

U.S. Geological Survey Mineral Resources Program, prepared in cooperation with Bureau of Reclamation, Bureau of Land Management, and Utah Department of Environmental Quality Division of Water Quality

Results of Mineral, Chemical, and Sulfate Isotopic Analyses of Water, Soil, Rocks, and Soil Extracts from the Pariette Draw Watershed, Uinta Basin, Utah

Open-File Report 2015-1132

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By Jean M. Morrison, Michele L.W. Tuttle, and Juli W. Fahy

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# **U.S. Department of the Interior** SALLY JEWELL, Secretary

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

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### **Conversion Factors**

Inch/Pound to SI

Ву	To obtain
Length	
2.54	centimeter (cm)
0.3048	meter (m)
1.609	kilometer (km)
Area	
0.004047	square kilometer (km²)
2.590	square kilometer (km²)
Volume	
3.785	liter (L)
0.003785	cubic meter (m <sup>3</sup> )
Mass	
28.35	gram (g)
0.4536	kilogram (kg)
Density	
0.01602	gram per cubic centimeter (g/cm³)
	Length 2.54 0.3048 1.609 Area 0.004047 2.590 Volume 3.785 0.003785 Mass 28.35 0.4536 Density

#### SI to Inch/Pound

Multiply	Ву	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
	Area	
square kilometer (km²)	247.1	acre
square kilometer (km²)	0.3861	square mile (mi <sup>2</sup> )
	Volume	
liter (L)	0.2642	gallon (gal)
cubic meter (m³)	0.0002642	million gallons (Mgal)
cubic hectometer (hm³)	810.7	acre-foot (acre-ft)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)
	Density	
gram per cubic centimeter (g/cm³)	62.4220	pound per cubic foot (lb/ft³)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:  $^{\circ}F=(1.8\times^{\circ}C)+32$ 

Temperature in degrees Fahrenheit (°F) may be converted to degrees Celsius (°C) as follows:  $^{\circ}C=(^{\circ}F-32)/1.8$ 

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

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

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

Concentrations of chemical constituents in water are given either in milligrams per liter  $(mg/L^{-1})$  or micrograms per liter  $(\mu g/L^{-1})$ .

# **Abbreviated Chemical Symbols**

Abbreviation	Chemical species
Ag	silver
Al	aluminum
As	arsenic
В	boron
Ba	barium
Be	beryllium
Bi	bismuth
Br	bromine
$\mathrm{C}_{\mathrm{total}}$	total carbon
$C_{\rm CO3}$	carbonate carbon
$\mathrm{C}_{\mathrm{org}}$	organic carbon
Ca	calcium
Cd	cadmium
Ce	cerium
Cl	chlorine
Co	cobalt
Cr	chromium
Cs	cesium
Cu	copper
DOC	dissolved organic carbon
Dy	dysprosium
Er	erbium
Eu	europium
Fe	iron
Ga	gallium
Gd	gadolinium
Ge	germanium
$HCO_3$	Bicarbonate
Hg	mercury
Но	holmium
In	indium
K	potassium
La	lanthanum
Li	lithium
Lu	lutetium
Mg	magnesium
Mn	manganese
Mo	molybdenum
N	nitrogen (as N)
Na	sodium
Nb	niobium
Nd	neodymium
Ni	nickel
$NO_3$	nitrate
${ m O}_{ m SO4}$	sulfate oxygen
P	phosphorous
Pb	lead
Pr	prasceodmium
Rb	rubidium
S	sulfur

# **Abbreviated Chemical Symbols—Continued**

Abbreviation	Chemical species
$S_{SO4}$	sulfate sulfur
Sb	antimony
Sc	scandium
Se	selenium
Sm	samarium
Sn	tin
$\mathrm{SO}_4$	sulfate
Sr	strontium
Ta	tantalum
Tb	terbium
Te	tellurium
Th	thorium
Ti	titanium
Tl	thallium
Tm	thulium
U	uranium
V	vanadium
W	tungsten
Y	yttrium
Zn	zinc
Zr	zirconium

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# Results of Mineral, Chemical, and Sulfate Isotopic Analyses of Water, Soil, Rocks, and Soil Extracts from the Pariette Draw Watershed, Uinta Basin, Utah

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#### Introduction

In 2010. Utah Department of Environmental Quality (DEQ) Division of Water Quality (UDWQ, 2010) determined that water quality in Pariette Draw was in violation of Federal and State water quality criteria for total dissolved solids (TDS), selenium (Se), and boron (B). The measure of total dissolved solids is the sum of all the major ion concentrations in solution and in this case, the dominant ions are sodium (Na) and sulfate (SO<sub>4</sub>), which can form salts like thenardite (Na<sub>2</sub>SO<sub>4</sub>) and mirabilite (Na<sub>2</sub>SO<sub>4</sub>·H<sub>2</sub>O). The Utah Department of Environmental Quality (2010) classified the contamination as natural background and from nonpoint sources related to regional lithology and irrigation practices. Although the daily loads of the constituents of concern and water chemistry have been characterized for parts of the watershed, little is known about the controls that bedrock and soil mineralogy have on salt, Se, and B storage and the water-rock interactions that influence the mobility of these components in ground and surface waters. Studies in the Uncompangre River watershed in Colorado by Tuttle and others (2014a, 2014b) show that salt derived from weathering of shale in a semiarid climate is stored in a variety of minerals that contribute solutes to runoff and surface waters based on a complex set of conditions such as water availability, geomorphic position (for example, topography controls the depth of salt accumulation in soils), water-table fluctuations, redox conditions, mineral dissolution kinetics, ion-exchange reactions, and secondary mineral formation. Elements like Se and B commonly reside in soluble salt phases, so knowledge of the behavior of salt minerals also sheds light on the behavior of associated contaminants.

The goal of this study was to establish a process-based understanding of salt, Se, and B behavior to address whether these contaminants can be better managed, or if uncontrollable natural processes will overwhelm any attempts to bring Pariette Draw into compliance with respect to recently established total maximum daily limits (TMDLs). We collected data to refine our knowledge about the role of rock weathering and soil formation in the transport and storage of salt in the

watershed and to show how salt is cycled under irrigated and natural conditions. Our approach was to sample rock, soils, and sediment on irrigated and natural terrain for mineralogical analysis to determine the residence of salt and associated Se and B, classify minerals as primary (related to rock formation) or secondary weathering products, and characterize mineral dissolution kinetics. Mineral and chemical analyses and selective extractions of rocks and soils provide useful information in understanding solute movement and mineral dissolution/formation. The resulting data are critical in determining residence of salt, Se, and B in weathered rock and soil and understanding the mobility during water-rock-soil interactions. This report summarizes our methods for sample and data collection and tabulates the mineral, chemical, and isotopic data collected.

#### Study Area

The Pariette Draw watershed (hydrologic unit code (HUC) #14060005) is 316 square miles (mi2) and is located in the Uinta Basin, northeastern Utah (Duchesne and Uintah Counties and the Uintah and Ouray Indian Reservation; fig. 1). The watershed is south of the city of Myton and is bordered by the Duchesne River (north), Tavaputs Plateau (south and west) and the Green River (east; Utah Department of Environmental Quality, 2010). Agriculture composes 4.2 percent of the land cover in the watershed and the remaining is primarily native inter-mountain basin shale badlands with extensive oil and gas development (Utah Department of Environmental Quality, 2010). Landownership is as follows: 68 percent Bureau of Land Management (BLM), 12 percent private, 9 percent School and Institutional Trust Lands Administration (SITLA), 7 percent tribal, and 4 percent U.S. Forest Service (Utah Department of Environmental Quality, 2010).

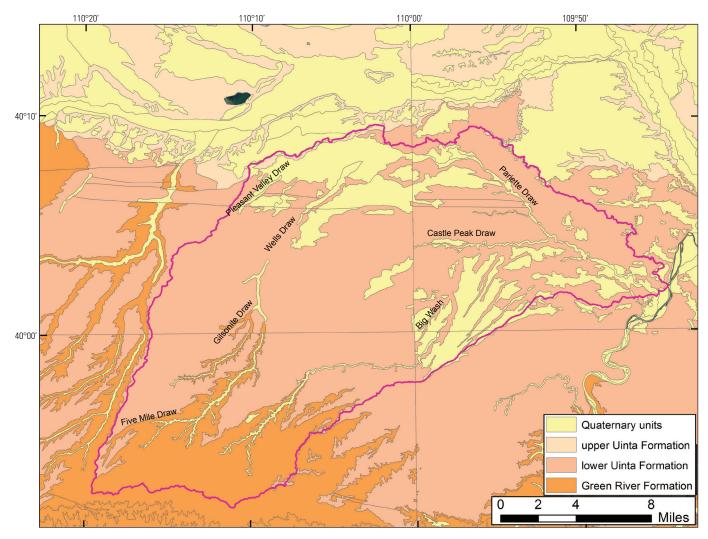
Pariette Draw is a tributary to the Green River and is perennial only because of irrigation in Pleasant Valley where agriculture occurs. All tributary drainages into Pariette Draw are ephemeral. Irrigation runoff provides water to Pariette

Figure 1. Location map for the Pariette Draw Watershed.

Wetlands, a series of BLM-managed wetlands located in the lower watershed just above the confluence with Green River (fig. 1). The wetlands provide habitat to abundant and diverse species of waterfowl. Because Se and B contamination can affect the health of waterfowl populations, the water quality in Pariette Draw is of major concern to wetland and wildlife managers.

In the northwestern portion of the upper watershed (Wells Draw and upper tributaries), the sandstone-limestone facies of the Tertiary Green River Formation underlie the lower Uinta Formation (fig. 2). This facies is a transitional unit which is light brown sandstone and siltstone interbedded with white to light gray marlstone and limestone. In the remainder of the watershed, the surface geology is the overlying lower Uinta Formation. The lower Uinta Formation consists of sandstone with minor conglomerates at its base and red and green claystone interbedded with sandstone at the top. Roll-front uranium deposits mined during the 1950s are hosted by the upper sandstone members of the lower Uinta Formation. The largest concentration of mining activity was in Castle Peak Draw near gilsonite mines

(397 pounds of U<sub>3</sub>O<sub>8</sub> from 1951 to 1954; Chenoweth, 1992). Formation of these roll-front deposits involved redox cycling in near-surface sandstone aquifers of fluvial or lacustrine origin similar to those deposited near the top of the lower Uinta Formation. The upper Uinta Formation, which crops out north of the study area, but has since been eroded in the watershed, is rich in bentonitic and swelling claystone likely derived from volcanic ash. The ash fall is thought to be the original source of uranium in the Pariette Draw roll-front deposits (Chenoweth, 1992). Volcanic ash is also known to contain selenium, vanadium, molybdenum, arsenic, and sulfur, elements that are often concentrated along with uranium in roll-front deposits that form as oxidized groundwater interacts with ash and solutes carried deeper into the aquifer (Harshman, 1974). When reducing conditions are encountered due to increased abundance of organic carbon, pyrite (FeS<sub>2</sub>) or hydrogen sulfide (H<sub>2</sub>S) gas, roll-front deposits form in halos around the reductant. Spatial separation of elements deposited is controlled by differential reduction potentials (Adler, 1974).



**Figure 2.** Simplified geologic map for the Pariette Draw Watershed (merged from four 30'x60' quadrangle geologic maps; Sprinkel, 2007; 2009; Utah Geologic Survey, 2009; Bryant, 2010). Magenta outline shows extent of the Pariette Draw Watershed (see location on fig. 1).

#### **Sampling**

Rock, soil, and surface and groundwater samples were collected throughout the Pariette Draw Watershed. Rocks were collected from 22 Green River Formation and lower Uinta Formation sites (table 1–1 in Appendix 1). Soil samples were collected at 69 sites (12 agricultural fields and 57 native soil sites). Water samples were collected from streams, canals, ponds, puddles in ephemeral streams, auger holes, springs, and seeps. Sample locations and number of samples collected at each site are in table 1.

Table 1. Number of samples collected and analyzed.

Sample type	Sample sites	Chemically analyzed	Mineralogically analyzed
Rock	22	65	19
Soil (bulk)	69	508	25
Extract	69	610	
Water	35	68	

#### **Rock and Soil Samples**

Rock sampling sites were selected to be representative of the three different geologic units in the watershed (upper Green River Formation and lower and upper portions of the lower Uinta Formation). Samples include upper Green River Formation limestones and sandstones, basal lower Uinta Formation sandstones, and upper lower Uinta Formation sandstones and interbedded red and green shales. White layers assumed to be volcanic ash were collected in all three units. Each sample was a grab sample composite across the lithologic unit of interest. When possible, the least-weathered rock was collected and any friable weathering crust was removed.

Soil was primarily collected by hand augering as deep as possible, up to a maximum of 9 feet. The soil was collected in 6-inch intervals and spread on a tarp, homogenized, and placed in plastic sample bags. In the laboratory, the bags were emptied onto plastic sheets and allowed to air dry. When dry, samples were disaggregated and sieved to less than 2 mm for

analysis of bulk chemistry, mineralogy, and saturation paste extracts. The location of rock and soil samples are shown in figures 3A and B.

#### Water Samples

Diverted irrigation and pond water, and stream water were collected as grab samples. Shallow groundwater was collected from auger holes in Pleasant Valley where the majority of agriculture occurs in the watershed. Samples from Pariette Draw below irrigated fields, standing water in ephemeral streambeds (puddles), shallow groundwater in wetland auger holes, and a natural spring were collected outside of Pleasant Valley. The location of water samples are shown in figure 4.

Water was collected with a syringe or a teflon bailer. Cation, anion, Se speciation, and bicarbonate splits were filtered (0.45 micrometer [ $\mu$ m]) in the field. Cation samples were acidified with nitric acid. Sulfate isotope samples were filtered in the laboratory. All samples were refrigerated during transport and prior to analysis.

#### **Soil Bulk Density Samples**

Three-inch sections of steel pipe (1.25 inches [in.] in diameter) were used to collect the bulk density samples. The volume of the samplers was determined in the laboratory by stretching tape over one end and setting that end in liquid wax to assure a water-tight seal. The samplers were then filled with water and the volume of the water measured. For each soil density sample, the pipe was pushed into the soil with as little disturbance as possible. The pipe was withdrawn, collecting all the material from the hole and then transferred to a plastic sample bag. The hole depth was then measured to check for compression. For surface samples, the pipe was inserted vertically. A 12 in. (30 centimeter [cm]) hole was then dug where the surface sample was taken and the second interval was sampled horizontally into the side of the hole. The third interval was sampled vertically from the bottom of the hole. Twenty-three interval samples (in triplicate) were taken at 9 sites (2 agricultural fields and 7 native soil sites). In the laboratory, samples were dried in an oven at 105 °C for 2 hours and

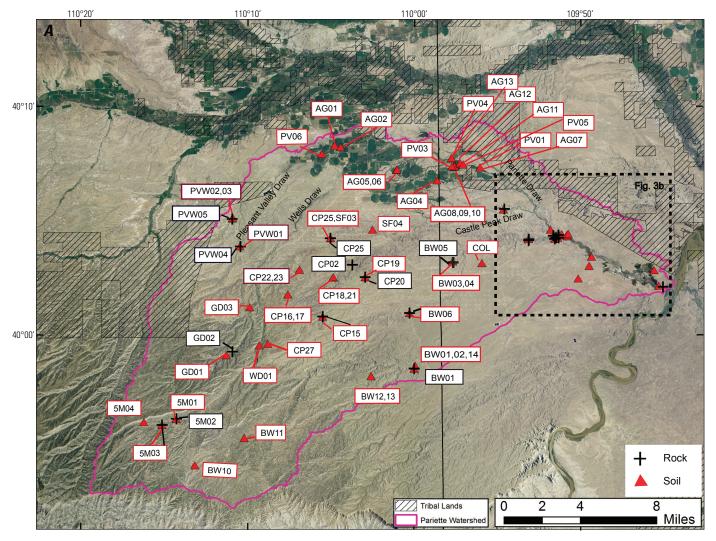


Figure 3. Map of rock and soil collection sites with A, showing entire watershed and B, a close up of the inset box in 3A.

then weighed. Outlier measurements generally occurred for samples with small pebbles or voids. In the results, densities without these outliers are also reported.

#### **Extract Preparation**

Saturated paste extracts were prepared by mixing the soil or disaggregated shale samples (dried and ground portion of less than 2 millimeters [mm] material) into a smooth paste with a consistency comparable to that of pudding. According to U.S. Department of Agriculture methods, "At saturation, the soil glistens as it reflects light, flows slightly when the container is tipped, and slides freely and cleanly from a spatula except for those soils with high clay content" (U.S. Department of Agriculture, 2004, p. 283). There should be no free water on top of the sample. Each sample requires a different amount of water to be added to achieve the proper mixture, and the amount of water added is reflected in the percent saturation. That number is the amount of water

added relative to the dry weight of the soil. For example, if a 200 gram (g) soil sample requires 100 milliliter (mL) of water to make a saturated paste, it has a 50 percent saturation. The pH is measured in the paste immediately prior to extraction of the solution. A sample of the paste is weighed, dried at 105 °C overnight, and reweighed to establish saturation percent. The extract solution is removed by means of vacuum filtration using a 2 mm filter paper and analyzed for conductivity. The filtrate is then filtered through a 0.45  $\mu$ m filter and split for further chemical analyses. Percent saturation can be used to calculate extract concentrations to a dry-weight basis to compare data among samples:

$$Cdw \equiv (Cex \times (\%S/100)) \tag{1}$$

where

*Cdw* is extract concentration on a dry-weight basis;

Cex is concentration in filtrate; and

%S is percent saturation.

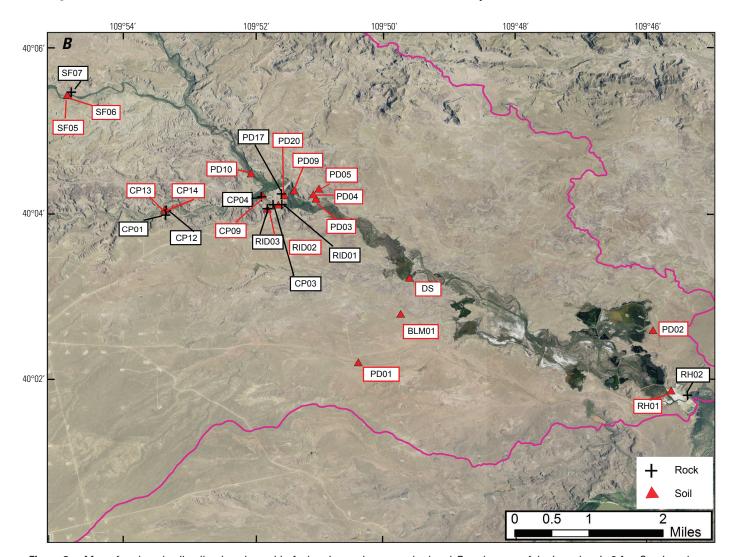


Figure 3. Map of rock and soil collection sites with A, showing entire watershed and B, a close up of the inset box in 3A.—Continued



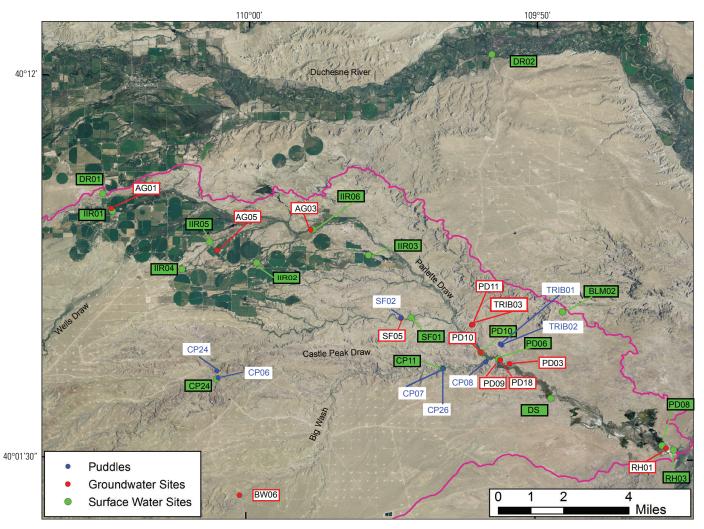


Figure 4. Map showing water collection sites.

#### **Analytical Methods**

#### Chemical Analyses of Solid Samples, Soil Extracts, and Water

SGS Mineral Services, Toronto, Canada, performed analyses of major and a suite of trace elements in the bulk rock and soil and also analyzed for additional trace elements on a selected split of rock and soil samples. Specialized analyses on solids such as istotopic analysis, mineralogy, and chemical anlyses of saturation paste extracts were performed in the U.S. Geological Survey laboratories, Denver, Colo. Table 2 summarizes the methods of analyses.

Water soluble salts and gypsum samples were placed in water and agitated for 30 minutes and then filtered (less than  $0.45 \mu m$ ). Gypsum samples were also heated in 6 normal

hydrochloric acid (N HCL) warmed to approximately 30 °C and filtered. The pH of the water solution was adjusted to less than 2. Barium chloride (10 weight percent [wt%]) solution was added to both the water and acid solutions until no further precipitation of barium sulfate (BaSO<sub>4</sub>) occurred. The solutions were allowed to stand overnight and were then filtered. The BaSO<sub>4</sub> collected was analyzed for the isotopic composition of sulfur and oxygen by methods in Kester and others (2011) and results reported as  $\delta^{34}$ S and  $\delta^{18}$ O.\(^1\) Oxygen compositions of the sulfate only were reported for samples extracted with water because of exchange of sulfate oxygen with water oxygen in acid extractions.

 $<sup>^{1}\</sup>delta$  (%0) =  $(R_{sample} - R_{standard})/(R_{standard}) \times 1000$ , where R is the ratio of  $^{34}S$ :  $^{32}S$  or  $^{18}O$ :  $^{16}O$ . The standard for sulfur is trolite from Cañon Diablo Meteorite (VCDT) and for oxygen, Standard Mean Oceaic Water (VSMOW).

Table 2. Analytical methods.

Analyses	Method	Abbreviation	Reference
Anions (extracts and water)	Ion chromotography	IC	2
As, Se (solids)	Hydride generation-atomic absorption spectroscopy	HY	1
As, Se (extracts and water)	Hydride generation-atomic absorption spectroscopy	HY	2
Bicarb. (extracts and water)	Titration	TITR	2
$C_{total}$ (solids)	Combustion	Total	1
$C_{CO3}(solids)$	Coulometric titration	Carbonate	1
$C_{org}$ (solids)	Difference ( $C_{total}$ - $C_{CO3}$ )	Organic	1
Cl (solids)	Ion selective electrode	ISE	1
$S_{total}$ (solids)	Combustion and infrared detection on LECO	Comb	1
Specific Cond. (extracts and water)	Electrial conductivity meter	ECe	2
Major element (solids)	4-acid digestion/inductively coupled plasma optical emission spectroscopy	OES	1,3
Major element (extracts and water)	Inductively coupled plasma optical emission spectroscopy	OES	2
рН	pH meter	pН	2
Refractory trace element (solids)	Sinter/inductively coupled plasma mass spectroscopy	S MS	1,4
Trace element (solids)	Inductively coupled plasma mass spectroscopy	MS	1,5
Trace element (extracts and water)	Inductively coupled plasma mass spectroscopy	MS	2

<sup>&</sup>lt;sup>1</sup>Analyzed in a commercial laboratory with quality assurance/quality control monitored by U.S. Geological Survey.

Mineralogy on a subset of rock and soil samples was determined by x-ray diffraction using methods from Eberl (2011). Samples were analyzed on a Scintag X-ray Diffractometer (XRD) from 5 to 65 degrees two-theta using Cu K-alpha radiation, with a step size of 0.02 degrees two theta, and a count time of two seconds per step using a scintillation counter. Quantitative mineralogy was calculated using RockJock (Eberl, 2011) and clay mineral identification was confirmed by collecting a less than 8  $\mu m$  fraction of each sample and preparing oriented slides. XRD scans were collected of the air-dried, ethylene glycolated, and two heat treatments (400 and 550 °C for 1 hour each) of the preparations ranging from 2 to 40 degrees two-theta using Cu K-alpha radiation, with a step size of 0.03 degrees two theta, and a count time of one second per step using a scintillation counter.

#### Results

Tabulated results are in the Appendix: table 1–2, water chemistry; table 1–3, rock mineralogy; table 1–4, rock chemistry; table 1–5, soil bulk density; table 1–6, soil mineralogy; table 1–7, bulk soil chemistry; table 1–8, sodium sinter/ICP-MS analyses on selected rock and soil samples; and table 1–9, soil extract chemistry. Metadata in tables 1–10—1–14 include additional analytical information including elements with censored values (less than the lower limit of determination, which is defined as 5 times the standard deviation of the blank; Taggart, 2002), duplicate analyses, and laboratory and field blanks.

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<sup>&</sup>lt;sup>2</sup>Analyzed in U.S. Geological Survey laboratories (Taggart, 2002). Quality assurance/quality control internally monitored.

<sup>&</sup>lt;sup>3</sup>Lithium metaborate fusion dissolved in dilute nitric acid.

<sup>&</sup>lt;sup>4</sup>Sodium peroxide sinter at 450 °C.

<sup>&</sup>lt;sup>5</sup>Hydrofluoric-, nitric-, perchloric-, and hydrochloric acid digestion.

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#### **Appendix 1**

(Available at http://dx.doi.org/10.3133/ofr20151132)

- **Table 1–1.** Sample site designation, location, and description.
- Table 1–2. Water chemical data.
- Table 1-3. Rock mineralogical data.
- Table 1-4. Rock chemical data.
- Table 1-5. Bulk soil density data.
- Table 1-6. Soil mineralogical data.
- Table 1-7. Bulk soil chemistry.
- Table 1-8. Bulk rock and soil chemistry (selected samples and selected elements by sodium peroxide sinter ICP-MS).
- Table 1-9. Soil extract chemistry.
- Table 1–10. Water analysis metadata.
- Table 1-11. Rock analysis metadata.
- Table 1-12. Bulk soil metadata.
- Table 1-13. Soil extract metadata.
- Table 1–14. Sulfate Isotope metadata.

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For more information concerning this publication, contact:

Director, USGS Crustal Geophysics and Geochemistry Science Center

Federal Center, Box 25046, MS 964

Denver, CO 80225 (303) 236-1373

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