Examination of Libby, Montana, Fill Material for Background Levels of Amphibole from the Rainy Creek Complex Using Scanning Electron Microscopy and X-Ray Microanalysis

By David T. Adams, William H. Langer, Todd M. Hoefen, Bradley S. Van Gosen, and Gregory P. Meeker

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Abstract

Natural background levels of Libby-type amphibole in the sediment of the Libby valley in Montana have not, up to this point, been determined. The purpose of this report is to provide the preliminary findings of a combined U.S. Geological Survey and U.S. Environmental Protection Agency designed study to constrain the natural background levels of fibrous amphiboles potentially derived from the nearby Rainy Creek Complex. The material selected for this study was sampled from three localities, two of which are active open-pit sand and gravel mines. Seventy samples were collected in total and examined using a scanning electron microscope equipped with an energy dispersive x-ray spectrometer. All samples contained varying amounts of feldspars, ilmenite, magnetite, quartz, clay minerals, pyroxene minerals, and non-fibrous amphiboles such as tremolite, actinolite, and magnesiohornblende. Of the 70 samples collected, only four had detectable levels of fibrous amphiboles compatible with those found in the Rainy Creek complex. The maximum concentraion, identified here, of the amphiboles potentially from the Rainy Creek Complex is 0.083 percent by weight.

Introduction

This Open-File Report provides preliminary findings of a study (U.S. Environmental Protection Agency, 2008) designed by the United States Environmental Protection Agency (EPA) and the U.S. Geological Survey (USGS). This report describes analytical results from samples collected from three localities in Libby, Mont. in June 2008 for the purpose of determining the local background levels of amphibole that could potentially be derived from the nearby Rainy Creek Complex (Boettcher, 1966, 1967). The amphiboles of interest were described in detail by Meeker and others (2003) and consist primarily of winchite, richterite, and tremolite based on the classification of Leake and others (1997). In this report we will refer to these amphiboles collectively as Libby-type amphibole (LA) for convenience.

The samples collected for the study represent the weakly consolidated sediments that were excavated from three Libby, Mont. sites from discrete layers of lake sediment deposited from glacial Lake Kootenai and Pleistocene terrace alluvium. These samples were analyzed to determine the background level of LA in the region, that is, the amount that could be attributed to prehistoric glacial processes. The background levels can help constrain the effects of anthropogenic contamination of soil with LA in the town of Libby and in surrounding areas. The results reported here are part of a larger study by USGS to constrain possible glacial contributions of Libby-type amphiboles to the region. The final report is currently in preparation for publication.

Background

The EPA Region 8 is conducting remediation activities to remove materials contaminated with fibrous and asbestiform amphibole derived from the former vermiculite mine located approximately 6 miles (mi) northeast of Libby, Mont. As part of that investigation, the EPA is determining background levels of LA and has requested the USGS to collect and analyze samples in the Libby area to determine if natural background levels of LA are present, which could be attributed to prehistoric glacial processes.

Sampling

In situ sampling took place in the sediment layers of two open pits (Sand Pit and Clay Pits) where sand and gravel material is actively mined. Sampling also was conducted in an undisturbed field (referred to as "Field") lying directly adjacent to Wooden Field, which was previously a source for fill material. In total, 70 samples were taken from discrete layers in the fill pit walls and from the Field. Many of these samples are shown in figures 1, 2, 3, and 4. Each sample was collected with a new, sterile, plastic sample scoop and placed into a clean plastic zip-top bag to prevent cross-contamination between sampled layers. For pit-wall samples, the surface of the outcrop was scraped back several centimeters to prevent surface contamination prior to collection. At the field location, the top-most organic layer (approximately 2 centimeters (cm) (0.08 inches (in)) was removed and discarded prior to sampling. Then a hole approximately 0.5 meters (m) (approximately 1.6 feet (ft)) in diameter was dug using a narrow-bladed shovel and a stainless steel auger to a depth of 55 to 73 cm (22 to 29 in). The hole walls were scraped back several centimeters using a sterile hand trowel to remove any contamination prior to sample collection. All non-disposable field equipment was thoroughly decontaminated between each use.

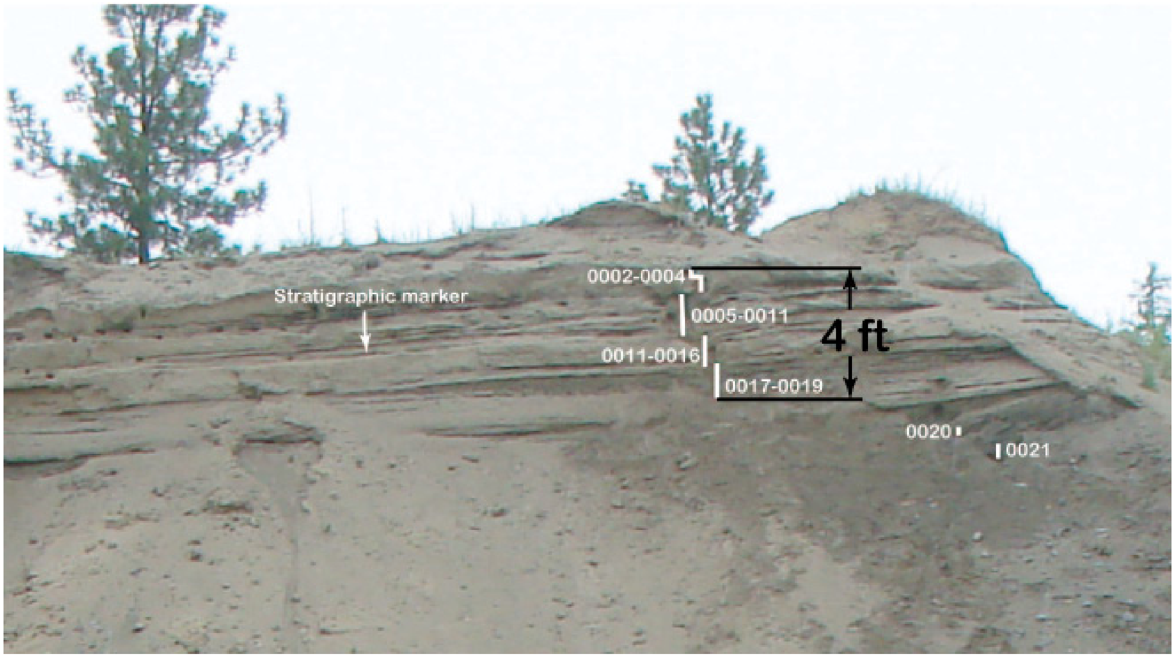

Figure 1. Upper Sand Pit. Samples 0002 through 0021 were obtained from this location.

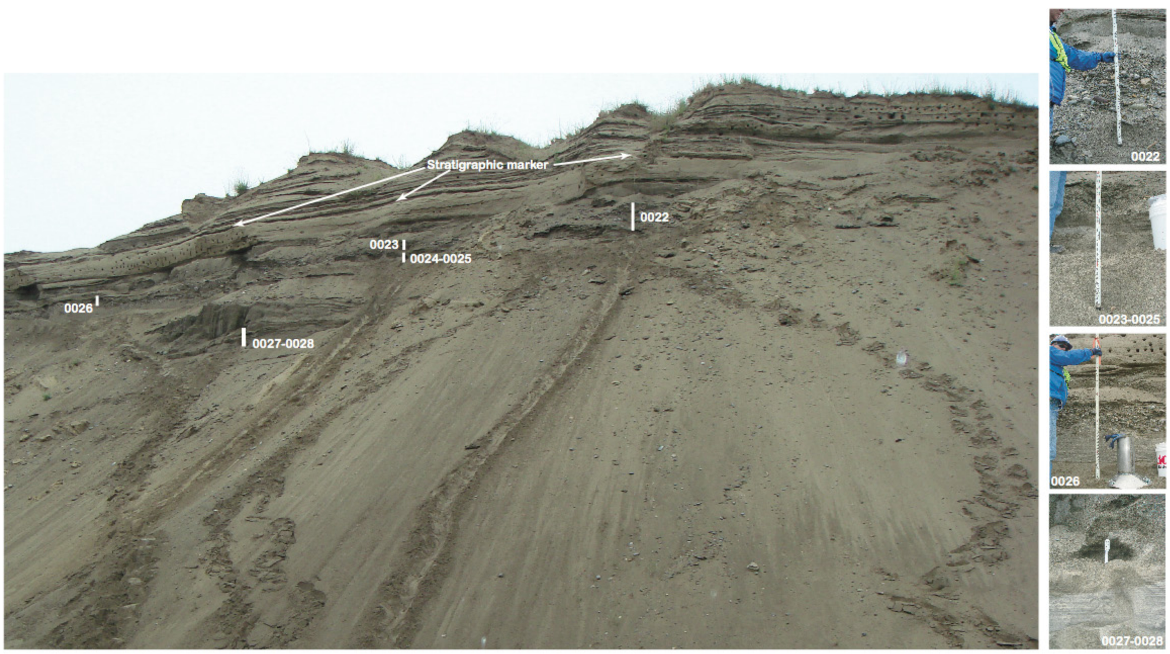

Figure 2. Upper Sand Pit. Samples 0022 through 0028 were obtained from this location.

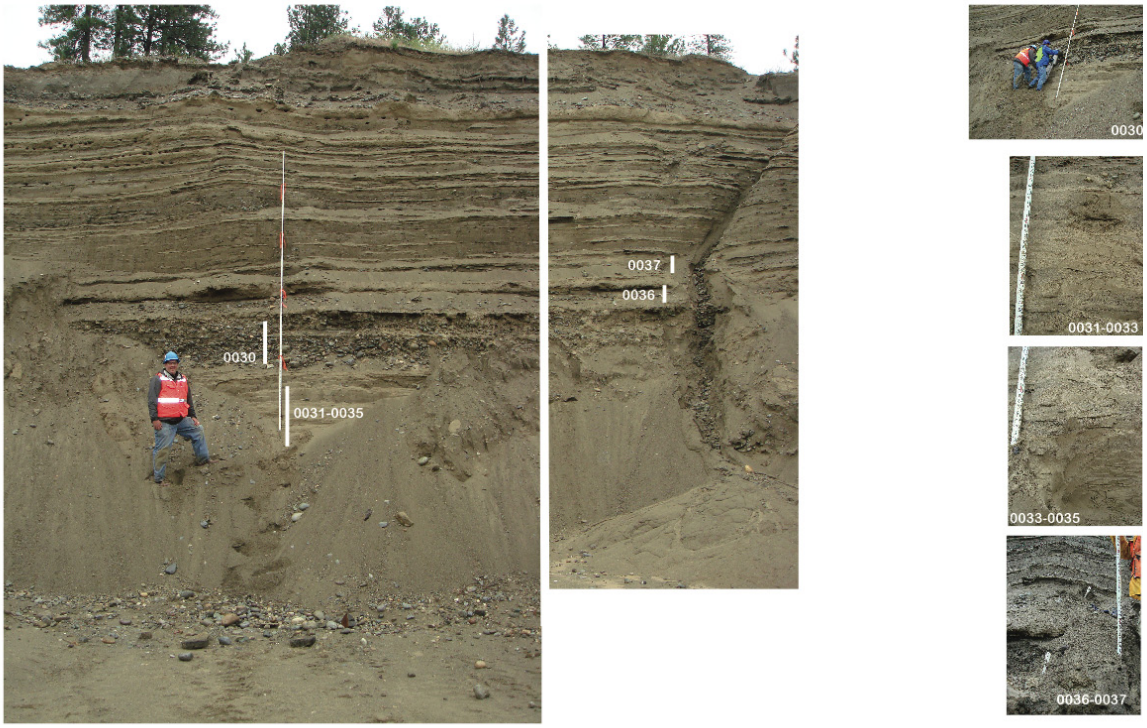

Figure 3. Lower Sand Pit. Samples 0030 through 0037 were collected at this location. Sample 0036 contains very low levels of LA (See table 1).

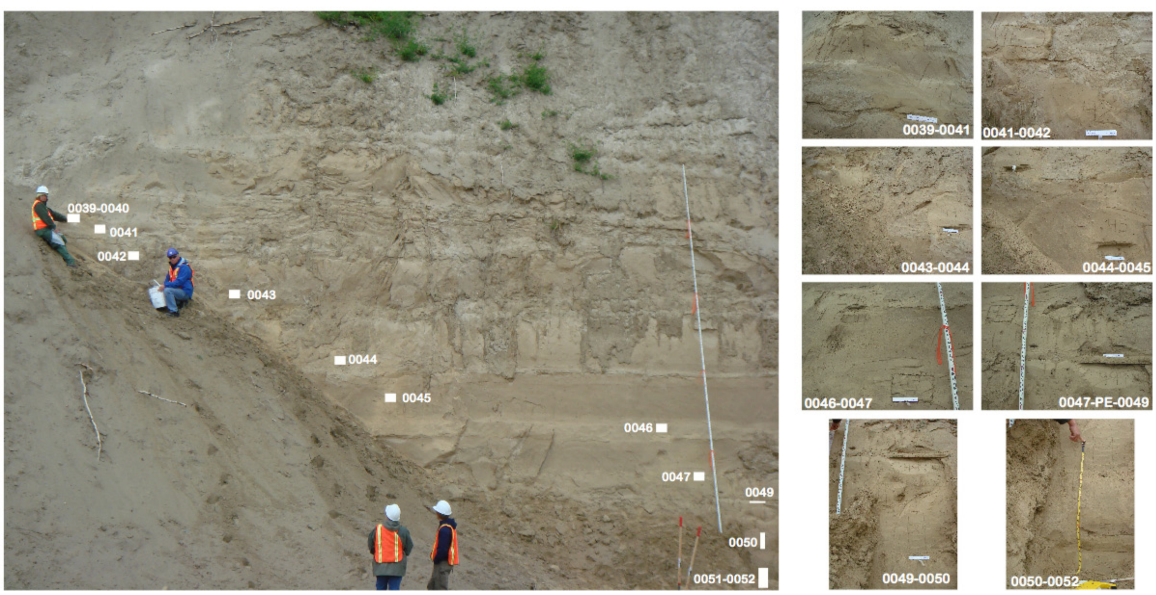

Figure 4. “Clay Pits”. Samples 0039 through 0052 were collected at this location. LA was found to be present in samples 0049 and 0051 (See table 1).

Sample Preparation

Samples were prepared using the methods described in Syracuse Research Corporation (2005) and are as follows:

- Drying: All of the samples collected were wet and required drying. To prevent cross-contamination, the samples were individually air dried in three fume hoods fitted with a HEPA (High-Efficiency Particulate Air) filter and stirred twice daily to facilitate drying. Minimum drying time was 3 days.
- Sample splits: Dried bulk samples were thoroughly mixed in their sampling bags by tumbling for 3 minutes (min). Each sample was then spilt using the cone and quarter technique (Gy, 1992); one of the quarters from each section was then sampled using a stainless steel spoon.
- Sample milling: Prior to milling any samples and between each sample, the ball mills were cleaned using several steps to avoid cross-contamination. Each mill was initially cleaned by milling quartz sand for 3 min and was then rinsed with isopropanol. After cleaning the mills, each was "pre-contaminated" with one heaping teaspoon of the sample, milled for 3 min, then rinsed with isopropanol. Once the mill was pre-contaminated, 3 heaping teaspoons of material were placed into the ball mill and milled for 3 min to ensure the breakup of larger particles and to homogenize the material. Milled samples were placed into individually labeled zip-top bags. After each sample was milled, the ball mill was cleaned with quartz sand and rinsed with isopropanol.
- Scanning Electron Microscope (SEM) Mount Preparation: Approximately 0.50 grams (g) of each milled sample were weighed inside a HEPA-filtered fume hood. Each sample was diluted with 125 milliliters (mL) of deionized (DI) water in an 800 mL beaker while being continuously stirred by a magnetic stir bar. A 45 microliter (µL) aliquot of the suspension material was extracted from the beaker with a 15-µL pipette fitted with a 200-µL pipette tip. The material suspension was seated onto a Whatman® Nuclepore™ 25 millimeter (mm) diameter, 0.2 micrometer (µm) pore size polycarbonate membrane filter using a Millipore® filtration apparatus and hand pump to ensure even distribution onto the filter. The filter was attached to an aluminum SEM stub using high-purity, ultra-thin Nisshin® carbon tabs. Two SEM stubs were created for each sample and each SEM stub was coated with a thin layer of carbon in a carbon evaporator to ensure the conductivity of the sample while under an electron beam in the SEM.

Results

Results of the study, along with sample and locality descriptions and global positioning system (GPS) coordinates, are summarized in table 1. Photographs of sample locations and pit walls are shown in figures 1–4. All samples contained varying amounts of pyroxene, quartz, feldspar, clay minerals, ilmenite, magnetite, rutile, and non-fibrous amphiboles (aspect ratio less than 3:1) such as tremolite, actinolite, and magnesiohornblende.

Table 1. Sample localities and notes.

[LA = Libby Amphibole, PE = Performance Evaluation, ND = Non-detect]

Sample no.	Height of center of sample above reference (ft)	Field description	Notes
Upper Sand Pit #1; 10 June 2008; N. 17594123.822 US ft; E. 1991161.433 US ft; Datum WGS 84, Zone 11 N; Reference Elevation Bottom of stadia rod: 2275 ft			
0002	5.1	Silty sand, cross bedded and ripples; root casts	
0003	4.8	Silty sand, cross bedded and ripples; root casts	
0004	4.5	Silty sand, cross bedded and ripples; root casts	
0005	4.3	Silty sand, cross bedded and ripples; root casts	
0006	4.1	Silty sand, cross bedded and ripples; root casts	
0007	3.9	Silty sand, cross bedded and ripples; root casts	
0008	3.7	Silty sand, cross bedded and ripples; root casts	
0009	3.6	Silty sand, cross bedded and ripples; root casts	
0010	3.4	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0011	3.3	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0012	3.2	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0013	3.0	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0014	2.7	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0015	2.4	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0016	2.2	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	
0017	1.9	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	17 & 18: duplicate samples
0018	1.9	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	17 & 18: duplicate samples
0019	1.1	Silt and very fine sand, subhorizontal layers, each layer about 2–4 mm	1-ft section sampled
0020	-1.0	Coarse sand with few pebbles; dipping foreset beds	
0021	-4.0	Coarse sand with few pebbles; dipping foreset beds	
Upper Sand Pit #2; 10 June 2008; N. 17593942.723 US ft; E. 1990995.142 US ft; Datum WGS 84, Zone 11 N; Reference Elevation Stratigraphic Marker 3.1 ft above base of stadia rod			
0022	-3.2	Pebble cobble gravel with coarse sand; rounded to subrounded; clast supported, no fabric, 3 ft lens	Bulk sample
0023	-4.5	Pebble cobble gravel with coarse sand; rounded to subrounded; clast supported, no fabric, 0.9 ft lens	Bulk sample; 40 ft south of Sample 22
0024	-5.3	Coarse sand, clean, massive	
0025	-6.3	Coarse sand, clean, massive	
Upper Sand Pit #3; 10 June 2008; N. 17593912.937 US ft; E. 1990977.450 US ft; Datum WGS 84, Zone 11 N; Reference Elevation Stratigraphic Marker 3.1 ft above base of stadia rod			
0026	-3.5	Cobble pebble gravel with coarse sand; rounded to subrounded; clast supported, imbricated, 1 ft lens	Bulk sample; 30 ft south of Sample 23

Table 1. Sample localities and notes.—Continued

Sample no.	Height of center of sample above reference (ft)	Field description	Notes
0027	-9.0	Coarse sand with pebbles, clean, horizontal layers, 2 mm thick; contains carbonized wood	16 ft south of Sample 23; 20 ft below bottom of gravel lens
0028	-10.0	Coarse sand with pebbles, clean, horizontal layers, 2 mm thick	
0029			PE sample
Lower Sand Pit #1; 10 June 2008; N. 17593553.394 US ft.; E. 1991723.413 US ft.; Datum WGS 84, Zone 11 N; Reference Elevation: 2177 ft			
0030	6.2	Cobble pebble gravel w/ coarse sand; rounded to subrounded; clast supported, no fabric, 4.5 ft lense	Bulk sample
0031	2.5	Fine sand, sub-horizontal, slight ripples	
0032	1.4	Fine sand, sub-horizontal	
0033	0.9	Fine sand, sub-horizontal	
0034	0.1	Sandy silt, massive	
0035	-0.5	Sandy silt, massive	LA – Lower Bound: 0.0591%, Upper Bound: 0.0815%
Lower Sand Pit #1; 10 June 2008; N. 17593553.394 US ft; E. 1991723.413 US ft; Datum WGS 84, Zone 11 N; Reference Elevation: 2177 ft			
0036	9.0	Clayey silt, 8 in thick, immediately above top of sample 30	30 ft east of stadia rod LA: Lower Bound: 0.059%, Upper Bound: 0.082%
0037	12.0	Pebbly sand	30 ft east of stadia rod
Clay Pits #1; 11 June 2008; N. 17584475.745 US ft; E. 1996916.646 US ft; Datum WGS 84, Zone 11 N; Reference Elevation: 2128 ft			
0038			PE sample
0039	20	Clayey silt	39 & 40 duplicate samples
0040	20	Clayey silt	39 & 40 duplicate samples
0041	18.5	Silty clay	
0042	17.0	Very fine sand, massive, iron concretions, fines upwards	
0043	15.5	Very fine sand, massive, iron concretions, fines upwards	8 ft south of 0042
0044	11.0	Massive silt with granular concretions	6 ft south of 0043
Clay Pits #2; 11 June 2008; N. 17584458.567 US ft; E. 1996916.373 US ft; Datum WGS 84, Zone 11 N; Reference Elevation: 2128 ft			
0045	8.5	Very fine sand, trace of silt, massive; numerous $CaCO_3$ concretions	3 ft south of 0044; 18.5 north of rod
0046	6	Sandy silt, massive	2.5 ft north of rod
0047	3.6	Silt to very fine sand; turbidity layer	1 ft north of rod
0048			PE sample

Table 1. Sample localities and notes.—Continued

Sample no.	Height of center of sample above reference (ft)	Field description	Notes
Clay Pits #3; 11 June 2008; N. 17584437.828 US ft; E. 1996918.196 US ft; Datum WGS 84, Zone 11 N; Reference Elevation: 2128 ft			
0049	2.2	Clay layer, 2.25 in thick	2.5 ft south of rod; LA: Lower bound: 0.023%, Upper bound: 0.050%
0050	-0.3	Silt, massive	2.5 ft south of rod
0051	-2.3	Clay, some silt; massive	2.5 ft south of rod; LA: Lower bound: ND, Upper bound: 0.037%
0052	-2.7	Clay, silty; massive	2.5 ft south of rod
Number in Ht. column for Field samples is sample interval in inches below reference elevation. Field #1; 11 June 2008; N. 17590529.663 US ft; E. 1987980.432 US ft; Datum WGS 84, Zone 11 N; Reference Elevation Land Surface Datum (LSD): 2070 ft			
0053	0–2	Organics	
0054	2–4	Cobble pebble gravel; A horizon	
0055	4–5	Cobble pebble gravel; B horizon	
0056	9–13	Cobble pebble gravel; B horizon	
0057	13–14	Cobble pebble gravel; B horizon	
0058	14–17	Cobble pebble gravel; B horizon	
0059	18–20	Cobble pebble gravel; B/C horizon transition	
0060			PE sample
0061	20–22	Cobble pebble gravel; B/C horizon transition	LA: Lower Bound 0.004%, Upper bound: 0.047%
0062	22–29	Cobble pebble gravel; B/C horizon transition	
Field #2; 11 June 2008; N. 17590457.826 US ft; E. 1987988.022 US ft; Datum WGS 84, Zone 11 N; Reference Elevation LSD: 2070 ft			
0063	0–4	Organics / A horizon	
0064			No sample taken
0065	4–7	Cobble pebble gravel; B horizon	
0066	7–10	Cobble pebble gravel; B horizon	
0067	10–13	Cobble pebble gravel; B horizon	
0068	13–16	Cobble pebble gravel; B horizon	67 & 68 duplicate samples
0069	13–16	Cobble pebble gravel; B horizon	
0070	16–29	Cobble pebble gravel; B/C horizon transition	
Field #3; 11 June 2008; N. 17590388.160 US ft; E. 1988008.336 US ft; Datum WGS 84, Zone 11 N; Reference Elevation LSD: 2070 ft			
0071	0–2	Organic	
0072	2–6	Cobble pebble gravel; A horizon	
0073	6–14	Cobble pebble gravel; B horizon	
0074	14–16	Cobble pebble gravel; B horizon	
0075	16–19	Cobble pebble gravel; B/C horizon	
0076	19–22	Cobble pebble gravel; B/C horizon	

Of the 70 samples collected and analyzed, only four samples contained amphibole compatible with amphibole derived from the once-active mine area of the Rainy Creek Complex (see Figure 6, Meeker and others, 2003). The detected LA was, in all cases, less than 0.1 percent by weight. The Sand Pit contained only one layer with detected LA and it is located in the lower portion of the pit in a clayey silt layer (elevation 2,286 ft). Two samples (elevations 2,126 ft and 2,130 ft) from the Clay Pits contain LA; these samples also came from the lower portion of the sampled wall at the base of the exposed cliff face and range in LA concentrations from non-detect to 0.05 percent (table 1). The final sample containing LA is from the Field, adjacent to the Wooden Field, where fill soil had been previously quarried. The sample containing LA from this locality was 20–22 in. below the surface and was at a concentration ranging from approximately 0.004 percent to approximately 0.047 percent by weight.

Conclusions

Seventy samples representing discrete individual stratigraphic layers were collected from three localities in or near the city of Libby, Mont. Each sample contained varying amounts of feldspars, ilmenite, magnetite, quartz, clay minerals, pyroxene minerals, and non-fibrous amphiboles such as tremolite, actinolite, and magnesiohornblende; this mineral assemblage is expected from the geology of this region. In general, background levels for LA are below detection limit except in four samples (Samples 0036, 0049, 0051, 0061). Measured concentrations of LA were: 0.059 percent to 0.082 percent at 2,186 ft elevation from the Lower Sand Pit; non-detect to 0.037 percent at 2,126 ft elevation; and 0.023 percent to 0.050 percent at 2,130 ft elevation from the Clay Pits; and 0.004 percent to 0.047 percent at 22 in below 2,070 ft elevation.

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