SEDIMENT QUALITY ASSESSMENT OF BLAIRS VALLEY LAKE, WASHINGTON COUNTY, MARYLAND AND FRANKLIN COUNTY, PENNSYLVANIA

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respective limit values. These benchmarks for freshwater sediments are based upon chronic, long-term impacts of contamination to benthic organisms (Buchman, 2008). The Lowest Effect Level (LEL) is a level of sediment contamination that can be tolerated by the majority of benthic organisms. The Severe Effect Level (SEL) is that at which pronounced disturbance of the sediment-dwelling community can be expected. This is the concentration that would be detrimental to the majority of the benthic community.   22

Acronyms and Abbreviations used in this report

Abbreviation	Description
Act Labs	Activation Laboratories, Inc.
Al, Ca, Fe, K, Na and Mg	major rock forming elements aluminum, calcium, iron, potassium, sodium and magnesium
As, Cd, Cr, Cu, Mn, Ni, Pb and Zn	trace elements arsenic, cadmium, chromium, copper, manganese, nickel, lead and zinc
BV	Blairs Valley Lake
°C	Celsius
C, N, S and P	carbon, nitrogen, sulfur and phosphorus
CNS	Carbon, Nitrogen and Sulfur (gas chromatography analysis)
cm	Centimeters
CRC	Canadian Research Council
DNR	Maryland Department of Natural Resources
EF	Enrichment Factor
EPA	Environmental Protection Agency
GIS	Geographical Information System
INAA	Instrumental Neutron Activation Analysis
HCl	Hydrochloric Acid
LEL	Lowest Effect Level
MGS	Maryland Geological Survey
mg/kg	Milligram per kilogram
ml	Milliliter
mmol/kg	Milli-mole per kilogram
NAD83	North America [Horizontal] Datum of 1983
NIST	National Institute of Standards and Technology
NOAA	National Oceanic and Atmospheric Administration
ppm	part per million
RAS	Resource Assessment Service (DNR)
RTK GPS	Real-Time Kinematic Global Positioning System
SAV	Submerged Aquatic Vegetation
SEL	Severe Effect Level
SRM	Standard Reference Material
TD-ICP	"near total" Digestion Inductively Coupled Plasma Spectrophotometer
TEA	Tidewater Ecosystem Assessment (DNR)
ug/g	microgram per gram (parts per million)
US SCS	U.S. Soil Conservation Service (in 1994, name changed to NRCS)
UTM	Universal Transverse Mercator (coordinate system)
WMA	Wildlife Management Area

Executive Summary

Maryland Geological Survey, in conjunction with other Maryland Department of Natural Resources programs, including Tidewater Ecosystem Assessment (TEA) and Fishing and Boating Services, was asked to perform sediment quality assessments of state-owned lakes in Maryland. The goal is to provide surficial sediment data for each lake to determine general sediment physical and chemical characteristics as a pre-assessment for potential future lake sediment dredging. Physical and chemical analysis of the sediments from each lake will pose as a baseline for the lake's sediment data. The sediment data will be incorporated into other study aspects, including fish populations, water quality, and submerged aquatic vegetation (SAV) abundance and distribution in order to generate overall lake health and status.

This report focuses on Blairs Valley Lake in Washington County, Maryland and Franklin County, Pennsylvania. The lake is 22 acres and is located within Indian Springs Wildlife Management Area (WMA). Ten surficial sediment samples were collected throughout the lake and coordinates were stored using Real-Time Kinematic Global Positioning System (RTK GPS). Sediment at each site was described and sampled for analysis. The analysis includes physical properties, including bulk density and grain size, and elemental analysis.

The elemental concentrations of 10 surficial sediments followed similar spatial patterns when plotted by station. The concentrations of major elements Al, Fe, K and Mg as well as the concentrations of trace metals Cu, Ni, Pb and Zn were quite similar to each other in profile-plot, and generally similar to clay content. The concentrations of trace metals As and Cr differed slightly from these others, perhaps because they are less affected by sorption in these environmental conditions. Compared to the relative eco-toxicological screening levels LEL, most trace metals were above the conservative LEL, but only Fe was above the higher SEL in three samples. No eco-toxicological harm is anticipated. Compared to the relative crustal abundance via the use of Fe-normalized enrichment factors, the elements As, Cs, Hf, Pb and Sb are enriched. However, these elements are often encountered in high concentrations in clay-rich sedimentary rocks and the sediments derived from them. No anomalies or outliers were identified.

The 10 collected samples throughout Blairs Valley Lake were similar to sediments of other lakes nearby, including New Germany Lake and Deep Creek Lake. No abnormalities were found in regard to the physical or chemical properties of the bottom sediments of Blairs Valley Lake.

Introduction

Study Area

Blairs Valley Lake (Figure 1) is a man-made lake fed from Little Conococheague Creek coming from the north within Washington County, Maryland and Franklin County, Pennsylvania. The 1ake is 22 acres and is located within Indian Springs Wildlife Management Area (WMA) and owned by the State of Maryland.

The Blairs Valley Lake watershed is located within the Ridge and Valley Physiographic Region of Maryland. The bedrock of this region contains strongly folded and faulted sedimentary rock and, in this section (west of Powell Mountain), is composed of shale and sandstone bedrock. Regional formations are mapped as the Martinsburg and Juniata formations.

Study Objectives

The objectives of this study are to:
1. Document sediment through the collection of grab samples from Blairs Valley Lake.
2. Analyze the samples for physical properties, including bulk density and grain size.
3. Process each sample for carbon, nitrogen and sulfur (CNS) and elemental analysis.

Methods

Field Methods

On September 10, 2019, MGS staff collected 10 sediment grab samples in Blairs Valley Lake (Figure 2). A 16-foot aluminum jon boat with an electric motor was used to collect the samples around the lake. Prior to sampling, using ESRI ArcMap 10.3, 10 proposed sample locations were placed throughout the lake to ensure full lake coverage. During sample collection, a TopCon Real-Time Kinematic Global Positioning System (RTK GPS) antenna and Carlson SurvPC software were used to locate each of the proposed locations and record the actual grab sample coordinates for each sample. Location coordinates were documented in UTM NAD83 Zone 18 State Plane, meters (Appendix A). Pictures were captured for each sample, displaying sediment properties and any present submerged aquatic vegetation (SAV). Pictures can also be found in Appendix A.

Sediment samples were collected with a hand-operated LaMotte stainless-steel dredge that sampled a bottom surface area of 19 centimeters (cm) x 14 cm and a mean sediment depth of 10 cm. Upon collection, samples were photographed, described and placed in Whirl-Pak^{TM} bags and kept cool until delivery to the MGS laboratory. Samples were refrigerated at 4° Celsius (C) until analysis. Descriptions of each sample along with sample coordinates can be found in Appendix A.

Figure 1. Blairs Valley Lake, view from sampling vessel.
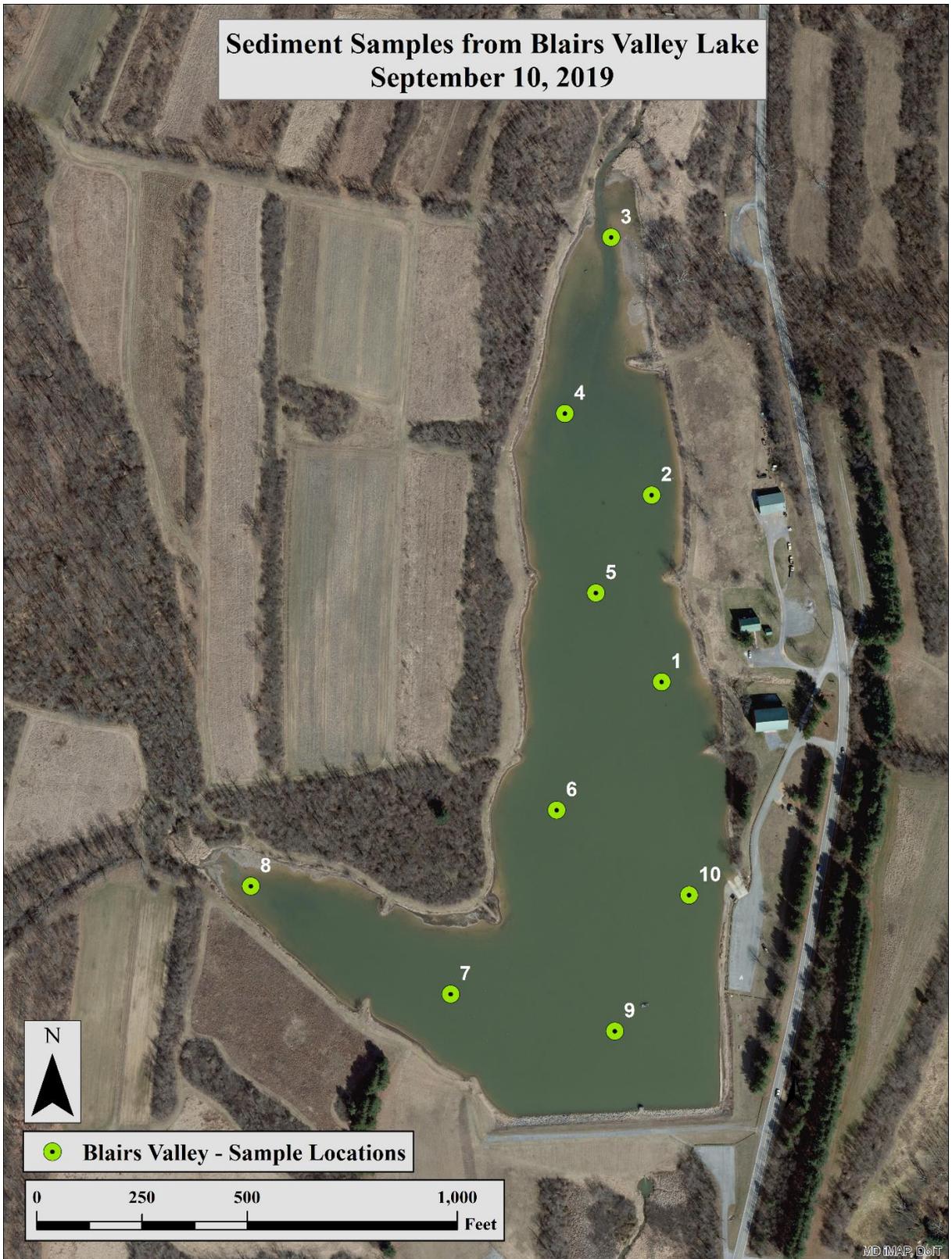
Figure 2. Map of grab sample locations.

Laboratory Methods

Grain Size

        Sediment grab samples were analyzed for water content, bulk density and grain size (sand, silt and clay contents, as well as gravel, when present). Two homogeneous splits of each sample were processed, one for bulk property analysis and the other for grain size characterization. Analyses were performed within one week of sample collection and all samples were stored at 4° C prior to analysis.

        Water content was determined by weighing 20-30 grams (g) of sediment. The sediment was then dried at 65° C and then re-weighed. Water content was calculated as the percentage of water weight to the weight of the wet sediment using Equation 1.

$$\%Water = \frac{W_w}{W_t} * 100$$

Equation 1

        where: W_w is the weight of water; and
                              W_t is the weight of wet sediment.

        Wet Bulk Density (ρ_B) is calculated from water content utilizing Equation 2 by assuming an average grain density (ρ_s) of 2.72 g/cm^3 and saturation of voids with water of density $\rho_w = 1.0$ g/cm³. This method was adopted from the work of Bennett and Lambert (1971):

$$\rho_B = \frac{W_t}{W_d \ / \ 2.72 + W_w}$$

Equation 2

        where: W_d is the weight of dry sediment.

        Gravel, sand, silt and clay contents were determined using the textural analysis detailed in Kerhin and others (1988). Grain size, in this report (Table 1), is given in phi units, a scale devised by Krumbein (1936) where phi is defined as the negative log (to the base 2) of the particle diameter (millimeters (mm)). For example, 4 phi corresponds to a particle with a diameter of $1/2^4$ mm (=1/16 mm, or 0.0625 mm or 62.5 microns).

        Grain size analysis consisted of cleaning the sediment samples in solutions of 10 percent hydrochloric acid (HCl) and 6 or 15 percent hydrogen peroxide (determined by water content) with subsequent rinsing with deionized water. This process removed soluble salts, carbonates and organic matter that could interfere with the dis-aggregation of the individual grains. The samples were then treated with a 0.26 percent solution of the dispersant sodium hexametaphosphate (($NaPO_3$)$_6$) to ensure that individual grains did not re-aggregate (flocculate) during pipette analysis.

        The separation of sand and silt-clay (mud) portions of the sample was accomplished by

wet-sieving through a 4-phi mesh sieve (0.0625 mm, U.S. Standard Sieve #230). The gravel-sand fraction (*i.e.* that portion of the sample not passing through the sieve) was dried and weighed, and saved for further analysis. The finer silt and clay-sized particles (*i.e.,* passing through the sieve) were suspended in a 1000 milliliter (ml) cylinder in a solution of 0.26 percent sodium hexametaphosphate. The suspension was agitated and, at specified times thereafter; 20 ml pipette withdrawals are made (Carver, 1971; Folk, 1974). The rationale behind this process is that larger particles settle faster than smaller ones (Stoke's law). By calculating the settling velocities for different sized particles, times for withdrawal can be determined at which all particles of a specified size will have settled past the point of withdrawal. Sampling times were calculated to permit the determination of the amount of particles corresponding to 4 phi (silt class) and 8 phi (clay class) particles in the suspension. Withdrawn samples were dried at 65°C and weighed. From these data, the percentages by dry weight of sand, silt and clay were calculated for each sample and classified according to Shepard (1954) and Pejrup (1988) nomenclatures (Figures 3 and 4). Sample weight loss due to cleaning was determined; the weight loss approximates the amount of non-clastic components in the sediment.
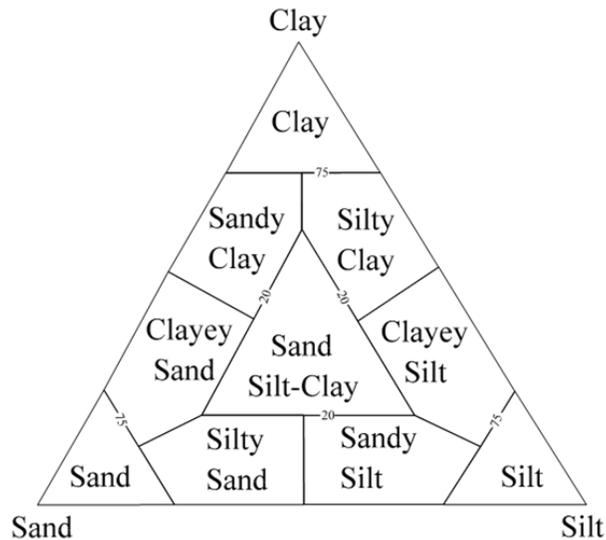
Figure 3. Shepard (1954) classification of sediment types. Sediment type classification is based on relative percentages of each size component (sand, silt, and clay).
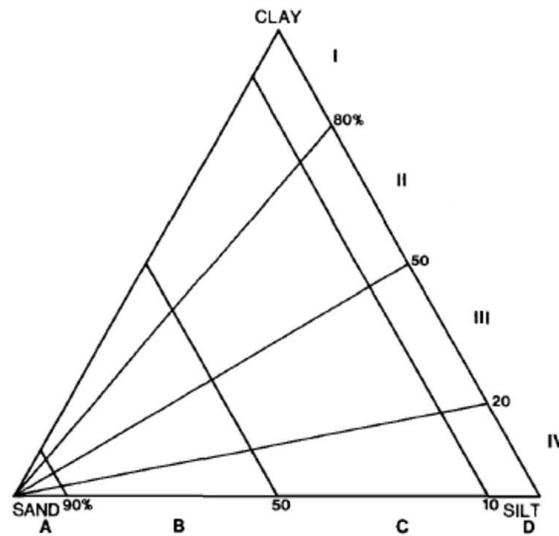
Figure 4. Pejrup's Diagram (1988) classification of sediment type.

Table 1. Sediment grain size definitions used in this study are based on the Wentworth (1922) scale. The term Mud is used to describe all particles smaller than sand (less than 0.0625 millimeters). The term Gravel is used to describe all rock fragment particles that are 2 millimeters or larger.

Descriptor	Grain Size (millimeters)	Class Sizes (phi)
Mud	< 0.0625	> 4
Clay	< 0.004	> 8
Silt	0.004 to 0.0625	> 4 to 8
Sand	0.0625 to 2	4 to -1
Very Fine Sand	0.0625 to 0.125	4 to 3
Fine Sand	0.125 to 0.25	3 to 2
Medium Sand	0.25 to 0.5	2 to 1
Coarse Sand	0.5 to 1	1 to 0
Very Coarse Sand	1 to 2	0 to -1
Gravel	2 to 4,096	-1 to -12
Granule	2 to 4	-1 to -2
Pebble	4 to 64	-1 to -6
Cobble	64 to 256	-6 to -8
Boulder	256 to 4,096	-8 to -12

Elemental Analysis

Samples were ground in-house to the point until 95 percent of the sample could pass through a 200-mesh sieve (0.074 mm screen opening). Each sample was divided, with a three to five gram portion of the sample submitted to Activation Laboratories Inc. (Act Labs), while the remainder of the sample was retained for in-house analysis of total carbon, nitrogen and sulfur (CNS).

The quality assurance and quality control of Act Labs has proved to meet MGS standards and requirements. Fifty elements (Ag, Al, As, Au, Ba, Be, Bi, Br, Ca, Cd, Ce, Co, Cr, Cs, Cu, Eu, Fe, Hf, Hg, Ir, K, La, Li, Lu, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Rb, S, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Th, Ti, U, V, W, Y, Yb and Zn) were analyzed. Samples were prepared and ground in-house and sent to Act Labs for analyses using both Instrumental Neutron Activation Analysis (INAA) and a four acid "near total" digestion technique followed by analysis on a "near total" Digestion Inductively Coupled Plasma Spectrophotometer (TD-ICP). In addition to the standards and blanks used by Act Labs, National Institute for Standards and Technology (NIST) Standard Reference Materials (SRMs) were inserted every eighth sample to run as blind, known sample analyses. Replicates of the Blairs Valley samples were also placed throughout the batch and were blindly run every fifth sample.

Carbon, Nitrogen and Sulfur Analysis

Sediments were analyzed by MGS for total carbon, nitrogen and sulfur (CNS) contents using a Carlo Erba NA1500 analyzer. This analyzer uses complete combustion of the sample followed by separation and analysis of the resulting gases by gas chromatographic techniques employing a thermal conductivity detector. The NA1500 Analyzer was configured for CNS analysis using the manufacturer's recommended settings. As a primary standard, sulfanilamide was used and was run after every eight unknown sediment samples. As a secondary standard, one of several NIST SRMs was run after every eight sediment samples. Blanks (tin capsules containing only vanadium pentoxide) were run at the beginning and end of the analyses each day. Replicates of every fifth sample were also run.

Results and Discussion

Physical Analysis

Based on the textural analyses of the 10 surficial sediment samples (representing the top 10 cm of the sediment column), the majority of the samples collected are fine-grained sediments, with an average textural content of 15% sand, 42% silt and 39% clay. Six samples fall within the clayey-silt and silt-clay classifications (Table 2). Four samples are classified as sand-silt-clay. Clay represents a major component of the collected sediments, which is not unexpected given the abundance of shale in the underlying formations within the watershed. Sand and gravel are relatively minor components in most samples. One sample (#10) was composed of almost 16% gravel, but was still classified as a sand-silt-clay. This sample was taken near the lake's boat ramp. Complete sample field descriptions, including coordinates and pictures for each sample can be found in Appendix A.

The physical and chemical behavior of sediment is influenced by its texture. Particle diameter reflects the energy environment in which the sediment was deposited. Generally, coarse grained sediments (i.e., sand and gravel) are found in higher energy environments, such as areas subjected to wave activity or high water currents, which tend to winnow out any fine grained sediment. Fine-grained sediments, which are transported further from the source and take a long time to settle, are usually found in areas that are not subjected to high waves or winds, or below the depth of wave motion, such as deeper areas in the central area of the lake, or coves that are

sheltered from high waves and winds. In Blairs Valley Lake, the sediment distribution follows this pattern (Figure 5). The finest-grained sediments (i.e., sediment with highest clay content) are in the deepest part of the lake, whereas sandier sediments are found in shallower depths and in the up-stream areas (excluding sample 10, which is believed to have additional gravel that was added during construction or maintenance of the boat ramp). Textural QA/QC analyses can be found in Appendix B.

Size also reflects the mineral composition of the sediments, which, in turn, is a product of the parent rock. Blairs Valley Lake can be found just west of Powell Mountain, in the more rugged area of the Ridge and Valley physiographic province in Maryland. This area in western Maryland, where the Blairs Valley Lake watershed lies, consists of sedimentary rock, namely shales and sandstones. Clay minerals are abundant in the lake since shale is a common parent rock. Shales are lithified mud deposits, composed of silt sized particles (mainly quartz) and clay minerals. Clay minerals are the remaining particles after the complete weathering of the shale. Clay particles are small, plate-like particles, with a diameter of less than two microns and have a relatively large surface area. Depending on the crystalline lattice, clays have an enormous capacity to incorporate both organic and metal cations onto the lattice surface, and water and organic compounds within lattice layers. These bound substances, in turn, contribute to the cohesiveness of the clays. Organic rich clays, in turn, support active benthic bacteria and plankton communities.

Blairs Valley Lake -- Sediment Samples

Sediment Data
Shepard Classificaton

○ Clayey-Silt
● Sand-Silt-Clay
● Silty-Clay
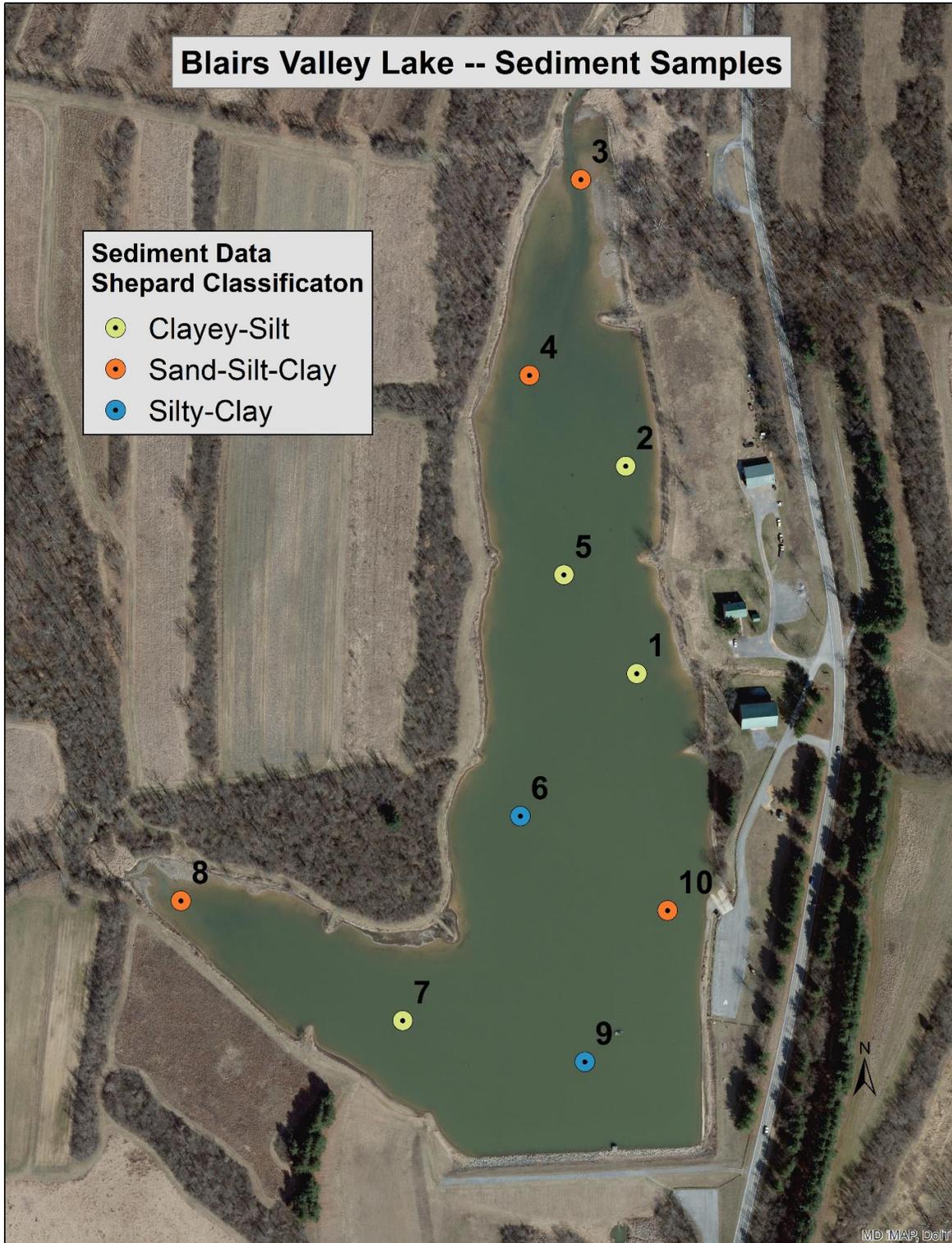
Figure 5. Distribution of sediment types based on Shepard (1954) classification. Deepest samples are #5, 6 and 7 which were collected at a depth of about 11 feet and sample 9, which was collected from a depth of about 18 feet (approximate depths were measured with rope while sample was retrieved).

Table 2. Summary table of physical properties result for Blairs Valley Lake.
[1]Approximate depths were measured with rope while the sample was retrieved.

Sample ID	% H2O	Bulk Density	% Gravel	% Sand	% Silt	% Clay	Shepard Class	Pejrup Class	Clay: Mud	Approx. Depth[1] (feet)
1	44.88	1.54	4.25	12.81	47.25	35.69	Clayey-Silt	C,III	0.43	8
2	47.29	1.50	3.11	14.23	41.92	40.74	Clayey-Silt	C,III	0.49	8
3	60.18	1.34	0.00	29.93	37.73	32.34	Sand-Silt-Clay	C,III	0.46	1.5
4	50.38	1.46	0.00	30.32	39.83	29.85	Sand-Silt-Clay	C,III	0.43	5
5	57.70	1.37	0.00	4.31	51.76	43.93	Clayey-Silt	D,III	0.46	11
6	57.91	1.36	0.30	0.30	42.36	57.05	Silty-Clay	D,II	0.57	11
7	49.15	1.47	0.00	1.17	51.67	47.16	Clayey-Silt	D,III	0.48	11
8	46.34	1.51	5.05	19.42	43.75	31.78	Sand-Silt-Clay	C,III	0.42	2
9	47.27	1.50	0.91	16.63	40.40	42.07	Silty-Clay	C,II	0.51	18
10	37.97	1.65	15.77	24.01	30.50	29.73	Sand-Silt-Clay	C,III	0.49	10

Carbon, Nitrogen, Sulfur and Phosphorus Analysis

Results of nitrogen (N), carbon (C), phosphorus (P) and sulfur (S) of each sample is shown in Table 3.

Table 3. Nitrogen, Carbon, Phosphorus and Sulfur results for Blairs Valley Lake.

Sample ID	% Nitrogen	% Carbon	% Phosphorus	% Sulfur
1	0.269	2.710	0.057	0.081
2	0.218	2.443	0.038	0.065
3	0.397	6.199	0.049	0.080
4	0.276	3.475	0.041	0.074
5	0.299	3.184	0.046	0.083
6	0.314	2.963	0.054	0.087
7	0.298	3.246	0.040	0.076
8	0.213	2.254	0.037	0.055
9	0.190	2.076	0.046	0.112
10	0.221	2.057	0.076	0.069

Total C contents measured in Blairs Valley Lake sediments range from 2.1% to 6.2% (dry weight), with a mean of 3.1%. These values are similar to that of the average C reported for New Germany Lake and Deep Creek Lake (Table 4).

Table 4. Comparison of total N, C, P and S in surficial sediment in Maryland freshwater reservoirs/lakes. Values given are percent dry sediment weight.

Reservoir/Lake	Physiographic Province	% N		% C		% P		%S	
		Avg.	Range	Avg.	Range	Avg.	Range	Avg.	Range
Loch Raven (Ortt et al, 1999)	Piedmont	0.32	0.24-0.40	3.17	2.53-3.94	0.16	0.12-0.19	0.057	0.0-0.15
Little Seneca Lake (Ortt et al., 2011)	Piedmont	0.29	0.18-0.34	3.25	2.07-5.06	0.08	0.06-0.14	0.11	0.04-0.15
Triadelphia Reservoir (Wells et al, 2007)	Piedmont	0.26	0.11-0.48	2.77	1.48-4.12	0.10	0.04-0.17	0.074	0.027-0.28
Deep Creek Lake (Wells and Ortt, 2011)	Piedmont	0.33	0.12-0.62	4.11	1.55-9.60	0.06	0.01-0.13	0.26	0- 0.98
Rocky Gorge Reservoir (Wells et al, 2007)	Appalachian Plateau	0.22	0.05-0.41	2.67	0.83-4.17	0.09	0.03-0.16	0.08	0.02-0.17
New Germany Lake (Ortt and Wells, 2009)	Appalachian Plateau	0.51	0.09-0.81	6.20	2.02-7.54	0.06	0.01-0.10	0.08	0.01-0.21
Blairs Valley Lake (this study)	**Ridge and Valley**	**0.27**	**0.19-0.40**	**3.06**	**2.06-6.20**	**0.05**	**0.04-0.08**	**0.08**	**0.05-0.11**
Herrington Lake (Sylvia et al, 2020)	Appalachian Plateau	0.38	0.12-0.56	5.12	1.33-6.61	0.07	0.05-0.11	0.10	0.03-0.15
St. Mary's Lake (Sylvia et al, 2020)	Coastal Plain	0.15	0.04-0.32	2.55	0.62-5.13	0.02	0.00-0.05	0.07	0.02-0.16

Total N measured in Blairs Valley Lake sediments average 0.3%, with values ranging from 0.2% to 0.4%. These values are within the same range as New Germany Lake and Deep Creek Lake, analogous lakes found in the Appalachian Plateau province, because of the similar bedrock types. Sources of N include atmospheric input, septic flow and fertilizers. As organic matter is "cycled through the natural system", relative proportions of P and, to a lesser degree, N increase as C decreases. Table 5 lists the Redfield ratios for N, C and P for different sources and in sediment from several Maryland fresh and marine environments. The ratios of C to N, C to P, and N to P in the Blairs Valley Lake sediments are calculated using total C.

Table 5. Comparison of mass ratios of C, N and P observed in different sample sources.

	C:N	C:P	N:P
Global forest litter (McGroddy *et al.*, 2004)	57.3	1166.1	20.4
Global forest foliage (McGroddy *et al.,* 2004)	37.1	470.0	12.7
Dried marsh plant (Wells *et al*., 2002)	32.3	711.2	21.7
Marsh sediments (Wells *et al*., 2002)	18.1	243.6	13
St. Mary's Lake (Sylvia *et al.*,2020)	16.7	158.4	9.5
Herrington Lake (Sylvia *et al.*,2020)	13.7	68.9	5.0
New Germany (Ortt *et al.*, 2009)	13.5	109.2	8.1
Dried algae (Wetzel, 1983)	13.3	40.0	3.0
Deep Creek Lake sediments (Wells and Ortt, 2011)	12.9	87.5	6.5
Rocky Gorge Reservoir (Wells *et a.l*, 2007)	12.5	31.2	2.5
Blairs Valley Lake (this study)	**11.1**	**66.2**	**5.8**
Triadelphia Reservoir (Wells *et a.l*, 2007)	11.1	29.2	2.6
Loch Raven (Ortt *et al.*, 1999)	10.1	19.9	2.0
Coastal Bays bottom sediments (Wells *et al.,* 1994)	7.0	65.1	9.3
Plankton (Redfield *et al.*, 1963)	5.7	41	7.2

Although total P does not directly undergo reduction-oxidation processes in sediments, its cycling within the lake is controlled, in part, by the redox state of certain metals, particularly S and Fe, and by the concentration of organic material (C). Sources of P include weathering of natural soils and rocks, runoff from agricultural land and seepage from septic systems. Phosphate (PO_4^{-3}) from fertilizers binds to soils, which erode during storm events adding suspended phosphate to streams that drain into the lake. Septic seepage may contribute phosphate in the form of orthophosphate and organic phosphorus. Unlike N and C, P has no gaseous form. Therefore, P does not cycle out of the system like N by way of denitrification or C by respiration. Thus P tends to accumulate in the sediments. Once in the sediments, P is slowly released into the interstitial water as organic material is oxidized. Free phosphate is rapidly bound to ferric oxyhydroxides and oxidized manganese which are found in the upper, oxidized layer of the sediments (i.e., oxidized flocculant layer on sediment surface). Deeper in the sediment column where anoxic conditions prevail and metals oxides have been reduced, P is released into the interstitial water and, if sulfide is low or absent, reacts with reduced forms of metals, particularly Fe, forming hydrous phosphates. However, if present, free sulfide will bind more readily to the reduced Fe and the phosphate remains free to diffuse upward to the oxidized layer where it is "captured" by excess ferric oxyhydroxides (FeOOH) and manganese oxides found in the upper sediment layer. If the overlying water column becomes anoxic, the "captured" P may be released in the overlying water column where it can contribute to increased algae/plankton production. The portion of total P active in this cycle includes the loosely sorbed phosphate, fresh, leachable, organic P and iron-bound phosphate. These available forms of P make up 40% to 50% of the total P in the upper one centimeter of sediments and are largely depleted below three centimeters in the sediment column (Jorgensen, 1996). Any P below this depth usually consists of the more stable forms, bound to clay minerals, or associated with apatite or calcium carbonate minerals, and become permanently buried in the sediments.

Total P measured in the sediments average 0.048%, with values ranging from 0.038% to 0.076%. These values are similar to those found in New Germany Lake and Deep Creek Lake (Table 5). Total P is often associated with the sediment clay content. P also shows a high correlation with Fe, due to binding to ferric oxyhydroxides (Table 6). When comparing the relative amounts of C, N and P in the Blairs Valley Lake sediments to those of dried algae, P appears to be the limiting nutrient; thus, mean C:P and N:P ratios are greater than those of dried algae.

Trace amounts of sulfur were measured in the sediments, averaging 0.08%. S concentrations in Blairs Valley Lake are similar to that found in New Germany Lake, Rocky Gorge and Triadelphia reservoir sediments, but lower than the sediments found in Deep Creek Lake.

Reduced S and dissolved sulfate (SO_4^{-2}) concentration is an important variable controlling P release from sediments (Caraco *et al.* 1989; Wetzel, 1983). The increased P release from sediments at higher sulfate concentrations may help explain why primary production in freshwater systems (with relatively low S concentrations) tends to be P limited, whereas in many saline systems (with high sulfate concentrations) production is often P sufficient. Sulfur also plays an important role in arsenic cycling (Fisher et al., 2008). In this case, S was not found to be strongly associated with any other element at statistical significance, although it was weakly associated with clay and Mn (Table 6).

Quality assurance and quality control (QA/QC) of C, N, S and P analysis can be found in Appendix B.

Table 6. Correlation matrix for textural, nutrient and target metal data based on all of the sediment samples collected at Blairs Valley Lake. The correlations were determined using a Pearson product-moment technique (Johnson and Wichern, 1982). Values listed in the table are Pearson correlation coefficients (r). Shaded values are statistically significant at the 95% confidence level ($p < 0.05$).

	H$_2$O	Sand	Silt	Clay	%N	%C	%S	%P	As	Cr	Cu	%Fe	Mn	Ni	Pb	Zn	%Al	%Ca	%K	%Mg
H$_2$O		-0.21	0.34	0.45	0.79	0.71	0.27	-0.37	-0.48	0.04	-0.10	-0.52	0.36	-0.07	-0.07	0.04	-0.35	0.82	-0.37	-0.37
Sand	-0.21		-0.71	-0.88	-0.02	0.32	-0.21	0.13	-0.17	-0.34	-0.15	-0.12	-0.07	-0.44	-0.67	-0.64	-0.55	-0.02	-0.40	-0.43
Silt	0.34	-0.71		0.49	0.17	-0.02	0.06	-0.55	-0.14	0.15	-0.37	-0.39	-0.20	0.01	0.24	0.15	-0.06	0.05	-0.23	-0.11
Clay	0.45	-0.88	0.49		0.16	-0.13	0.43	-0.16	-0.02	0.19	0.16	-0.04	0.22	0.37	0.60	0.59	0.45	0.26	0.32	0.30
%N	0.79	-0.02	0.17	0.16		0.91	0.05	0.00	-0.51	-0.09	0.23	-0.22	0.54	0.20	0.06	0.11	-0.26	0.90	-0.28	-0.22
%C	0.71	0.32	-0.02	-0.13	0.91		0.03	-0.13	-0.61	-0.34	-0.01	-0.42	0.38	-0.12	-0.31	-0.27	-0.58	0.86	-0.55	-0.52
%S	0.27	-0.21	0.06	0.43	0.05	0.03		0.11	0.01	0.06	0.00	-0.24	0.42	-0.02	0.23	0.19	0.03	0.20	-0.03	-0.10
%P	-0.37	0.13	-0.55	-0.16	0.00	-0.13	0.11		0.47	0.39	0.83	0.86	0.62	0.51	0.47	0.48	0.60	0.00	0.64	0.61
As	-0.48	-0.17	-0.14	-0.02	-0.51	-0.61	0.01	0.47		0.64	0.33	0.61	-0.22	0.39	0.16	0.38	0.60	-0.61	0.71	0.53
Cr	0.04	-0.34	0.15	0.19	-0.09	-0.34	0.06	0.39	0.64		0.28	0.50	0.23	0.24	0.45	0.44	0.47	-0.15	0.47	0.39
Cu	-0.10	-0.15	-0.37	0.16	0.23	-0.01	0.00	0.83	0.33	0.28		0.79	0.61	0.84	0.52	0.78	0.75	0.18	0.78	0.81
%Fe	-0.52	-0.12	-0.39	-0.04	-0.22	-0.42	-0.24	0.86	0.61	0.50	0.79		0.33	0.58	0.56	0.55	0.79	-0.24	0.82	0.80
Mn	0.36	-0.07	-0.20	0.22	0.54	0.38	0.42	0.62	-0.22	0.23	0.61	0.33		0.27	0.53	0.40	0.22	0.68	0.15	0.23
Ni	-0.07	-0.44	0.01	0.37	0.20	-0.12	-0.02	0.51	0.39	0.24	0.84	0.58	0.27		0.54	0.91	0.82	0.00	0.80	0.85
Pb	-0.07	-0.67	0.24	0.60	0.06	-0.31	0.23	0.47	0.16	0.45	0.52	0.56	0.53	0.54		0.72	0.73	0.07	0.55	0.61
Zn	0.04	-0.64	0.15	0.59	0.11	-0.27	0.19	0.48	0.38	0.44	0.78	0.55	0.40	0.91	0.72		0.88	0.01	0.81	0.88
%Al	-0.35	-0.55	-0.06	0.45	-0.26	-0.58	0.03	0.60	0.60	0.47	0.75	0.79	0.22	0.82	0.73	0.88		-0.30	0.96	0.96
%Ca	0.82	-0.02	0.05	0.26	0.90	0.86	0.20	0.00	-0.61	-0.15	0.18	-0.24	0.68	0.00	0.07	0.01	-0.30		-0.33	-0.29
%K	-0.37	-0.40	-0.23	0.32	-0.28	-0.55	-0.03	0.64	0.71	0.47	0.78	0.82	0.15	0.80	0.55	0.81	0.96	-0.33		0.94
%Mg	-0.37	-0.43	-0.11	0.30	-0.22	-0.52	-0.10	0.61	0.53	0.39	0.81	0.80	0.23	0.85	0.61	0.88	0.96	-0.29	0.94	

Elemental Analysis

Laboratory results are located in Appendix C.

Table 7 lists summary statistics for those metals having reported threshold limits listed in NOAA Screening Quick Reference Tables (SQuiRTs) (Buchman, 2008). Most elements listed in Table 7 are above background levels for freshwater sediments. Because the Blairs Valley Lake samples were analyzed using a near total decomposition method (four-acid digestion), caution is warranted when comparing the resulting concentration values for some metals to screening limits given in the NOAA tables. The values listed in the NOAA tables are based on EPA sample digestion methods which direct partial decomposition of sediment samples and thus reflect that portion of any element that may become biologically available/mobile under environmental conditions. For example, the NOAA tables list background levels in soil/sediments for Al as 0.26% which reflects the average Al which is biologically available. However, Act Labs' results for Al range from 1.06% to 7.33%, reflecting total recovery of the element by the stronger digestion method used. Al is a major component of most minerals found in native rock and soils. Likewise, average concentrations of Fe and Mn exceed the NOAA background levels for the same reasons given for Al.

Table 7. Summary statistics of select metal concentration measured in Blairs Valley Lake sediments. All values are ppm unless otherwise indicated. For comparison, benchmark levels for freshwater sediments are included along with the number of lake samples exceeding the respective limit values. These benchmarks for freshwater sediments are based upon chronic, long-term impacts of contamination to benthic organisms (Buchman, 2008). The Lowest Effect Level (LEL) is a level of sediment contamination that can be tolerated by the majority of benthic organisms. The Severe Effect Level (SEL) is that at which pronounced disturbance of the sediment-dwelling community can be expected. This is the concentration that would be detrimental to the majority of the benthic community.

	As	Cd	Cr	Cu	Fe (%)	Mn	Ni	Pb	Zn
Average	12.6	< 0.3	65	35	3.85	470	41	34	119
Std. Dev.	3.0	0.2	10	5	0.45	120	5	4	15
min	7.9	< 0.3	52	28	3.41	305	33	28	98
max	18.5	0.4	88	45	4.94	640	48	41	148
Background[1]	1.1	0.3	13	25	1.8	400	9.9	17	35
LEL	6	0.6	26	16	2	460	16	31	120
SEL	33	10	110	110	4	1100	75	250	820
#>Background	10	1	10	10	10	6	10	10	10
#>LEL	10	0	10	10	10	5	10	7	5
#>SEL	0	0	0	0	3	0	0	0	0

[1] Background as provided in NOAA SQuiRTs for freshwater sediments (Buchman, 2008).

The two screening levels considered here were the freshwater LEL and SEL. The freshwater LEL is a level of sediment concentration that can be tolerated by the majority of benthic organisms. The SEL is the concentration that would be detrimental to the majority of the benthic community. Derivations of these screening limits are explained in the reference, but briefly: a survey of at least 20 aquatic species presence is performed, and the LEL corresponds to 5th percentile concentrations and the SEL corresponds to the 95% percentile of concentrations where the benthic community is observed to exist.

While most metals of concern (As, Cd, Cr, Cu, Ni, Pb and Zn) are above more conservative LEL in most sediments (Table 7), only Fe exceeds the higher SEL value. The average Fe content of the Blairs Valley sediments was 3.85 ppm, and three of the sediment samples were above the SEL of 4 ppm. These three sediment samples were stations 1, 6 and 10, located in the central portion of the lake, at approximate depths between 8 and 11 ft. However, as stated above, these elemental concentrations were generated with a more vigorous total digestion than were the screening concentrations in the SQuiRTs tables, and represent a concentration greater than that which would be environmentally available through mineral decomposition in natural settings. Therefore, no ecotoxicological harm is anticipated.

As was shown in Table 6, Cu showed a strong association with Fe at statistical significance, while several other metals (including As, Cr, Ni, Pb and Zn) showed less strong correlations with Fe (correlation coefficients ranging from 0.50 to 0.61). Many of the metals show significant correlations amongst themselves and with certain "major" ions found in greater abundance in rock forming minerals and the sediments derived from them. For instance, Fe is significantly correlated with Cu at the 95% confidence interval, as well as with major rock formers Al, K and Mg. To examine some of the associations more closely, plots of the elemental

concentrations by station number can be found in Figure 6. Concentrations are shown in milli-moles per kilogram (mmol/kg, where a milli-mole is Avogadro's number multiplied by 1000). On these plots, the profiles of major rock forming elements Al, Fe, K and Mg are shown in the upper panel. These 4 elements follow a similar profile; local low concentrations located at station 3 and locally high concentrations located at stations 6 and 10. The similarity in profile implies either a common source (from shale weathering) or a common process which redistributes and deposits these elements together. The clay content is shown as a dashed line. Although the correlation between clay and these elements did not achieve statistical significance in a correlation matrix, general similarity may be observed in the profile. Shown next is the same plot by-station number for several trace metals (Cu, Ni, Pb and Zn). These four trace metals are found at lower abundance than the major rock forming elements, but the by-station number profile for these elements is generally similar to that of the first panel. Local high concentrations were again observed at stations 6 and 10. Local lows for these trace metals were found at stations 2 (for Cu, Ni and Zn) and at both stations 3 and 8 for Pb, which differs slightly from the major ion distribution. Lastly is shown a profile plot for metals As and Cr. These last two trace metals were more similar to each other and both distinct from the elemental profiles of previously discussed elements. For As and Cr, the local low concentrations were observed at station 3, but the local high was at station 5 (not 6).

From these observations, some preliminary interpretations are offered. It is hypothesized that the major rock forming elements (here Al, Fe, K and Mg) share a common source, and that source is likely to be weathered shales, greywackes, and sandstones encountered in the local country rock. Several of the trace metals (here Cu, Ni, Pb, and Zn) also followed a similar pattern. Local country rock has been mapped and described as the Martinsburg and Juniata formations, which are layered shales, greywackes, siltstones, carbonaceous claystones and/or muddy sandstones (Drake and Epstein, 1967). The clay component of these sedimentary units is itself composed of sericite (i.e. weathered muscovite mica and illite clay) and chlorite. The mineralogical composition of muscovite is $KAl_2(AlSi_3O_{10})(F,OH)_2$, the composition of illite is $(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$, and the composition of chlorite is $(Mg,Fe)_3(Si,Al)_4O_{10}(OH)_2 \cdot (Mg,Fe)_3(OH)_6$. Chlorite is shown as Mg-rich chlorite, but Fe, Ni and Mn-rich end members exist. These shale minerals would therefore be reasonably inferred sources of Al, Fe, K and Mg. The trace metals Cu, Pb and Zn may be absorbed to these mica and clay minerals, and Ni may be either absorbed or incorporated in chlorite. The trace metals Cu, Ni, Pb and Zn are likely to exist as positively charged ions at circum-neutral pH and transitional redox (declining dissolved oxygen) environment, and to adhere to negatively charged clay particle surfaces. Lastly, the by-station profiles for As and Cr were similar to each other and both differed slightly from the other trace metals in that the local high was at station 5, which was not the clay content maximum. As is often found associated with S, however, there was low S content in these sediments, and As is often found associated with Fe in soils. In circum-neutral and transitional to reducing sedimentary environments, dissolved As^{3+} may exist as an oxyanion with a negative charge ($H_2AsO_3^-$) or as an uncharged species. These forms of As would be less affected by sorption since they would be less attracted to a negatively charged clay surface than would be a positively charged ion. Cr is found associated with clays and clay-rich sedimentary rocks and as well as found associated with Fe and organic matter in soils. Dissolved Cr also commonly exists as an oxyanion, with a solubility minimum for both the Cr^{6+} and Cr^{3+} species encountered between pH 5.5 and 8, meaning that they are less mobile at common surface water

and pore-water pH (Kabata-Pendias 2011). It may be that the distribution of As and Cr is slightly different from the other trace metals since they are less affected by sorption onto clay materials as long as they are present in their negatively charged form.
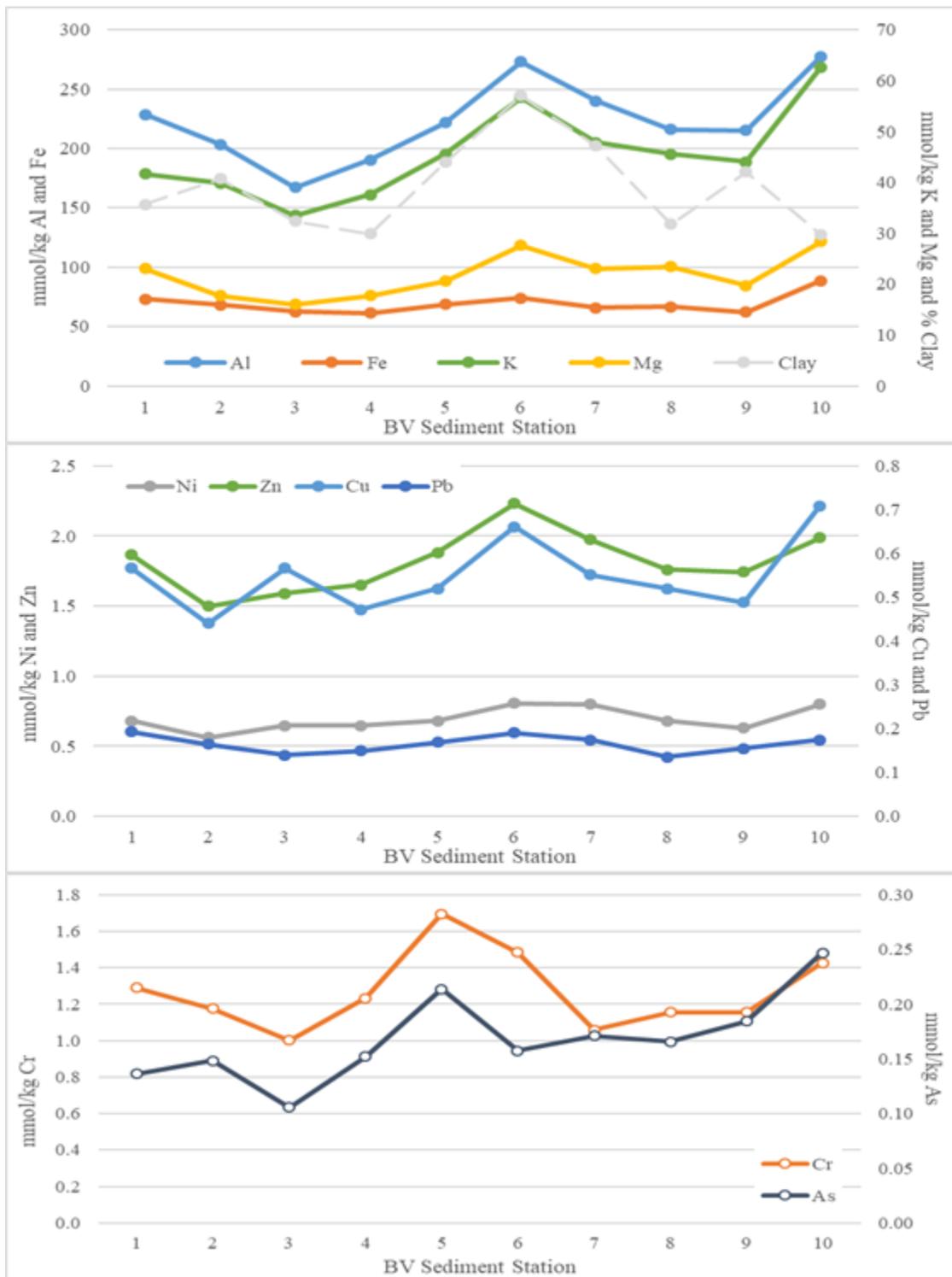
Figure 6. Concentration profile plots of several elements by Blairs Valley station number, shown with clay content. Concentrations are in percent for clay, and in milli-moles per kilogram for the elements shown.

Enrichment Factors

Because of the wide range of sediment types analyzed, comparisons of absolute metal concentrations between the surficial sediments are difficult. Therefore, metal concentrations are discussed in terms of enrichment factors (EF). The enrichment factor approach normalizes a concentration of an element, or a ratio of concentrations of elements in the sediment, to those found in a standardized material like average crustal rock. The use of enrichment factors also allows for comparisons of sediments from different environments and the comparisons of sediments whose trace metal contents were obtained by different analytical techniques (Cantillo, 1982; Hill *et al*., 1990; Sinex and Helz, 1981). However, the use of enrichment factors to assess metal data does not entirely eliminate the influence of textural variation.

Enrichment factor is defined as:

$$EF_{(x)} = X/Fe_{(sample)} / X/Fe_{(reference)}$$

Equation 3

where: $EF_{(x)}$ is the enrichment factor for the metal X;

$X/Fe_{(reference)}$ is the ratio of the concentrations of metal X to Fe in a reference material, such as an average continental crust rock.

Fe is chosen as the element for normalizing because anthropogenic sources for Fe are small compared to natural sources (Helz, 1976). Taylor's (1964) average continental crust is used as the reference material. Average crustal abundance data may not be representative of Blairs Valley Lake sediments because there is a higher proportion of clay and silt in the sediments compared to the average crustal rock. However, abundance data is useful as a relative indicator when comparing the data with other studies.

The average EF values for most metals are within the range of values obtained for other freshwater lakes and reservoirs in Maryland (Table 8). The sediments in Blairs Valley Lake are significantly enriched (*i.e*., EF>3) in As, Cs, Hf, Pb and Sb with respect to average continental crust rock. With Blairs Valley Lake falling within the Ridge and Valley Physiographic Region of Maryland, its bedrock is mainly strongly folded and faulted sedimentary rock and, in this section (west of Powell Mountain), is comprised of shale and sandstone bedrock. Regional formations around Blairs Valley Lake are mapped as the Martinsburg and Juniata formations. Blairs Valley Lake can be compared to Deep Creek Lake and New Germany Lake because all the lakes have similar depositional environments, although the western lakes lie within the Appalachian Plateau. The EF values are most similar to those reported for New Germany Lake and Deep Creek Lake, and since they are in similar sedimentary bedrocks, it can be expected that these lakes will be similar in geochemistry. The enrichment factor values for As, Cs, Hf, Pb and Sb are all near or above and EF of 3 for the three lakes. The highest EF value for As was located at station BV-5, which was also the highest un-normalized (ppm or mmol/kg) concentration of As. But local maxima for the other high EF elements Cs, Hf, Pb and Sb were each distributed differently throughout the lake, without a general pattern This may be due to several factors. There may be more than one source of the elements. The elements may have different geochemical behaviors during and after deposition in the lake. These elements are enriched relative to crustal abundance, but when compared relative to the ranges for argillaceous (clay-rich) sedimentary rocks (Kabata-Pendias 2011), the concentrations of As, Cs, Pb and Sb were

within range, and the concentration of Hf was approximately 1.5 times higher than the high concentration of the range, although high concentrations were reported in sandstones rich in zircon.

In summary, the sediments at Blairs Valley Lake contained concentrations of As, Cs, Hf, Pb and Sb with enrichment values above 3, when compared to crustal abundance. However, these EFs were quite similar to New Germany and Deep Creek Lakes, and the un-normalized concentrations were typical of the ranges encountered in shales and other clay-rich rocks.

Table 8. Comparisons of average enrichment factors in several Maryland freshwater reservoirs/lakes. Enrichment factors are relative to the average earth's crust (Taylor, 1964.)

Element	Loch Raven (Ortt *et al.*, 1999)	Triadelphia Reservoir (Wells *et al.* 2007)	Rocky Gorge Reservoir (Wells *et al.*, 2007)	New Germany Lake (Ortt *et al.*, 2009)	Deep Creek Lake (Wells *et al.*, 2011)	Blairs Valley Lake (this study)	Herrington Lake (Sylvia, *et al.*, 2020)	St. Mary's Lake (Sylvia, *et al.*, 2020)
Cd	0.28	4.93	1.89	0.9	8.90	2.47	11.81	
Cr	1.62		0.93	1.05	1.19	0.96	0.99	1.61
Cu	0.85	0.83	0.95	0.52	0.62	0.93	0.47	0.61
Mn	1.25	1.09	1.45	0.50	0.94	0.72	0.89	0.70
Ni	0.86	0.80	0.77	0.69	0.91	0.80	0.83	0.65
Pb	4.35	4.26	3.83	3.46	5.24	4.00	3.49	5.94
Zn	2.87	1.98	1.92	2.62	4.09	2.49	4.49	2.51
Al		0.78	1.27	1.24	0.94	1.07	1.02	1.52
As			3.61	9.13	15.61	10.20	8.79	8.98
Ce			2.41	1.88	2.21	2.19	1.77	0.00
Co		1.74	1.17	0.87	2.13	1.44	1.88	1.31
Cs			1.87	2.99	3.54	3.14	2.44	3.96
Eu			2.56	1.83	2.16	1.62	1.21	3.23
Hf			3.79	6.77	7.48	4.26	3.65	
Sb			3.19	10.64	24.41	5.65	5.26	11.27
Th		0.08	1.64	1.91	1.78	1.68	1.37	3.22
Ti			1.21	0.94	1.05	0.69	0.81	1.44
U			1.64	3.10	2.32	2.46	1.76	4.01
V		1.09	1.00	0.78	0.85	0.71	0.71	0.67
Y		2.25	2.29	1.48	1.39	1.42	1.14	3.36

Summary and Conclusions

In September 2019, 10 surficial sediment samples were collected from Blairs Valley Lake and based on the textural analyses of the samples, the majority of the samples collected are fine-grained sediments, with an average textural content of 15% sand, 42% silt and 39% clay. This area in western Maryland, where the Blairs Valley Lake watershed lies, is comprised of sedimentary rock, namely shales and sandstones. Clay minerals are abundant in the lake since shale is a common parent rock. Shales are lithified mud deposits, composed of silt sized particles (mainly quartz) and clay minerals which are the end product of weathering of other minerals.

Total C contents measured in Blairs Valley Lake sediments ranged from 2.1% to 6.2% (dry weight), with a mean of 3.1%. Total N measured in the Lake's sediment averaged 0.3%, with values ranging from 0.2% to 0.4%. Total P measured in the sediments average 0.048%, with values ranging from 0.038% to 0.076%. Concentrations of C, N and P were similar to that found in comparable lakes, including Deep Creek Lake and New Germany Lake.

The elemental concentrations of 10 surficial sediments followed similar spatial patterns when plotted by station. The concentrations of major elements Al, Fe, K and Mg as well as the concentrations of trace metals Cu, Ni, Pb and Zn were quite similar to each other in profile-plot, and generally similar to clay content. The concentrations of trace metals As and Cr differed slightly from these others, perhaps because they are less affected by sorption in these environmental conditions. Compared to the relative eco-toxicological screening levels LEL, most trace metals were above the conservative LEL, but only Fe was above the higher SEL in three samples. No eco-toxicological harm is anticipated. Compared to the relative crustal abundance via the use of Fe-normalized enrichment factors, the elements As, Cs, Hf, Pb and Sb are enriched. However, these elements are often encountered in high concentrations in clay-rich sedimentary rocks and the sediments derived from them. No anomalies or outliers were identified.

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Appendix A

Field Descriptions, Sample Coordinates and Pictures

Sample Number	Approx. Water Depth (ft.)*	Northing (NAD83 State Plane, meters)	Easting (NAD83 State Plane, meters)	Description
				Table A-1. Sample descriptions and coordinates, with approximate water depth. *Approx. depths were collected as a reference.
1	8	319232	226021	5Y 4/1 (olive gray) no SAV, no roots, 3 cm soft soupy watery slightly gritty clayey mud over firmer drier lumpy silty mud
2	8	319225	226156	Similar to sample #1 but with less grit, and more clay at depth. 5Y 4/1 (olive gray) no SAV, no roots, 3 cm soft soupy watery very slight grit clayey mud over firmer drier lumpy silty clayey mud
3	1.5	319196	226344	5Y 4/1 (olive gray) soft soupy watery silty mud with lots of detritus organic material (leaves, sticks, debris) throughout, gassy, presence of SAV (potentially hydrilla)
4	5	319162	226216	5Y 4/1 (olive gray) soft soupy watery silty mud, slightly firm at depth, some detritus throughout but less than in sample #3, somewhat gassy, no SAV
5	11	319185	226085	5Y 4/1 (olive gray) soft soup watery smooth not gritty and gassy, no roots, no SAV
6	11	319156	225927	5Y 4/1 (olive gray) soft soupy watery clayey mud over very firm drier dense lumpy silty mud with concretions
7	11	319079	225794	5Y 4/1 (olive gray) slightly firm to very firm at depth, smooth not gritty silt mud, few organics, very slightly gassy, lumpy
8	2	318934	225872	5Y 4/1 (olive gray) mottled with 5Y 4/1 (olive gray) at depth, upper 4-5 cm soft soupy watery silty mud over slightly firm lumpy gritty gassy fine-sandy mud, some SAV, no obvious roots, some debris/detritus, lots of SAV in core (potentially hydrilla)
9	18	319198	225767	5Y 4/1 (olive gray) very slightly gritty lumpy gassy soft soupy clayey mud mottled with N1.5 (black to grayish black) concretions, some organics throughout, very slightly firming with depth
10	10	319252	225866	5Y 4/1 (olive gray) surface ~4 cm soft soupy watery gritty sandy mud, over firmer slightly dry lumpy very slightly gritty silty mud, no gas, no SAV, very little detritus

Sample 1

Sample 2

Sample 3

Sample 4

Sample 5

Sample 6

Sample 7

Sample 8

Sample 9

Sample 10

Appendix B

QA/QC

Textural Analyses

Although the techniques used to determine grain size are based on traditional analytical methods developed for the sedimentology laboratory, some analytical error is inherent to the techniques. For example, results can be affected by level of technician skill and/or changes in laboratory conditions (such as sudden temperature changes). Furthermore, there is no standard reference material available that includes the broad range of particle sizes and shapes contained in natural sediment. To maximize consistency of textural analysis, several "checks" are used to monitor results. The calculated sand, silt, clay and gravel (when present) percentages are checked against 1) sample field descriptions; 2) calculated water contents; and 3) calculated weight loss of sample during processing. These comparisons are made to determine if the size components match the visual description of the sample and/or fall within an expected classification with respect to water content and weight loss. Any discrepancy is "flagged" and the results are reviewed further to determine if re-analysis is warranted.

Carbon, Nitrogen and Sulfur Analyses

Table B-1. Results of nitrogen, carbon and sulfur analyses of the standard reference materials (SRMs) compared to the certified or known values. MGS values were obtained by averaging the results of all SRM analyses run during this study.

Element	NIST SRM 8704 Buffalo River Sediment			NIST SRM 1646a Estuarine Sediment			NIST SRM 2702 Inorganics in Marine Sediment			Canadian SRM MESS 4 Marine Sediment		
	NIST Values[1]	MGS Results	% Recovery	NIST Values[2]	MGS Results	% Recovery	NIST Values[3]	MGS Results	% Recovery	NIST Values[4]	MGS Results	% Recovery
Total Nitrogen (% dry weight)	0.188	0.179 +/-0.006	95.3	0.058	0.056 +/-0.004	96.4	0.25	0.253 +/-0.007	101.1	0.142	0.138+/-0.010	96.8
Carbon (% dry weight)	3.351 +/-0.017	3.235 +/-0.054	96.6	0.583	0.563 +/-0.017	95.9	3.36	3.167 +/-0.091	94.2	1.79	1.780+/-0.103	99.5
Sulfur (% dry weight)	0.261	0.206 +/-0.060	78.91	0.352 +/- 0.004	0.347 +/-0.017	98.6	1.5	1.577 +/-0.069	105.12	0.158 +/-0.020	0.169+/-0.019	106.2

[1] For NIST 8704, the value for carbon is certified by NIST. The value of nitrogen and sulfur was obtained from repeated analyses in-house and by other laboratories (Haake Buchler Labs and U.S. Dept. of Agriculture).

[2] For NIST SRMs 1646a, the value for sulfur are certified values reported by NIST; nitrogen and carbon values were obtained from repeated analyses in-house and by Act Labs.

[3] For NIST SRM 2702, the value for sulfur are information values reported by NIST; nitrogen and carbon values were obtained from repeated analyses in-house and by Act Labs.

[4] For Canadian SRM, the value for sulfur is certified from the National Research Council Canada (NRCC). The value for carbon is an information value reported by the NRCC. The value for nitrogen was obtained from repeated analyses in-house.

Appendix C

Elemental Analysis Results and QA/QC

Table C-1. Elements (analytes) reported in this study include 50 elements analyzed by Act Labs. Methods abbreviations: High Temp. Combustion-GC: High Temperature combustion, following by Gas Chromatography; TD-ICP: Total Digestion followed by Inductively Coupled Plasma Spectrometry; INAA: Instrumental Neutron Activation Analysis.

Element	Symbol	Reporting Unit	Detection Limit	Analysis Method	Element	Symbol	Reporting Unit	Detection Limit	Analysis Method
Aluminum	Al	%	0.01	TD-ICP	Mercury	Hg	ppm	1	INAA
Antimony	Sb	ppm	0.1	INAA	Molybdenum	Mo	ppm	1	TD-ICP
Arsenic	As	ppm	0.5	INAA	Neodymium	Nd	ppm	5	INAA
Barium	Ba	ppm	50	INAA	Nickel	Ni	ppm	1	INAA / TD-ICP
Beryllium	Be	ppm	1	TD-ICP	Phosphorus	P	%	0.001	TD-ICP
Bismuth	Bi	ppm	2	TD-ICP	Potassium	K	%	0.01	TD-ICP
Bromine	Br	ppm	0.5	INAA	Rubidium	Rb	ppm	15	INAA
Cadmium	Cd	ppm	0.3	TD-ICP	Samarium	Sm	ppm	0.1	INAA
Calcium	Ca	%	0.01	TD-ICP	Scandium	Sc	ppm	0.1	INAA
Cerium	Ce	ppm	3	INAA	Selenium	Se	ppm	3	INAA
Cesium	Cs	ppm	1	INAA	Silver	Ag	ppm	0.3	INAA / TD-ICP
Chromium	Cr	ppm	2	INAA	Sodium	Na	%	0.01	INAA
Cobalt	Co	ppm	1	INAA	Strontium	Sr	ppm	1	TD-ICP
Copper	Cu	ppm	1	TD-ICP	Sulfur	S	%	0.01	TD-ICP
Europium	Eu	ppm	0.2	INAA	Tantalum	Ta	ppm	0.5	INAA
Gold	Au	ppb	2	INAA	Terbium	Tb	ppm	0.5	INAA
Hafnium	Hf	ppm	1	INAA	Thorium	Th	ppm	0.2	INAA
Iridium	Ir	ppb	5	INAA	Tin	Sn	%	0.01	INAA
Iron	Fe	%	0.01	INAA	Titanium	Ti	%	0.01	TD-ICP
Lanthanum	La	ppm	0.5	INAA	Tungsten	W	ppm	1	INAA
Lead	Pb	ppm	3	TD-ICP	Uranium	U	ppm	0.5	INAA
Lithium	Li	ppm	1	TD-ICP	Vanadium	V	ppm	2	TD-ICP
Lutetium	Lu	ppm	0.05	INAA	Ytterbium	Yb	ppm	0.2	INAA
Magnesium	Mg	%	0.01	TD-ICP	Yttrium	Y	ppm	1	TD-ICP
Manganese	Mn	ppm	1	TD-ICP	Zinc	Zn	ppm	1	INAA / TD-ICP

Table C-2. Blairs Valley Lake sediment elemental data. All values are ppm (ug/g) unless indicated otherwise.

Station	Ag	Al%	As	Au	Ba	Be	Bi	Br	Ca%	Cd	Ce	Co	Cr	Cs	Cu	Eu	Fe%
1	0.4	6.15	10.2	<2	590	2	<2	7.8	0.19	<0.3	99	26	67	5	36	1.1	4.07
2	<0.3	5.47	11.1	<2	500	2	<2	4.3	0.18	<0.3	85	23	61	6	28	1.3	3.8
3	<0.3	4.5	7.9	14	500	2	<2	10.5	0.28	0.3	76	21	52	4	36	1.2	3.48
4	<0.3	5.12	11.4	<2	950	2	<2	7.8	0.17	<0.3	81	21	64	7	30	1.1	3.41
5	<0.3	5.97	16	<2	810	2	<2	6.1	0.2	0.4	100	23	88	7	33	1.8	3.83
6	<0.3	7.34	11.8	<2	785	2.5	<2	11.8	0.225	0.3	104	28.5	77	10	42	1.7	4.125
7	<0.3	6.45	12.8	<2	920	2	<2	8.9	0.18	<0.3	95	23	55	9	35	1.3	3.68
8	<0.3	5.81	12.4	<2	510	2	<2	<0.5	0.15	<0.3	78	22	60	4	33	1.3	3.72
9	<0.3	5.79	13.8	<2	760	2	<2	7.5	0.16	<0.3	85	29	60	6	31	1.2	3.47
10	0.3	7.47	18.5	<2	740	3	<2	7.2	0.15	<0.3	89	29	74	6	45	1.2	4.94

Table C-2 (cont). Blairs Valley Lake sediment elemental data. All values are ppm (ug/g) unless indicated otherwise.

Station	Hf	Hg	Ir	%K	La	Li	Lu	Mg%	Mn	Mo	Na%	Nd	Ni	Pb	Rb	S%
1	9	<1	<5	1.63	40.9	48	0.4	0.56	610	<1	0.25	41	40	40	134	0.06
2	9	<1	<5	1.56	41.1	54	0.46	0.43	372	<1	0.28	34	33	34	134	0.05
3	10	<1	<5	1.31	31.8	42	0.37	0.39	612	<1	0.23	26	38	29	76	0.07
4	10	<1	<5	1.47	52	46	0.35	0.43	380	<1	0.22	22	38	31	85	0.06
5	9	<1	<5	1.78	62.8	52	0.44	0.5	480	<1	0.28	58	40	35	142	0.07
6	6.5	<1	<5	2.215	69	62.5	0.375	0.67	630.5	<1	0.23	38	47.5	39.5	171.5	0.075
7	9	<1	<5	1.87	63.5	53	0.33	0.56	350	<1	0.36	64	47	36	105	0.06
8	8	<1	<5	1.78	53.6	48	0.41	0.57	305	<1	0.36	39	40	28	105	0.04
9	9	<1	<5	1.72	57.6	50	0.41	0.48	447	<1	0.2	31	37	32	120	0.1
10	6	<1	<5	2.45	60.4	49	0.36	0.69	508	<1	0.11	45	47	36	113	0.06

Table C-2 (cont). Blairs Valley Lake sediment elemental data. All values are ppm (ug/g) unless indicated otherwise.

Station	Sb	Sc	Se	Sm	Sn%	Sr	Ta	Tb	Th	Ti%	U	V	W	Y	Yb	Zn
1	0.8	13.1	<3	6.9	<0.02	53	<0.5	<0.5	11.6	0.28	5.2	77	<1	29	3.2	122
2	0.6	12.3	<3	8.1	<0.02	56	<0.5	<0.5	11.1	0.24	3.1	59	<1	34	3.3	98
3	0.6	9.7	<3	6.7	<0.02	54	<0.5	<0.5	8.9	0.26	5.1	56	<1	33	3	104
4	0.8	11.4	<3	6.8	<0.02	54	<0.5	1.3	10.4	0.14	4.3	49	<1	32	3.3	108
5	0.9	13.7	<3	8	<0.02	64	<0.5	<0.5	13.7	0.29	5.6	62	<1	33	3.8	123
6	0.7	15.8	<3	8	<0.02	67.5	<0.5	0.75	10.8	0.315	4.35	81	<1	33	3.4	145
7	0.5	14.4	<3	7.9	<0.02	62	<0.5	<0.5	11.9	0.22	4.7	55	<1	32	3.4	129
8	0.9	13.2	<3	7.1	<0.02	53	<0.5	<0.5	10.3	0.13	4.1	46	<1	30	3.2	115
9	0.9	12.3	<3	6.7	<0.02	57	<0.5	<0.5	10.2	0.41	4.9	74	<1	32	3.2	114
10	1	16	<3	6.2	<0.02	45	<0.5	<0.5	10.9	0.4	3.4	107	<1	28	3.1	130

Table C-3. Results of analyses of Standard Reference Material (NIST SRM #8704 - Buffalo River Sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Silver	Ag	ppm	0.3			0.5	0.1	
Gold	Au	ppb	2			6	6	
Aluminum	Al	%	0.01	6.1	0.18	5.68	0.07	93.1
Arsenic	As	ppm	0.5	17		19.9	1.8	117.3
Barium	Ba	ppm	50	413	13	577	93	139.6
Beryllium	Be	ppm	1			2	0	
Bismuth	Bi	ppm	2			< 2		
Bromide	Br	ppm	0.5			8.8	0.9	
Calcium	Ca	%	0.01	2.641	0.083	2.71	0.01	102.7
Cadmium	Cd	ppm	0.3	2.94	0.29	3.1	0.0	105.4
Cerium	Ce	ppm	3	66.5	2	64	8	96.2
Cobalt	Co	ppm	1	13.57	0.43	14	3	103.2
Chromium	Cr	ppm	2	121.9	3.8	128	13	104.7
Cesium	Cs	ppm	1	5.83	0.12	8	1	131.5
Copper	Cu	ppm	1			91	2	
Europium	Eu	ppm	0.2	1.31	0.038	1.2	0.1	89.1
Iron	Fe	%	0.01	3.97	0.1	4.28	0.15	107.8
Hafnium	Hf	ppm	1	8.4	1.5	8	1	99.2
Mercury	Hg	ppm	1			1	1	
Iridium	Ir	ppb	5			< 5		
Potassium	K	%	0.01	2.001	0.041	1.92	0.04	96.1
Lanthanum	La	ppm	0.5			30.6	2.6	
Lithium	Li	ppm	1			47	0	
Lutetium	Lu	ppm	0.05			0.42	0.05	
Magnesium	Mg	%	0.01	1.2	0.018	1.15	0.01	95.6
Manganese	Mn	ppm	1	544	21	554	11	101.8
Molybdenum	Mo	ppm	1			2	1	
Sodium	Na	%	0.01	0.553	0.015	0.66	0.06	118.7
Neodymium	Nd	ppm	5			26	8	
Nickel	Ni	ppm	1	42.9	3.7	44	1	103.3
Phosphorus	P	%	0.001			0.081	0.005	
Lead	Pb	ppm	3	150	17	155	5	103.1
Rubidium	Rb	ppm	15			117	36	
Sulfur	S	%	0.01			0.35	0.01	
Antimony	Sb	ppm	0.1	3.07	0.32	2.9	0.2	94.5
Scandium	Sc	ppm	0.1	11.26	0.19	11.7	0.4	104.2
Selenium	Se	ppm	0.1			< 3		
Samarium	Sm	ppm	0.1			6.1	0.3	
Tin	Sn	%	0.01			0.02	0.03	
Strontium	Sr	ppm	1			132	1	
Tantatum	Ta	ppm	0.5			< 0.5		

Table C-3 con't. Results of analyses of Standard Reference Material (NIST SRM #8704 - Buffalo

River Sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Terbium	Tb	ppm	0.5			< 0.5		
Thorium	Th	ppm	0.2	9.07	0.16	9.7	0.6	106.6
Titanium	Ti	%	0.01	0.457	0.02	0.40	0.01	88.3
Uranium	U	ppm	0.5	3.09	0.13	3.2	0.2	104.6
Vanadium	V	ppm	2	94.6	4	83	4	88.1
Tungsten	W	ppm	1			< 1		
Yttrium	Y	ppm	1			23	1	
Ytterbium	Yb	ppm	0.2			3.1	0.2	
Zinc	Zn	ppm	1	408	15	403	13	98.8

Table C-4. Results of analyses of Standard Reference Material (NIST SRM #1646a- Estuarine Sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Silver	Ag	ppm	0.3			0.2	0.2	
Gold	Au	ppb	2			4	4	
Aluminum	Al	%	0.01	2.297	0.018	2.22	0.03	96.8
Arsenic	As	ppm	0.5	6.23	0.21	10.0	2.5	160.0
Barium	Ba	ppm	50			223	188	
Beryllium	Be	ppm	1			< 1		
Bismuth	Bi	ppm	2			< 2		
Bromide	Br	ppm	0.5			58.9	1.8	
Calcium	Ca	%	0.01	0.519	0.02	0.59	0.01	114.3
Cadmium	Cd	ppm	0.3	0.148	0.007	< 0.3		
Cerium	Ce	ppm	3			36	2	
Cobalt	Co	ppm	1			7	1	
Chromium	Cr	ppm	2	40.9	1.9	42	6	101.9
Cesium	Cs	ppm	1			< 1		
Copper	Cu	ppm	1	10.01	0.34	10	0	99.9
Europium	Eu	ppm	0.2			0.6	0.2	
Iron	Fe	%	0.01	2.008	0.039	2.21	0.08	109.9
Hafnium	Hf	ppm	1			12	1	
Mercury	Hg	ppm	1			< 1		
Iridium	Ir	ppb	5			< 5		
Potassium	K	%	0.01	0.864	0.016	0.87	0.01	100.7
Lanthanum	La	ppm	0.5			19.7	4.5	
Lithium	Li	ppm	1			19	1	
Lutetium	Lu	ppm	0.05			0.17	0.06	
Magnesium	Mg	%	0.01	0.388	0.009	0.39	0.01	101.4
Manganese	Mn	ppm	1	234.5	2.8	237	6	101.2
Molybdenum	Mo	ppm	1			< 1		
Sodium	Na	%	0.01	0.741	0.017	0.78	0.06	105.3
Neodymium	Nd	ppm	5			15	8	
Nickel	Ni	ppm	1			25	1	
Phosphorus	P	%	0.001	0.027	0.001	0.027	0.000	100.0
Lead	Pb	ppm	3	11.7	1.2	10	1	88.3
Rubidium	Rb	ppm	15			8	14	
Sulfur	S	%	0.01	0.352	0.004	0.35	0.01	99.4
Antimony	Sb	ppm	0.1			0.2	0.1	
Scandium	Sc	ppm	0.1			4.9	0.1	
Selenium	Se	ppm	0.1	0.193	0.028	< 3		
Samarium	Sm	ppm	0.1			3.3	0.1	
Tin	Sn	%	0.01			< 2		
Strontium	Sr	ppm	1			73	1	
Tantatum	Ta	ppm	0.5			< 0.5		
Terbium	Tb	ppm	0.5			< 0.5		

Table C-4 con't. Results of analyses of Standard Reference Material (NIST SRM #1646a- Estuarine Sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Thorium	Th	ppm	0.2			5.5	0.3	
Titanium	Ti	%	0.01	0.456	0.021	0.43	0.06	95.0
Uranium	U	ppm	0.5			2.1	0.7	
Vanadium	V	ppm	2	44.84	0.76	34	14	76.6
Tungsten	W	ppm	1			< 1		
Yttrium	Y	ppm	1			10	0	
Ytterbium	Yb	ppm	0.2			1.4	0.2	
Zinc	Zn	ppm	1	48.9	1.6	50	1	101.6

Table C-5. Results of analyses of Standard Reference Material (NIST SRM #2702- Inorganics in marine sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Silver	Ag	ppm	0.3	0.622	0.078	1.2	0.1	192.9
Gold	Au	ppb	2			17	20	
Aluminum	Al	%	0.01	8.41	0.22	7.63	0.26	90.7
Arsenic	As	ppm	0.5	45.3	1.8	49.8	2.6	110.0
Barium	Ba	ppm	50	397.4	3.2	170	151	42.8
Beryllium	Be	ppm	1			3	0	
Bismuth	Bi	ppm	2			6	2	
Bromide	Br	ppm	0.5			65.8	3.4	
Calcium	Ca	%	0.01	0.343	0.024	0.35	0.01	103.0
Cadmium	Cd	ppm	0.3	0.817	0.011	0.9	0.1	106.1
Cerium	Ce	ppm	3	123.4	5.8	117	8	94.8
Cobalt	Co	ppm	1	27.76	0.58	30	6	109.3
Chromium	Cr	ppm	2			324	28	
Cesium	Cs	ppm	1			6	2	
Copper	Cu	ppm	1	117.7	5.6	115	5	97.4
Europium	Eu	ppm	0.2			2.0	0.3	
Iron	Fe	%	0.01			7.78	0.24	
Hafnium	Hf	ppm	1			10	2	
Mercury	Hg	ppm	1	0.4474	0.0069	< 1		
Iridium	Ir	ppb	5			< 5		
Potassium	K	%	0.01	2.054	0.072	1.91	0.15	93.2
Lanthanum	La	ppm	0.5	73.5	4.2	64.5	5.4	87.8
Lithium	Li	ppm	1			77	2	
Lutetium	Lu	ppm	0.05			0.55	0.15	
Magnesium	Mg	%	0.01	0.990	0.074	0.92	0.02	93.3
Manganese	Mn	ppm	1	1757	58	1653	35	94.1
Molybdenum	Mo	ppm	1	10.8	1.6	9	1	83.3
Sodium	Na	%	0.01	0.681	0.020	0.75	0.05	109.6
Neodymium	Nd	ppm	5			63	14	
Nickel	Ni	ppm	1	75.4	1.5	77	2	102.1
Phosphorus	P	%	0.001	0.1552	0.0066	0.133	0.012	85.9
Lead	Pb	ppm	3	132.8	1.1	126	4	94.6
Rubidium	Rb	ppm	15	127.7	8.8	89	78	69.4
Sulfur	S	%	0.01			1.50	0.09	
Antimony	Sb	ppm	0.1	5.6	0.24	4.9	0.7	86.9
Scandium	Sc	ppm	0.1	25.9	1.1	24.1	1.1	93.2
Selenium	Se	ppm	0.1	4.95	0.46	< 3		
Samarium	Sm	ppm	0.1			10.6	0.7	
Tin	Sn	%	0.01	31.6	2.4	< 0.02		
Strontium	Sr	ppm	1	119.7	3.0	118	4	98.9
Tantatum	Ta	ppm	0.5			3.4	3.4	
Terbium	Tb	ppm	0.5			< 0.5		

Table C-5 con't. Results of analyses of Standard Reference Material (NIST SRM #2702- Inorganics in marine sediment) submitted as blind unknowns with the Blairs Valley Lake surficial samples. Also given are the method detection limits for each element reported by Act Labs, Inc.

Analyte	Symbol	Unit	Detection Limit	Certified value	Std dev	Act Labs Results		
						Average	Std dev	% recovery
Thorium	Th	ppm	0.2	20.51	0.96	19.5	0.1	95.1
Titanium	Ti	%	0.01	0.884	0.082	0.81	0.05	91.3
Uranium	U	ppm	0.5			9.0	1.4	
Vanadium	V	ppm	2	357.6	9.2	327	12	91.5
Tungsten	W	ppm	1			1	2	
Yttrium	Y	ppm	1			34	1	
Ytterbium	Yb	ppm	0.2			3.9	0.3	
Zinc	Zn	ppm	1	485.3	4.2	473	10	97.4

Table C-6. Results of Act Labs analyses of Reference material for the TD-ICP method. Act Labs' measure values compared to the certified values. Reference materials include USGS geochemical exploration references.

Analyte Symbol	Unit Symbol	Detection Limit	USGS DNC-1a Dolerite		USGS DNC-1a Dolerite		USGS SBC-1 Shale		Method Blank
			Measured value	Certified value	Measured value	Certified value	Measured value	Certified value	
Ag	ppm	0.3							< 0.3
Cu	ppm	1	97	100	97	100	32	31	< 1
Cd	ppm	0.3					0.4	0.4	< 0.3
Mo	ppm	1					< 1	2	< 1
Pb	ppm	3	< 3	6.3	< 3	6.3	30	35	< 3
Ni	ppm	1	248	247	248	247	87	83	< 1
Zn	ppm	1	59	70	60	70	187	186	< 1
S	%	0.01							< 0.01
Al	%	0.01							< 0.01
Be	ppm	1					3	3.2	< 1
Bi	ppm	2					< 2	0.7	< 2
Ca	%	0.01	7.63	8.21	7.76	8.21			< 0.01
K	%	0.01							< 0.01
Li	ppm	1	5	5.2	6	5.2	181	163	< 1
Mg	%	0.01							< 0.01
Mn	ppm	1							2
P	%	0.001							< 0.001
Sr	ppm	1	136	144	138	144	185	178	< 1
Ti	%	0.01	0.27	0.29	0.28	0.29	0.5	0.51	< 0.01
V	ppm	2	133	148	135	148	207	220	< 2
Y	ppm	1	16	18	16	18	34	36.5	< 1