

# **MAPPING THE SOIL GEOCHEMISTRY AND MINERALOGY OF THE CONTERMINOUS UNITED STATES—EVOLUTION AND SUCCESSFUL COMPLETION OF A CONTINENTAL-SCALE, GOVERNMENT-SPONSORED SURVEY**

David B. Smith<sup>1</sup>, Laurel G. Woodruff<sup>2</sup>, William F. Cannon<sup>3</sup>, Federico Solano<sup>3</sup>

<sup>1</sup>U.S. Geological Survey, Denver, CO 80225, USA; [dsmith@usgs.gov](mailto:dsmith@usgs.gov)

<sup>2</sup>U.S. Geological Survey, St. Paul, MN 55112, USA

<sup>3</sup>U. S. Geological Survey, Reston, VA 20192, USA

## **Introduction**

In 2002, the U.S. Geological Survey (USGS) established a project to conduct a low-density soil geochemical and mineralogical survey of the United States (U.S). The project was initially part of the North American Soil Geochemical Landscapes Project, a joint effort among the USGS, the Geological Survey of Canada, and the Mexican Geological Survey to conduct a similar project for the whole of North America. Each of the three agencies would be responsible for funding and carrying out the project in its respective country. The focus of this paper is only on the USGS project.

## **Project Development**

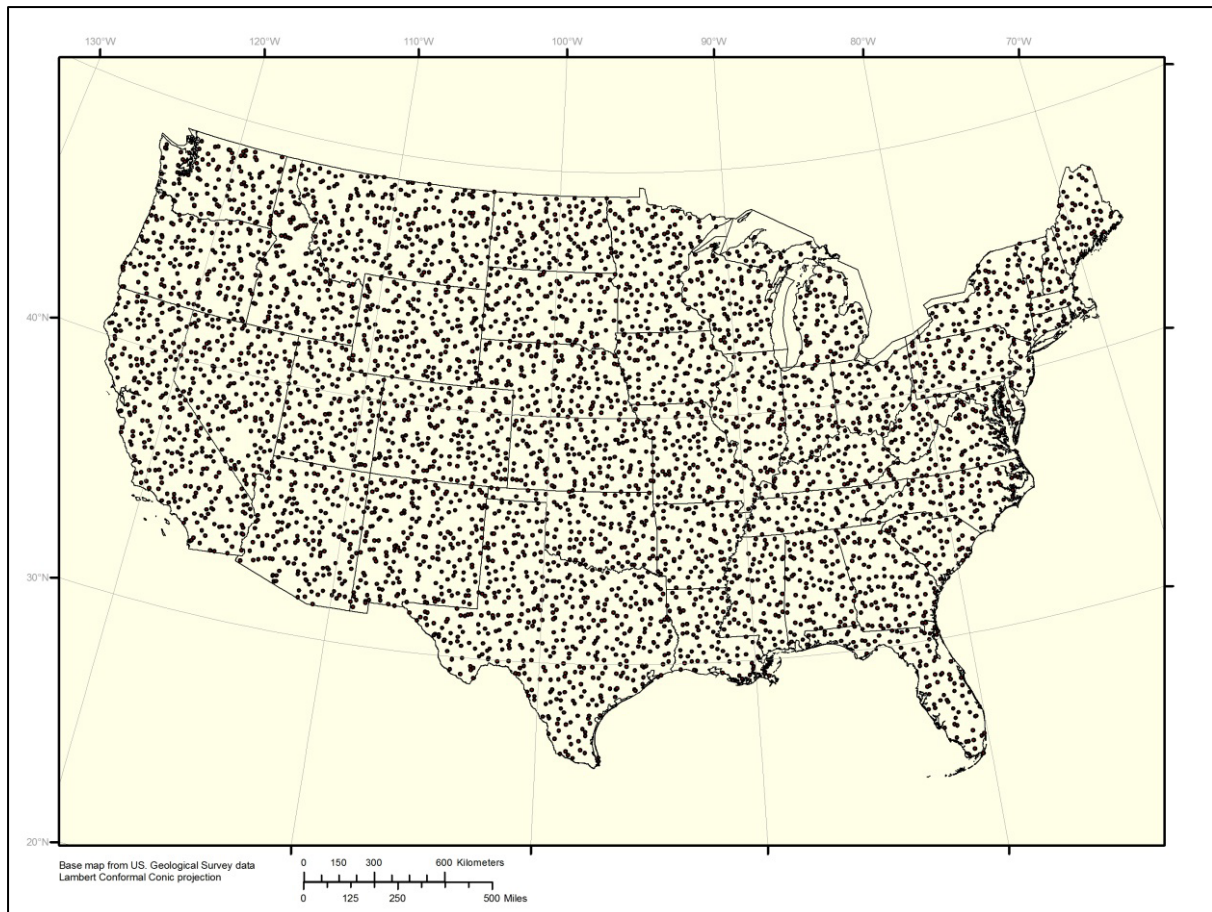
There was a well-established need for a new national-scale soil geochemical survey of the U.S. Prior to the current project, the best national-scale soil geochemical data set was that generated by a project conducted from 1961 to 1975. That project collected soil samples from 1,323 sites (1 site per 6,000 km<sup>2</sup>) throughout the conterminous U.S. At each site, a sample was collected from a depth of about 20 cm and the <2-mm fraction was analysed by methods available from the USGS laboratories at that time. A total of 50 elements were reported, but 30 of those elements were determined by semi-quantitative methods (Shacklette et al. 1971; Boerngen & Shacklette 1981; Shacklette & Boerngen 1984; Gustavsson et al. 2001; Smith et al. 2013a). This data set proved to be so popular that a similar survey was conducted in Alaska (Gough et al. 1984, 1988, 2005). However, by the late 1990s, it was obvious that this data set was no longer adequate for the growing number of users needing information on the background concentration of potentially toxic elements in soil.

The first step in the development of the new project was to engage the stakeholder community to obtain input on how such a national-scale survey should be designed, what the sampling protocols should be, and what analytical methods were preferred. In 2003, a workshop was held in Denver, Colorado to establish this communication. The workshop attracted 112 participants representing 43 stakeholder entities from Federal and state governmental agencies, academia, the medical and public health community, and environmental consultancies. The recommendations from this workshop provided the foundation for this project from 2003 to the present.

The recommendations from the workshop were so involved that a 3-year pilot study was conducted (2004–2007) to test and refine the recommended sampling and analytical protocols and to optimize field logistics. The results from this pilot phase were published as 21 papers in a special issue of *Applied Geochemistry* in August 2009. Several modifications were made to the protocols recommended by the 2003 workshop and sampling for the full national-scale soil geochemical survey was initiated in the summer of 2007.

## **Survey Design**

The target sampling sites were selected on the basis of a generalized random tessellation stratified design at a density of 1 site per 1,600 km<sup>2</sup> (4,857 sites). If the original target site could not be accessed for any reason, an alternative site was chosen as close as possible to the original site and matching as closely as possible the landscape and soil characteristics of the original site. The sampling crews were also provided with guidelines for avoiding areas of obvious contamination (e.g., no sample should be collected within 200 m of a major highway). Figure 1 shows the location of the 4,857 sites sampled during the project.



**Figure 1. Map showing the location of the 4,857 sampling sites in the conterminous United States.**

## Sampling Protocols

The final sampling 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.

## Sample Preparation and Analytical Methods

Each sample was air-dried at ambient temperature, disaggregated, and sieved to <2 mm. Splits of the <2-mm material were archived and are available for future investigations. The <2-mm material was then crushed to <150  $\mu\text{m}$  prior to chemical analysis. Concentrations of Al, Ca, Fe, K, Mg, Na, S, Ti, Ag, Ba, Be, Bi, Cd, Ce, Co, Cr, Cs, Cu, Ga, In, La, Li, Mn, Mo, Nb, Ni, P, Pb, Rb, Sb, Sc, Sn, Sr, Te, Th,

Tl, U, V, W, Y, and Zn were determined by a combination of inductively coupled plasma–atomic absorption spectrometry and inductively coupled plasma–mass spectrometry. The sample was decomposed using a near-total four-acid (hydrochloric, nitric, hydrofluoric, and perchloric) digestion at a temperature between 125 and 150 °C. Mercury was determined by cold-vapor atomic absorption spectrometry after dissolution in a mixture of nitric and hydrochloric acids. For analysis of arsenic, 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 analysed by hydride-generation atomic absorption spectrometry. Selenium was determined by hydride-generation atomic absorption spectrometry after dissolution in a mixture of nitric, hydrofluoric, and perchloric acids. Total carbon was determined by the use of an automated carbon analyser. 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. Quality control protocols included randomization of samples prior to analysis, insertion of international reference materials, insertion of project standards, and insertion of analytical duplicates of project samples.

All A-horizon and C-horizon samples were analysed by X-ray diffraction, and the percentages of major mineral phases were calculated using a Rietveld refinement method. 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- $\mu$ m sieve and lightly pressed into back-loaded sample mounts. Samples were analysed on a PANalytical X'Pert PRO Materials Research Diffractometer using Cu K $\alpha$  radiation to collect digital data continuously from 3° to 70° 2 $\theta$  (scan speed = 0.0567° 2 $\theta$  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 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 the reliability of this method was done by analysing standard mixtures of pure mineral phases prepared in-house and statistically evaluating the data.

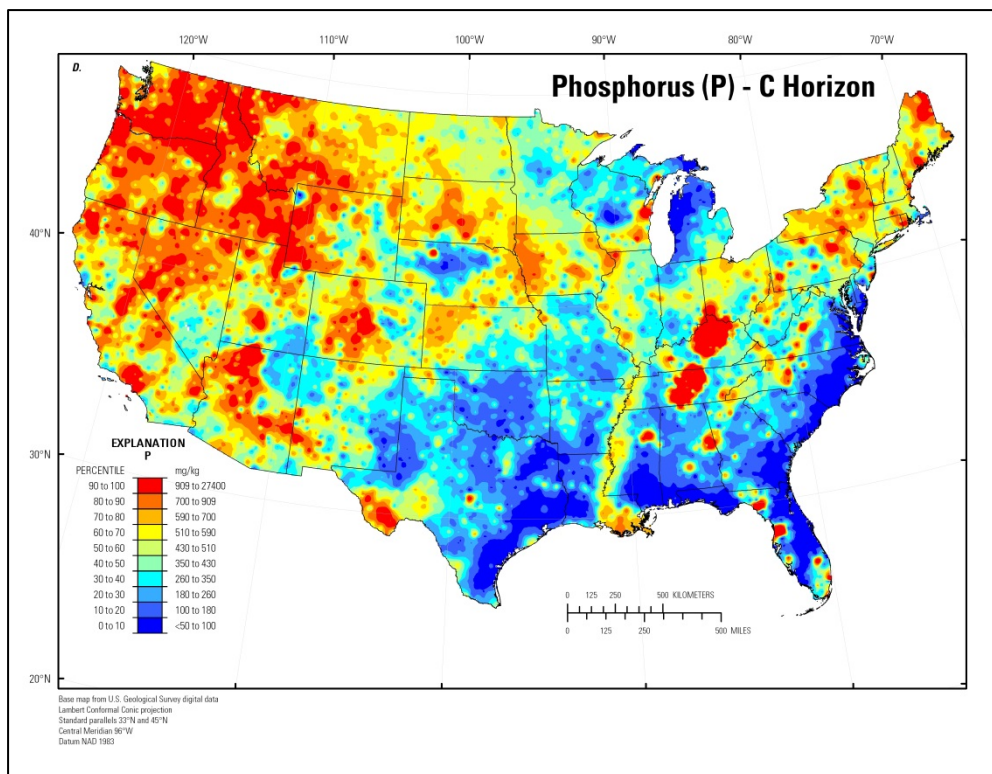
## **Project Completion and Continuing Research**

Sampling was completed in late 2010 with chemical and mineralogical analysis completed in May 2013. The geochemical and mineralogical data were published as USGS Data Series 801 (<http://pubs.usgs.gov/ds/801/>) in 2013 (Smith et

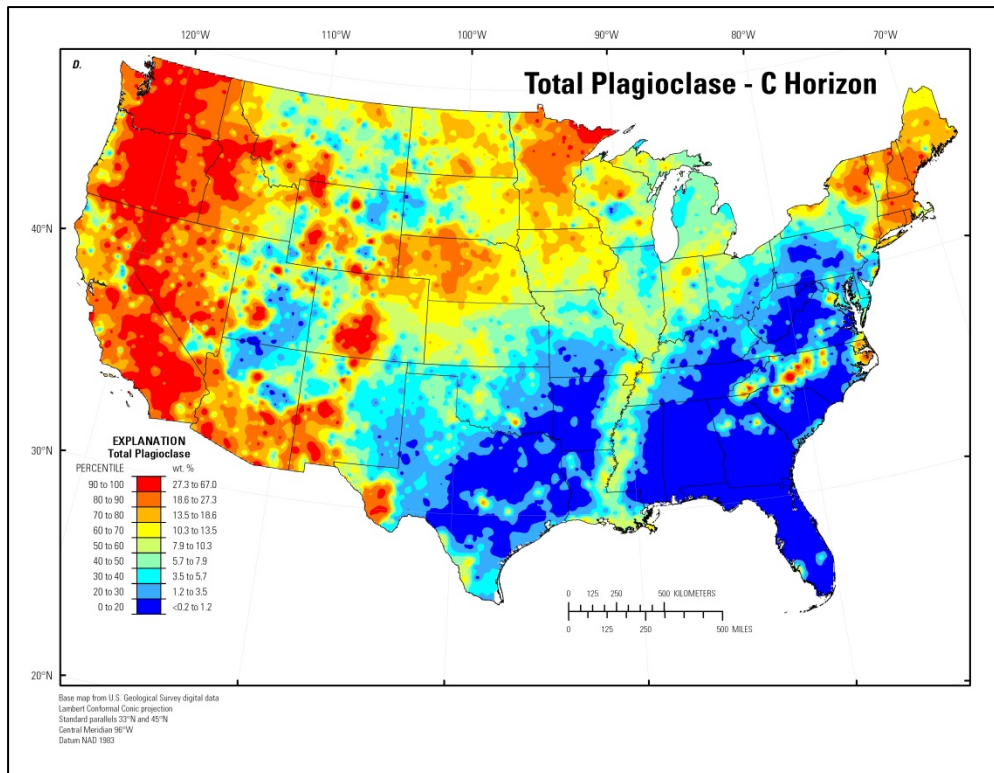


al. 2013b). This report also contains (1) detailed descriptions of the sampling protocols, analytical methods, and quality control protocols; (2) statistical summaries for the geochemical and mineralogical analyses; and (3) tables showing the statistical summaries of analysis of reference materials and analytical duplicates. The geochemical and mineralogical maps were published as USGS Open-File Report 2014-1082 (<http://pubs.usgs.gov/of/2014/1082/>) in 2014 (Smith et al. 2014). A website (<http://mrddata.usgs.gov/soilgeochemistry/#/summary>) was also established in 2014 where all the maps and statistical graphics can be viewed and the maps can be downloaded in a variety of formats. These formats include georeferenced TIFF files that can be exported into any GIS software and .KML files that can be opened in Google Earth. Figures 2 and 3 show examples of the geochemical and mineralogical maps produced.

With the publication of the data and maps, the emphasis for the next few years will be on interpretation of the geochemical and mineralogical patterns delineated by the data and using the sample archives as a resource for additional research. Current studies focus on topics ranging from global climate change to forensic applications of soil geochemical data.



**Figure 2. Map showing the distribution of phosphorus in the soil C horizon, conterminous U.S.**



**Figure 3. Map showing the distribution of total plagioclase in the soil C horizon, conterminous U.S.**

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