



# **Soil Geochemical Data for the Wyoming Landscape Conservation Initiative Study Area**

By David B. Smith and Karl J. Ellefsen

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## Conversion Factors

SI to Inch/Pound

Multiply	By	To obtain
Length		
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
Area		
square kilometer (km <sup>2</sup> )	247.1	acre
Mass		
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)

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

$$^{\circ}\text{F}=(1.8\times^{\circ}\text{C})+32$$

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## Abstract

In 2008 and 2010, soil samples were collected at 175 sites throughout the Wyoming Landscape Conservation Initiative study area in southwest Wyoming. These samples, representing a density of 1 site per 440 square kilometers, were collected from a depth of 0–5 cm and analyzed for a suite of more than 40 major and trace elements following a near-total multi-acid extraction. In addition, soil pH, electrical conductivity, total nitrogen, total and organic carbon, and sodium adsorption ratio were determined. The resulting data set provides a baseline for detecting changes in soil composition that might result from natural processes or anthropogenic activities. This report describes the sampling and analytical protocols used, and makes available all the soil geochemical data generated in the study.

## Introduction

As discussed in Bowen and others (2009a, b), the Wyoming Landscape Conservation Initiative (WLCI) was initiated in 2007 primarily in response to concerns about threats posed to the state's world-class wildlife resources by increasing energy development in southwest Wyoming. The WLCI, in partnership with Federal, State, and local agencies and with participation from public and private entities, industry, and landowners, seeks to assess and enhance aquatic and terrestrial habitats at a landscape scale, while facilitating responsible energy and other types of development. The role of the U.S. Geological Survey (USGS) as a partner in the WLCI is to provide multidisciplinary scientific and technical support to WLCI partners and to advance the scientific understanding of southwest Wyoming's ecosystems.

One of the major work activities performed by the USGS is targeted monitoring and research, and one of the tasks included in this activity is long-term monitoring. For long-term monitoring, USGS scientists developed a landscape-scale monitoring framework using a spatially balanced design with random site selection to represent the WLCI landscape. This framework provides a means for detecting changes in land use, vegetation, priority habitats, populations of priority species, and soil geochemistry across the region. The purpose of this report is to provide information regarding the soil geochemistry component and to make available the soil geochemical data generated in the WLCI study area.

## Sample-Collection and Sample-Preparation Protocols

A generalized random tessellation stratified design was used to select 175 target sites for soil sampling within the WLCI study area. This represented a density of approximately 1 site per 440 square kilometers ( $\text{km}^2$ ). The actual site from which a sample was collected was chosen from the most representative landscape as near as possible to the target site while also taking into consideration the following guidelines:

1. No sample should be collected from within 200 meters (m) of a major highway.
2. No sample should be collected closer than 50 m to a rural road.
3. No sample should be collected closer than 100 m to a building or structure.

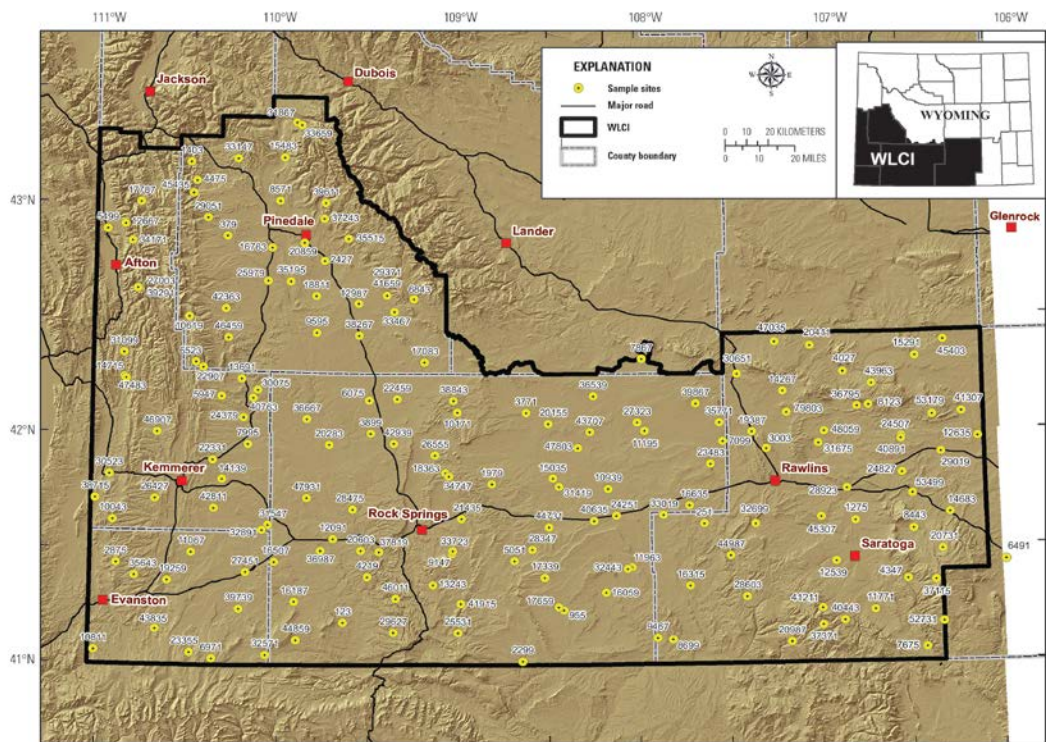


Figure 1. Wyoming Landscape Conservation Initiative (WLCI) study area showing the locations of 175 soil samples collected for chemical analysis.

At each site, a sample of soil was collected from a depth of 0 to 5 centimeters (cm) after removing loose plant debris (if any) from the ground surface. This particular sample medium was selected because this near-surface material is the portion of the soil with which humans and most animals come into contact most often. Geochemical information for this layer is critical for evaluation of a soil pathway through which potentially toxic elements could enter the bodies of both humans and animals. It is also considered to be the portion of the soil most likely to indicate the influence of human activities such as energy development or industrialization. The soil surface, or 0 cm, is considered to be the top boundary of the first soil layer that can support plant/root growth. For mineral soils, the surface is generally the air-earth interface; for vegetated mineral soils, it is the upper boundary of the first layer that can support root growth; for organic soils, the surface is considered to be the upper boundary of the first layer that supports root growth, excluding freshly fallen plant litter, but including litter that has compacted and begun to decompose.

The samples were air dried at ambient temperature, disaggregated, and sieved through a 2-millimeter (mm) stainless steel screen. Material less than 2 mm in size was crushed to less than 150 micrometers ( $\mu\text{m}$ ) in a ceramic mill and thoroughly mixed to ensure homogeneity. The crushed samples were randomized prior to chemical analysis to avoid confusing spatial variation with any possible systematic bias within a given analytical technique. This randomization does not eliminate a systematic error, but the error is effectively transformed into one that is random with respect to geographic location (Tidball, 1984).

## Analytical Protocols

The prepared samples were sent to a USGS contract geochemical laboratory for major and trace element analysis. The concentrations of the elements aluminum (Al), calcium (Ca), iron (Fe), potassium (K), magnesium (Mg), sodium (Na), sulfur (S), titanium (Ti), silver (Ag), barium (Ba), beryllium (Be), bismuth (Bi), cadmium (Cd), cerium (Ce), cobalt (Co), chromium (Cr), cesium (Cs), copper (Cu), gallium (Ga), indium (In), lanthanum (La), lithium (Li), manganese (Mn), molybdenum (Mo), niobium (Nb), nickel (Ni), phosphorus (P), lead (Pb), rubidium (Rb), antimony (Sb), scandium (Sc), tin (Sn), strontium (Sr), tellurium (Te), thorium (Th), thallium (Tl), uranium (U), vanadium (V), tungsten (W), yttrium (Y), and zinc (Zn) were determined by inductively coupled plasma–atomic emission spectrometry (ICP-AES) by a method similar to Briggs (2002) and inductively coupled plasma–mass spectrometry (ICP-MS) by a method similar to Briggs and Meier (2002). A sample of 0.25 grams (g) was decomposed using a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids at a temperature between 125 and 150 °C.

The four-acid digestion results in effectively a total dissolution of most mineral constituents in soil. However, it does not fully dissolve some of the more refractory or resistant minerals. Examples of such incomplete dissolution include Ba in barite, Cr in chromite, Ti in rutile, Sn in cassiterite, Al in corundum, and rare earth elements in monazite (Briggs, 2002). An aliquot of the digested sample was aspirated into the ICP-AES instrument and the ICP-MS instrument and the concentrations of the optimal elements were determined. The ICP-AES method is best for the major elements, sulfur, and elements with relatively high concentrations not requiring a low detection limit, and the ICP-MS method is optimal for trace elements requiring lower limits of determination near or below their crustal abundance and elements not determined by ICP-AES. The lower limits of determination (LLD) are listed in table 1.

Table 1. Elements determined by ICP-MS and ICP-AES. [LLD, lower limits of determination; ICP-MS, inductively coupled plasma–mass spectrometry; ICP-AES, inductively coupled plasma–atomic emission spectrometry]

Element	LLD
Aluminum	0.01%
Calcium	0.01%
Iron	0.01%
Potassium	0.01%
Magnesium	0.01%
Sodium	0.01%
Phosphorous	50 mg/kg
Titanium	0.01%
Silver	1 mg/kg
Barium	5 mg/kg
Beryllium	0.1 mg/kg
Bismuth	0.04 mg/kg
Cadmium	0.1 mg/kg
Cerium	0.05 mg/kg
Cobalt	0.1 mg/kg
Chromium	1 mg/kg
Cesium	0.05 mg/kg
Copper	0.5 mg/kg
Gallium	0.05 mg/kg
Indium	0.02 mg/kg
Lanthanum	0.5 mg/kg
Lithium	1 mg/kg
Manganese	5 mg/kg
Molybdenum	0.05 mg/kg
Niobium	0.1 mg/kg
Nickel	0.5 mg/kg
Lead	0.5 mg/kg
Rubidium	0.2 mg/kg
Sulfur	0.01%
Antimony	0.05 mg/kg
Scandium	0.1 mg/kg
Tin	0.1 mg/kg
Strontium	0.5 mg/kg
Tellurium	0.1 mg/kg
Thallium	0.1 mg/kg
Thorium	0.2 mg/kg
Uranium	0.1 mg/kg
Vanadium	1 mg/kg
Tungsten	0.1 mg/kg
Yttrium	0.1 mg/kg
Zinc	1 mg/kg



Mercury (Hg) was determined by treating 0.1 g of sample with a mixture of nitric and hydrochloric acids and heating for 30 minutes (min). Once cooled, solutions of sulfuric acid, potassium permanganate, and potassium persulfate were added followed by sodium chloride-hydroxylamine sulfate. The final solution was reduced by stannous chloride and analyzed on an atomic absorption spectrometer. This method is a modification of that published by the U.S. Environmental Protection Agency (1994). The LLD is 0.02 milligrams per kilogram (mg/kg).

Selenium (Se) was determined by heating 0.25 g of sample with a combination of nitric, hydrofluoric and perchloric acids. After the solution was cooled, hydrochloric and nitric acids were added, then the solution was heated and cooled again. The sample was diluted and analyzed using hydride generation atomic absorption spectrometry in a method similar to Hageman and Brown (2002). The LLD is 0.2 mg/kg.

For analysis of arsenic (As), the sample was fused in a mixture of sodium peroxide and sodium hydroxide at 750 °C. The fused mixture was then dissolved in hydrochloric acid and analyzed by hydride-generation atomic absorption spectrometry in a method similar to Hageman and Brown (2002). The LLS is 0.6 mg/kg.

Total carbon was determined by the use of an automated carbon analyzer. A sample of 0.25 g was combusted in an oxygen atmosphere at 1,370 °C to oxidize carbon to carbon dioxide (CO<sub>2</sub>). Moisture and dust were removed and the CO<sub>2</sub> gas was measured by a solid state infrared detector in a method similar to Brown and Curry (2002). The LLD is 0.05 percent.

Carbonate carbon was determined as CO<sub>2</sub> by coulometric titration. A sample of 0.1 g was treated with hot 2N perchloric acid and the evolved CO<sub>2</sub> was passed into a cell containing a solution of monoethanolamine. The CO<sub>2</sub>, quantitatively absorbed by the monoethanolamine, was coulometrically titrated using platinum and silver/potassium-iodide electrodes in a method similar to Brown and others (2002). The LLD is 0.05 percent. The measured percent of CO<sub>2</sub> was converted to percent of carbonate carbon by multiplying the percent by the weight fraction of carbon in CO<sub>2</sub> (0.273). The concentration of organic carbon was then calculated as the difference between total carbon and carbonate carbon.

Splits of the material less than 2 mm in size were provided to Colorado State University's Soil, Water, and Plant Testing Laboratory for analysis of total nitrogen (N) content, soil pH, electrical conductivity, and sodium adsorption ratio (SAR). Total N was determined on a carbon-hydrogen-nitrogen (CHN) analyzer by combusting an aliquot of the soil sample at approximately 950 °C and determining the percent N in a thermal conductivity cell (Bremner, 1996). Soil pH and electrical conductivity were determined on a saturated soil paste using the methods described by Richards (1954) and Rhoades (1996), respectively. The SAR is defined as

$$\text{SAR} = [\text{Na}^+]/([\text{Ca}^{2+}] + [\text{Mg}^{2+}]/2)^{1/2} \quad (1)$$

where [Na<sup>+</sup>], [Ca<sup>2+</sup>], and [Mg<sup>2+</sup>] are the concentrations of these cations, expressed as milliequivalents per liter (meq/L), in a saturation extract (Sumner and Miller, 1996; Natural Resources Conservation Service, 1996).

## Quality Assurance and Quality Control

Quality assurance (QA) is mainly the concern of the analytical laboratory. The various components of the QA plan include standard operating procedures, instrument logs, training records, data acceptance and rejection criteria, and laboratory audits. Unlike the unquantifiable QA element, the quality control (QC) element measures, by an analytical method, the accuracy and precision of the data produced. The accuracy and precision are established through the analysis of reference materials (RMs) and sample replicates, respectively.

The samples taken through the analytical process in the USGS laboratories received QC checks on three separate levels. The first level involved QC assessment by the USGS contract laboratory. In the next level, quality was assessed by the USGS QC officer, followed by the third-level assessment by the USGS principal investigator for the WLCI soil geochemistry study.

The USGS contract laboratory is accredited to the International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) 17025 standard, which includes both QA and QC protocols. The QC is monitored by analyzing an RM with every batch of 48 samples. The RM most often used is a syenite rock standard (SY-3) developed by the Canadian Centre for Mineral and Energy Technology (Govindaraju, 1989). Shewhart Control Charts (Taylor, 1987) are generated for the RM analyses and reviewed with every report as part of the internal quality audits.

The accuracy for elements determined by ICP-MS and ICP-AES, and for total C and carbonate C was considered acceptable if recovery was within the range of 85–115 percent at five times the LLD. The accuracy for Hg and Se was considered acceptable if recovery was within 80–120 percent at five times the LLD.

At the second tier, the USGS QC officer assessed precision and accuracy on the basis of five RMs that were inserted between every batch of 50 samples. The soil RMs used in this study were SRM 2709 and SoNE-1. SRM 2709, a soil from the San Joaquin Valley of central California, is a certified RM available from the National Institute of Standards and Technology (NIST) and is used to assess both accuracy and precision (National Institute of Standards and Technology, 2003). The acceptance criteria for accuracy are the same as those itemized earlier as used by the contract laboratory.

SoNE-1 is a USGS in-house, non-certified soil RM prepared specifically for the North American Soil Geochemical Landscapes Project. It was collected from the Sharpsburg Soil Series (Natural Resources Conservation Service, 1997) in Lancaster County, Nebr., and is used in this study to assess only precision. The precision for elements determined by ICP-MS and ICP-AES, and for total C and carbonate C was considered acceptable if the calculated relative standard deviation (RSD) of duplicate samples is no greater than 15 percent. The precision for Hg, As, and Se was considered acceptable if the calculated RSD of duplicate samples was no greater than 20 percent.

The USGS principal investigator for the WLCI soil geochemistry study initiated the final QC tier, which included two blind SoNE-1 RMs inserted in each batch of 18 samples. In general, one RM was inserted into the first half of the batch and one RM into the second half. The acceptance criteria for accuracy and precision are those stated in the previous paragraph.

Colorado State University's Soil, Water, and Plant Testing Laboratory maintains its own rigorous QC program using appropriate standard reference materials to determine the accuracy of each technique and to estimate precision through analytical duplicates.

## Description of Data Tables

Table 2 shows a statistical summary of the major and trace element data generated from the WLCI soil samples. Table 3 presents the complete analytical results from both the USGS and Colorado State University laboratories for the WLCI soils.

Table 2. Statistical summary for geochemical data on 0–5-cm soils in the WLCI study area. [n = 175; WLCI, Wyoming Landscape Conservation Initiative; LLD, lower limit of determination; Min, minimum; Q1, first quartile; Max, maximum; Q3, third quartile; MAD, median absolute deviation]

Element	Concentration units	Number of samples below LLD	Min	Q1	Median	Q3	Max	MAD
Aluminum	%	0	2.11	4.38	5.27	6.24	8.09	1.36
Calcium	%	0	0.21	0.82	1.62	3.02	10.3	1.39
Iron	%	0	0.29	1.56	1.95	2.35	4.47	0.59
Potassium	%	0	0.83	1.62	1.88	2.15	3.87	0.34
Magnesium	%	0	0.07	0.49	0.73	1.06	2.42	0.43
Sodium	%	0	0.08	0.58	1.05	1.48	2.66	0.67
Sulfur	%	4	<0.01	0.02	0.03	0.04	0.29	0.015
Titanium	%	0	0.04	0.15	0.19	0.24	0.47	0.059
Arsenic	mg/kg	1	<0.6	3.2	5.2	7.2	30.1	3.0
Silver	mg/kg	175	<1	<1	<1	<1	<1	0
Barium	mg/kg	0	220	553	660	851	2,390	200
Beryllium	mg/kg	0	0.6	1.3	1.5	1.8	2.5	0.4
Bismuth	mg/kg	1	<0.04	0.15	0.20	0.27	0.60	0.089
Cadmium	mg/kg	4	<0.1	0.2	0.3	0.5	5.7	0.15
Cerium	mg/kg	0	18.3	45.6	59.1	70.6	161	19.1
Cobalt	mg/kg	0	0.8	5.9	7.6	9.8	24.9	2.7
Chromium	mg/kg	0	3	31.5	42	50	287	13.3
Cesium	mg/kg	138	<5	<5	<5	5	11	0
Copper	mg/kg	0	1.4	10.6	14.7	19.3	47.9	6.5
Gallium	mg/kg	0	5.6	11.0	13.6	16.0	22.2	3.7
Mercury	mg/kg	31	<0.01	0.01	0.02	0.02	0.15	0.015
Indium	mg/kg	20	<0.02	0.03	0.03	0.04	0.06	0.01
Lanthanum	mg/kg	0	10.1	17.8	23.2	36.8	75.8	9.8
Lithium	mg/kg	0	3	17	23	29	72	8.9
Manganese	mg/kg	0	63	323	460	619	2,000	214
Molybdenum	mg/kg	0	0.15	0.61	0.94	1.27	7.38	0.49
Niobium	mg/kg	0	1.5	6.0	7.7	9.4	25.8	2.5
Nickel	mg/kg	0	1.7	11.2	14.1	17.5	115	4.6
Phosphorous	mg/kg	0	150	555	800	1,030	8,590	341
Lead	mg/kg	0	10.6	16.2	19.2	21.4	32.2	3.7
Rubidium	mg/kg	0	40.9	68.0	79.3	91.6	140	18.1
Antimony	mg/kg	0	0.09	0.40	0.52	0.67	2.53	0.22
Scandium	mg/kg	0	1.5	5.0	6.3	8.0	15.4	2.2
Selenium	mg/kg	110	<0.2	<0.2	<0.2	0.3	4.2	0
Tin	mg/kg	0	0.4	1.0	1.2	1.5	2.8	0.44

Strontium	mg/kg	0	50.1	120	160	364	706	93.4
Tellurium	mg/kg	175	<0.1	<0.1	<0.1	<0.1	<0.1	0
Thorium	mg/kg	0	4.4	7.4	9.4	11.2	32.5	2.8
Thallium	mg/kg	0	0.2	0.4	0.5	0.6	1.5	0.15
Uranium	mg/kg	0	0.8	1.7	2.1	2.6	9.6	0.74
Vanadium	mg/kg	0	8	41	53	68	196	21
Tungsten	mg/kg	0	0.1	0.6	0.8	1.1	6.9	0.3
Yttrium	mg/kg	0	3.4	12.0	14.3	16.3	45.3	3.1
Zinc	mg/kg	0	12	42	55	71	210	20.8
Total Carbon	%	0	0.16	0.89	1.73	3.30	18.3	14.2
Carbonate								
Carbon	%	18	<0.0030	0.01	0.07	0.75	3.28	0.099
Organic								
Carbon	%	0	0.16	0.54	0.96	2.66	18.3	0.85
Total Nitrogen	%	0	0.0036	0.079	0.11	0.24	1.10	0.076







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