), Olympic National Park, near Eldon, Washington, September 11, 2009.

Summary

This report summarizes atmospheric deposition, water quality, lake sediment, and diatom data collected for the U.S. Geological Survey-National Park Service Pacific Northwest Critical Loads Project. Deposition data were collected using a passive bulk deposition collector, and water quality and sediment cores were collected from a field-assembled inflatable pontoon boat during the summers of 2008–10. High-elevation lakes in the national parks had low ionic strength and low nutrients, as indicated by water-quality data. The sediment data from this report will be used to age date several lake cores and historical diatom community reconstructions will be used to determine if a critical load for nitrogen exists at these lakes. The critical load for nitrogen is defined as the level of nitrogen deposition at which shifts in diatom communities occur that could potentially alter the ecosystem functioning at these lakes.

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Appendix A . Quality-Assurance and Quality-Control Data

Quality-assurance and quality-control data were collected for lake surface-water quality, sediment quality, and data from passive deposition collectors for this project. All quality-control data collected for this project are summarized in this appendix.

Lake Surface Water Quality-Control Data

Several quality-control samples were collected throughout this project and included field and laboratory replicates, and field blanks. These data are presented in tables A1 and A2. Generally, most replicate and blank data are within the project data-quality objectives:

- **Field and laboratory replicates.**—The relative percent difference is less than 10 percent for duplicates with concentrations of greater than 0.5 mg/L and less than 20 percent for duplicates with concentrations of less than 0.5 mg/L. The absolute difference is less than 0.050 for samples with concentrations of less than 0.1 mg/L.

- **Field Blanks.**—Values are less than the method detection limit for target analytes.

However, for a few parameters, the absolute difference or relative percent difference criteria were not met. These included replicates for chlorophyll-*a* and ANC at Hidden Lake in MORA, chlorophyll-a at Milk Lake in OLYM, and potassium at Hidden Lake in NOCA. No nutrient parameters exceeded data quality objectives for the project. Because the focus of this project was on effects of nutrients, the few parameters that did not meet data quality objectives will not affect any conclusions made based on the water-quality data.

A single field blank was collected at Snow Lake and analyzed for all parameters measured in the surface water of study lake sites. In all cases, the determined value was less than the method reporting limit for that parameter (table A2).

Surface Sediment Quality-Control Data

Several field and laboratory replicates were analyzed for carbon and nitrogen and all were within the established data quality objectives. For carbon and nitrogen content, relative percent difference limits were 15 percent or less (table A3). For sediment $\delta^{15}N$, an absolute difference of 1.0 per mil or less was achieved for the single replicate collected at Milk Lake.

Additionally, high and low organic content standard reference samples (SRSs) were submitted to the laboratory for analysis of percent nitrogen and carbon. Standard reference samples were obtained from Elemental Microanalysis. Certified values for percent carbon were 1.26 (low SRS) and 6.1 (high SRS), and for percent nitrogen were 0.1 (low SRS) and 0.46 (high SRS). The relative percent differences for all SRSs were within 7 percent or less of the known values for carbon and nitrogen.

Passive Deposition Collector Quality-Control Data

Deposition data collected for this project used a novel technique involving deployment of passive bulk deposition collectors developed by the USFS Pacific Southwest Research Station (PSRS) laboratory (Fenn and others, 2002). The process involves deploying in the field an ion exchange resin (IER) column attached to a funnel. Ions are adsorbed from solution to the resin as rainwater passes through the IER column. After field deployment, these IER columns are extracted in the laboratory and the extractant solution is analyzed for nitrogen and sulfur species. PSRS laboratory personnel supplied all IER columns, performed column extractions, and analyzed extractant solutions for all project samples.

Because these types of analyses typically are not run by the USGS NWQL, extensive testing of the USFS laboratory performance was done. All quality-control samples supplied to the USFS laboratory are documented and the procedure used to correct for background signal and to correct for percent recovery of the column extraction process to arrive at the final masses (table 2) is described.

Each step of the process for determining total nitrogen and sulfur deposition at the field sites was tested during this study.

Table A1. Field and laboratory replicate data for lake surface-water quality, Mount Rainier, North Cascades, and Olympic National Parks, Washington, 2008–10.

[All samples collected from upper 1 meter of the water column over the deepest part of the lake. Relative percent difference was calculated as the absolute difference of the sample and replicate or expected value, divided by the average of these two values, which was then multiplied by 100. Ranges in percent relative difference and absolute difference represents the range based on assuming that the replicate value was zero, or the value at which the data was censored. Results in **bold** did not meet the project's data quality objectives. **Abbreviations**: °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25 degrees Celsius; $CaCO_3$, calcium carbonate; N, nitrogen; P, phosphorus; mg/L, milligram per liter; µg/L, microgram per liter; E, estimated; <, less than; –, not applicable]

Site identifier	Quality-control sample type	Parameter	Sample value	Replicate value	Relative percent difference	Absolute difference
Hidden MORA	Field replicate	Specific conductance (µS/cm)	15.8	17.0	7.2	1.18
		pH	6.7	7.2	7.6	0.53
		Ammonia (mg/L as N)	<0.01	<0.01	0.0	–
		Nitrate plus nitrite (mg/L as N)	0.001	<0.001	0–200	0.0–0.001
		Orthophosphate (mg/L as P)	0.001	0.001	0.0	0
		Total nitrogen (mg/L as N)	0.06	0.06	0.0	0
		Total phosphorous (mg/L as P)	0.004	0.004	0.0	0
		Chlorophyll *a* (µg/L)	**0.7E**	**0.8E**	**13.3**	**0.1**
		Pheophytin *a* (µg/L)	0.21E	0.20E	4.9	0.010
		Acid neutralizing capacity (ANC) (mg/L as $CaCO_3$)	**2.3**	**3.3**	**35.7**	**1.0**
		Chloride (mg/L)	0.243	0.230	5.5	0.013
		Sulfate (mg/L)	0.331	0.330	0.3	0.001
		Silica (mg/L)	5.94	5.87	1.2	0.071
		Fluoride (mg/L)	<0.08	<0.08	0.0	–
		Calcium (mg/L)	2.07	2.04	1.4	0.030
		Magnesium (mg/L)	0.195	0.198	1.5	0.003
		Sodium (mg/L)	0.717	0.710	1.0	0.007
		Potassium (mg/L)	0.241	0.210	13.7	0.031
		Iron (µg/L)	3.24E	<6.0	60–200	3.24–2.76
		Manganese (µg/L)	0.782	1.00	24.5	0.218
Copper	Field Replicate	Ammonia (mg/L as N)	<0.01	<0.01	0.0	–
		Nitrate plus nitrite (mg/L as N)	<0.001	<0.001	0.0	–
		Orthophosphate (mg/L as P)	<0.001	<0.001	0.0	–
		Total nitrogen (mg/L as N)	0.07	0.05	33.3	0.02
		Total phosphorous (mg/L as P)	0.004	0.004	0.0	0
Milk	Field Replicate	Chlorophyll *a* (µg/L)	**0.28**	**0.20**	**33.3**	**0.08**
		Pheophytin *a* (µg/L)	<0.1	<0.1	0.0	–
Hidden NOCA	Field Replicate	Chloride (mg/L)	0.146	0.140	4.2	0.006
		Sulfate (mg/L)	0.215	0.180	17.7	0.035
		Silica (mg/L)	0.956	0.970	1.5	0.014
		Fluoride (mg/L)	<0.08	<0.08	0.0	–
		Calcium (mg/L)	0.705	0.650	8.1	0.055
		Magnesium (mg/L)	0.031	0.030	3.3	0.001
		Sodium (mg/L)	0.148	0.160	7.8	0.012
		Potassium (mg/L)	**0.173**	**0.120**	**36.2**	**0.053**
		Iron (µg/L)	<4.0	<4.0	0.0	–
		Manganese (µg/L)	0.252	0.300	17.4	0.048
Hidden MORA	Laboratory replicate	Total nitrogen (mg/L as N)	0.07	0.06	15.4	0.01
		Total phosphorous (mg/L as P)	0.005	0.004	22.2	0.001
Lower Thornton	Laboratory replicate	Total nitrogen (mg/L as N)	0.1	0.1	0.0	0
Stiletto	Laboratory replicate	Total nitrogen (mg/L as N)	0.27	0.27	0.0	0
Milk	Laboratory replicate	Ammonia (mg/L as N)	<0.01	<0.01	0.0	–
		Nitrate plus nitrite (mg/L as N)	<0.001	<0.001	0.0	–
		Orthophosphate (mg/L as P)	<0.001	<0.001	0.0	–

Table A2. Field blank data for lake surface water quality, Snow Lake, Mount Rainier National Park, Washington, October 7, 2009.

[Blank water source was obtained from the U.S. Geological Survey National Field Supply Service. **Abbreviations**: N, nitrogen; P, phosphorus; mg/L, milligram per liter; µg/L, microgram per liter; <, less than; E, estimated]

Parameter	Sample value	Reporting Level
Ammonia (mg/L as N)	<0.01	0.01
Nitrate plus nitrite (mg/L as N)	<0.001	0.001
Orthophosphate (mg/L as P)	<0.001	0.001
Total nitrogen (mg/L as N)	<0.01	0.01
Total phosphorous (mg/L as P)	<0.002	0.002
Chloride (mg/L)	0.077E	0.12
Sulfate (mg/L)	<0.18	0.18
Silica (mg/L)	<0.058	0.058
Fluoride (mg/L)	<0.08	0.08
Calcium (mg/L)	<0.044	0.044
Magnesium (mg/L)	<0.016	0.016
Sodium (mg/L)	<0.1	0.10
Potassium (mg/L)	<0.064	0.064
Iron (µg/L)	<6.0	6.0
Manganese (µg/L)	<0.2	0.20

Table A3. Field and laboratory replicate, and standard reference sample data for lake surface sediment, Mount Rainier, North Cascades, and Olympic National Parks, Washington.

[Sediment samples were collected from the upper 2 cm of lake bottom near the deepest part of the lake. Relative percent difference was calculated as the absolute difference of the sample and replicate or expected value, divided by the average of these two values, which was then multiplied by 100. **Abbreviations**: SRS, standard reference sample; ^{15}N, nitrogen isotope 15; N, nitrogen; ‰, per mil; –, not applicable]

Sample identifier	Quality-control sample type	Parameter	Sample value	Replicate or expected value	Relative percent difference
Milk Lake	Field replicate	Total nitrogen (percent)	0.2	0.2	2.4
		Total carbon (percent)	2.0	2.0	0.5
		δ^{15}N (‰)	0.150	1.030	–
Stiletto	Laboratory replicate	Total nitrogen (percent)	2.0	1.9	0.6
		Total carbon (percent)	22.4	22.4	0.0
Milk Lake	Laboratory replicate	Total nitrogen (percent)	0.2	0.2	12.4
		Total carbon (percent)	2.0	2.2	11.5
High SRS	Laboratory replicate	Total nitrogen (percent)	0.4	0.5	4.2
		Total carbon (percent)	6.3	6.3	0.2
Low SRS	Standard reference sample	Total nitrogen (percent)	0.095	0.10	5.1
		Total carbon (percent)	1.18	1.26	6.6
High SRS	Standard reference sample	Total nitrogen (percent)	0.44	0.46	4.4
		Total carbon (percent)	6.3	6.1	3.4

Laboratory Analyses of Nitrogen and Sulfur

The PSRS laboratory used an ion chromatograph to analyze ammonia, nitrate, and sulfate in the extraction solutions following established methods for use with IER columns (Fenn and others, 2002, 2009; Simkin and others, 2004). To test the accuracy of the laboratory methods, a series of SRSs obtained from the USGS Branch of Quality Systems were sent to the USFS laboratory for analysis of various parameters. The results of these analyses are listed in table A4.

Generally, the PSRS laboratory accurately determined concentrations of ammonia, nitrate, and sulfate in the SRS samples. The relative percent difference for three samples of ammonia ranged from 1 to 12 percent, and for three samples of nitrate ranged from 0 to 3 percent (table A4). The determination of sulfate for three precipitation SRS samples also was acceptable, ranging from 7 to 10 percent. Only one blank water sample was analyzed and results were within acceptable ranges for this project (table A4).

Column Sorption and Laboratory Extraction Efficiency of Nitrogen and Sulfur

To test the effectiveness of the IER columns provided by the USFS PSRS laboratory, we conducted two laboratory tests on the columns. The first test was to examine how efficient the IER columns were at removing ions from solution as water passed through the columns. The second test was used to determine the efficiency of the laboratory extraction procedure. For these tests, two spike solutions, containing a high and low concentration of target analytes, were made in the USGS Washington Water Science Laboratory in Tacoma, Washington. Each spike was introduced into triplicate columns, for a total of three high and 3 low spike columns.

Table A4. Concentrations, most probable values, and relative percent differences of standard reference samples and blank water provided to the U.S. Forest Service Pacific Southwest Research Station (PSRS) laboratory..

[All samples analyzed at the U.S. Forest Service Pacific Southwest Research Station laboratory. Most probable values are the results from the autumn 2008 inter-laboratory comparison administered by the U.S. Geological Survey Branch of Quality Systems (BQS). **Sample identifier:** Identifiers in parenthesis represent the sample number assigned by BQS. Blank water was obtained from the U.S. Geological Survey Washington Water Science Center laboratory, reverse osmosis system. Relative percent difference was calculated as the absolute difference of the laboratory result and most probable value, divided by the average of these two values, which was then multiplied by 100 to get a percentage. **Abbreviations:** SRS, standard reference sample; N, nitrogen; mg/L, milligram per liter; –, parameter was not present in the SRS, therefore not analyzed; NA, not applicable]

Sample identifier	Laboratory result			Most probable value			Relative percent difference		
	Ammonia (mg/L as N)	Nitrate (mg/L as N)	Sulfate (mg/L)	Ammonia (mg/L as N)	Nitrate (mg/L as N)	Sulfate (mg/L)	Ammonia (mg/L as N)	Nitrate (mg/L as N)	Sulfate (mg/L as SO^4)
Low nutrient SRS (N-99)	0.101	0.233	–	0.090	0.230	–	12	1	–
	0.101	0.230	–	0.090	0.230	–	12	0	–
	0.101	0.230	–	0.090	0.230	–	12	0	–
High nutrient SRS (N-100)	0.786	0.725	–	0.761	0.704	–	3	3	–
	0.778	0.727	–	0.761	0.704	–	2	3	–
	0.770	0.725	–	0.761	0.704	–	1	3	–
Precipitation SRS (P-48)	–	–	0.310	–	–	0.290	–	–	7
	–	–	0.260	–	–	0.290	–	–	10
	–	–	0.270	–	–	0.290	–	–	7
Blank water	0.016	0.000	0.003	NA	NA	NA	NA	NA	NA

Column Sorption Efficiency

For the first test of the extraction process of the IER columns, the two spike solutions were passed through unused columns in the USGS Washington Water Science Laboratory. A 100-mL solution of low and high spike solutions containing ammonia, nitrate, and sulfate were passed through three columns, and the effluent solutions were collected in clean bottles for analysis. These water samples were sent to the PSRS laboratory for analysis of ammonia, nitrate, and sulfate. Results from these analysis show that almost 100 percent of each constituent was removed from solution by the IER columns (table A5). Blank water also was passed through three unused columns in triplicate to test for possible contamination from resin materials. Results from the blank water showed a slight amount of ammonia that could result from leaching of the columns, or from the source water itself. The blank water source for these tests was from the reverse osmosis system in the USGS Tacoma laboratory, and was not tested independently for contamination. Despite this small amount of ammonia contamination, the contamination levels are much lower than levels for columns that were deployed in the field and are not expected to influence interpretation of the field collected IER data. Nitrate and sulfate were detected in some blank samples, but at lower levels than those for ammonia.

Table A5. Laboratory results and removal by column for water samples passed through the ion exchange columns in the laboratory.

[All samples analyzed at the U.S. Forest Service Pacific Southwest Research Station laboratory. Spike solutions were prepared in the U.S. Geological Survey (USGS) Washington Water Science Center laboratory; concentrations verified by submitting subsamples to National Water Quality Laboratory for analysis. Low spike contained 39.5 mg/L as N ammonia and nitrate and 117 mg/L sulfate. High spike contained 202 mg/L as N of ammonia, 196 mg/L as N for nitrate, and 585 mg/L for sulfate. Data are from water samples collected after passing though ion exchange columns in the laboratory. Blank water was obtained from the USGS Washington Water Science Center laboratory reverse osmosis system. **Removal by column:** Calculated by subtracting the measured concentration from the known concentration, divided by the known concentration times 100. **Abbreviations**: N, nitrogen; S, sulfur; mg/L, milligram per liter; –, no data, solution contained no N or S]

Sample description	Laboratory result			Removal by column (percent)		
	Ammonia (mg/L as N)	Nitrate (mg/L as N)	Sulfate (mg/L)	Ammonia	Nitrate	Sulfate
Low N and S spike	0.016	0.000	0.003	99.96	100.0	100.0
	0.008	0.000	0.010	99.98	100.0	99.99
	0.016	0.000	0.003	99.96	100.0	100.0
High N and S spike	0.016	0.000	0.003	99.99	100.0	100.0
	0.008	0.005	0.007	100.0	100.0	100.0
	0.016	0.005	0.007	99.99	100.0	100.0
Blank water	0.016	0.005	0.007	–	–	–
	0.016	0.002	0.003	–	–	–
	0.016	0.000	0.000	–	–	–

Laboratory Extraction Efficiency

Test two of the IER columns utilized the same spiked columns from the first efficiency test to determine how well the PSRS laboratory was at extracting the spiked columns. Data from this test was used to calculate a percent recovery used in further analyses. Six spiked columns (three high and three low) were extracted in the PSRS laboratory and the resulting data was used to determine the extracted mass, expected mass, and percent recovery from spike columns for nitrogen and sulfur and shown in table A6. The extracted mass was calculated by multiplying the extractant concentration by the volume of extraction solution for each column (100-150 mL). The expected mass was calculated by multiplying the concentration of nitrogen and sulfur from spike solutions by the spike solution volume (100 mL) that was added to the columns prior to laboratory extraction. Percent recovery was calculated by dividing extracted mass by expected mass, and then multiplying by 100 to express as a percent. Percent recovery for the low spike columns was highest for ammonia, and similar for nitrate and sulfate (table A6). Percent recovery for the high spike samples was lower for ammonia, but similar or more efficient for nitrate and sulfate when compared to the low spike columns. Because the extracted mass of field-deployed columns was typically less than the mass of the low spike columns, the average percent recovery from the low spike columns was used to correct extracted masses presented in table 2. These average percent recoveries for the low spike columns for ammonia, nitrate, and sulfate are 96, 90, and 90 percent, respectively.

Table A6. Extracted mass, expected mass, and percent recovery calculations of nitrogen and sulfate from spiked resin columns.

[Extracted mass is calculated from concentration data received from U.S. Forest Service Pacific Southwest Research Station (PSRS) laboratory times the volume of extraction solution. Expected mass is calculated from the concentration and volume of spike solution added to laboratory columns. Spike solutions were prepared in the U.S. Geological Survey Washington Water Science Center laboratory; concentrations verified by submitting subsamples to National Water Quality Laboratory for analysis. All resin columns were extracted by the U.S. Forest Service PSRS laboratory. Values in **bold** were used to correct for extraction efficiency in order to calculate final masses (table 2). **Abbreviations**: N, nitrogen; mg, milligram]

Sample identifier	Extracted mass			Expected mass			Percent recovery		
	Ammonia (mg as N)	Nitrate (mg as N)	Sulfate (mg)	Ammonia (mg as N)	Nitrate (mg as N)	Sulfate (mg)	Nitrogen as ammonia	Nitrogen as nitrate	Sulfate
Low spike	3.79	3.64	9.05	3.94	3.95	11.74	96	92	77
	3.69	3.52	10.92	3.94	3.95	11.74	94	89	93
	3.86	3.52	11.62	3.94	3.95	11.74	98	89	99
High spike	16.67	16.96	52.57	20.20	19.69	58.52	83	86	90
	12.54	17.40	57.32	20.20	19.69	58.52	62	88	98
	15.70	19.67	63.66	20.20	19.69	58.52	78	100	109
Low spike average	3.78	3.56	10.53	3.94	3.95	11.74	**96**	**90**	**90**
High spike average	14.97	18.01	57.85	20.20	19.69	58.52	74	91	99

Field and Laboratory Blank Columns

Fenn and others (2002) showed that IER columns have the potential to release contamination from the ion exchange resins, likely as a result of repeated exposure in the field to hot, sunny days and cool nights. A control column (with both ends capped and sealed) was deployed in the field during the same period as the exposed columns, as described by Fenn and others (2002). These control columns, or field blank columns, were sent to the PSRS laboratory for extraction of nitrogen and sulfur in order to make background corrections to field-exposed columns. Laboratory and field blanks generally showed similar levels of contamination for ammonia, nitrate, and sulfate (table A7). Extracted mass from blanks was low for ammonia and nitrate, but ranged from 0.28 to 0.69 mg for sulfate. Average mass of ammonia, nitrate, and sulfate from field blanks were 0.02, 0.03 and 0.41 mg, respectively. These average masses were used to background-correct field-exposed columns for calculation of final extracted masses presented in table 2.

Table A7. Extracted masses of ammonia, nitrate, and sulfate from laboratory and field blank columns, Mount Rainier, North Cascades, and Olympic National Parks, Washington.

[**Extracted mass:** Calculated from concentration data received from U.S. Forest Service Pacific Southwest Research Station (PSRS) laboratory times the volume of extraction solution. All resin columns were extracted by the U.S. Forest service PSRS laboratory. Values in **bold** were used for background corrections to field-exposed columns to calculate final masses (table 2). **Abbreviations**: N, nitrogen; mg, milligram; –, no sample, control columns not deployed at Milk and PJ Lakes]

Sample description	Extracted mass		
	Ammonia (mg as N)	Nitrate (mg as N)	Sulfate (mg)
Laboratory blank	0.00	0.05	0.36
	0.01	0.09	0.40
	0.00	0.03	0.32
	0.01	0.07	0.36
	0.00	0.04	0.33
	0.00	0.05	0.43
Mount Rainier National Park			
Eunice field blank	0.02	0.02	0.29
Hidden MORA Lake field blank	0.00	0.06	0.28
Shriner Lake field blank	0.00	0.03	0.40
Snow Lake field blank	0.00	0.03	0.35
North Cascades National Park			
Copper Lake field blank	0.02	0.02	0.46
Hidden NOCA Lake field blank	0.01	0.07	0.36
Lower Thornton Lake field blank	0.00	0.02	0.69
Stiletto Lake field blank	0.05	0.05	0.30
Olympic National Park			
Heather Lake field blank	0.05	0.03	0.45
Hoh Lake field blank	0.00	0.02	0.54
Milk Lake field blank	–	–	–
PJ Lake field blank	–	–	–
Field blank average	**0.02**	**0.03**	**0.41**

Summary

Quality-assurance procedures and quality-control data were collected for lake water and sediment quality using passive deposition collectors. Overall, most concentrations in samples collected from water and sediment met or exceeded the data quality objectives. For estimating deposition, average masses for nitrogen and sulfur from field blank columns were used to correct field-exposed columns for background conditions at the lake study sites. Additionally, percent recovery data from the low spike columns were used to correct for extraction efficiency of all field-deployed columns.

Overall, the data presented indicate that the environmental data collected for this study were of good quality; future interpretation of the data should not be compromised as a result of data quality.

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