Geochemical and Mineralogical Data for Soils of the Conterminous United States

Data Series 801

Front cover.   *Left*, Harvey Thorleifson (Minnesota State Geologist) collects soil sample at site 7880 in Minnesota; photograph by Richard Lively, Minnesota Geological Survey. *Center*, Site 11369 in Vermont; photograph by Laurel Woodruff, U.S. Geological Survey. *Right*, James Kilburn (U.S. Geological Survey) collecting soil sample at site 9643 in Kansas; photograph by John Horton, U.S. Geological Survey. *Background*, Site 10457 in Minnesota; photograph by Harvey Thorleifson, Minnesota Geological Survey.

Back cover.   *Left*, Site 828 in New York; photograph by Suzanne Nicholson, U.S. Geological Survey. *Upper right*, Harvey Thorleifson (Minnesota State Geologist) collects sample at site 968 in Minnesota; photograph by Richard Lively, Minnesota Geological Survey. *Middle right*, James Kilburn (U.S. Geological Survey) collecting soil sample in New Mexico during pilot study phase of project; photograph by Harley King, U.S. Geological Survey. *Lower right*, Helen Folger (U.S. Geological Survey) collecting soil sample at site 1500 in West Virginia; photograph by Damon Bickerstaff, U.S. Geological Survey.

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SALLY JEWELL, Secretary

U.S. Geological Survey
Suzette M. Kimball, Acting Director

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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)
meter (m)	1.094	yard (yd)
Area		
square kilometer (km^2)	247.1	acre
square kilometer (km^2)	0.3861	square mile (mi^2)
Mass		
kilogram (kg)	2.205	pound, avoirdupois (lb)
milligram (mg)	0.0000353	ounce, avoirdupois (oz)

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

$$°F=(1.8×°C)+32$$

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Abstract

In 2007, the U.S. Geological Survey initiated a low-density (1 site per 1,600 square kilometers, 4,857 sites) geochemical and mineralogical survey of soils of the conterminous United States as part of the North American Soil Geochemical Landscapes Project. Sampling and analytical protocols were developed at a workshop in 2003, and pilot studies were conducted from 2004 to 2007 to test and refine these recommended protocols. The final sampling protocol for the national-scale survey included, at each site, a sample from a depth of 0 to 5 centimeters, a composite of the soil A horizon, and a deeper sample from the soil C horizon or, if the top of the C horizon was at a depth greater than 1 meter, from a depth of approximately 80–100 centimeters. The <2-millimeter fraction of each sample was analyzed for a suite of 45 major and trace elements by methods that yield the total or near-total elemental content. The major mineralogical components in the samples from the soil A and C horizons were determined by a quantitative X-ray diffraction method using Rietveld refinement. Sampling in the conterminous United States was completed in 2010, with chemical and mineralogical analyses completed in May 2013. The resulting dataset provides an estimate of the abundance and spatial distribution of chemical elements and minerals in soils of the conterminous United States and represents a baseline for soil geochemistry and mineralogy against which future changes may be recognized and quantified. This report (1) describes the sampling, sample preparation, and analytical methods used; (2) gives details of the quality control protocols used to monitor the quality of chemical and mineralogical analyses over approximately six years; and (3) makes available the soil geochemical and mineralogical data in downloadable tables.

Introduction

Soil is a critical component of the Earth's life support system. It plays a vital role in (1) determining human health and ecosystem integrity; (2) supporting food production; (3) water storage and ground water recharge; and (4) global biogeochemical cycles of carbon, phosphorus, nitrogen, and other essential nutrients in the environment. Soil is also the medium into which many communities dispose of solid and liquid wastes from households, agricultural operations, and industrial processes. Through ingestion, inhalation, or dermal absorption, soil can be a pathway for potentially toxic chemicals of natural or human origin to enter the human body (Oliver, 1997; Abrahams, 2002, 2012; Plumlee and Ziegler, 2003). In addition, soil is the primary source of biologically active trace elements that reach humans through the food chain (Oliver, 1997). Although soil is important, our knowledge about the concentration and spatial distribution of naturally occurring elements in the soils of North America is remarkably limited (Smith and others, 2013). At present, there is neither a common soil geochemical database for the continent of North America nor a sound understanding of the processes that might control the variation in elemental composition at the continental scale (Smith, 2009).

In 2001, the Directors of the U.S. Geological Survey (USGS), the Geological Survey of Canada (GSC), and the Mexican Geological Survey (Servicio Geológico Mexicano, SGM) jointly recognized the need to establish a soil geochemical database for the continent of North America. These three agencies subsequently established the North American Soil Geochemical Landscapes Project (NASGLP) to address this issue. A workshop was held in 2003 to obtain stakeholder input on the project's optimal design; it attracted 112 attendees representing 42 different stakeholder entities. One outcome of the workshop was a set of recommendations for sample collection protocols and analytical techniques for the proposed continental-scale soil geochemical survey. Smith and others (2011, 2012) document the history and evolution of the project from 2001 to 2010.

Pilot studies at two very different scales were carried out from 2004 to 2007. One of the pilot studies was conducted at a continental-scale and consisted of sampling and analysis of soils at approximately 40-kilometer (km) intervals along two transects across Canada, the United States, and Mexico (Smith and others, 2005). The purpose of this continental-scale pilot study was to test and refine the sampling and analytical protocols developed at the 2003 workshop (Smith and others, 2011, 2012) and to optimize field logistics. A pilot

study at a more regional scale—a 20,000-square-kilometer (km^2) area of northern California—was conducted as a model for higher-resolution, process-oriented follow-up studies that might be performed on areas of interest selected from the low-density continental-scale mapping. The results of these pilot studies were published as 21 papers in a special issue of "Applied Geochemistry" in August 2009 (Bern, 2009; Cannon and Horton, 2009; Chiprés and others, 2009 a, b; Eberl and Smith, 2009; Garrett, 2009; Garrett and others, 2009; Goldhaber and others, 2009; Griffin and others, 2009; Grunsky and others, 2009; Holloway and others, 2009; Klassen, 2009; McCafferty and Van Gosen, 2009; Morman and others, 2009; Morrison and others, 2009; Reeves and Smith, 2009; Smith and others, 2009; Tuttle and Breit, 2009; Tuttle and others, 2009; Wanty and others, 2009; Woodruff and others, 2009). Smith and others (2009) and Chiprés and others (2009a) give details of the design, sample collection, and analytical protocols for the continental-scale pilot study, and Goldhaber and others (2009) provide similar information for the northern California regional-scale pilot study.

Sampling for the full national-scale soil geochemical and mineralogical survey of the conterminous United States began in 2007 and was completed in 2010. Chemical and mineralogical analyses of the samples were completed in May 2013. The current report (1) describes the sampling, sample preparation, and analytical methods used; (2) gives details of the quality control protocols used to monitor the quality of chemical and mineralogical analyses generated over approximately six years; and (3) makes available the soil geochemical and mineralogical data in downloadable tables.

Selection of Sample Sites

In 2006, a small workshop devoted exclusively to sample site selection for the NASGLP was convened in Denver, Colorado. Thirteen attendees representing the USGS, GSC, U.S. Environmental Protection Agency, Centers for Disease Control and Prevention, Minnesota Geological Survey, Natural Resources Conservation Service, Savannah River Ecology Laboratory, Agriculture and Agri-Food Canada, and Environment Canada participated in the workshop. The consensus recommendation from this group was to use a generalized random tessellation stratified (GRTS) design to select target sites that represented a density of approximately 1 site per 1,600 km^2 (4,857 sites for the conterminous United States). The GRTS design produces a spatially balanced set of sampling points without adhering to a strict grid-based system. Its attributes have been fully described in technical publications (Stevens and Olsen, 2000, 2003, 2004; Olsen, 2005), and routines for implementing the design are readily available.

If a target site was inaccessible for any reason during the sampling program, the field crew would select an alternative site as close as possible to the original site with the landscape

and soil characteristics as similar to the original site as possible. The following guidelines were also used in the site selection process to ensure that samples were not collected from obviously contaminated areas:

1. No sample should be collected within 200 meters (m) of a major highway.
2. No sample should be collected within 50 m of a rural road.
3. No sample should be collected within 100 m of a building or structure.
4. No sample should be collected within 5 km downwind of active major industrial activities such as power plants or smelters.

Sampling Protocols

Participants in the 2003 workshop (Smith and others, 2012) recommended that sampling at each site should be based primarily on soil horizons rather than on constant depth intervals. Sampling by horizon provides data on discrete soil genetic units, whereas depth-interval sampling mixes different genetic units in an uncontrolled and largely unknown manner. Stakeholders from the public health sector, however, strongly supported collection of surface soil from a depth of 0 to 5 centimeters (cm) because it is the portion of the soil profile with which humans most often come into contact during their daily activities.

The final protocols used for the national-scale survey were a combination of depth-based and horizon-based sampling. Ideally, the following samples were collected at each site: (1) soil from a depth of 0 to 5 cm; (2) a composite of the soil A horizon (the uppermost mineral soil); and (3) a sample from the soil C horizon (generally partially weathered parent material) or, if the top of the C horizon was deeper than 1 m, a sample from about 80 to 100 cm. In addition, a separate sample of surface soil (0–5 cm) was collected at each site for the determination of selected soil pathogens, and separate samples of all three sample types were collected at 10 percent of the sites for further microbial characterization studies (the microbiological aspects of the project will not be discussed in this report). The sampling manual provided to field crews is shown in appendix 1.

Samples were collected by state beginning in 2007 with the last sample collected in late 2010 (fig. 1). Sampling in 2007 (the six New England states and New York) was conducted by USGS personnel. Most of the sampling during the final three years was conducted by teams of students chosen for their academic course work in soil science and participation on their university's soil judging team. Nineteen students representing twelve universities participated in this sampling program. Samples in North Dakota and South Dakota were collected by staff of the U.S. Department of Agriculture's Natural Resources Conservation Service. The Pennsylvania Geological Survey, the Conservation and Survey Division of
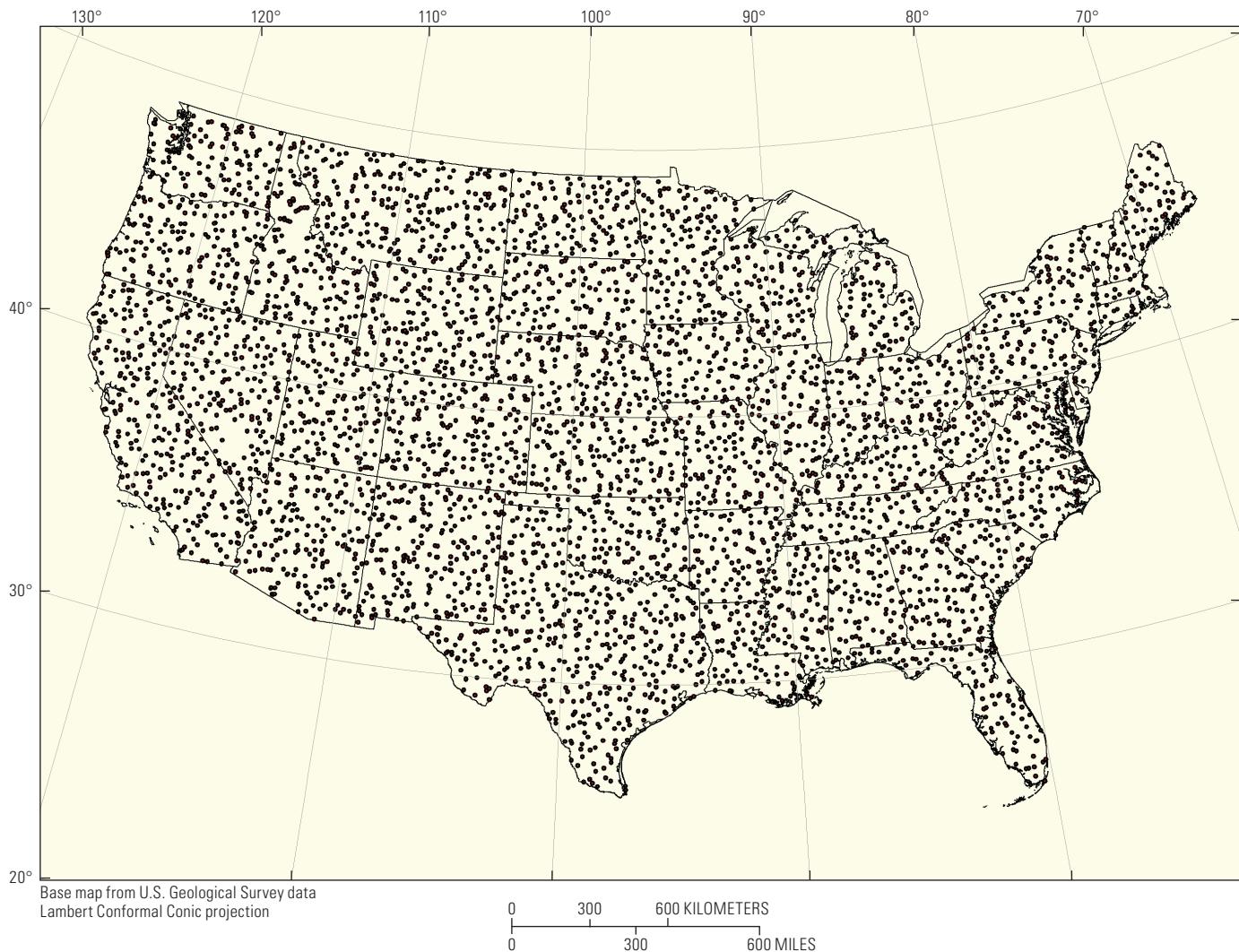
Figure 1.   Map showing the location of 4,857 soil sampling sites in the conterminous United States.

the University of Nebraska's School of Natural Resources, and the Minnesota Geological Survey collected samples in their respective states.

Not all sample types were collected at each site. For some urban sites (for example, city parks or private yards), only the surface sample (0–5 cm) was collected for fear of digging into buried utilities or sprinkler systems. In addition, a small number of samples were lost in shipping, so some sites have only one or two sample types analyzed and shown in the data tables.

Sample Preparation and Chemical Analysis

All samples were shipped to the USGS laboratories in Denver, Colorado, where they were prepared and submitted for analysis by a USGS contract laboratory in the order they were collected, by state. As a result of this process, chemical analyses were carried out from late 2007 to early 2013. For large

geochemical surveys like this one, the ideal course of action is to submit the samples for chemical analysis in a single batch after all samples have been collected in order to avoid bias in the chemical data caused by changes during the several years of the collection phase, such as changes in analytical instruments or analysts. The year-to-year budget process in the USGS, however, dictated that samples had to be submitted on a yearly basis. All samples within a given state 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 regard to geographic location within a state.

Each sample was air-dried at ambient temperature, disaggregated, and sieved to <2 millimeters (mm). The <2-mm material was then crushed to <150 micrometers (µm) prior to chemical analysis. Concentrations of 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 adapted from Briggs (2002) and by inductively coupled plasma–mass spectrometry (ICP-MS) by a method adapted from Briggs and Meier (2002). The sample was decomposed using a near-total four-acid (hydrochloric, nitric, hydrofluoric, and perchloric) digestion at a temperature between 125 and 150 ºC. The lower limits of determination (LLD) are shown in table 1. Mercury (Hg) was determined by cold-vapor atomic absorption spectrometry after dissolution in a mixture of nitric and hydrochloric acids in a modification of the method published by the U.S. Environmental Protection Agency (1994). The LLD is 0.01 milligram Hg per kilogram (mg Hg/kg). For analysis of arsenic (As), the sample was fused in a mixture of sodium peroxide and sodium hydroxide at 750 ºC. The fused mixture was then dissolved in hydrochloric acid and analyzed by hydride-generation atomic absorption spectrometry in a method similar to Hageman and others (2002). The LLD was 0.6 mg As/kg. Selenium (Se) was determined by hydride-generation atomic absorption spectrometry after dissolution in a mixture of nitric, hydrofluoric, and perchloric acids (Hageman and others, 2002). The LLD was 0.2 mg Se/kg. Total carbon (C) was determined by the use of an automated carbon analyzer. The sample was combusted in an oxygen atmosphere at 1,370 ºC to oxidize C to carbon dioxide (CO_2). The CO_2 gas was measured by a solid state infrared detector in a method similar to Brown and Curry (2002). The LLD is 0.01 percent C. The concentration of organic carbon was calculated by subtracting the amount of inorganic (carbonate) carbon (determined from the mineralogical data for the carbonate minerals calcite, dolomite, and aragonite) from the total carbon concentration.

Statistical summaries for the chemical analyses of surface soils (0–5 cm), the soil A horizon, and the soil C horizon are given in tables 2–4, respectively.

Table 1. Lower limits of determination (LLD) for elements determined by inductively coupled plasma–mass spectrometry (ICP-MS) and inductively coupled plasma–atomic emission spectrometry (ICP-AES).

[wt. %, weight percent; mg/kg, milligrams per kilogram]

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

Table 2.   Statistical summary for chemical analyses of surface soil samples collected from a depth of 0 to 5 centimeters, conterminous United States.

[LLD, lower limit of determination; Min, minimum; Q1, 1st quartile; Q3, 3rd quartile; Max, maximum; MAD, median absolute deviation; wt. %, weight percent; mg/kg, milligrams per kilogram; <, less than; ND, not determined]

Element	Units	Number of samples	Number of samples below LLD	Min	Q1	Median	Mean	Q3	Max	MAD	Standard deviation
Ag	mg/kg	4,841	4,828	<1	<1	<1	<1	<1	7.7	ND	ND
Al	wt. %	4,841	0	0.02	3.20	4.67	4.59	6.00	15.3	2.06	2.12
As	mg/kg	4,841	56	<0.6	3.1	5.2	6.4	7.6	830	3.3	16.7
Ba	mg/kg	4,841	0	5	329	510	518	683	4,770	262	288
Be	mg/kg	4,841	97	<0.1	0.9	1.3	1.3	1.7	17.3	0.6	0.8
Bi	mg/kg	4,841	121	<0.04	0.11	0.16	0.34	0.23	694	0.09	10
Ca	wt. %	4,841	57	<0.01	0.31	0.76	1.59	1.71	32.8	0.82	2.66
Cd	mg/kg	4,841	1,054	<0.1	0.1	0.2	0.3	0.3	76.8	0.15	1.4
Ce	mg/kg	4,841	0	0.65	36.2	51.1	52.1	63.6	415	20.2	26.1
Co	mg/kg	4,841	24	<0.1	4.4	7.7	8.9	11.1	216	4.9	7.9
Cr	mg/kg	4,841	6	<1	18	30	36	41	4,120	18	89
Cs	mg/kg	4,841	3,954	<5	<5	<5	<5	<5	97	ND	ND
Cu	mg/kg	4,841	2	<0.5	8.8	14.4	17.9	20.9	996	8.7	22.1
Fe	wt. %	4,841	8	<0.01	1.28	1.95	2.14	2.66	13.3	1.02	1.39
Ga	mg/kg	4,841	0	0.10	7.45	11.0	11.1	14.7	45.1	5.4	5.4
Hg	mg/kg	4,841	367	<0.01	0.01	0.02	0.05	0.04	56.4	0.01	0.8
In	mg/kg	4,841	1,017	<0.02	0.02	0.03	0.04	0.05	4.54	0.01	0.08
K	wt. %	4,841	24	<0.01	0.99	1.49	1.46	1.88	5.44	0.65	0.74
La	mg/kg	4,841	5	<0.5	18.0	25.7	26.0	31.9	239	10.2	13.0
Li	mg/kg	4,841	18	<1	13	20	21	27	300	10.4	14
Mg	wt. %	4,841	50	<0.01	0.22	0.46	0.58	0.74	13.6	0.39	0.61
Mn	mg/kg	4,841	16	<5	290	492	612	791	7,780	353	529
Mo	mg/kg	4,841	11	<0.05	0.51	0.78	1.04	1.14	75.7	0.46	2.10
Na	wt. %	4,841	200	<0.01	0.29	0.69	0.81	1.10	6.41	0.61	0.68
Nb	mg/kg	4,841	2	<0.1	6.0	8.5	9.3	11.1	80.1	3.7	5.9
Ni	mg/kg	4,841	23	<0.5	7.8	13.5	17.7	19.8	1,890	8.9	45.2
P	mg/kg	4,841	34	<50	360	580	660	840	9,120	356	488
Pb	mg/kg	4,841	2	<0.5	13.5	18.1	25.8	23.9	12,400	7.4	185
Rb	mg/kg	4,841	14	<0.2	45.0	65.2	66.2	84.4	299	29.2	34.3
S	wt. %	4,841	436	<0.01	0.02	0.03	0.05	0.04	16.1	0.01	0.40
Sb	mg/kg	4,841	34	<0.05	0.37	0.57	0.83	0.80	482	0.31	7.33
Sc	mg/kg	4,841	20	<0.1	3.8	6.1	6.8	8.4	42.3	3.4	4.7
Se	mg/kg	4,841	2,154	<0.2	<0.2	0.2	0.3	0.4	6.9	0.1	0.3
Sn	mg/kg	4,841	9	<0.1	0.9	1.3	1.6	1.8	88.9	0.6	2.6
Sr	mg/kg	4,841	0	0.5	63.4	121	159	203	2,620	96.5	162
Te	mg/kg	4,841	4,684	<0.1	<0.1	<0.1	<0.1	<0.1	50.5	ND	ND
Th	mg/kg	4,841	6	<0.2	5.2	7.6	8.0	9.9	78.3	3.6	4.8
Ti	wt. %	4,841	2	<0.01	0.18	0.24	0.27	0.32	2.47	0.10	0.17
Tl	mg/kg	4,841	276	<0.1	0.3	0.4	0.4	0.5	8.8	0.1	0.3
U	mg/kg	4,841	6	<0.1	1.4	2.0	2.1	2.6	102	0.9	1.9
V	mg/kg	4,841	12	<1	33	53	60	74	530	31	43
W	mg/kg	4,841	72	<0.1	0.5	0.8	1.3	1.1	1,150	0.4	18
Y	mg/kg	4,841	0	0.2	9.8	14.4	14.8	18.4	191	6.4	8.6
Zn	mg/kg	4,841	5	<1	36	58	66	80	11,700	33	176

Table 3.   Statistical summary for chemical analyses of samples from the soil A horizon, conterminous United States.

[LLD, lower limit of determination; Min, minimum; Q1, 1st quartile; Q3, 3rd quartile; Max, maximum; MAD, median absolute deviation; wt. %, weight percent; C_Inorg, inorganic carbon (carbonate); C_Org, organic carbon; mg/kg, milligrams per kilogram; <, less than; ND, not determined]

Element	Units	Number of samples	Number of samples below LLD	Min	Q1	Median	Mean	Q3	Max	MAD	Standard deviation
Ag	mg/kg	4,813	4,799	<1	<1	<1	<1	<1	14	ND	ND
Al	wt. %	4,813	0	0.01	3.22	4.71	4.65	6.12	15.6	2.15	2.15
As	mg/kg	4,813	67	<0.6	3.1	5.2	6.6	7.8	1,110	3.4	19.6
Ba	mg/kg	4,813	0	6	331	513	522	686	4,850	261	289
Be	mg/kg	4,813	89	<0.1	0.9	1.3	1.4	1.7	22.1	0.6	0.8
Bi	mg/kg	4,813	118	<0.04	0.11	0.16	0.22	0.23	129	0.09	1.87
C_Total	wt. %	4,810	0	0.04	0.96	1.78	3.01	3.15	60.2	1.44	4.60
C_Inorg	wt. %	4,801	3,623	<0.2	<0.2	<0.2	0.3	<0.2	8.6	ND	ND
C_Org	wt. %	4,810	10	0	0.84	1.55	2.75	2.80	60.1	1.26	4.58
Ca	wt. %	4,813	57	<0.01	0.31	0.74	1.61	1.68	29.7	0.82	2.76
Cd	mg/kg	4,813	1,146	<0.1	0.1	0.2	0.3	0.3	46.6	0.1	0.8
Ce	mg/kg	4,813	2	<0.05	36.9	51.7	52.7	65.1	487	20.9	26.2
Co	mg/kg	4,813	34	<0.1	4.6	7.8	9.1	11.3	184	5.0	8.2
Cr	mg/kg	4,813	12	<1	18	31	37	42	3,850	18	89
Cs	mg/kg	4,813	3,942	<5	<5	<5	<5	<5	97	ND	ND
Cu	mg/kg	4,813	4	<0.5	9.1	14.8	19.9	21.8	5,090	9.2	75.5
Fe	wt. %	4,813	6	<0.01	1.30	1.99	2.19	2.75	13.9	1.08	1.46
Ga	mg/kg	4,813	0	0.08	7.54	11.2	11.2	15.0	40.8	5.5	5.3
Hg	mg/kg	4,813	386	<0.01	0.01	0.02	0.04	0.04	8.24	0.01	0.17
In	mg/kg	4,813	1,017	<0.02	0.02	0.03	0.04	0.05	4.61	0.01	0.07
K	wt. %	4,813	26	<0.01	0.98	1.50	1.46	1.90	5.10	0.65	0.74
La	mg/kg	4,813	4	<0.5	18.2	25.7	25.9	31.9	205	10.1	12.7
Li	mg/kg	4,813	13	<1	13	20	22	27	315	10	14
Mg	wt. %	4,813	41	<0.01	0.21	0.46	0.59	0.75	13.3	0.39	0.64
Mn	mg/kg	4,813	14	<5	289	498	622	797	6,850	365	542
Mo	mg/kg	4,813	7	<0.05	0.50	0.78	1.02	1.13	70.3	0.46	1.75
Na	wt. %	4,813	182	<0.01	0.28	0.69	0.81	1.10	6.60	0.61	0.68
Nb	mg/kg	4,813	1	<0.1	6.1	8.6	9.4	11.2	96.8	3.7	6.0
Ni	mg/kg	4,813	9	<0.5	7.9	13.8	18.5	20.0	2,310	8.9	54.4
P	mg/kg	4,813	42	<50	340	550	632	810	7,650	341	466
Pb	mg/kg	4,813	1	<0.5	13.2	17.8	22.2	23.2	2,200	7.3	46.6
Rb	mg/kg	4,813	14	<0.2	44.7	65.8	66.4	84.8	461	29.7	34.7
S	wt. %	4,813	583	<0.01	0.02	0.03	0.06	0.04	16.6	0.01	0.6
Sb	mg/kg	4,813	25	<0.05	0.37	0.57	0.84	0.80	630	0.33	9.1
Sc	mg/kg	4,813	10	<0.1	3.9	6.1	6.9	8.5	48.9	3.4	4.8
Se	mg/kg	4,813	2,116	<0.2	<0.2	0.2	0.3	0.4	8.3	0.1	0.3
Sn	mg/kg	4,813	6	<0.1	0.9	1.3	1.6	1.7	375	0.6	5.9
Sr	mg/kg	4,813	2	<0.5	64	122	163	204	7,080	98	196
Te	mg/kg	4,813	4,655	<0.1	<0.1	<0.1	<0.1	<0.1	9.6	ND	ND
Th	mg/kg	4,813	6	<0.2	5.3	7.7	8.1	10.0	84.1	3.6	4.7
Ti	wt. %	4,813	0	0.01	0.18	0.24	0.27	0.31	2.76	0.10	0.18
Tl	mg/kg	4,813	276	<0.1	0.3	0.4	0.4	0.5	11.5	0.1	0.3
U	mg/kg	4,813	6	<0.1	1.4	2.0	2.1	2.6	105	0.9	1.9
V	mg/kg	4,813	10	<1	33	54	61	76	524	31	44
W	mg/kg	4,813	61	<0.1	0.5	0.8	1.1	1.1	299	0.4	5.5
Y	mg/kg	4,813	0	0.2	10.0	14.6	15.0	18.7	254	6.4	8.8
Zn	mg/kg	4,813	6	<1	36	59	64	81	2,130	34	60

Table 4.   Statistical summary for chemical analyses of samples from the soil C horizon, conterminous United States.

[LLD, lower limit of determination; Min, minimum; Q1, 1st quartile; Q3, 3rd quartile; Max, maximum; MAD, median absolute deviation; C_Inorg, inorganic carbon (carbonate); C_Org, organic carbon; wt. %, weight percent; mg/kg, milligrams per kilogram; <, less than; ND, not determined]

Element	Units	Number of samples	Number of samples below LLD	Min	Q1	Median	Mean	Q3	Max	MAD	Standard deviation
Ag	mg/kg	4,780	4,769	<1	<1	<1	<1	<1	3.0	ND	ND
Al	wt. %	4,780	0	0.02	3.93	5.40	5.44	6.88	18.6	2.18	2.31
As	mg/kg	4,780	73	<0.6	3.4	5.7	7.0	8.4	397	3.7	9.7
Ba	mg/kg	4,780	0	5	343	506	542	701	9,360	265	380
Be	mg/kg	4,780	86	<0.1	1.0	1.4	1.5	1.9	31.6	0.6	0.9
Bi	mg/kg	4,780	238	<0.04	0.11	0.16	0.19	0.23	8.41	0.09	0.22
C_Total	wt. %	4,777	4	<0.01	0.22	0.62	1.32	1.65	43	0.71	2.17
C_Inorg	wt. %	4,773	2,903	<0.2	<0.2	<0.2	0.7	0.6	10.6	ND	ND
C_Org	wt. %	4,777	114	0	0.18	0.38	0.72	0.71	43	0.4	1.8
Ca	wt. %	4,780	183	<0.01	0.26	1.05	2.63	3.36	32.3	1.45	3.99
Cd	mg/kg	4,780	2,149	<0.1	<0.1	0.1	0.18	0.2	36.4	0.07	0.58
Ce	mg/kg	4,780	0	0.5	37.4	52.2	55.5	68.1	914	22.7	33.9
Co	mg/kg	4,780	24	<0.1	5.0	8.4	10.1	12.3	316	5.3	9.9
Cr	mg/kg	4,780	19	<1	17	30	39	45	4,620	21	107
Cs	mg/kg	4,780	3,470	<5	<5	<5	<5	5.0	144	ND	ND
Cu	mg/kg	4,780	14	<0.5	9.2	15.1	19.8	23.0	2,540	9.9	41.7
Fe	wt. %	4,780	6	<0.01	1.57	2.34	2.62	3.28	15.3	1.25	1.67
Ga	mg/kg	4,780	0	0.13	9.28	13.1	13.3	16.9	50.4	5.63	5.86
Hg	mg/kg	4,780	752	<0.01	0.01	0.02	0.028	0.03	1.75	0.01	0.053
In	mg/kg	4,780	687	<0.02	0.03	0.04	0.04	0.05	4.39	0.01	0.07
K	wt. %	4,780	34	<0.01	1.04	1.53	1.51	1.96	5.67	0.68	0.76
La	mg/kg	4,780	4	<0.5	18.7	26.1	27.3	33.8	283	11.3	15.6
Li	mg/kg	4,780	7	<1	16	24	27	33	280	12	18
Mg	wt. %	4,780	41	<0.01	0.32	0.61	0.82	1.06	16.8	0.52	0.84
Mn	mg/kg	4,780	23	<5	202	392	504	646	12,000	323	564
Mo	mg/kg	4,780	11	<0.05	0.51	0.83	1.20	1.27	94.7	0.53	2.64
Na	wt. %	4,780	214	<0.01	0.27	0.70	0.82	1.14	5.54	0.64	0.69
Nb	mg/kg	4,780	2	<0.1	6.3	9.2	10.2	12.3	289	4.4	8.2
Ni	mg/kg	4,780	8	<0.5	9.8	16.7	22.6	24.9	2,870	10.8	66.8
P	mg/kg	4,780	119	<50	220	430	508	640	27,400	311	626
Pb	mg/kg	4,780	4	<0.5	11.1	14.9	16.6	19.2	681	6.1	18.5
Rb	mg/kg	4,780	3	<0.2	47.0	67.2	69.8	89.5	267	31.7	35.5
S	wt. %	4,780	1,061	<0.01	0.01	0.02	0.11	0.03	16.2	0.01	0.75
Sb	mg/kg	4,780	83	<0.05	0.36	0.58	0.70	0.82	40.6	0.34	1.08
Sc	mg/kg	4,780	9	<0.1	4.8	7.3	8.4	10.4	70.8	4.0	5.8
Se	mg/kg	4,780	2,718	<0.2	<0.2	<0.2	0.28	0.3	7.5	ND	0.4
Sn	mg/kg	4,780	18	<0.1	1.0	1.4	1.5	1.8	30.9	0.6	1.0
Sr	mg/kg	4,780	1	<0.5	71.2	142	189	232	10,900	115	278
Te	mg/kg	4,780	4,567	<0.1	<0.1	<0.1	<0.1	<0.1	6.1	ND	ND
Th	mg/kg	4,780	3	<0.2	5.8	8.3	8.8	11.0	55.9	3.9	4.8
Ti	wt. %	4,780	2	<0.01	0.18	0.25	0.29	0.34	3.42	0.12	0.20
Tl	mg/kg	4,780	204	<0.1	0.3	0.5	0.5	0.6	4.3	0.15	0.2
U	mg/kg	4,780	6	<0.1	1.5	2.1	2.3	2.8	63	1.0	1.9
V	mg/kg	4,780	5	<1	41	64	73	92	1,080	37	53
W	mg/kg	4,780	140	<0.1	0.6	0.9	1.24	1.3	199	0.44	3.7
Y	mg/kg	4,780	0	0.2	10.7	15.6	16.5	20.3	288	7.0	11.2
Zn	mg/kg	4,780	17	<1	33	54	58	76	653	31	40

Quality Assurance and Quality Control

The International Organization for Standardization (ISO) defines quality assurance (QA) as all those planned and systematic actions necessary to provide adequate confidence that an entity will fulfill requirements for quality (Hoyle, 2009). The various components of the QA plan include standard operating procedures, instrument logs, training records, data acceptance and rejection criteria, and laboratory audits. The ISO defines quality control (QC) as the operational techniques and activities that are used to fulfill requirements for quality (Hoyle, 2009). The QC components involve quantitative assessments of the accuracy of the analytical data produced.

In a major geochemical mapping project such as this, the quality of chemical analyses is of utmost importance. Reimann and others (2008) recommend the following five QC procedures:

- Collection and analysis of field duplicates;
- Randomization of samples prior to analysis;
- Insertion of international reference materials;
- Insertion of project standards; and
- Insertion of analytical duplicates of project samples.

In this project, field duplicates were not collected. This step was done during the pilot studies (Smith and others, 2009) and reported on by Garrett (2009). Based on the results of the pilot studies, it was felt that the additional collection of field duplicates during the national-scale study would not add significantly to the QC analysis and, therefore, was not worth the added expense. The remaining four QC procedures were carried out fully. Note that for the purposes of the present quality assessment, data reported as below the LLD were given a value of one-half the LLD, as per standard practice (Reimann and others, 2008).

Trueness and Precision

The terms trueness and precision are used to describe the accuracy of an analytical method. Trueness is defined as the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value (International Organization for Standardization, 1994). Trueness is generally measured in terms of bias, which is defined as the difference between the expectation of the test results and an accepted reference value (International Organization for Standardization, 1994).

To estimate trueness, one or more reference materials (RMs) are analyzed with the project samples. In this project, trueness estimation was done on three separate levels. The USGS contract laboratory monitored QC by analyzing a RM with every batch of 48 samples. The RM most often used was

a syenite rock standard (SY-3) developed by the Canadian Centre for Mineral and Energy Technology (Govindaraju, 1989). Shewhart control charts (Taylor, 1987) were generated for the RM analyses and were reviewed with every report as part of the internal quality audits. At the second tier, the USGS QC officer inserted at least one RM between every batch of 20–30 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) (National Institute of Standards and Technology, 2003). SoNE-1 is a non-certified USGS in-house RM prepared as an internal project standard specifically for the North American Soil Geochemical Landscapes Project. It was collected from the Sharpsburg soil series (Natural Resources Conservation Service, 2008) in Lancaster County, Nebraska, and was analyzed 153 times in one batch by the USGS contract laboratory to establish consensus values for all of the elements analyzed except Hg, which was analyzed only 5 times to determine a consensus value. The USGS principal investigator for the current study (D.B. Smith) initiated the final QC tier, which included the insertion of two blind SoNE-1 RMs in each batch of 20–30 samples. In addition, internal project standards from two other continental-scale geochemical surveys—the National Geochemical Survey of Australia (Caritat and Cooper, 2011) and EuroGeoSurveys' Geochemical Mapping of Agricultural and Grazing Land Soils (GEMAS) Project in Europe (Reimann and others, 2012)—were analyzed to determine comparability of the U.S. data with these surveys.

Tables 5 and 6 show the results for the analyses of NIST SRM 2709 and SoNE-1, respectively. Bias was calculated in each table by dividing the mean concentration of the analyzed RMs by the certified or recommended value (SRM 2709) or the consensus value (SoNE-1) for the element in question and expressing the result as a percentage, where 100 percent means a perfect match (no bias). These two tables demonstrate that bias is specific to the RM being used. Analyzing the same element in two different RMs will generally yield a different bias.

Precision is defined as the closeness of agreement between independent test results obtained under stipulated conditions (International Organization for Standardization, 1994). It depends only on the distribution of random errors and has no relationship to the "true" concentration of a chemical element. This parameter is generally assessed by repeated analyses of a RM (tables 5 and 6) or by replicate analyses of real project samples (table 7). In regard to the latter method, the USGS contract laboratory inserted duplicate samples at random intervals at an approximate rate of 1 duplicate sample per 80 regular samples. Precision is usually expressed in terms of imprecision. It is estimated through the standard deviation of the test results, usually adjusted for the mean, and expressed as the coefficient of variation (CV, also known as the relative standard deviation) in percent. A low CV indicates a high precision. In tables 5 and 6, the CV for an element is calculated

Table 5.   Results from chemical analyses of National Institute of Standards and Technology (NIST) Certified Reference Material 2709 compared with certified or recommended values for determined elements (arranged by increasing coefficient of variation).

[CV, coefficient of variation; %, percent; wt. %, weight percent; mg/kg, milligrams per kilogram; ICP-AES, inductively coupled plasma–atomic emission spectrometry; ICP-MS, inductively coupled plasma–mass spectrometry; CVAAS, cold-vapor atomic absorption spectrometry; HG-AAS, hydride-generation atomic absorption spectrometry; ND, not determined; NIST 2709 results in square brackets are not certified but are only recommended concentrations]

Element	Units	NIST 2709 Certified or recommended value	NIST 2709 Measured values, this study					
			Number of analyses	Method	Mean	Standard deviation	CV %	Bias %
Hg	mg/kg	1.40 ± 0.08	124	CVAAS	1.38	0.05	3.6	98.6
Fe	wt. %	3.50 ± 0.11	125	ICP-AES	3.36	0.13	3.9	96
Mn	mg/kg	538 ± 17	125	ICP-AES	518	21	4.1	96.3
Mg	wt. %	1.51 ± 0.05	125	ICP-AES	1.45	0.06	4.1	96
Al	wt. %	7.50 ± 0.06	125	ICP-AES	7.05	0.30	4.3	94
Ca	wt. %	1.89 ± 0.05	125	ICP-AES	1.84	0.08	4.3	97.4
Sr	mg/kg	231 ± 2	125	ICP-AES	219	10	4.6	94.8
Ba	mg/kg	968 ± 40	125	ICP-AES	928	44	4.7	95.9
P	mg/kg	620 ± 50	125	ICP-AES	622	32	5.1	100.3
Na	wt. %	1.16 ± 0.03	125	ICP-AES	1.12	0.06	5.4	96.6
Ni	mg/kg	88 ± 5	125	ICP-AES	72.9	4.0	5.5	82.8
Li	mg/kg	ND	125	ICP-AES	54	3	5.6	ND
Th	mg/kg	[11]	125	ICP-MS	10.6	0.60	5.7	96.4
K	wt. %	2.03 ± 0.06	125	ICP-AES	1.92	0.12	6.3	94.6
Ti	wt. %	0.342 ± 0.024	125	ICP-AES	0.30	0.02	6.7	87.7
Rb	mg/kg	[96]	125	ICP-MS	94.6	6.4	6.8	98.5
Y	mg/kg	[18]	125	ICP-MS	14.7	1.0	6.8	81.7
Zn	mg/kg	106 ± 3	125	ICP-AES	102	7	6.9	96.2
La	mg/kg	[23]	125	ICP-MS	21.8	1.5	6.9	94.8
U	mg/kg	[3]	125	ICP-MS	2.9	0.2	6.9	96.7
Ce	mg/kg	[42]	125	ICP-MS	43.2	3.0	6.9	103
Se	mg/kg	1.57 ± 0.08	125	HG-AAS	1.4	0.1	7.1	89.2
S	wt. %	0.089 ± 0.002	125	ICP-AES	0.096	0.007	7.3	108
Co	mg/kg	13.4 ± 0.7	125	ICP-MS	13.4	1.0	7.5	100
Pb	mg/kg	18.9 ± 0.5	125	ICP-MS	18.5	1.4	7.6	97.9
Cu	mg/kg	34.6 ± 0.7	125	ICP-AES	33.0	2.5	7.6	95.4
Ga	mg/kg	[14]	125	ICP-MS	16.2	1.4	8.6	116
V	mg/kg	112 ± 5	125	ICP-AES	112	10	8.9	100
Sc	mg/kg	[12]	125	ICP-MS	11.9	1.1	9.2	99.2
Nb	mg/kg	ND	125	ICP-MS	8.5	0.8	9.4	ND
Mo	mg/kg	[2.0]	125	ICP-MS	2.10	0.2	9.5	105
Tl	mg/kg	0.74 ± 0.05	125	ICP-MS	0.6	0.06	10.0	81.1
In	mg/kg	ND	125	ICP-MS	0.05	0.005	10.0	ND
Cd	mg/kg	0.38 ± 0.01	125	ICP-MS	0.4	0.04	10.0	105
Cs	mg/kg	[5.3]	121	ICP-MS	5.9	0.6	10.2	111
Bi	mg/kg	ND	125	ICP-MS	0.29	0.03	10.3	ND
Cr	mg/kg	130 ± 4	125	ICP-AES	95	10	10.5	73.1
W	mg/kg	[2]	125	ICP-MS	1.9	0.2	10.5	95
Sb	mg/kg	7.9 ± 0.6	125	ICP-MS	6.61	0.77	11.6	83.7
C_Total	wt. %	[1.2]	62	Combustion	1.22	0.15	12.3	102
As	mg/kg	17.7 ± 0.8	124	HG-AAS	18.6	2.3	12.4	105
Sn	mg/kg	ND	125	ICP-MS	1.6	0.2	12.5	ND
Be	mg/kg	ND	125	ICP-MS	3.8	1	26.3	ND
Ag	mg/kg	0.41 ± 0.03	125	ICP-MS	ND	ND	ND	ND
Te	mg/kg	ND	125	ICP-MS	ND	ND	ND	ND

Table 6.   Results from chemical analyses of the U.S. Geological Survey internal project standard SoNE-1 compared with consensus values for determined elements (arranged by increasing coefficient of variation).

[CV, coefficient of variation; %, percent; wt. %, weight percent; mg/kg, milligrams per kilogram; ICP-AES, inductively coupled plasma–atomic emission spectrometry; ICP-MS, inductively coupled plasma–mass spectrometry; CVAAS, cold-vapor atomic absorption spectrometry; HG-AAS, hydride-generation atomic absorption spectrometry; WDXRF, wavelength-dispersive X-ray fluorescence spectrometry; <, less than; ND, not determined]

Element	Units	SoNE-1 Consensus values				SoNE-1 Measured values				CV %	Bias %
		Number of analyses	Method	Mean	Standard deviation	Number of analyses	Method	Mean	Standard deviation		
C_Total	wt. %	153	Combustion	1.94	0.18	686	Combustion	1.83	0.04	2.2	94.3
Mn	mg/kg	153	ICP-AES	639	59	1,479	ICP-AES	597	25	4.2	93.4
Al	wt. %	153	WDXRF	6.44	0.22	1,479	ICP-AES	6.10	0.27	4.4	94.7
Fe	wt. %	153	WDXRF	2.92	0.02	1,479	ICP-AES	2.80	0.13	4.6	95.9
Ba	mg/kg	153	ICP-AES	768	58	1,479	ICP-AES	737	35	4.7	96.0
Ca	wt. %	153	WDXRF	0.65	0.02	1,479	ICP-AES	0.62	0.03	4.8	95.4
Mg	wt. %	153	WDXRF	0.64	0.03	1,479	ICP-AES	0.61	0.03	4.9	95.3
Na	wt. %	153	WDXRF	0.75	0.06	1,479	ICP-AES	0.73	0.04	5.5	97.3
Sr	mg/kg	153	ICP-AES	130	6	1,479	ICP-AES	128	7	5.5	98.5
P	mg/kg	153	ICP-AES	510	100	1,479	ICP-AES	522	33	6.3	102
Hg	mg/kg	5	CVAAS	0.03	0.00	1,479	CVAA	0.03	0.002	6.7	100
Ni	mg/kg	153	ICP-MS	30.9	1.8	1,479	ICP-AES	27.5	1.9	6.9	89.0
Y	mg/kg	153	ICP-MS	26.4	5.2	1,479	ICP-MS	21.3	1.5	7.0	80.7
K	wt. %	153	WDXRF	1.86	0.06	1,479	ICP-AES	1.83	0.13	7.1	98.4
La	mg/kg	153	ICP-MS	38.2	1.6	1,479	ICP-MS	36.7	2.6	7.1	96.1
Li	mg/kg	153	ICP-MS	29.5	2.1	1,479	ICP-AES	28	2	7.1	94.9
Zn	mg/kg	153	ICP-MS	87	6	1,479	ICP-AES	81	6	7.4	93.1
As	mg/kg	153	HG-AAS	10.8	0.9	1,479	HG-AAS	10.5	0.8	7.6	97.2
Th	mg/kg	153	ICP-MS	12.8	0.14	1,479	ICP-MS	11.8	0.9	7.6	92.2
Ce	mg/kg	153	ICP-MS	74	4	1,479	ICP-MS	73.2	5.6	7.7	98.9
Sc	mg/kg	153	ICP-MS	9.3	1.1	1,479	ICP-MS	8.7	0.7	8.0	93.5
Co	mg/kg	153	ICP-MS	12.5	1.1	1,479	ICP-MS	12.3	1.0	8.1	98.4
Tl	mg/kg	153	ICP-MS	0.75	0.05	1,479	ICP-MS	0.7	0.06	8.6	93.3
Rb	mg/kg	153	ICP-MS	94.8	8.9	1,479	ICP-MS	90.3	8.2	9.1	95.2
V	mg/kg	153	ICP-MS	107	9	1,479	ICP-AES	99	9	9.1	92.5
Ti	wt. %	153	WDXRF	0.38	0.04	1,479	ICP-AES	0.30	0.028	9.3	78.9
Ga	mg/kg	153	ICP-MS	15	1.5	1,479	ICP-MS	14.6	1.4	9.6	97.3
Cr	mg/kg	153	ICP-MS	67	8	1,479	ICP-AES	51	5	9.8	76.1
In	mg/kg	153	ICP-MS	ND	ND	1,479	ICP-MS	0.05	0.005	10.0	ND
U	mg/kg	153	ICP-MS	3.23	0.52	1,479	ICP-MS	3.0	0.3	10.0	92.9
Cu	mg/kg	153	ICP-MS	24	2	1,479	ICP-AES	22.1	2.3	10.4	92.1
Mo	mg/kg	153	ICP-MS	1.28	0.02	1,479	ICP-MS	1.20	0.14	11.7	93.8
Be	mg/kg	153	ICP-MS	1.9	0.13	1,479	ICP-MS	1.7	0.2	11.8	89.5
Sb	mg/kg	153	ICP-MS	1.4	0.2	1,479	ICP-MS	1.35	0.16	11.9	96.4
Se	mg/kg	153	ND	ND	ND	1,479	HG-AAS	0.5	0.06	12.0	ND
Bi	mg/kg	153	ND	ND	ND	1,479	ICP-MS	0.28	0.035	12.5	ND
Cd	mg/kg	153	ND	ND	ND	1,479	ICP-MS	0.4	0.05	12.5	ND
Nb	mg/kg	153	ICP-MS	14.2	2.9	1,479	ICP-MS	12.2	1.6	13.1	85.9
S	wt. %	153	ND	ND	ND	1,479	ICP-AES	0.03	0.004	13.3	ND
Pb	mg/kg	153	ICP-MS	25.6	1.6	1,479	ICP-MS	22.4	3.6	16.1	87.5
Sn	mg/kg	153	ICP-MS	2.2	0.8	1,479	ICP-MS	1.7	0.3	17.6	85.0
W	mg/kg	153	ND	ND	ND	1,479	ICP-MS	1.2	0.25	20.8	ND
Ag	mg/kg	153	ND	ND	ND	1,479	ICP-MS	ND	ND	ND	ND
Cs	mg/kg	153	ICP-MS	5	0.3	1,479	ICP-MS	ND	ND	ND	ND
Te	mg/kg	153	ND	ND	ND	1,479	ICP-MS	ND	ND	ND	ND

Table 7. Precision expressed as coefficient of variation (CV) calculated from analytical duplicates for determined elements (arranged by increasing CV, or decreasing precision).

[LLD, lower limit of determination; Min, minimum; Max, maximum; %, percent; wt. %, weight percent; mg/kg, milligrams per kilogram; <, less than; ND, not determined]

Element	Units	Number of duplicate pairs	Number of values below LLD	Min	Max	Mean	Standard deviation for duplicates	CV %
Total C	wt. %	105	0	0.05	28.2	2.31	0.1	2.9
Al	wt. %	176	0	0.10	13.6	5.38	0.18	3.3
Fe	wt. %	176	0	0.04	10.8	2.50	0.08	3.3
Hg	mg/kg	176	19	<0.02	1.48	0.05	0.002	3.4
Li	mg/kg	176	2	<1	190	26.1	1.0	4.0
Na	wt. %	176	6	<0.01	2.83	0.85	0.04	4.3
Y	mg/kg	176	0	0.3	70.9	17.2	0.75	4.3
Mg	wt. %	176	2	<0.01	7.37	0.76	0.033	4.3
Mn	mg/kg	176	0	5	3,280	591	27.7	4.7
P	mg/kg	176	4	<50	5,250	618	30.2	4.9
Ca	wt. %	176	3	<0.01	19.2	1.98	0.10	5.1
K	wt. %	176	0	0.04	3.81	1.58	0.082	5.2
La	mg/kg	176	0	1.6	104	29.0	1.5	5.3
Ce	mg/kg	176	0	3.17	208	57.3	3.08	5.4
Ga	mg/kg	176	0	0.3	30.2	13.2	0.73	5.5
Pb	mg/kg	176	0	0.9	123	19.0	1.10	5.8
Ti	wt. %	176	0	0.03	1.70	0.30	0.019	6.2
Rb	mg/kg	176	0	1.8	189	74.2	4.66	6.3
Ni	mg/kg	176	1	<0.5	176	21.7	1.45	6.7
Ba	mg/kg	176	0	23	1,300	571	38	6.7
Sc	mg/kg	176	0	0.1	29.9	8.2	0.56	6.9
Co	mg/kg	176	2	<0.1	41.9	10.4	0.75	7.2
Sb	mg/kg	176	1	<0.05	6.41	0.85	0.06	7.4
Tl	mg/kg	176	16	<0.1	1.7	0.49	0.037	7.5
As	mg/kg	171	4	<0.6	33.2	7.0	0.5	7.6
U	mg/kg	176	0	0.1	26.4	2.50	0.21	8.2
Cr	mg/kg	176	0	1	239	40	3.3	8.2
Cu	mg/kg	176	2	<0.5	237	22	1.8	8.3
Nb	mg/kg	176	0	0.7	61.7	10.7	0.90	8.4
Th	mg/kg	176	0	0.5	46.5	9.2	0.81	8.8
Mo	mg/kg	176	0	0.09	20.7	1.14	0.10	8.9
In	mg/kg	176	40	<0.02	0.13	0.042	0.004	9.1
Sr	mg/kg	176	0	3.5	1,640	192	18.0	9.3
Be	mg/kg	176	0	0.1	9.7	1.6	0.15	9.4
Bi	mg/kg	176	0	0.04	2.01	0.21	0.02	9.4
Se	mg/kg	174	143	0.2	2.3	0.39	0.037	9.5
Cd	mg/kg	176	68	<0.1	1.4	0.27	0.027	10.1
S	wt. %	176	0	0.010	5.0	0.092	0.009	10.2
Cs	mg/kg	176	251	<5	46	5.3	0.54	10.2
Zn	mg/kg	176	2	<1	239	64.4	7.89	12.2
Sn	mg/kg	176	0	0.1	5.4	1.5	0.19	12.3
V	mg/kg	176	0	1	405	74.1	9.15	12.3
W	mg/kg	176	4	<0.1	16.9	1.2	0.22	18.1
Te	mg/kg	176	340	<0.1	0.6	<0.1	ND	ND
Ag	mg/kg	176	352	<1	<1	<1	ND	ND

by dividing the standard deviation by the mean and expressing this value as a percentage. In table 7, the CV for an element is calculated by first determining the squared difference for each duplicate pair of samples. The sum of these values is then divided by the number of samples. The square root of this value is the standard deviation. The CV is then calculated by dividing the standard deviation by the overall mean of the samples. It is important to note that these duplicate analyses do not represent just repeat instrumental determinations on previously prepared sample extracts. Such determinations give a false sense of high precision because the natural inhomogeneity of the sample material is not considered. The analytical duplicates in this study involve separate splits of the same sample that undergo repeated execution of the entire method from the point of time at which the samples reach the laboratory (sample digestion followed by chemical analysis).

In general, no serious quality problems were detected. No single batch of samples was found where the RMs inserted by the QC officer and the principal investigator both deviated beyond normal variation. As expected, elements known to occur in minerals that present dissolution problems for the four-acid digestion showed the largest negative deviation (bias) from the accepted true value. For example, Ti had a bias of 78.9 percent for SoNE-1, and Cr had a bias of 73.1 percent for SRM 2709 and 76.1 percent for SoNE-1.

Mineralogical Analysis

All A-horizon and C-horizon samples were analyzed by X-ray diffraction (XRD), and the percentages of major mineral phases were calculated using a Rietveld refinement method. Statistical summaries of the results are shown in tables 8 and 9. Splits of the <2-mm fraction were used for analysis. Zinc oxide (ZnO, 10 weight percent) was added to each sample as an internal standard, which allows calculation of the amorphous component (portion of sample that is not quantified by the diffraction technique). The sample-ZnO mixture was ground for 3 minutes in isopropyl alcohol using a micronizing mill and agate beads. Dried samples were disaggregated by passing through a 400-μm sieve and lightly pressed into back-loaded sample mounts. Samples were analyzed on a PANalytical X'Pert PRO Materials Research Diffractometer using Cu Kα radiation to collect digital data continuously from 3° to 70° 2θ (scan speed = 0.0567° 2θ per second). PANalytical HighScore Plus software version 2.2a was used for pattern processing, mineral phase identification, and Rietveld quantitative mineral analysis. Rietveld refinements simultaneously adjust the percentage of each identified phase to achieve the best least squares fit between the observed

Table 8. Statistical summary for mineralogical analyses of samples from the soil A horizon, conterminous United States.

[Min, minimum; Q1, 1st quartile; Q3, 3rd quartile; Max, maximum; wt. %, weight percent; K, potassium; Å, angstrom; <0.2, less than the detection limit. Values under the detection limit of 0.2% were replaced by 0.1% to calculate quartiles, median, and mean]

Mineral component	Units	Number of samples	Number of samples below detection	Min	Q1	Median	Mean	Q3	Max
Quartz	wt. %	4,802	11	<0.2	33.8	48.5	49.9	65.5	100.0
Total K feldspar	wt. %	4,802	686	<0.2	1.4	4.7	5.6	8.0	41.9
Total plagioclase	wt. %	4,802	682	<0.2	3.5	8.6	11.7	16.2	70.5
Total feldspar	wt. %	4,802	298	<0.2	6.4	14.7	17.3	24.1	79.6
Total 14 Å clays	wt. %	4,802	3,042	<0.2	<0.2	<0.2	1.3	1.5	28.0
Total 10 Å clays	wt. %	4,802	563	<0.2	3.6	7.1	7.4	10.7	45.8
Kaolinite	wt. %	4,802	3,529	<0.2	<0.2	<0.2	1.3	0.6	43.7
Total clays	wt. %	4,802	351	<0.2	4.7	9.1	9.9	13.6	68.9
Gibbsite	wt. %	4,802	4,752	<0.2	<0.2	<0.2	<0.2	<0.2	12.9
Calcite	wt. %	4,802	3,692	<0.2	<0.2	<0.2	1.7	<0.2	69.8
Dolomite	wt. %	4,802	4,317	<0.2	<0.2	<0.2	0.5	<0.2	57.2
Aragonite	wt. %	4,802	4,669	<0.2	<0.2	<0.2	<0.2	<0.2	41.9
Total carbonates	wt. %	4,802	3,475	<0.2	<0.2	<0.2	2.2	0.4	71.5
Analcime	wt. %	4,802	4,743	<0.2	<0.2	<0.2	<0.2	<0.2	9.6
Heulandite	wt. %	4,802	4,746	<0.2	<0.2	<0.2	<0.2	<0.2	29.3
Total zeolites	wt. %	4,802	4,687	<0.2	<0.2	<0.2	<0.2	<0.2	29.3
Gypsum	wt. %	4,802	4,759	<0.2	<0.2	<0.2	0.2	<0.2	84.7
Talc	wt. %	4,802	4,776	<0.2	<0.2	<0.2	<0.2	<0.2	20.0
Hornblende	wt. %	4,802	4,034	<0.2	<0.2	<0.2	0.4	<0.2	33.8
Serpentine	wt. %	4,802	4,782	<0.2	<0.2	<0.2	<0.2	<0.2	17.9
Hematite	wt. %	4,802	4,534	<0.2	<0.2	<0.2	<0.2	<0.2	12.8
Goethite	wt. %	4,802	4,736	<0.2	<0.2	<0.2	<0.2	<0.2	15.0
Pyroxene	wt. %	4,802	4,655	<0.2	<0.2	<0.2	0.4	<0.2	34.4
Pyrite	wt. %	4,802	4,796	<0.2	<0.2	<0.2	<0.2	<0.2	0.6
Other	wt. %	4,802	4,767	<0.2	<0.2	<0.2	<0.2	<0.2	24.9
Amorphous	wt. %	4,802	274	<0.2	10.8	19.1	19.7	26.9	90.4

diffractogram and the experimental diffractogram calculated as the combined contributions of each individual phase. The refinements include calculations that correct for preferred orientation of phyllosilicate minerals and account for variations in peak shape.

Evaluation of Method

Quantification of soil mineralogy by X-ray diffraction is a difficult analytical task largely because of the highly weathered nature of many of the phases within the samples. Reliable quantification of soil mineralogy must incorporate into the calculation not only all crystalline phases, but all poorly crystalline clay phases and amorphous phases as well. This is especially challenging for a reconnaissance study such as this current analysis of nearly 10,000 samples, which had to be completed within a limited time frame. Given these constraints, which required a fast and efficient technique utilizing just a single XRD analysis for each sample, we chose full-pattern calculation by Rietveld refinement as the most appropriate method to achieve this task. However, limitations inherent to this method restrict the reliability of the analysis of clay phases, disordered clay phases, and amorphous components.

Quality Control Protocols

Evaluation of the reliability of this method was done by analyzing standard mixtures of pure mineral phases prepared in-house and statistically evaluating the data. Standard ST1001 contained quartz and ZnO. Standard ST1003 contained quartz, ZnO, orthoclase, plagioclase (albite), illite, and calcite. Each of these standards was included in each batch run of 45 unknowns to evaluate for instrumental drift. A series of additional standard mineral mixtures prepared in-house that contained from 2 to 6 phases of common rock-forming minerals were analyzed separately from 5 to 10 times to evaluate dolomite, chlorite, muscovite, and amorphous content. Rock standard USGS G-2 (Flanagan, 1969, 1976) was also analyzed to test the method, even though it is a crystalline rock matrix.

The overall average accuracy reported in terms of absolute difference between actual and calculated percentages for all phases and standards included in this study is 2 percent. Precision, reported in absolute terms of average standard deviation, yields an average analytical uncertainty (1σ) of ±0.4 percent for ST1001, ±0.5 percent for ST1003, and ±1 percent overall for other prepared mixtures. The results for ST1001 and ST1003 are shown in table 10.

Table 9. Statistical summary for mineralogical analyses of samples from the soil C horizon, conterminous United States.

[Min, minimum; Q1, 1st quartile; Q3, 3rd quartile; Max, maximum; wt. %, weight percent; K, potassium; Å, angstrom; <0.2, less than the detection limit. Values under the detection limit of 0.2% were replaced by 0.1% to calculate quartiles, median, and mean]

Mineral component	Units	Number of samples	Number of samples below detection	Min	Q1	Median	Mean	Q3	Max
Quartz	wt. %	4,773	9	<0.2	30.5	44.0	46.2	62.0	99.4
Total K feldspar	wt. %	4,773	705	<0.2	1.1	3.7	5.0	6.6	45.2
Total plagioclase	wt. %	4,773	830	<0.2	2.4	7.9	11.1	15.5	67.0
Total feldspar	wt. %	4,773	390	<0.2	4.7	12.7	16.0	22.4	80.1
Total 14 Å clays	wt. %	4,773	2,237	<0.2	<0.2	0.7	2.6	4.2	44.1
Total 10 Å clays	wt. %	4,773	436	<0.2	4.2	7.3	8.2	10.9	65.1
Kaolinite	wt. %	4,773	2,770	<0.2	<0.2	<0.2	2.8	2.5	79.9
Total clays	wt. %	4,773	162	<0.2	7.1	11.7	13.5	17.3	86.3
Gibbsite	wt. %	4,773	4,651	<0.2	<0.2	<0.2	0.3	<0.2	30.4
Calcite	wt. %	4,773	2,941	<0.2	<0.2	<0.2	3.5	3.0	84.1
Dolomite	wt. %	4,773	3,902	<0.2	<0.2	<0.2	1.5	<0.2	81.4
Aragonite	wt. %	4,773	4,734	<0.2	<0.2	<0.2	<0.2	<0.2	65.3
Total carbonates	wt. %	4,773	2,796	<0.2	<0.2	<0.2	5.0	5.0	84.1
Analcime	wt. %	4,773	4,758	<0.2	<0.2	<0.2	<0.2	<0.2	9.2
Heulandite	wt. %	4,773	4,717	<0.2	<0.2	<0.2	<0.2	<0.2	38.0
Total zeolites	wt. %	4,773	4,702	<0.2	<0.2	<0.2	<0.2	<0.2	38.0
Gypsum	wt. %	4,773	4,564	<0.2	<0.2	<0.2	0.5	<0.2	96.5
Talc	wt. %	4,773	4,747	<0.2	<0.2	<0.2	<0.2	<0.2	16.4
Hornblende	wt. %	4,773	3,933	<0.2	<0.2	<0.2	0.5	<0.2	62.6
Serpentine	wt. %	4,773	4,764	<0.2	<0.2	<0.2	<0.2	<0.2	26.7
Hematite	wt. %	4,773	4,457	<0.2	<0.2	<0.2	<0.2	<0.2	13.5
Goethite	wt. %	4,773	4,589	<0.2	<0.2	<0.2	<0.2	<0.2	14.1
Pyroxene	wt. %	4,773	4,626	<0.2	<0.2	<0.2	0.4	<0.2	33.6
Pyrite	wt. %	4,773	4,770	<0.2	<0.2	<0.2	<0.2	<0.2	0.4
Other	wt. %	4,773	4,703	<0.2	<0.2	<0.2	0.2	<0.2	35.9
Amorphous	wt. %	4,773	345	<0.2	8.4	17.5	17.8	25.3	95.2

Table 10. Summary statistics for two standard samples used throughout mineralogical analyses (ST1001 and ST1003). Each was prepared by precise weighing of pure mineral standards.

[ZnO, zinc oxide]

| | ST1001 | | ST1003 | | | | | |
	Quartz	ZnO	Quartz	Orthoclase	Plagioclase	Illite	Calcite	ZnO
Actual	90	10	45.5	18.0	17.5	5.0	5.0	9.0
Mean	90.04	9.91	47.36	20.0	16.7	2.7	4.7	8.8
Median	90.13	9.84	47.21	20.0	16.7	2.6	4.8	8.4
Standard deviation	0.42	0.37	0.82	0.5	0.3	0.5	0.4	0.7
Minimum	88.46	8.83	45.74	18.7	15.56	0.22	3.83	7.47
Maximum	91.17	10.85	51.68	22	18.33	4.58	5.68	9.93
Count	276	276	220	220	220	220	220	220

Two duplicate samples were analyzed within each standard analytical run of 45 samples for a total of 668 duplicate pairs. A duplicate pair consists of using the same prepared powdered sample to make two X-ray mounts, measuring two diffractograms, and making two calculations of mineral percentages. Correlation coefficients for major mineral phases are shown in table 11.

Minimum Detection Limit

Peaks determined to be significant (above statistical noise) were used for phase identification, and these phases were subsequently incorporated into the Rietveld calculations. A minimum 2nd derivative method was used to determine peak significance. Rietveld calculations showed diminished correlations between duplicate analyses below specific weight percentages for each phase. Statistical evaluation of in-house standards showed that standard deviations taken as a percentage of the mean for all phases increase significantly as concentrations decrease to <1 percent, indicating that counting error is probably the primary limiting factor for the precision of this method. Setting a precise minimum detection limit (MDL) is difficult considering the complexity and variability of the mineral assemblages among samples and the variability

of peak overlap in the complex diffraction spectra of various soils. Under the best circumstances, the smallest diffraction peaks that are both automatically determined by the Rietveld program and visible on inspection of the diffractogram produce calculated concentrations as low as 0.2 percent. We consider this percentage as the absolute minimum detection limit. However, it is likely that some mineral phases were inadvertently not identified by the method in cases of severe peak overlap.

Minimum Precision Limit

Inspection of the data from duplicate analyses shows that the correlation between duplicate sets of analyses decreases and becomes insignificant at low concentrations. For major minerals, it is possible to approximate the minimum concentrations for which precision is adequate based on significance of correlations. Those minimum concentration values, here called the minimum precision limit (MPL), are shown in table 11. Although we report concentrations down to the MDL, users should be aware that concentration values below the MPL are poorly reproducible. For less common minerals, for which there are few duplicate pairs, we have not quantified a MPL concentration, but we suggest that values below a concentration of 1 percent are poorly reproducible.

Clay Minerals

The reliability of quantitative clay determinations—including those of smectites, mixed-layer clays, and soil phyllosilicates—is diminished by the lack of well-defined structural data necessary for Rietveld refinement. Additional weathering-related complications include hydration effects, interstratification, stacking disorders, and lattice substitution, which contribute to peak width broadening and to peak shifting from published values. Evaluation of clay phases to the highest degree of reliability requires the preparation of additional specialized mounts for each unknown, which is beyond the scope of this reconnaissance effort. Further, the unknowns here were analyzed as randomly oriented powders, minimizing basal clay intensities.

Table 11. Pearson correlation coefficients for duplicate mineralogical analyses of A-horizon (329 pairs) and C-horizon (339 pairs) samples and the minimum precision limit (MPL) for each mineral.

[K, potassium; Å, angstrom. Minimum precision limit is defined as the minimum concentration for which precision is adequate]

Mineral	Correlation A	Correlation C	MPL (weight percent)
Quartz	0.999	0.998	1%
Calcite	0.998	0.998	0.5%
Dolomite	0.981	0.997	0.5%
K-feldspar	0.918	0.923	2%
Plagioclase	0.986	0.989	2%
10Å clay	0.897	0.835	3%
14–15 Å clay	0.809	0.886	2%
Kaolinite	0.913	0.968	2%
Hornblende	0.980	0.963	1%

Generally, all peaks that appeared in the range of about 14–15 angstroms (Å) were identified as either smectite, chlorite, or vermiculite and were included in the refinements. Given the uncertainty of the exact clay phase structures present in these highly weathered samples, each of the phases quantified within that range was reported in the same grouping (14–15 Å clays).

All XRD patterns were visually inspected, and a small number of unknowns showed peaks near 12.5 Å, which did not match any catalogued structural data applicable for Rietveld refinement. In order to accurately identify this phase, we treated the <2-μm size fraction using glycolation and heating techniques and performed XRD analyses on a variety of mount orientations. These minerals were variously identified as sepiolite, Na-montmorillonite, and hydrobiotite in different samples.

Amorphous Content

An internal standard (10 percent by weight ZnO) was added to each standard and unknown to allow calculation of the amorphous content. The addition of an internal standard may introduce dilution and absorption effects, but it is a necessary requirement to calculate the total amorphous content from background intensity by Rietveld analysis. The Rietveld method normalizes the sum of the mass fractions of all crystalline phases to unity, so if amorphous material is present in the soil sample, the amount of ZnO calculated will have a higher mass fraction than the actual ZnO added to the soil sample. In those cases, percentages are recalculated so that ZnO equals 10 percent; as a result, the sum of quantified phases is less than 100 percent. The difference between that sum and 100 percent is reported as the amorphous (unaccounted) content.

Use of XRD for quantification is generally less reliable for amorphous content than for crystalline phases. Even a completely crystalline sample will have some background intensity resulting from such factors as sample imperfection, fluorescence, scatter, and instrumental and optical error. Determination of amorphous content in soils is further complicated by the effects of matrix and compositional variation, enhancement and absorption effects, contribution of amorphous material created during sample preparation, and wide variation between amorphous material types. The presence of amorphous content in a soil sample can be attributed to a variety of sources, including organic material, glass, poorly-ordered phases, or nano-scale crystals insufficient in size to contribute to Bragg reflections. Each of these amorphous types may contribute different relative amounts to background intensity. We have not attempted to isolate the effects of them but, rather, report a total amorphous content as the percentage of each sample that was not quantified as a crystalline phase.

Data Tables

All the geochemical and mineralogical data for the conterminous United States generated in this study are available in downloadable tables in both Excel (.xls) and text (.txt, tab delimited) formats. There are separate tables for surface soils collected from a depth of 0 to 5 cm (appendix 2a [.xls] and 2b [.txt]), the soil A horizon (appendix 3a [.xls] and 3b [.txt]), and the soil C horizon (appendix 4a [.xls] and 4b [.txt]). Each table for each sample medium contains both geochemical and mineralogical data. There are an equal number of rows (samples) and columns (attributes) in each table, and the order of samples and attributes is exactly the same in each table. This construction facilitates the user's ability to merge the files for the purpose of calculating element ratios or differences between the three sample types. As a result, however, some cells in appendix 2 are left blank, indicating that this parameter was not determined; some cells in appendixes 2, 3, and 4 are shown as N.S., indicating that this particular sample type was not collected or was lost in shipping; and some cells in appendixes 2, 3, and 4 are shown as INS, indicating there was insufficient sample material to analyze for a particular parameter.

The formal metadata is provided in appendix 5 and provides a detailed description of each table.

Acknowledgments

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Appendix 1. U.S. Geological Survey soil sampling manual for the North American Soil Geochemical Landscapes Project

The Soil Sampling Manual was provided to all field crews engaged in collecting soil samples during this project and provides the basis for standardization of the sampling protocols. See report text for details about the study. The manual is shown as it was provided to the sampling crews; some minor editorial changes have been made, but it has not been formatted to conform to U.S. Geological Survey publication standards.

Appendix 1_NASGLP-Soil-sampling-manual.pdf

Appendix 2. Geochemical and mineralogical data for samples of surface soils collected from a depth of 0 to 5 centimeters in the conterminous United States

Appendix_2a_Top5_18Sept2013.xls
Appendix_2b_Top5_18Sept2013.txt

Appendix 3. Geochemical and mineralogical data for samples from the soil A horizon in the conterminous United States

Appendix_3a_Ahorizon_18Sept2013.xls
Appendix_3b_Ahorizon_18Sept2013.txt

Appendix 4. Geochemical and mineralogical data for samples from the soil C horizon in the conterminous United States

Appendix_4a_Chorizon_18Sept2013.xls
Appendix_4b_Chorizon_18Sept2013.txt

Appendix 5. Metadata for geochemical and mineralogical data for soils of the conterminous United States

Appendix_5_Metadata.pdf
Appendix_5_Metadata_FAQ.pdf