November 25, 2002

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Certificate of Analysis
MCLinc Project #: WES001009
X-Ray Diffraction and Scanning Electron Microscopy Characterization

The soil samples were received at Materials and Chemistry Laboratory, Inc. (MCLinc) in good condition on November 15, 2002 and were given MCLinc identification codes as presented in Table 1.

Table 1. Sample Identification Cross-Reference

Weston Sample Number	MCLinc Sample Number
2BH038-BS-020-R	02-0866
2BH018-BS-025-0	02-0867
2BH018-BS-050-R	02-0868
2BH018-BS-018-0	02-0869

MCLinc prepared the samples for examination by X-Ray Diffraction (XRD) and Scanning Electron Microscopy with Energy-Dispersive Spectroscopy (SEM-EDS). Imaging techniques used for SEM-EDS facilitate locating deposits of heavy metals, such as uranium, and identifying elements associated (co-located) with the heavy metals. XRD is used to identify the crystalline phases present in the sample. The samples consist primarily of quartz (SiO_2) with minor mica and some feldspar and calcite.

Samples 02-0866 and 02-0869 had the greatest gross radioactivity (as monitored with a Ludlum beta/gamma survey meter). These as-received samples evidenced weak XRD lines suggestive of uranium oxide, probably U_3O_8. In order to facilitate more definitive identification, the heavy mineral components in these samples were concentrated with use of a density gradient. The XRD of the concentrated samples displayed an improvement in the intensities for several confirmatory peaks not previously well defined. A review of the data (summarized in Table 2) indicates that both U_3O_8 (sometimes referred to as "pitchblende") and UO_2 (uraninite) are present in these samples. The data in Table 2 agree within experimental uncertainty with the reference values compiled by the JCPDS International Center for Diffraction Data for major diffraction lines, d(Å), in the cited compounds. The experimental diffractogram is complex, and additional minor uranium oxide phases and complexes with iron oxyhydroxide may also be present. However, there is no indication in these samples for uranophane ($Ca(UO_2)_2(SiO_3OH)_2 \cdot 5H_2O$).

The identified phases are low solubility (and low mobility) reduced forms of uranium, commonly present in uranium ore. Uraninite is the main ore mineral in many U deposits; it is very sparingly soluble at normal pH values, especially in reducing groundwaters. In nature, pitchblende is found only in veins of hydrothermal origin and usually contains no thorium and only traces of rare earths. (Katz, J.J.; Rabinowitch, E. (1951). *The Chemistry of Uranium. Part I.* McGraw-Hill.) Synthetic forms of UO_2 and U_3O_8 are important intermediate products in nuclear fuel production [Wymer, R.G.; Vondra, B.L. (1981). Light *Water Reactor Nuclear Fuel Cycle*, CRC press].

Table 2. Comparison of Literature (JCPDS) and Experimental d(Å) XRD Values for Select Phases in Soil Samples

UO_2		$B\text{-}U_3O_8$	
JCPDS 5-0550	Sample Value	JCPDS 23-1460	Sample Value
3.157 *	3.14	8.67	8.72
2.73	2.70	4.15 *	4.15
1.93	1.93	3.53	3.53
1.65	1.65	3.35	3.33
1.58	1.57	2.69	2.69
1.255	1.255	2.60	2.62
1.223	1.225	2.072	2.071
		2.004	1.97

* Most intense line (I/Io = 100%) for pure compound.

SEM-EDS was used to verify the chemical association and morphology of the uranium-bearing components in the samples. The samples are dominated by quartz with minor feldspar, consistent with the XRD results. Samples 02-0866 and 02-0869 contained a relatively high proportion of uranium-bearing grains. Sample 02-0867 contained relatively few uranium-bearing grains, but a significantly greater number of lead-containing grains (about 15 times the number of U grains). Sample 02-0868 contained relatively few uranium-bearing grains. All of the uranium-bearing grains examined evidenced uranium and oxygen, with minor contribution by calcium and silicon components.

Thank you for choosing MCLinc. If you have any questions or need additional information, please contact us at (865) 576-4138.

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W.D Bostick, Ph.D. Date
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October 15, 2002

Mr. David Pohl
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Addendum
MCLinc Project # WES000873
X-Ray Diffraction and Scanning Electron Microscopy Characterization

This memorandum provides some additional detail requested by you on the subject project. As reported by Dr. Bob Stevenson in his Certificate of Analysis, dated 12 August 2002, the uranium-bearing mineral phases identified in the submitted soil sample[1] were uranophane ($Ca(UO_2)_2(SiO_3OH)_2 \cdot 5H_2O$) and metastudite ($UO_4 \cdot 2H_2O$). Uranium in the soil sample was relatively abundant, and the predominant quartz phase served as an internal standard for the XRD peak positions. The two uranium-bearing crystalline phases were comparable in abundance, with somewhat more intense lines observed for the uranophane phase.

Uranophane forms as an oxidative alteration or weathering product of uraninite (UO_2). It is the most abundant uranyl mineral found in nature and possibly the most common U mineral after uraninite. Uranophane precipitates from near neutral to alkaline groundwaters that contain dissolved Si and Ca. It is moderately insoluble in most groundwaters, and thus it displays relatively low mobility in the geosphere.[2]

Metastudite has been found in nature as a pale yellow, nonfluorescent mineral phase. Synthetic metastudite is prepared by the addition of hydrogen peroxide to mildly acidic solutions containing uranyl ion; this reaction is sometimes used within the nuclear fuel cycle to precipitate and recover uranium from leach or waste solutions.[3] Metastudite has a minimal solubility at pH values near 4; however, at near-neutral or alkaline pH values it is considered a relatively soluble compound of uranium.

We hope that this additional information is useful to your groundwater modeling and mobility calculations. We look forward to receiving additional samples for characterization.

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[1] Weston COC ID: 29; WO #03886-518-013-0270
[2] The calculated solubility limit for uranophane as a function of pH is illustrated in Figure 2 of the reference URL www.wmsym.org/wm97proceedings/sess27/27-04.htm

[3] See URL www.radsafe.com/solubility/intro.htm