

Appendix H  
Derivation of a Site-Specific  
Oral Bioavailability Adjustment Factor  
for Arsenic in Soil

# Appendix H

## Derivation of a Site-Specific Oral Bioavailability Adjustment Factor for Arsenic in Soil

### Background

Oral bioavailability is a measure of the amount of a constituent that is absorbed into the body after ingestion exposure. Some constituents are absorbed almost completely (100 percent bioavailability) when ingested in their pure, soluble form. Others may pass through the body largely unabsorbed. The oral bioavailability of soil-bound arsenic largely depends on the rate at which it dissociates from the soil matrix in the gastrointestinal (GI) tract. Soil-bound arsenic is usually absorbed by the GI tract to a lesser degree than when in the pure, soluble form. This reduced absorption results from the affinity between arsenic and the soil matrix, the low solubility of the chemical form of arsenic associated with the soil, or both. The issue of bioavailability of arsenic is especially important at mining, milling, and smelting sites. This is because the arsenic at these sites often exists, at least in part, as a poorly soluble sulfide, and may also occur in particles of inert or insoluble material. These factors collectively tend to reduce the bioavailability of arsenic. Thus, the bioavailability of arsenic from Iron King Mine – Humboldt Smelter Superfund Site (the Site) soil is expected to be low for constituents that are tightly bound within the soil matrix and/or are in a form that is insoluble in the GI tract under physiological conditions.

When animals are exposed to arsenic, the degree of absorption from the GI tract is reflected in the degree of toxicity. Because the toxicity values used for risk assessment are derived from studies where humans are exposed to arsenic in drinking water containing a totally soluble form of arsenic, overestimates of risk can result if site soil contains a form that is less bioavailable than the form in these studies. Given these findings, the risk assessment presented in this report adjusts oral exposure to account for this reduced bioavailability.

This appendix describes the approach and methods used to identify a site-specific oral bioavailability adjustment factor for arsenic from soil at the Site. The approach used is consistent with the U.S. Environmental Protection Agency (EPA) *Guidance for Evaluating the Oral Bioavailability of Metals in Soils for Use in Human Health Risk Assessment* (EPA, 2007a) and *Framework for Metals Risk Assessment* (EPA, 2007b).

The site-specific bioavailability adjustment factor was identified using the following steps, as described in the following subsections:

- **Step 1.** Measure the *in vitro* bioaccessible fraction of arsenic from 72 soil samples using laboratory extraction tests.
- **Step 2.** Measure the *in vivo* bioavailable fraction of arsenic from a limited but representative number of soil samples using live animals.
- **Step 3.** Establish the relationship between the bioavailable and bioaccessible fractions of arsenic.
- **Step 4.** From Steps 1 through 3, identify a sitewide high-end estimate of the bioavailable fraction of arsenic.

## Step 1. *In Vitro* Bioaccessibility Evaluation

The ability of inorganic arsenic in soil at the site to be extracted under laboratory conditions that simulate physiological conditions (for example, gastric pH, buffering, temperature, stomach emptying time, etc.) should approximate the relative bioavailability from the site soil. This measured extracted fraction is referred to as bioaccessible arsenic.

The portion of total arsenic that is extractable under these conditions is referred to as *in vitro* bioaccessible arsenic (IVBA), where:

$$\text{IVBA (\%)} = (\textit{in vitro} \text{ extractable arsenic mg/kg soil} \div \text{total arsenic mg/kg soil}) \times 100.$$

For the remedial investigation (RI), IVBA measurements were obtained from a total of 72 soil samples collected across the Site from a combination of residential and non-residential areas (gulch areas, Humboldt Smelter area, and main tailings pile [MTP]). The soil samples were extracted according to EPA Method 9200.2-86 *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil* dated April 2012. Sample extractions and analyses were conducted by a combination of the EPA Region 9 Laboratory and ACZ Laboratory in Steamboat Springs, Colorado. Standard Operating Procedure (SOP) 256, used by the Region 9 Laboratory, is provided here in Attachment H-1.

The soil samples were homogenized, dried at 37 degrees Celsius (°C), and then sieved to less than (<) 250 micrometers (µm). A 1-gram aliquot of sample was rotated end-over-end in 100 milliliters (ml) of 0.4M glycine at pH 1.5 for 1 hour in an environmental chamber controlled at 37 °C. The extract was filtered immediately (within 30 minutes) and held at 0 to 6 °C until digestion and analysis for arsenic by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) (EPA, 2007c). The laboratory reports for all *in vitro* bioaccessibility extractions are provided in Attachment H-2. The <250-µm size fraction was used because this particle size is representative of that which adheres to children's hands (EPA, 2000).

The test results are summarized in Table H-1, which lists the total arsenic, the extractable arsenic, and the estimated bioaccessible fraction for each soil sample. The total arsenic concentrations in the test samples ranged from 29 milligrams per kilogram (mg/kg) to 6,899 mg/kg, spanning the range of levels typically seen during the RI. The bioaccessible fraction of arsenic does not appear to be concentration-dependent with respect to total arsenic. The bioaccessible fraction results from sample locations 515 (57.3 percent) and HSJ-583 (53.6 percent) were the highest seen. However, these two samples were in locations where known efflorescent salts have been observed during site investigations; they were therefore considered outliers and were not used to estimate the sitewide bioavailable fraction of arsenic (Step 4).

## Step 2. *In Vivo* Mouse Bioavailability Evaluation

Laboratory mouse assays were conducted in 2011 and 2014 to determine the actual bioavailability of arsenic in live animals (*in vivo*) for a limited number of Site soil samples<sup>1</sup>. Due to the expense and time constraints for these studies, only a limited number of samples (a total of nine) were tested. From these same samples, the IVBA was also measured. The overall objective was to establish a defensible, site-specific relationship between *in vitro* bioaccessibility and *in vivo* bioavailability of arsenic in the Site soil.

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<sup>1</sup> *In vivo* bioavailability studies with juvenile swine have also been conducted using Site soil from locations HSJ-583 and IJK-583, and the comparison with results from mice is discussed elsewhere (Bradham et al., 2011; EPA, 2012b).

The results of the *in vivo* assays are expressed in terms of relative bioavailability (RBA). RBA is calculated as the ratio of the bioavailability of arsenic from a soil-amended diet to the bioavailability of arsenic from a diet containing sodium arsenate (which is expected to be completely available). RBA is commonly expressed on a percentage basis:

$$\text{RBA (\%)} = \left( \frac{\text{arsenic bioavailability from IKM diet}}{\text{arsenic bioavailability from sodium arsenate diet}} \right) \times 100.$$

Soil samples were sent to EPA’s Office of Research and Development (ORD) for *in vivo* bioavailability testing, which was conducted by Karen Bradham (ORD, Research Triangle Park, North Carolina). The RBA of arsenic was estimated for nine surface soil samples with arsenic concentrations ranging from 39 to 6,899 mg/kg, using the repeated dose steady-state urinary excretion fraction method (Bradham et al., 2011, 2013, 2015). During the test, 4- to 6-week-old female C57BL/6 mice were fed diets containing the test soil or sodium arsenate. The test soil and sodium arsenate groups typically consisted of 12 mice that were housed in metabolic cages containing three mice per cage. The test soil was mixed into the powdered AIN-93G purified rodent diet to achieve a 1 percent (w/w) soil:diet ratio. Mice received the diets for a period of 10 days, during which urine and feces were collected daily. Arsenic concentrations in diet, soil, urine, and feces were determined by Instrumental Neutron Activation Analysis (INAA). Daily arsenic dosages were estimated from measurements of daily diet consumption. Arsenic RBA was estimated as the ratio of the urinary excretion fraction (UEF) for soil arsenic and sodium arsenate treatment groups, where the UEF was the cumulative urinary arsenic (µg) excretion divided by the cumulative arsenic dose (µg).

The RBA and IVBA results of the paired *in vivo* and *in vitro* tests are provided in Table H-2, along with the total arsenic concentrations in the soil samples tested. These results were used for the regression analysis to identify the site-specific relationship between IVBA and RBA (Step 3), as described in the next subsection.

## Step 3. Regression Analysis of Bioavailable and Bioaccessible Fractions

The RBA and IVBA results of the paired *in vivo* and *in vitro* tests from Step 3 were subjected to linear regression analysis to determine the degree of correlation between the data sets, and to identify the mathematical relationship describing the correspondence. The regression results are depicted on Figure H-1. The results indicate that there is strong correlation between the RBA and IVBA results, with a coefficient of determination (R-squared) of 0.8. This indicates that IVBA can serve as a good predictor for bioavailability of arsenic in soil at the Site. The linear regression relationship between IVBA and RBA is described as follows:

$$\%RBA = 0.5498(\%IVBA) + 7.6236 \quad (R^2 = 0.8034)$$

This relationship allows for transformation of all the IVBA results from the 72 Site soil samples where bioaccessibility has been measured. Figure H-2 shows the locations for the IVBA samples at the Site. The value shown with each location is the predicated bioavailability (transformed result after site-specific regression) for that location. Sample points in residential yards are colored yellow; points in non-residential areas are colored green; and the two efflorescent salt samples are colored purple. The strong site-specific regression adds to the defensibility of the bioavailability estimates and risk calculations for the RI, as does the large number of points where IVBA has been measured, and the substantial amount of data from actual residential yards (point of exposure).

## Step 4. Selection of Site-Specific Bioavailability Adjustment Factor for Arsenic in Soil

As previously indicated, the IVBA (and hence the predicted oral bioavailability) across the site does not appear to be concentration-related (Table H-1). Moreover, a comparison of residential soil and non-residential soil results does not indicate systematic differences between these property types. The bioavailability value is arguably well-constrained. Given these observations, the data from the entire data set were pooled to identify a site-specific oral bioavailability adjustment factor for Site soil.

Table H-3 provides the 50<sup>th</sup>, 90<sup>th</sup>, 95<sup>th</sup>, and 99<sup>th</sup> percentile values for the estimated bioavailable fraction for the pooled data set. To provide a health-conservative estimate of site-specific bioavailability, the 95<sup>th</sup> percentile value of 22.5 percent was selected as the site-specific oral bioavailability adjustment factor for use in the human health risk assessment (Section 9 of the RI main report).

The site-specific oral bioavailability adjustment factor of 22.5 percent is used to adjust the oral exposure from total arsenic measured in all soil and sediment samples included in the human health and ecological risk assessments. These results indicate that the forms of arsenic in soil at the Site are of relatively low bioavailability, when compared to the EPA default value of 60 percent (EPA, 2012a).

It should be noted that the oral bioavailability adjustment factor derived herein is intended to be a site-specific value, and is not intended for general use at other sites.

## References

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Tables

TABLE H-1

**Oral Bioaccessibility of Arsenic in Soil***Iron King Mine – Humboldt Smelter Superfund Site, Dewey-Humboldt, Yavapai County, Arizona*

<b>Sample</b>	<b>Date Sampled</b>	<b>Current Property Type</b>	<b>Total Arsenic (mg/kg)</b>	<b>Extractable Arsenic<sup>a</sup> (mg/kg)</b>	<b>Bioaccessible Arsenic (percent)</b>
IJK-525-0-2	8/20/2008	Non-residential	6,899	NR	17.5
HSJ 501-0-2	9/4/2008	Non-residential	173	NR	6.80
OSF-118-1	9/18/2008	Residential	244	NR	18.1
HSJ-583	5/2/2009	Non-residential <sup>b</sup>	280	NR	53.6
IJK-583	5/2/2009	Non-residential	4,495	NR	8.80
417	4/30/2013	Non-residential	2,550	369	14.5
431	4/30/2013	Non-residential	447	105	23.5
442	4/30/2013	Non-residential	2,990	37.9	1.27
451	4/30/2013	Non-residential	585	138	23.6
467	4/30/2013	Non-residential	1,480	170	11.5
477	4/30/2013	Non-residential	3,580	84.4	2.36
485	4/30/2013	Non-residential	4,180	267	6.39
486	4/30/2013	Non-residential	1,750	107	6.11
513	4/29/2013	Non-residential	888	186	20.9
515	4/29/2013	Non-residential <sup>b</sup>	3,960	2,270	57.3
527	4/29/2013	Non-residential	6,730	312	4.64
621	4/30/2013	Non-residential	310	12.4	4.00
642	5/1/2013	Non-residential	240	ND	<1.0
647	5/1/2013	Non-residential	190	6.40	3.37
648	5/1/2013	Residential	220	16.0	7.27
669	5/1/2013	Residential	305	31.4	10.3
701	5/2/2013	Non-residential	841	125	14.9
750	5/1/2013	Non-residential	29	1.70	5.86
753	5/1/2013	Non-residential	300	110	36.7
820	5/1/2013	Non-residential	660	112	17.0
861	5/1/2013	Non-residential	497	28.1	5.65
865	5/1/2013	Non-residential	649	62.7	9.66
873	5/1/2013	Non-residential	680	77.4	11.4
879	5/1/2013	Non-residential	892	116	13.0
978	5/1/2013	Non-residential	240	ND	<1.0
979	4/29/2013	Non-residential	480	15.0	3.13
980	4/29/2013	Non-residential	3,700	510	13.8
13330WellsSt	7/11/2013	Residential	441	95.9	21.7
13336WellsSt	7/11/2013	Residential	387	84.2	21.8
GulchYard	7/11/2013	Non-residential	330	43.0	13.0
106-04	2/26/2014	Residential	250	23.0	9.20
108-03	2/24/2014	Residential	420	110	26.2
109-11	2/19/2014	Residential	170	47.0	27.6
126-14	2/27/2014	Residential	180	13.0	7.22
2014-08	1/31/2014	Residential	310	45.0	14.5
2216-02	3/5/2014	Residential	280	17.0	6.07
2324-03	2/5/2014	Residential	230	42.0	18.3
2328-02	2/5/2014	Residential	780	130	16.7
2408-01	3/10/2014	Residential	220	24.0	10.9
2410-03	3/10/2014	Residential	290	28.0	9.66
2426-09	2/5/2014	Residential	340	18.0	5.29
2519-10	3/10/2014	Residential	160	20.0	12.5
2523-05	2/19/2014	Residential	170	31.0	18.2



TABLE H-1

**Oral Bioaccessibility of Arsenic in Soil***Iron King Mine – Humboldt Smelter Superfund Site, Dewey-Humboldt, Yavapai County, Arizona*

<b>Sample</b>	<b>Date Sampled</b>	<b>Current Property Type</b>	<b>Total Arsenic (mg/kg)</b>	<b>Extractable Arsenic<sup>a</sup> (mg/kg)</b>	<b>Bioaccessible Arsenic (percent)</b>
2602-09	2/13/2014	Residential	140	24.0	17.1
2615-03	2/20/2014	Residential	1,200	49.0	4.08
2743D-11	2/24/2014	Residential	650	39.0	6.00
2755-07	2/22/2014	Residential	150	11.0	7.33
2808-15	2/21/2014	Residential	410	15.0	3.66
2901-06	2/26/2014	Residential	160	15.0	9.38
3004-08	3/3/2014	Residential	260	51.0	19.6
3005-18	3/4/2014	Residential	230	52.0	22.6
GAL-01	2/28/2014	Non-residential	1,300	41.0	3.15
GAL-02	2/28/2014	Non-residential	170	7.10	4.18
GAL-03	2/28/2014	Non-residential	710	24.0	3.38
GAL-04	2/28/2014	Non-residential	2,700	930	34.4
GAL-04 FD	2/28/2014	Non-residential	2,500	920	36.8
GAL-05	2/28/2014	Non-residential	650	66.0	10.2
MTP-01	2/27/2014	Non-residential	5,100	170	3.33
MTP-02	2/27/2014	Non-residential	4,300	400	9.30
MTP-03	2/27/2014	Non-residential	310	59.0	19.0
MTP-04	2/27/2014	Non-residential	1,800	340	18.9
MTP-05	2/27/2014	Non-residential	1,300	390	30.0
MTP-06	2/27/2014	Non-residential	2,100	260	12.4
MTP-07	2/27/2014	Non-residential	1,000	230	23.0
MTP-08	2/27/2014	Non-residential	1,500	360	24.0
MTP-09	2/27/2014	Non-residential	2,700	650	24.1
MTP-10	2/27/2014	Non-residential	890	110	12.4

Notes:

<sup>a</sup> Gastric-phase extraction using EPA Method 9200.2-86 (0.4 M glycine, pH 1.5).<sup>b</sup> Known efflorescent salts have been observed at this location, and these results were not used for the regression analysis.

mg/kg = milligrams per kilogram

ND = not detected

NR = not reported

TABLE H-2

**Test Results of In Vivo Oral Bioavailability of Arsenic from Iron King Mine Soil***Iron King Mine – Humboldt Smelter Superfund Site, Dewey-Humboldt, Yavapai County, Arizona*

Sample	Total Arsenic <sup>a</sup> (mg/kg)	<i>In vivo</i>		<i>In vitro</i>	
		Relative Bioavailable Arsenic <sup>b</sup> (percent)	± Standard Deviation (percent)	Bioaccessible Arsenic <sup>c</sup> (percent)	± Standard Deviation (percent)
OSF-118-1	244	15.9	1.8	18.1	0.4
HSJ 501-0-2	173	14.4	1.6	6.8	0.8
IJK-525-0-2	6,899	15.0	1.4	17.5	0.6
HSJ-583	280	40.9	2.6	53.6	0.2
IJK-583	4,495	14.8	1.5	8.8	0.1
IK B10-4	448	17	0.7	25	0.6
IK B10-5	195	18.6	4.2	27.3	0.5
IK B10-6/7	446	17.2	1.2	22	0.3
HSJ-504-02	39	22.9	8	17.5	1.1

## Notes:

<sup>a</sup> Determined by Instrumental Neutron Activation Analysis.<sup>b</sup> From in vivo mouse assays (Bradham et al., 2011).<sup>c</sup> Gastric phase extraction using EPA Method 9200.2-86 (0.4 M glycine, pH 1.5).

mg/kg = milligrams per kilogram

TABLE H-3

**Estimates of Oral Bioavailability of Arsenic in Soil***Iron King Mine – Humboldt Smelter Superfund Site, Dewey-Humboldt, Yavapai County, Arizona*

<b>Sample</b>	<b>Date Sampled</b>	<b>Current Property Type</b>	<b>Bioaccessible Arsenic<sup>a</sup> (percent)</b>	<b>Bioavailable Arsenic<sup>b</sup> (percent)</b>
IJK-525-0-2	8/20/2008	Non-residential	17.5	17.2
HSJ 501-0-2	9/4/2008	Non-residential	6.80	11.4
OSF-118-1	9/18/2008	Residential	18.1	17.6
IJK-583	5/2/2009	Non-residential	8.80	12.5
417	4/30/2013	Non-residential	14.5	15.6
431	4/30/2013	Non-residential	23.5	20.5
442	4/30/2013	Non-residential	1.27	8.3
451	4/30/2013	Non-residential	23.6	20.6
467	4/30/2013	Non-residential	11.5	13.9
477	4/30/2013	Non-residential	2.36	8.9
485	4/30/2013	Non-residential	6.39	11.1
486	4/30/2013	Non-residential	6.11	11.0
513	4/29/2013	Non-residential	20.9	19.1
527	4/29/2013	Non-residential	4.64	10.2
621	4/30/2013	Non-residential	4.00	9.8
642	5/1/2013	Non-residential	<1.0	<8.2
647	5/1/2013	Non-residential	3.37	9.5
648	5/1/2013	Residential	7.27	11.6
669	5/1/2013	Residential	10.3	13.3
701	5/2/2013	Non-residential	14.9	15.8
750	5/1/2013	Non-residential	5.86	10.8
753	5/1/2013	Non-residential	36.7	27.8
820	5/1/2013	Non-residential	17.0	17.0
861	5/1/2013	Non-residential	5.65	10.7
865	5/1/2013	Non-residential	9.66	12.9
873	5/1/2013	Non-residential	11.4	13.9
879	5/1/2013	Non-residential	13.0	14.8
978	5/1/2013	Non-residential	<1.0	<8.2
979	4/29/2013	Non-residential	3.13	9.3
980	4/29/2013	Non-residential	13.8	15.2
13330WellsSt	7/11/2013	Residential	21.7	19.6
13336WellsSt	7/11/2013	Residential	21.8	19.6
GulchYard	7/11/2013	Non-residential	13.0	14.8
106-04	2/26/2014	Residential	9.20	12.7
108-03	2/24/2014	Residential	26.2	22.0
109-11	2/19/2014	Residential	27.6	22.8
126-14	2/27/2014	Residential	7.22	11.6
2014-08	1/31/2014	Residential	14.5	15.6
2216-02	3/5/2014	Residential	6.07	11.0
2324-03	2/5/2014	Residential	18.3	17.7
2328-02	2/5/2014	Residential	16.7	16.8
2408-01	3/10/2014	Residential	10.9	13.6
2410-03	3/10/2014	Residential	9.66	12.9
2426-09	2/5/2014	Residential	5.29	10.5
2519-10	3/10/2014	Residential	12.5	14.5
2523-05	2/19/2014	Residential	18.2	17.6
2602-09	2/13/2014	Residential	17.1	17.0
2615-03	2/20/2014	Residential	4.08	9.9
2743D-11	2/24/2014	Residential	6.00	10.9

TABLE H-3

**Estimates of Oral Bioavailability of Arsenic in Soil***Iron King Mine – Humboldt Smelter Superfund Site, Dewey-Humboldt, Yavapai County, Arizona*

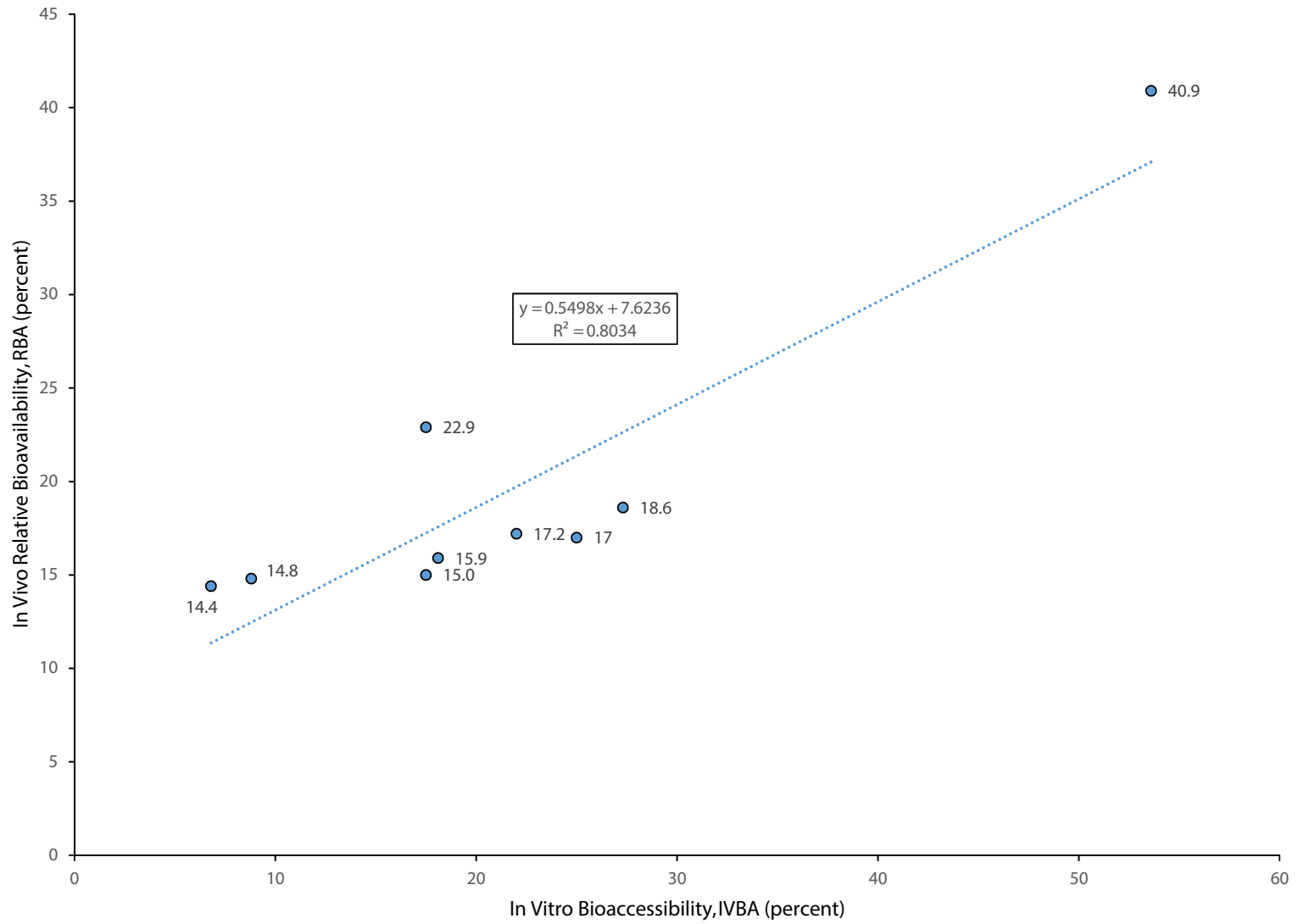
<b>Sample</b>	<b>Date Sampled</b>	<b>Current Property Type</b>	<b>Bioaccessible Arsenic<sup>a</sup> (percent)</b>	<b>Bioavailable Arsenic<sup>b</sup> (percent)</b>
2755-07	2/22/2014	Residential	7.33	11.7
2808-15	2/21/2014	Residential	3.66	9.6
2901-06	2/26/2014	Residential	9.38	12.8
3004-08	3/3/2014	Residential	19.6	18.4
3005-18	3/4/2014	Residential	22.6	20.1
GAL-01	2/28/2014	Non-residential	3.15	9.4
GAL-02	2/28/2014	Non-residential	4.18	9.9
GAL-03	2/28/2014	Non-residential	3.38	9.5
GAL-04 FD	2/28/2014	Non-residential	36.8	27.9
GAL-05	2/28/2014	Non-residential	10.2	13.2
MTP-01	2/27/2014	Non-residential	3.33	9.5
MTP-02	2/27/2014	Non-residential	9.30	12.7
MTP-03	2/27/2014	Non-residential	19.0	18.1
MTP-04	2/27/2014	Non-residential	18.9	18.0
MTP-05	2/27/2014	Non-residential	30.0	24.1
MTP-06	2/27/2014	Non-residential	12.4	14.4
MTP-07	2/27/2014	Non-residential	23.0	20.3
MTP-08	2/27/2014	Non-residential	24.0	20.8
MTP-09	2/27/2014	Non-residential	24.1	20.9
MTP-10	2/27/2014	Non-residential	12.4	14.4
			50th percentile	13.9
			90th percentile	20.6
			<b>95th percentile</b>	<b>22.5</b>
			99th percentile	27.8

## Notes:

<sup>a</sup> Gastric phase extraction using EPA Method 9200.2-86 (0.4 M glycine, pH 1.5).<sup>b</sup> Estimated from bioaccessible arsenic using regression equation (see Figure H-1): %RBA = 0.5498(%IVBA) + 7.6236 (R<sup>2</sup> = 0.8034).

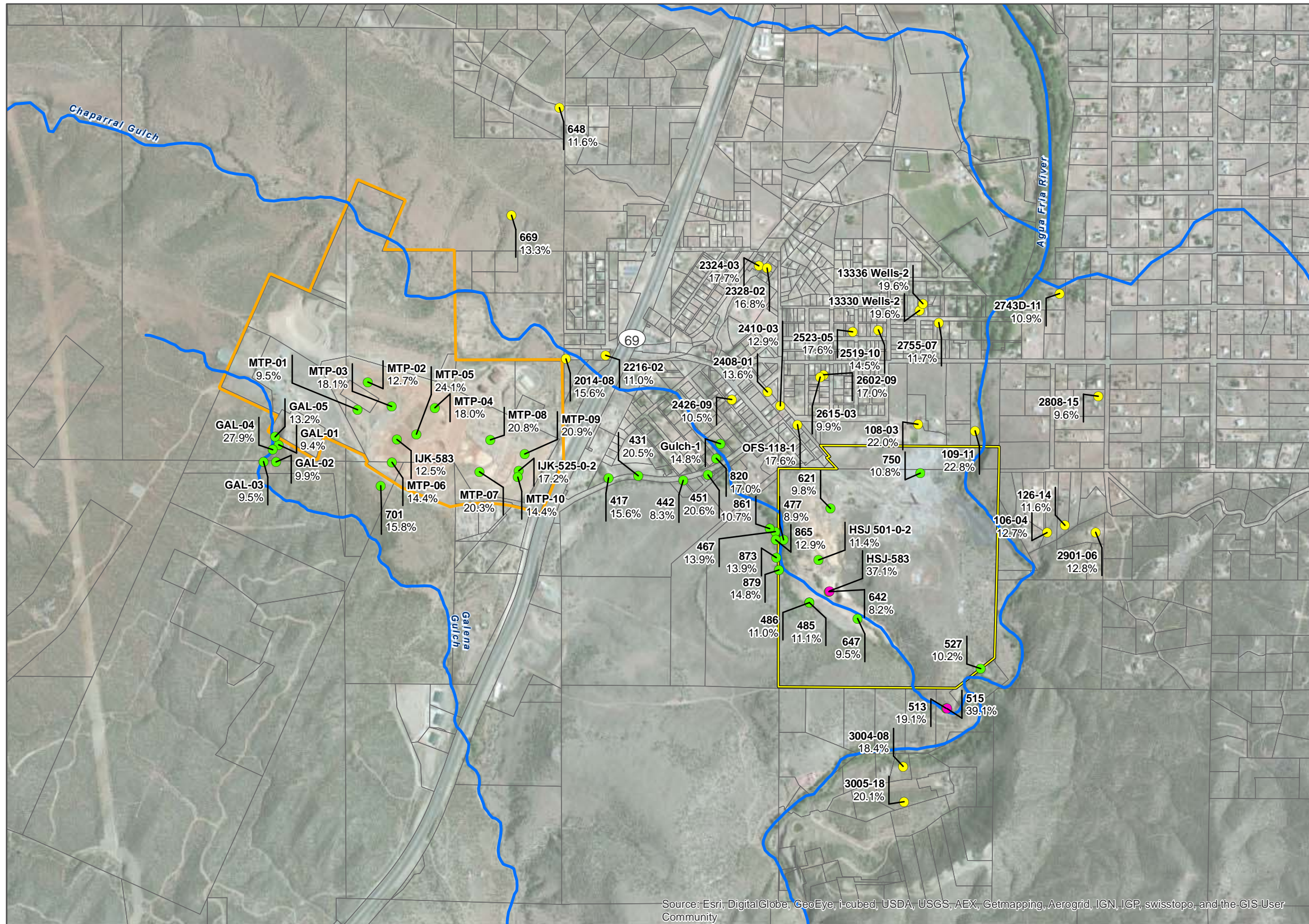
Bold = Bioavailability adjustment factor used for the Iron King Mine Remedial Investigation Human Health Risk Assessment.

Figures



**Figure H-1**  
**Correlation Between Arsenic IVBA and RBA for Soil**  
*Iron King Mine – Humboldt Smelter Superfund Site*  
*Dewey-Humboldt, Yavapai County, Arizona*



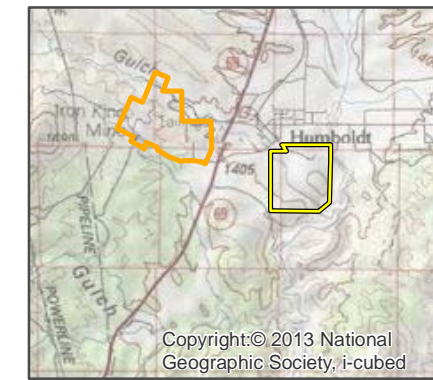
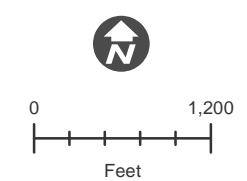


Parcels Yavapai County  
 Iron King Mine  
 Humboldt Smelter  
 Streams

**Sample Locations**

- Known Efflorescent Salts
- Non-Residential
- Residential

Sample Location — 109-11  
 Bioavailability AS (%) — 22.8%



**Figure H-2**  
**Estimated Bioavailability of Arsenic in Soil**  
**(Based on Site-Specific Regression)**  
 Iron King Mine – Humboldt Smelter Superfund Site  
 Dewey-Humboldt, Yavapai County, Arizona






Attachment H-1  
Standard Operating Procedure 256  
EPA Region 9 Laboratory




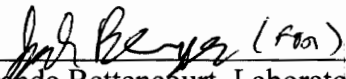
USEPA REGION 9 LABORATORY  
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 256  
BIOACCESSIBILITY EXTRACTION

Revision 1  
Effective Date: June 3, 2011

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## 1 SCOPE AND APPLICABILITY

This SOP provides procedures for the preparation of samples for metals analysis based on EPA 9200.1-86, *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil*, November 2008. The *in vitro* bioaccessibility assay provides a rapid and relatively inexpensive alternative to *in vivo* assays for predicting relative bioavailability (RBA) of lead and other metals in soils and soil-like materials. The method is based on the concept that metal solubilization in gastrointestinal fluid is likely to be an important determinant of bioavailability *in vivo*. The method measures the extent of metal solubilization in an extraction solvent that resembles gastric fluid. The user is cautioned that excess phosphate in the sample medium may result in the formation of pyromorphite ( $\text{Pb}_5(\text{PO}_4)_3\text{Cl}$ ) during the extraction (i.e. the procedure may not be suitable to phosphate-amended soils if lead is the metal of interest). Deviations from the reference method are described in Appendix A.

The digestion and analysis of bioaccessibility extracts prepared using this SOP is detailed in EPA Region 9 Laboratory SOPs 515 (mercury), 407 (digestion for metals), and 503 (metals analysis).

## 2 METHOD SUMMARY

A soil or soil-like material is homogenized, dried at 37 °C, and then sieved. A 1 gram aliquot of sample is rotated end-over-end in 100 mL of 0.4M glycine at pH 1.5 for 1 hour in an environmental chamber controlled at 37 °C. The extract is filtered immediately (within 30 minutes) and held at 0 to 6 °C until digestion and analysis.

## 3 DEFINITIONS

Absolute Bioavailability (ABA) – The ratio of the amount of metal absorbed compared to the amount ingested.

Analytical Sample – Any sample in which metals analysis is being determined, excluding standards, method blanks, or QC reference samples.

Extraction Blank – An aliquot of extraction fluid placed into an extraction vessel and treated exactly as a sample.

Laboratory Control Sample (LCS) – An aliquot of extraction fluid to which known quantities of the method analytes are added with the elements of interest and placed into an extraction vessel. The LCS is treated exactly like a sample and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making

accurate and precise measurements. The LCS is also known as a laboratory fortified blank (LFB).

Laboratory Information Management System (LIMS) – The Element Database.

Method Blank (MB) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample. The MB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment. The MB is also known as a laboratory reagent blank (LRB).

Matrix Duplicate (MD) – A duplicate aliquot of an analytical sample. The MD is treated exactly like a sample and its purpose is to determine laboratory precision. The MD is also known as the laboratory duplicate (LD or DUP).

Matrix Spike (MS) – An aliquot of an analytical sample to which known quantities of the method analytes are added in the laboratory. The MS is treated exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations. The MS is also known as a laboratory fortified matrix (LFM) sample.

Relative Bioavailability (RBA) – The ratio of the absolute bioavailability of a metal present in some test material compared to the absolute bioavailability of the metal in some appropriate reference material.

Reagent Blank – An aliquot of the extraction fluid.

Sample Delivery Group (SDG) – A group of twenty samples or less from a project that is sent to the laboratory for analysis.

Solid Sample – For the purpose of this method, a sample taken from the matrices classified as soil, sludge, solid or sediment.

Standard Reference Material (SRM) – A solid material containing a concentration of analyte that is certified and documented by a reputable supplier. This procedure uses an SRM as the LCS for solids.

#### **4 SAFETY & HEALTH**

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

#### 4.1 Physical Hazards

Heat stress is associated with working at elevated temperatures such as the 37 °C required in the environmental chamber. Analysts are cautioned to wear light clothing under PPE and to take frequent breaks. Use of the “buddy system” is encouraged.

#### 4.2 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. This procedure uses concentrated hydrochloric acid (see note in Section 7.2). Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN for additional information.

#### 4.3 Equipment and Instruments

Follow the manufacturer’s safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

- Areas of high, lethal voltages exist within the rotary agitation apparatus. Never touch parts of the apparatus which are not intended for access by the operator.
- Remove or secure any dangling articles, such as neckties, necklaces, badges, chains or pendants, which may get caught in the rotary agitation apparatus. Secure ponytails and long hair so as to not get caught within the rotary agitation apparatus.

#### 4.4 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Environmental Management System* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the volume of any reagent described in Sections 7.2 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure.

Reduce the toxicity of waste by purchasing lower concentration stock reagents and solutions to replace neat chemicals whenever possible. **However, the analyst must not change the concentrations of reagents specifically designated in this SOP.**

#### 4.5 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure* and City of Richmond Discharge Permit. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or designees.

This procedure generates the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-regulated Waste	Not applicable
Bioaccessibility extraction waste (water, glycine, hydrochloric acid)	Hazardous Waste	Toxic, check pH to determine corrosive properties
Regulated Solid Waste (soil samples)	Hazardous Waste	Toxic

## 5 SAMPLE HANDLING AND PRESERVATION

### 5.1 Containers and Required Sample Volume

Samples should be collected in glass bottles, jars, or plastic bags. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis and

minimize waste disposal. An 8-oz jar is generally sufficient to meet these requirements.

## 5.2 Internal Chain-of-Custody

Verify sample IDs and dates and times of collection against the chain-of-custody form.

Update the LIMS database internal custody form when sample containers are moved from the designated sample location. Change the container disposition to “active out” and the location to the appropriate room number. At the end of the day, return sample containers to the “Home” locations. Update the LIMS database using the “return to home location” feature and update container disposition to “available in”. Verify that your initials are recorded whenever you update the LIMS custody information.

## 5.3 Sample Storage

No preservatives or special storage conditions are required. Retain samples for 60 days after the final analytical report is sent to the data user.

Filtered extracts are stored at  $> 0$  and  $\leq 6$  °C until they are digested.

## 5.4 Holding Time

Digestion of the filtered extracts should be conducted within 1 week of extraction.

# 6 INTERFERENCES

1. Excess phosphate in the sample may result in interference with lead analysis (i.e., the assay is not suited to phosphate-amended soils).
2. The dissolution of metals from a test material into the extraction fluid depends on a number of variables including extraction fluid composition, temperature, time, agitation, solid/fluid ratio, and pH. Any deviations from conditions specified in this SOP must be documented in the LIMS WO memo field.
3. Samples containing soluble forms of lead (i.e., lead acetate, lead oxide, lead carbonate) exceeding 50,000 ppm will saturate the extraction fluid and lead chloride crystals will precipitate. If this is suspected, the sample aliquot must be reduced to  $0.50 \pm 0.01$  g. Saturation of the extraction fluid with other metals is also possible, but has not been investigated.

4. The pH electrode is critical to this procedure. It is not subject to interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity. The analyst must monitor and correct for the following sources of error.
  - Sodium error at pH levels greater than 10 can be reduced or eliminated by using a “low sodium error” electrode.
  - Coatings of oily material or particulate matter can impair pH electrode response time. These coatings may be removed by gently wiping or washing the electrode with detergent. An additional treatment with dilute hydrochloric acid (1+9) may be necessary to remove any remaining film.
  - The sample temperature affects pH measurement in two manners:
    - a. The electrode response will vary with changes in temperature. Using a pH meter with ATC can minimize this variability.
    - b. Sample pH is dependent upon the sample temperature, i.e., the true pH of a sample is different at different temperatures. Consequently, the sample temperature must be recorded as part of the pH measurement process.

## 7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. All equipment, reagents, standards, and supplies must meet the technical and QC requirements of the reference method. Substitutions may be made provided that they are documented and equivalency is maintained.

### 7.1 Instruments and Equipment

- Analytical balance, capable of weighing accurately to  $\pm 0.001\text{g}$ , integrated with PC-based software for data collection and the LIMS for reporting purposes.
- ASTM Class 1 Weights, or equivalent
- Drying oven, set at  $37\text{ }^{\circ}\text{C}$
- Dessicator
- Environmental chamber, capable of maintaining  $37 \pm 2\text{ }^{\circ}\text{C}$
- Magnetic stirrer
- pH Meter and associated equipment accurate to  $\pm 0.05$  units at  $25\text{ }^{\circ}\text{C}$ :
  - a. Orion Expandable IonAnalyzer EA 940 pH meter, or equivalent
  - b. Orion Combination pH Electrode Model 91-56, or equivalent
  - c. Automatic Temperature Compensation probe, Epoxy Body, Beckman No. 592373, or equivalent



- Rotary Agitation Apparatus – Six-port or more, capable of end-over-end rotation at  $30 \pm 2$  rpm. Associated Design and Mfg. Co. Model 3740-6 or 3740-12, or equivalent
- Thermometer, capable of measuring to  $37 \pm 2$  °C

## 7.2 Reagents

Reagents may contain elemental impurities which affect analytical data. Only high-purity reagents that conform to the American Chemical Society (ACS) specifications should be used. If the purity of a reagent is in question, analyze for contamination.

Record all chemical and reagent preparations in the LIMS.

NOTE: Hydrochloric acid presents various hazards and is moderately toxic and extremely irritating to skin and mucus membranes. Use this reagent in a fume hood whenever possible. The use of heavy gloves is recommended when working with hydrochloric acid.

- Glycine, ACS Reagent Grade or better, suitable for trace metals analysis
- Hydrochloric acid (HCl), concentrated, ACS Reagent Grade or better, suitable for trace metals analysis
- Reagent water – All references to reagent water in this SOP refer to laboratory deionized water as described in EPA Region 9 Laboratory SOP 825.

## 7.3 Standards

Record all standards and standard preparations in the LIMS.

### 7.3.1 Solid Laboratory Control Sample (LCS or BS)

Use solid materials from the National Institute of Standards and Testing (NIST), if available.

- Standard Reference Material 2711a Montana II Soil is suitable for lead.
- Certified Reference Material from RTC, Trace Metals - Sandy Loam 8, is suitable for mercury.

SRMs for other elements must be determined on a project specific basis. Follow the manufacturer's supplied expiration date for all SRMs.

### 7.3.2 Stock Standards

Stock standard solutions are purchased from a reputable supplier such as Spex CertiPrep, Ultra Scientific, or Inorganic Ventures. These solutions have a shelf life of one year or longer and come with NIST traceable documentation.

- Mercury Matrix Spike Standard. Use Perkin Elmer Pure Plus, 10 mg/L, or equivalent.
- 6010 Matrix Spike A Standard. Use Inorganic Ventures CLPP-SPK-SET, or equivalent.

Analyte	CLPP-SPK-1, mg/L	CLPP-SPK-2, mg/L	CLPP-SPK-3, mg/L
Al	2,000	-	-
Sb	-	500	-
As	-	-	2,000
Ba	2,000	-	-
Be	50	-	-
Cd	-	-	50
Cr	200	-	-
Co	500	-	-
Cu	250	-	-
Fe	1,000	-	-
Pb	-	-	500
Mn	500	-	-
Ni	500	-	-
Se	-	-	2,000
Ag	50	-	-
Tl	-	-	2,000
V	500	-	-
Zn	500	-	-

The analyst may select the appropriate solutions from the above list dependent upon project requirements (e.g., projects requiring analysis for lead only will need CLPP-SPK-3). Use of single-element solutions is also allowed.

### 7.4 Supplies

- Bottle Extraction Vessel – High Density Polyethylene (HDPE), wide mouth, 2 L volume, manufactured by Nalgene, P/N 16125-118; or equivalent
- Bottle Extraction Vessel – High Density Polyethylene (HDPE), wide mouth, 125-mL volume
- Disposable Syringe Filters, 0.45-  $\mu$ m cellulose acetate

- Disposable Syringes with Luer-lock fittings, 20 cc
- Graduated cylinders, 100-mL
- Magnetic stirrer bars
- Plastic or Teflon wash bottle
- Polypropylene Digestion Vessels, Environmental Express P/N SC475, or equivalent
- Sieve, stainless steel, US Series 70 (212  $\mu\text{m}$ )
- Volumetric flask, 1000-mL
- Volumetric pipet, 100-mL

## 8 ANALYTICAL PROCEDURES

### 8.1 Sample Preparation

1. Prepare and homogenize soil and sediment samples according to SOP 150, *Soil and Sediment Homogenization*.
2. Set the drying oven at 37 °C according to the manufacturer's instructions. Daily prior to use, verify the oven temperature through the digital readout on the oven and document in the temperature log book.
3. Take a sufficient amount of the homogenized samples and place in an oven at 37 °C until dry. The length of time required to dry depends on the physical condition of the samples. Overnight drying is usually sufficient.
4. Remove the samples from the oven and place them in a desiccator to cool for at least 30 minutes.
5. Sieve the samples using the stainless steel US Series 70 (212  $\mu\text{m}$ ) sieve.
6. Sample is now ready for extraction.

### 8.2 Preparation of Extraction Fluid, 0.4 M Glycine

Approximately a day before the extraction fluid is to be prepared the environmental chamber #1 in Room 308 must be adjusted to  $37 \pm 2$  °C. Contact a technical director to arrange to have the chamber reserved and properly adjusted.

Larger volumes of extraction fluids may be prepared as required for the number of samples to be extracted. Simply multiply the quantities provided below and record the volume prepared in the extraction logbook. Care must be taken to ensure adequate mixing of large volumes to make certain that a stable pH has been reached prior to recording the reading.

These instructions result in approximately 1 liter of extraction fluid. The analyst may increase the volume prepared to meet the needs of the project.

1. To 0.9 liter reagent water, add 30.03 g of glycine, bring the solution to a final volume of 1 L and mix.
2. Adjust pH of solution to  $1.50 \pm 0.05$  with concentrated hydrochloric acid (approximately 25 mL). Stir with a magnetic stirrer until the pH stabilizes. Record the volume of hydrochloric acid used. Note that it is not yet necessary to record pH as the final adjustment will be made in the environmental chamber (see below).
3. Place the solution in the environmental chamber #1 in Room 308 where the temperature was set to  $37 \pm 2$  °C. Making sure that a thermometer is set inside the chamber to monitor the room temperature.
4. Keep the solution in the chamber until its temperature reaches  $37 \pm 2$  °C.
5. Inside the environmental chamber, following SOP 580 *pH in Aqueous Samples*, standardize the pH meter with pH 4 and 7 buffers. Include pH 2 buffer as part of the CCV check. Use pH 4 buffer as a second source buffer. All buffer temperature must be maintained at  $37 \pm 2$  °C.
6. Adjust pH of solution to  $1.50 \pm 0.05$  with concentrated hydrochloric acid if necessary. Stir with a magnetic stirrer until the pH stabilizes.
7. Record the additional volume of acid used, pH meter ID, thermometer serial number, and pH in the pH Analysis Run Log, Bioaccessibility Extraction Logbook, and the LIMS reagent comments field.
8. If the pH of the extraction fluid is adjusted more than 24 hours before use, the pH must be checked prior to extraction.

### 8.3 Bioaccessibility Extraction Procedure

All extraction vessels, glassware and utensils used for the extraction procedure must be washed with soapy water, acid rinsed, and rinsed with reagent water.

Generate sample extraction labels using the LIMS or use a pressure sensitive labeling tape to label the vessels.

Prepare a LIMS benchsheet for each batch of samples to be extracted before beginning with any preparations. Refer to Section 9.2 for the appropriate QC samples and frequency.

1. Approximately a day before the extraction fluid is to be prepared the environmental chamber #1 in Room 308 must be adjusted to  $37 \pm 2$  °C. Contact a technical director to arrange to have the chamber reserved and properly adjusted.
2. Make sure that a thermometer is set inside the chamber to monitor the temperature of the room. Record room temperature in the extraction log and / or benchsheet.
3. Weigh  $1.00 \pm 0.05$  g of the dried and sieved sample into a 125-mL wide mouth HDPE bottle. Care should be taken to ensure that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle. Record weight in the benchsheet and / or extraction log.

NOTE: Steps 4 to 9 below must be completed in the environmental chamber so that the extraction solution remains at  $37 \pm 2$  °C. Analysts must be aware of heat stress and take frequent breaks while still assuring timely completion of these steps.

4. Measure  $100 \pm 0.5$  mL of the extraction fluid using a volumetric pipet or graduated cylinder and transfer to the 125-mL bottle. Record volume in the benchsheet and / or extraction log.
5. Hand-tighten each bottle cap and shake/invert to ensure that no leakage occurs and that no soil is caked on the bottom of the bottle.
6. Place approximately 6 of the 125-mL bottle inside the 2-L wide mouth HDPE vessel.
7. Verify the rotary agitator turning rate prior to each use (must be  $30 \pm 2$  rpm).
8. Clamp the 2-L vessels to the TCLP rotator, making sure each vessel is secure and the lids are tightly fastened.
9. Rotate the vessels for 1 hour. Record the analyst, date, and thermometer serial number, as well as the time and temperature at the beginning (setup) and end of the extraction (final) in the Bioaccessibility Extraction logbook. The temperature in the room should be maintained at  $37 \pm 2$  °C.

NOTE: While the samples are rotating, make certain that syringes, filters, and labeled clean 50-mL digestion vessels are prepared for filtration as this step must be completed within 30 minutes of the end of sample rotation.

10. When the extraction is complete, remove the 2-L extraction vessels from the rotator to a lab cart and exit the environmental chamber.

11. Remove the 125-mL bottles from the 2-L vessels and place upright on the bench top to allow the soil to settle to the bottom for a few minutes.
12. Use a disposable 20 cc syringe with Luer-Lok attachment to draw extract directly from the 125-mL bottle. Attach a 0.45-  $\mu\text{m}$  cellulose acetate disk filter to the syringe and filter the extract into a labeled clean 50-mL digestion vessel. Repeat as necessary to filter approximately 50 mL.
13. Record the time the extracts are filtered. The total time elapsed from rotation to filtering should not exceed 1 hour and 30 minutes. If the elapsed time exceeded 1 hour and 30 minutes, the test must be repeated.
14. Measure the pH of the remaining fluid in the 125-mL bottle the same day of extraction. Record pH reading in the pH log and/or extraction log. If the pH is not within  $\pm 0.5$  units of the starting pH (1.5), the sample must be discarded and the sample re-extracted following the same procedure above.
  - If the second test results in a decrease in pH of greater than 0.5 units, this reading will be recorded and the extract will be used for analysis.
  - If the second test results in an increase in pH of greater than 0.5 units, the test must be repeated a third time, but the extractor must be stopped at specific intervals and the pH manually adjusted to pH of 1.5 with drop-wise addition of HCl (adjustments at 5, 10, 15, and 30 minutes into the extraction).
15. The filtered extracts are stored at  $> 0$  and  $\leq 6$  °C until they are digested.
16. Digestion of the filtered extracts should be conducted within 1 week of extraction.
17. Filtered extracts are digested following EPA Region 9 Laboratory SOP 407, *Preparation of Leachate Procedure Extracts for Metals Analysis* or analyzed by EPA Region 9 Laboratory SOP 515, *Determination of Mercury in Water by CVAA Spectrometry*.

#### 8.4 Maintenance

General laboratory maintenance is suitable for this method. The analyst should be aware of the following issues:

- Observation of the rotary agitation apparatus at the beginning of each extraction period (or prior to beginning the entire process if the apparatus has been unused for a significant period of time) should provide adequate feedback regarding performance. A stop watch can be used to confirm rotation at  $30 \pm 2$  rpm.

## 9 QUALITY CONTROL

The EPA Region 9 Laboratory operates a formal quality control program. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

### 9.1 Demonstration of Capability

To be trained in this procedure, an analyst must read and follow the current revision of this SOP and document this by signing the SOP Signature Log. Additionally, the analyst must, prior to extracting samples under this procedure and annually thereafter, successfully extract a precision and accuracy study. No MDL study is required for this procedure.

### 9.2 Batch QC

Refer to Appendix B for a summary of batch QC requirements and criteria.

#### 9.2.1 Method Blank

One extraction fluid blank must be extracted with each batch of 20 or fewer field samples. Identify this as BLK1 in the LIMS benchsheet.

#### 9.2.2 Reagent Blank

An aliquot of the extraction fluid that was not extracted or filtered must be digested once per batch of 20 or fewer field samples. Identify this as BLK2 in the LIMS benchsheet and add a note in the comment field indicating that the fluid was not extracted or filtered.

#### 9.2.3 Laboratory Control Sample

One spiked extraction fluid blank must be extracted with each batch of 20 or fewer field samples. A spike level of 1 mg/L lead or 0.0002 mg/L (0.2 µg/L) mercury is appropriate. If other elements are to be determined consult project specific requirements.

#### 9.2.4 Matrix Duplicate

One matrix duplicate must be extracted for each 10 field samples in an SDG.

#### 9.2.5 Matrix Spike

One matrix spike must be extracted for each 10 field samples in an SDG. A spike level of 5 mg/L lead or 0.0002 mg/L mercury is appropriate. If other elements are to be determined consult project specific requirements.

#### 9.2.6 Solid Reference Material

Two SRMs must be extracted with each batch of 20 or fewer field samples. The SRM must be element specific (i.e. assigned per project depending upon the metals to be determined in the extracts).

## 10 DOCUMENTATION

### 10.1 Bioaccessibility Extraction Logbook

The Bioaccessibility extraction logbook requires the following information: analyst initials (setup and final), date and time (setup and final), thermometer serial number, room temperature in °C (setup and final), extraction fluid preparation, EPA ID, Lab ID, sample weight, amount of extraction fluid used (mL), extract pH, pH meter ID, and any comments. The logbook is maintained in Room 302.

### 10.2 pH Analysis Run Log

Entries are made for the pH buffer solutions, project number, SDG number, slope of the calibration, lab sample IDs, client sample IDs, sample volume in mL, time of analysis, pH result, analyst initials, date of analysis, and comments, if any.

### 10.3 Standards

All standards (MS, LCS, and SRM) are recorded in the LIMS. A copy of each Analytical Standard Record associated with sample analysis must be included in the data package.

### 10.4 Reagents

Record all reagents used for each analytical batch in the LIMS.

### 10.5 SOP Distribution and Acknowledgement

After approval, distribute an electronic copy of the final SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. (The Laboratory QC Database is contains a list of assigned analysts for each SOP). All approved



Region 9 Laboratory SOPs are maintained in the LotusNotes database in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Laboratory QC Database.

#### 10.6 SOP Revisions

Revisions to this SOP are summarized in Appendix C.

## 11 REFERENCES

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USEPA Region 9 Laboratory. SOP 880, *Demonstration of Capability*.

U.S. Environmental Protection Agency, EPA 9200.1-86, *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil*, November 2008.

U.S. Environmental Protection Agency, EPA 2007a, *Guidance for Evaluating the Oral Bioavailability of Metals in Soils for Use in Human Health Risk Assessment*. OSWER 9285.7-80.

U.S. Environmental Protection Agency, EPA 2007b, *Estimation of Relative Bioavailability of Lead in Soil and Soil-like Materials Using in Vivo and in Vitro Methods*. OSWER 9285.7-77.

**APPENDIX A.**  
**DEVIATIONS FROM THE REFERENCE METHOD**

1. The EPA Region 9 Laboratory uses an environmental chamber in place of a water bath to control the temperature during extraction.
2. The reference procedure uses spike levels of 10 mg/L lead in the extract; this SOP requires spiking the LCS at 1 mg/L and the MS at 5 mg/L for lead. The lower spike levels reflect the sensitivity of the analytical system used for determination of lead.
3. This SOP specifies extract storage temperature of  $> 0$  and  $\leq 6$  °C, while the reference method specifies 4 °C.
4. This SOP specifies that filtered extract be digested within one week from extraction while the reference method specifies that extracts be analyzed within one week from extraction and does not specify that extracts must be digested.
5. This SOP broadens the scope of metals that may be determined in the extract. The user is cautioned that the method has not been validated for the additional elements.

**APPENDIX B.**  
**QUALITY CONTROL MEASURES AND CRITERIA**

<b>Parameter</b>	<b>Frequency</b>	<b>Criteria</b>
MB	1 per batch	< 50 µg/L Lead *
LCS	1 per batch	85 - 115% Recovery
Matrix Spike	1 per 10 samples	75 - 125% Recovery
Matrix Duplicate	1 per 10 samples	± 20% RPD
SRM (see NOTE)	2 per batch	± 10% RPD
Reagent Blank	1 per batch	< 25 µg/L Lead *

\* Blank acceptance criterion is element specific. See project specific requirements for other elements.

NOTE: According to the reference method, NIST SRM 2711a yields an average recovery of 84.4% for lead. If the observed recovery is outside 75 to 95% consult the Chemistry Technical Director.



Attachment H-2  
Laboratory Reports

## Final Iron King IVBA Speciation Report

### Iron King Soil Samples

US EPA's Region 9 shipped three new Iron King soil samples to USEPA's Office of Research and Development (ORD) for bioavailability testing, which was conducted by Karen Bradham (ORD, Research Triangle Park, NC). After collecting weights for the soil containers and contents, the soils were blended and spread out in drying trays. The trays containing the soil were placed in an air-drying oven and dried for ~ 5 days at < 40 °C and sample weights were collected subsequent to air-drying. The soil was then added to a vibrating 2 mm stainless steel sieve screen to remove any large chunks of aggregated soil. Material remaining on the screen was disaggregated using a gloved hand and rescreened. The soil was sieved to < 250 µm to maximize the quantity of soil for bioavailability studies. The soil was passed through a riffler five times and aliquots were collected in pre-cleaned 250 mL high-density polyethylene bottles. The soil samples were extracted according to EPA Method 9200.2-86 *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil* dated April 2012 with the following exceptions: duplicate extractions of each soil sample were conducted (duplicate samples are only required once per batch according to EPA Method 9200.2-86). Region 9 selected two Iron King samples (BKG-201-02 and HSJ-504-02) for bioavailability testing that had been archived under chain-of-custody at EPA, RTP NC.

Dr. Bradham provided samples (via chain of custody) to:

- Dr. David Thomas (USEPA, ORD) for in vivo mouse assays (Bradham et al 2011) and total arsenic analysis by Instrumental Neutron Activation Analysis (INAA) at North Carolina State University's Nuclear Reactor Program;
- Dr. Kirk Scheckel (USEPA, ORD) for arsenic speciation, which was examined using the Materials Research Collaborative Access Team's (MRCAT) beamline 10-ID, Sector 10 at the Advanced Photon Source (APS), Argonne National Laboratory (ANL), Argonne, IL. **Speciation results for the 5 new Iron King soils are listed in Table 2 and the speciation results for the original Iron King soils are listed in Table 3.**

After conducting in vivo mouse assays and obtaining total arsenic tissue results by Instrumental Neutron Activation Analysis, we discovered that the tissue concentrations for samples BKG-201-02 and HSJ-504-02 were lower than the reporting limit for INAA (0.2 µg/g soil). In order to obtain tissue values for these two soils, we completed an extensive study to develop a microwave assisted digest method and analysis method for the tissue samples. Analysis of the sample digests was completed by USEPA Method 6020A (ICP-MS). The microwave assisted digest method development and analysis included blanks, blank spikes, matrix spikes, and NIST SRM biological samples (for microwave assisted digestion method). All microwave assisted digestion and analysis QCs were within acceptable quality assurance parameters as described in USEPA Solid Waste methods guidelines. The RBA values listed in Table 1 for BKG-201-02 and HSJ-504-02 are based on results of microwave assisted digestion and ICP-MS analysis. All other soil samples and tissue samples were above the INAA reporting limit. A strong correlation for the RBA and IVBA values was observed for

the Iron King samples ( $R^2 = 0.80$ ), indicating good agreement between the bioavailability and bioaccessibility of the soil samples.

*Table 1. Total arsenic soil concentrations, %RBA values, and %IVBA values for Iron King soils (data for soils listed in **bold** in the table were recently developed).*

Soil ID	[As] <sup>a</sup> (mg/kg)	%RBA ± S.D. (%)	%IVBA <sup>b</sup> ± S.D. (%)
OSF-118-1	244	15.9 ± 1.8	18.1 ± 0.4
HSJ 501-0-2	173	14.4 ± 1.6	6.8 ± 0.8
IJK-525-0-2	6899	15 ± 1.4	17.5 ± 0.6
HSJ-583	280	40.9 ± 2.6	53.6 ± 0.2
IJK-583	4495	14.8 ± 1.5	8.8 ± 0.1
IK B10-4	448	17 ± 0.7	25 ± 0.6
IK B10-5	195	18.6 ± 4.2	27.3 ± 0.5
IK B10-6/7	446	17.2 ± 1.2	22 ± 0.3
HSJ-504-02	39	22.9 ± 8	17.5 ± 1.1
BKG-201-02	10	29.9 ± 10.7	5.7 ± 0.1

<sup>a</sup> Determined by Instrumental Neutron Activation Analysis

<sup>b</sup> Gastric phase extraction using EPA Method 9200.2-86 (0.4 M glycine, pH 1.5).



Graph 1: Correlation between RBA and IVBA for 9 Iron King samples (Sample BKG-201-02 was removed as an outlier from regression due to low As concentration).

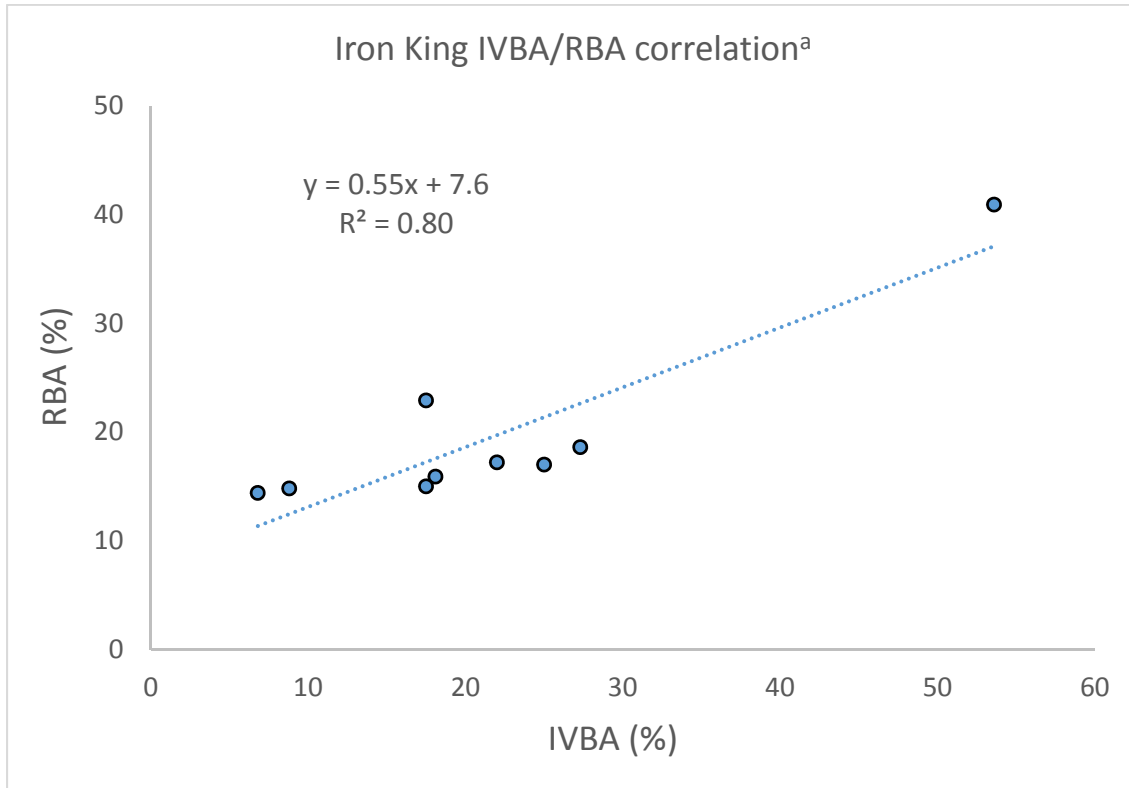


Table 2. Arsenic solid phase speciation results for new Iron King mine site soils.

sample	%						reduced chi squared
	As(III) sorbed	As (III) mineral	As (III) total	As (V) sorbed	As (V) mineral	As (V) total	
IK B10-4	-	-	0	53.3	46.7	100	0.000261
IK B10-5	-	-	0	100	-	100	0.000132
IK B10-6	8.5	-	8.5	91.5	-	91.5	0.000254
HSJ-504-02	-	-	0	48.2	51.8	100	0.001614
BKG-201-02	*not measured, concentration too low for sufficient signal*						

Table 3. Arsenic solid phase speciation results for original Iron King mine site soils.

soil ID	source <sup>a</sup>	Arsenic Speciation*					$\chi^2$ red <sup>c</sup>
		As <sup>b</sup> (mg/kg)	Arsenate (As <sup>V</sup> )		Arsenite (As <sup>III</sup> )		
			Sorbed As(V) (%)	Scorodite (%)	Realgar (%)	Arsenopyrite (%)	
1	OSF-118-1	244	96.2	3.8	-	-	0.002
2	HSJ 501-0-2	173	66.8	33.2	-	-	0.002
3	IJK-525-0-2	6899	18.3	47.1	-	34.6	0.001
4	HSJ-583	280	79.5	20.5	-	-	0.007
5	IJK-583	4495	67.6	32.4	-	-	0.011

<sup>a</sup> Source of arsenic contaminated soil

<sup>b</sup> Determined by Instrumental Neutron Activation Analysis

<sup>c</sup> Reduced chi-squared values

\* Determined by linear combination of As XAS



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 7/30/2013

**Subject:** Analytical Testing Results - Project R13S75  
SDG: 13155D

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine April-May 2013 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

**Analyses included in this report:**

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Metals by ICP

Percent Solids



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R13S75

**Project:** Iron King Mine April-May 2013 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 13155D

**Reported:** 07/30/13 16:23

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
642	1306007-01	Soil	05/01/13 00:00	06/04/13 13:30
647	1306007-02	Soil	05/01/13 00:00	06/04/13 13:30
648	1306007-03	Soil	05/01/13 00:00	06/04/13 13:30
750	1306007-04	Soil	05/01/13 00:00	06/04/13 13:30
753	1306007-05	Soil	05/01/13 00:00	06/04/13 13:30
978	1306007-06	Soil	05/01/13 00:00	06/04/13 13:30
979	1306007-07	Soil	04/29/13 14:21	06/04/13 13:30
980	1306007-08	Soil	04/29/13 15:13	06/04/13 13:30
Chap-01-surface	1306007-09	Sediment	04/29/13 13:43	06/04/13 13:30
Chap-02-surface	1306007-10	Sediment	04/29/13 13:47	06/04/13 13:30
Chap-03-surface	1306007-11	Sediment	04/29/13 13:50	06/04/13 13:30

**SDG ID 13155D**

Samples were digested using EPA method SW3050 for "Total" metals.

**Work Order(s)**

**1306007**



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<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:23
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1306007-01</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>642</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		240		2	mg/kg wet	B13F059	06/13/13	06/17/13	6010C/SOP503
Lead		360		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-02</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>647</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		190		2	mg/kg wet	B13F059	06/13/13	06/17/13	6010C/SOP503
Lead		190		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-03</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>648</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		220		2	mg/kg wet	B13F059	06/13/13	06/17/13	6010C/SOP503
Lead		180		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-04</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>750</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		29		2	mg/kg wet	B13F059	06/13/13	06/17/13	6010C/SOP503
Lead		1,100		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-05</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>753</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic	RE1	300		2	mg/kg wet	B13F059	06/13/13	07/03/13	6010C/SOP503
Lead		290		3	"	"	"	06/17/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-06</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>978</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic	RE1	240		2	mg/kg wet	B13F059	06/13/13	07/03/13	6010C/SOP503
Lead		440		3	"	"	"	06/17/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-07</b>								<b>Soil - Sampled: 04/29/13 14:21</b>
<b>Sample ID:</b>	<b>979</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic	RE1	480		2	mg/kg wet	B13F059	06/13/13	07/03/13	6010C/SOP503
Lead		730		3	"	"	"	06/17/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-08</b>								<b>Soil - Sampled: 04/29/13 15:13</b>
<b>Sample ID:</b>	<b>980</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic	RE1	3,700		2	mg/kg wet	B13F059	06/13/13	07/03/13	6010C/SOP503
Lead		5,700		3	"	"	"	06/17/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-09</b>								<b>Sediment - Sampled: 04/29/13 13:43</b>
<b>Sample ID:</b>	<b>Chap-01-surface</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Aluminum		9,800		130	mg/kg dry	B13F059	06/13/13	06/17/13	6010C/SOP503
Antimony		1.4	Cl, J	2.5	"	"	"	"	6010C/SOP503
Arsenic	RE1	150		2.5	"	"	"	07/03/13	6010C/SOP503
Barium	RE1	58		6.3	"	"	"	"	6010C/SOP503
Beryllium		0.37		0.13	"	"	"	06/17/13	6010C/SOP503
Cadmium		3.1		0.63	"	"	"	"	6010C/SOP503
Calcium		3,500		130	"	"	"	"	6010C/SOP503



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<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:23
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
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**Lab ID: 1306007-09** **Sediment - Sampled: 04/29/13 13:43**

**Sample ID: Chap-01-surface** **Metals by EPA 6000/7000 Series Methods**

Chromium		13		1.3	mg/kg dry	B13F059	06/13/13	06/17/13	6010C/SOP503
Cobalt		9.3		2.5	"	"	"	"	6010C/SOP503
Copper		440		5.1	"	"	"	"	6010C/SOP503
Iron		28,000		130	"	"	"	"	6010C/SOP503
Lead		69		3.8	"	"	"	"	6010C/SOP503
Magnesium	RE1	4,900		63	"	"	"	07/03/13	6010C/SOP503
Manganese		490		6.3	"	"	"	06/17/13	6010C/SOP503
Molybdenum		ND	U	6.3	"	"	"	"	6010C/SOP503
Nickel		12		6.3	"	"	"	"	6010C/SOP503
Potassium		1,500		630	"	"	"	"	6010C/SOP503
Selenium		3.6		2.5	"	"	"	"	6010C/SOP503
Silver		1.1	C1, J	1.3	"	"	"	"	6010C/SOP503
Sodium	RE1	190		63	"	"	"	07/03/13	6010C/SOP503
Thallium		ND	U	6.3	"	"	"	06/17/13	6010C/SOP503
Vanadium		40		2.5	"	"	"	"	6010C/SOP503
Zinc		630		10	"	"	"	"	6010C/SOP503

**Sample ID: Chap-01-surface** **Conventional Chemistry Parameters by APHA/EPA Methods**

% Solids		79		1	%	B13F065	06/13/13	06/14/13	3550C/SOP460
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**Lab ID: 1306007-10** **Sediment - Sampled: 04/29/13 13:47**

**Sample ID: Chap-02-surface** **Metals by EPA 6000/7000 Series Methods**

Aluminum		17,000		150	mg/kg dry	B13F059	06/13/13	06/17/13	6010C/SOP503
Antimony		2.4	C1, J	3	"	"	"	"	6010C/SOP503
Arsenic	RE1	210		3	"	"	"	07/03/13	6010C/SOP503
Barium	RE1	140		7.5	"	"	"	"	6010C/SOP503
Beryllium		0.44		0.15	"	"	"	06/17/13	6010C/SOP503
Cadmium		3.4		0.75	"	"	"	"	6010C/SOP503
Calcium		7,200		150	"	"	"	"	6010C/SOP503
Chromium		19		1.5	"	"	"	"	6010C/SOP503
Cobalt		17		3	"	"	"	"	6010C/SOP503
Copper		940		6	"	"	"	"	6010C/SOP503
Iron		41,000		150	"	"	"	"	6010C/SOP503
Lead		210		4.5	"	"	"	"	6010C/SOP503
Magnesium	RE1	7,300		75	"	"	"	07/03/13	6010C/SOP503
Manganese		820		7.5	"	"	"	06/17/13	6010C/SOP503
Molybdenum		ND	U	7.5	"	"	"	"	6010C/SOP503
Nickel		21		7.5	"	"	"	"	6010C/SOP503
Potassium		2,000		750	"	"	"	"	6010C/SOP503
Selenium		6.3		3	"	"	"	"	6010C/SOP503
Silver		3.9		1.5	"	"	"	"	6010C/SOP503
Sodium	RE1	170		75	"	"	"	07/03/13	6010C/SOP503



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<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:23
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1306007-10</b>								<b>Sediment - Sampled: 04/29/13 13:47</b>
<b>Sample ID:</b>	<b>Chap-02-surface</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Thallium		ND	U	7.5	mg/kg dry	B13F059	06/13/13	06/17/13	6010C/SOP503
Vanadium		58		3	"	"	"	"	6010C/SOP503
Zinc		1,200		12	"	"	"	"	6010C/SOP503
<b>Sample ID:</b>	<b>Chap-02-surface</b>								<b>Conventional Chemistry Parameters by APHA/EPA Methods</b>
% Solids		67		1	%	B13F065	06/13/13	06/14/13	3550C/SOP460
<b>Lab ID:</b>	<b>1306007-11</b>								<b>Sediment - Sampled: 04/29/13 13:50</b>
<b>Sample ID:</b>	<b>Chap-03-surface</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Aluminum		12,000		120	mg/kg dry	B13F059	06/13/13	06/17/13	6010C/SOP503
Antimony		2.7		2.3	"	"	"	"	6010C/SOP503
Arsenic	RE1	290		2.3	"	"	"	07/03/13	6010C/SOP503
Barium	RE1	92		5.8	"	"	"	"	6010C/SOP503
Beryllium		0.36		0.12	"	"	"	06/17/13	6010C/SOP503
Cadmium		1.9		0.58	"	"	"	"	6010C/SOP503
Calcium		7,800		120	"	"	"	"	6010C/SOP503
Chromium		13		1.2	"	"	"	"	6010C/SOP503
Cobalt		13		2.3	"	"	"	"	6010C/SOP503
Copper		470		4.6	"	"	"	"	6010C/SOP503
Iron		36,000		120	"	"	"	"	6010C/SOP503
Lead		150		3.5	"	"	"	"	6010C/SOP503
Magnesium	RE1	5,600		58	"	"	"	07/03/13	6010C/SOP503
Manganese		450		5.8	"	"	"	06/17/13	6010C/SOP503
Molybdenum		ND	U	5.8	"	"	"	"	6010C/SOP503
Nickel		14		5.8	"	"	"	"	6010C/SOP503
Potassium		1,900		580	"	"	"	"	6010C/SOP503
Selenium		6.1		2.3	"	"	"	"	6010C/SOP503
Silver		1.8		1.2	"	"	"	"	6010C/SOP503
Sodium	RE1	270		58	"	"	"	07/03/13	6010C/SOP503
Thallium		ND	U	5.8	"	"	"	06/17/13	6010C/SOP503
Vanadium		58		2.3	"	"	"	"	6010C/SOP503
Zinc		570		9.3	"	"	"	"	6010C/SOP503
<b>Sample ID:</b>	<b>Chap-03-surface</b>								<b>Conventional Chemistry Parameters by APHA/EPA Methods</b>
% Solids		86		1	%	B13F065	06/13/13	06/14/13	3550C/SOP460



**United States Environmental Protection Agency  
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<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:23
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
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Batch B13F059 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 06/13/13 Analyzed: 06/17/13  
Metals by EPA 6000/7000 Series Methods - Quality Control

**Blank (B13F059-BLK1)**

Aluminum	ND	U		100 mg/kg wet						
Antimony	ND	U		2 "						
Arsenic	ND	U		2 "						
Beryllium	ND	U		0.1 "						
Cadmium	ND	U		0.5 "						
Calcium	ND	U		100 "						
Chromium	ND	U		1 "						
Cobalt	ND	U		2 "						
Copper	ND	U		4 "						
Iron	ND	U		100 "						
Lead	ND	U		3 "						
Manganese	ND	U		5 "						
Molybdenum	ND	U		5 "						
Nickel	ND	U		5 "						
Potassium	ND	U		500 "						
Selenium	ND	U		2 "						
Silver	ND	U		1 "						
Thallium	ND	U		5 "						
Vanadium	ND	U		2 "						
Zinc	ND	U		8 "						

**Blank (B13F059-BLK2)**

Barium	ND	U		5 mg/kg wet						
Magnesium	ND	U		50 "						
Sodium	ND	U		50 "						

**Matrix Spike (B13F059-MS1)**

Source: 1306007-01

Arsenic	663			2 mg/kg wet	396	238	107	75-125		20
Lead	453			3 "	99.0	359	95	75-125		20

**Matrix Spike Dup (B13F059-MSD1)**

Source: 1306007-01

Arsenic	696			2 mg/kg wet	396	238	116	75-125	5	20
Lead	472			3 "	99.0	359	115	75-125	4	20

**Reference (B13F059-SRM1)**

Aluminum	123			100 mg/kg wet	115		107	47.6-152		
Antimony	61.8			2 "	66.1		93	41.2-158		
Arsenic	284			2 "	254		112	60.9-139		
Beryllium	4.97			0.1 "	4.91		101	61.2-139		
Cadmium	10.7			0.5 "	10.9		98	70.6-128		
Calcium	47,300			100 "	44300		107	68.6-132		
Chromium	28			1 "	27.2		103	68.3-132		





**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13155D
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:23
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
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**Batch B13F059 - 3050B Sld Acid Dig - Metals by 6010**

**Prepared: 06/13/13 Analyzed: 06/17/13**

**Metals by EPA 6000/7000 Series Methods - Quality Control**

**Reference (B13F059-SRM1)**

Cobalt	37.3			2 "	37.5		100	64.7-135		
Copper	1,560			4 "	1770		88	74.6-126		
Iron	6,740			100 "	6480		104	66.2-134		
Lead	58.9			3 "	57.0		103	72.8-127		
Manganese	62.7			5 "	61.1		103	68.2-132		
Nickel	16.3			5 "	16.3		100	55.2-145		
Potassium	ND	U		500 "	39.8			0-215		
Selenium	10.3			2 "	10.0		103	41-159		
Silver	6.1			1 "	5.91		103	45.8-154		
Thallium	9.61			5 "	9.52		101	30.5-169		
Vanadium	19.2			2 "	17.6		109	65.9-135		
Zinc	47.3			8 "	47.6		99	43.2-157		

**Reference (B13F059-SRM2)**

Barium	ND	U		5 mg/kg wet	1.60			62.5-138		
Magnesium	28,000			50 "	29300		96	70.2-130		
Sodium	ND	U		50 "	72.6			0-298		

**Batch B13F065 - Solids, Dry Weight (Prep) - Solids, Dry Weight**

**Prepared: 06/13/13 Analyzed: 06/14/13**

**Conventional Chemistry Parameters by APHA/EPA Methods - Quality Control**

**Blank (B13F065-BLK1)**

% Solids	ND	U		1 %						
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**Duplicate (B13F065-DUP1)**

**Source: 1306007-10**

% Solids	68			1 %		67			2	20
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**Project Manager:** Zi Zi Searles

**Project Number:** R13S75

**Project:** Iron King Mine April-May 2013 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 13155D

**Reported:** 07/30/13 16:23

**Qualifiers and Comments**

J The reported result for this analyte should be considered an estimated value.

C1 The reported concentration for this analyte is below the quantitation limit.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 7/30/2013

**Subject:** Analytical Testing Results - Project R13S75  
SDG: 13155D

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine April-May 2013 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

**Analyses included in this report:**

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Analysis of In Vitro Gastric Extracts by ICP



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13155D
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:33
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
642	1306007-01	Soil	05/01/13 00:00	06/04/13 13:30
647	1306007-02	Soil	05/01/13 00:00	06/04/13 13:30
648	1306007-03	Soil	05/01/13 00:00	06/04/13 13:30
750	1306007-04	Soil	05/01/13 00:00	06/04/13 13:30
753	1306007-05	Soil	05/01/13 00:00	06/04/13 13:30
978	1306007-06	Soil	05/01/13 00:00	06/04/13 13:30
979	1306007-07	Soil	04/29/13 14:21	06/04/13 13:30
980	1306007-08	Soil	04/29/13 15:13	06/04/13 13:30

**SDG ID 13155D**

Samples were analyzed using an in-vitro bioaccessibility leaching method. Calculated bio-accessibilities are as follows:

Sample ID	Lab ID	Analyte	In-vitro mg/kg	Total mg/kg	%bio-access.
642	1306007-01	Arsenic	ND	240	<1%
642	1306007-01	Lead	14	360	3.9%
647	1306007-02	Arsenic	6.4	190	3.4%
647	1306007-02	Lead	3.8	190	2.0%
648	1306007-03	Arsenic	16	220	7.3%
648	1306007-03	Lead	12	180	6.7%
750	1306007-04	Arsenic	1.7	29	5.9%
750	1306007-04	Lead	570	1100	51.8%
753	1306007-05	Arsenic	110	300	36.7%
753	1306007-05	Lead	ND	290	<1%
978	1306007-06	Arsenic	ND	240	<1%
978	1306007-06	Lead	9.8	440	2.2%
979	1306007-07	Arsenic	15	480	3.1%
979	1306007-07	Lead	130	730	17.8%
980	1306007-08	Arsenic	510	3700	13.8%
980	1306007-08	Lead	32	5700	0.6%

**Work Order(s)**

**1306007**



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13155D
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:33
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1306007-01</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>642</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		ND	U	2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		14		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-02</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>647</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		6.4		2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		3.8		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-03</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>648</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		16		2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		12		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-04</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>750</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	1.7	C1, J	2	mg/kg	B13G071	07/18/13	07/19/13	6010C/SOP503
Lead		570		3	"	B13F077	07/09/13	07/11/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-05</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>753</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		110		2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		ND	U	3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-06</b>								<b>Soil - Sampled: 05/01/13 00:00</b>
<b>Sample ID:</b>	<b>978</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	ND	U	2	mg/kg	B13G071	07/18/13	07/19/13	6010C/SOP503
Lead		9.8		3	"	B13F077	07/09/13	07/11/13	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-07</b>								<b>Soil - Sampled: 04/29/13 14:21</b>
<b>Sample ID:</b>	<b>979</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		15		2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		130		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1306007-08</b>								<b>Soil - Sampled: 04/29/13 15:13</b>
<b>Sample ID:</b>	<b>980</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		510		2	mg/kg	B13F077	07/09/13	07/11/13	6010C/SOP503
Lead		32		3	"	"	"	"	6010C/SOP503



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Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13155D
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 07/30/13 16:33
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B13F077 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>										
<b>Prepared: 07/09/13 Analyzed: 07/11/13</b>										
<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>										
<b>Blank (B13F077-BLK1)</b>										
Arsenic	1.2	C1, J		2 mg/kg						
Lead	ND	U		3 "						
<b>Blank (B13F077-BLK2)</b>										
Arsenic	ND	U		2 mg/kg						
Lead	ND	U		3 "						
<b>LCS (B13F077-BS1)</b>										
Arsenic	402			2 mg/kg	400		100	80-120		200
Lead	102			3 "	100		102	80-120		200
<b>Duplicate (B13F077-DUP1) Source: 1306007-05</b>										
Arsenic	110			2 mg/kg		110			0.4	20
Lead	ND	U		3 "		ND				20
<b>Matrix Spike (B13F077-MS1) Source: 1306007-05</b>										
Arsenic	1,770			2 mg/kg	2000	110	83	75-125		20
Lead	395			3 "	500	ND	79	75-125		20
<b>Reference (B13F077-SRM1)</b>										
Arsenic	56.4			2 mg/kg	89.3		63	0-200		
Lead	1,070			3 "	1300		82	0-200		
<b>Reference (B13F077-SRM2)</b>										
Arsenic	56.3			2 mg/kg	89.0		63	0-200		
Lead	1,060			3 "	1300		81	0-200		
<b>Batch B13G071 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>										
<b>Prepared: 07/18/13 Analyzed: 07/19/13</b>										
<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>										
<b>Blank (B13G071-BLK1)</b>										
Arsenic	ND	U		2 mg/kg						
<b>Blank (B13G071-BLK2)</b>										
Arsenic	ND	U		2 mg/kg						
<b>LCS (B13G071-BS1)</b>										
Arsenic	390			2 mg/kg	400		97	80-120		200
<b>Reference (B13G071-SRM1)</b>										
Arsenic	55.8			2 mg/kg	89.1		63	0-200		
<b>Reference (B13G071-SRM2)</b>										
Arsenic	56.2			2 mg/kg	89.3		63	0-200		



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R13S75

**Project:** Iron King Mine April-May 2013 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 13155D

**Reported:** 07/30/13 16:33

**Qualifiers and Comments**

J The reported result for this analyte should be considered an estimated value.

C1 The reported concentration for this analyte is below the quantitation limit.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 8/16/2013

**Subject:** Analytical Testing Results - Project R13S75  
SDG: 13210B

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine April-May 2013 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

**Analyses included in this report:**

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Metals by ICP





United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 15:12
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
13330WellsSt	1307058-01	Soil	07/11/13 15:03	07/26/13 15:50
13336WellsSt	1307058-02	Soil	07/11/13 15:56	07/26/13 15:50
GulchYard	1307058-03	Soil	07/11/13 11:00	07/26/13 15:50

**SDG ID 13210B**

**Work Order(s)**

**1307058**

Total metals: Soil samples were dried at 37 degree C then sieved through a No. 70 (212um) screen prior to preparation and analysis.



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 15:12
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
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**Lab ID: 1307058-01** **Soil - Sampled: 07/11/13 15:03**

**Sample ID: 13330WellsSt** **Metals by EPA 6000/7000 Series Methods**

Aluminum		20,000		100	mg/kg wet	B13H062	08/12/13	08/13/13	6010C/SOP503
Antimony		11		2	"	"	"	"	6010C/SOP503
Arsenic		440		2	"	"	"	"	6010C/SOP503
Barium		210		5	"	"	"	"	6010C/SOP503
Beryllium		0.54		0.10	"	"	"	"	6010C/SOP503
Cadmium		6.3		0.50	"	"	"	"	6010C/SOP503
Calcium		12,000		100	"	"	"	"	6010C/SOP503
Chromium		31		1	"	"	"	"	6010C/SOP503
Cobalt		11		2	"	"	"	"	6010C/SOP503
Copper		230		4	"	"	"	"	6010C/SOP503
Iron		41,000		100	"	"	"	"	6010C/SOP503
Lead		1,400		3	"	"	"	"	6010C/SOP503
Magnesium		6,100		50	"	"	"	"	6010C/SOP503
Manganese		670		5	"	"	"	"	6010C/SOP503
Molybdenum		ND	U	5	"	"	"	"	6010C/SOP503
Nickel		23		5	"	"	"	"	6010C/SOP503
Potassium		5,300		500	"	"	"	"	6010C/SOP503
Selenium		3.8		2	"	"	"	"	6010C/SOP503
Silver		11		1	"	"	"	"	6010C/SOP503
Sodium		390		50	"	"	"	"	6010C/SOP503
Thallium		ND	U	5	"	"	"	"	6010C/SOP503
Vanadium		54		2	"	"	"	"	6010C/SOP503
Zinc		1,400		8	"	"	"	"	6010C/SOP503

**Lab ID: 1307058-02** **Soil - Sampled: 07/11/13 15:56**

**Sample ID: 13336WellsSt** **Metals by EPA 6000/7000 Series Methods**

Aluminum		17,000		100	mg/kg wet	B13H062	08/12/13	08/13/13	6010C/SOP503
Antimony		7.8	J, Q4	2	"	"	"	"	6010C/SOP503
Arsenic		390		2	"	"	"	"	6010C/SOP503
Barium		190		5	"	"	"	"	6010C/SOP503
Beryllium		0.56		0.10	"	"	"	"	6010C/SOP503
Cadmium		7.1		0.50	"	"	"	"	6010C/SOP503
Calcium		9,400		100	"	"	"	"	6010C/SOP503
Chromium		30		1	"	"	"	"	6010C/SOP503
Cobalt		10		2	"	"	"	"	6010C/SOP503
Copper		240		4	"	"	"	"	6010C/SOP503
Iron		37,000		100	"	"	"	"	6010C/SOP503
Lead		1,400		3	"	"	"	"	6010C/SOP503
Magnesium		5,400	J, Q4	50	"	"	"	"	6010C/SOP503
Manganese		550		5	"	"	"	"	6010C/SOP503
Molybdenum		ND	U	5	"	"	"	"	6010C/SOP503



**United States Environmental Protection Agency  
Region 9 Laboratory**

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Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 15:12
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1307058-02</b>								<b>Soil - Sampled: 07/11/13 15:56</b>
<b>Sample ID:</b>	<b>13336WellsSt</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Nickel		24		5	mg/kg wet	B13H062	08/12/13	08/13/13	6010C/SOP503
Potassium		5,400		500	"	"	"	"	6010C/SOP503
Selenium		3.5		2	"	"	"	"	6010C/SOP503
Silver		9.7		1	"	"	"	"	6010C/SOP503
Sodium		310		50	"	"	"	"	6010C/SOP503
Thallium		ND	U	5	"	"	"	"	6010C/SOP503
Vanadium		46		2	"	"	"	"	6010C/SOP503
Zinc		1,700		8	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1307058-03</b>								<b>Soil - Sampled: 07/11/13 11:00</b>
<b>Sample ID:</b>	<b>GulchYard</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Aluminum		19,000		100	mg/kg wet	B13H062	08/12/13	08/13/13	6010C/SOP503
Antimony		2.9		2	"	"	"	"	6010C/SOP503
Arsenic		330		2	"	"	"	"	6010C/SOP503
Barium		130		5	"	"	"	"	6010C/SOP503
Beryllium		0.41		0.10	"	"	"	"	6010C/SOP503
Cadmium		2.2		0.50	"	"	"	"	6010C/SOP503
Calcium		9,400		100	"	"	"	"	6010C/SOP503
Chromium		25		1	"	"	"	"	6010C/SOP503
Cobalt		13		2	"	"	"	"	6010C/SOP503
Copper		71		4	"	"	"	"	6010C/SOP503
Iron		45,000		100	"	"	"	"	6010C/SOP503
Lead		400		3	"	"	"	"	6010C/SOP503
Magnesium		6,600		50	"	"	"	"	6010C/SOP503
Manganese		560		5	"	"	"	"	6010C/SOP503
Molybdenum		ND	U	5	"	"	"	"	6010C/SOP503
Nickel		18		5	"	"	"	"	6010C/SOP503
Potassium		3,400		500	"	"	"	"	6010C/SOP503
Selenium		3.4		2	"	"	"	"	6010C/SOP503
Silver		2.3		1	"	"	"	"	6010C/SOP503
Sodium		140		50	"	"	"	"	6010C/SOP503
Thallium		ND	U	5	"	"	"	"	6010C/SOP503
Vanadium		79		2	"	"	"	"	6010C/SOP503
Zinc		540		8	"	"	"	"	6010C/SOP503



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 15:12
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
---------	--------	-----------------------	--------------------	-------	-------------	---------------	------	-------------	-----	-----------

Batch B13H062 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 08/12/13 Analyzed: 08/13/13  
 Metals by EPA 6000/7000 Series Methods - Quality Control

**Blank (B13H062-BLK1)**

Aluminum	ND	U		100 mg/kg wet						
Antimony	ND	U		2 "						
Arsenic	ND	U		2 "						
Barium	ND	U		5 "						
Beryllium	ND	U		0.1 "						
Cadmium	ND	U		0.5 "						
Calcium	ND	U		100 "						
Chromium	ND	U		1 "						
Cobalt	ND	U		2 "						
Copper	ND	U		4 "						
Iron	ND	U		100 "						
Lead	ND	U		3 "						
Magnesium	ND	U		50 "						
Manganese	ND	U		5 "						
Molybdenum	ND	U		5 "						
Nickel	ND	U		5 "						
Potassium	ND	U		500 "						
Selenium	ND	U		2 "						
Silver	ND	U		1 "						
Sodium	ND	U		50 "						
Thallium	ND	U		5 "						
Vanadium	ND	U		2 "						
Zinc	ND	U		8 "						

**Matrix Spike (B13H062-MS1)**

**Source: 1307058-02**

Aluminum	17,700	Q10		100 mg/kg wet	385	16,900	<b>204</b>	75-125		20
Antimony	30.4			2 "	96.2	7.8	<b>24</b>	75-125		20
Arsenic	769			2 "	385	387	99	75-125		20
Barium	550			5 "	385	191	93	75-125		20
Beryllium	9.61			0.1 "	9.62	0.556	94	75-125		20
Cadmium	15.8			0.5 "	9.62	7.09	91	75-125		20
Calcium	11,300	Q10		100 "	1920	9,400	98	75-125		20
Chromium	65.2			1 "	38.5	29.6	93	75-125		20
Cobalt	96.7			2 "	96.2	10.2	90	75-125		20
Copper	285	Q10		4 "	48.1	238	97	75-125		20
Iron	37,200	Q10		100 "	192	36,700	<b>243</b>	75-125		20
Lead	1,480	Q10		3 "	96.2	1,380	112	75-125		20
Magnesium	7,340			50 "	1920	5,440	99	75-125		20
Manganese	628	Q10		5 "	96.2	551	80	75-125		20
Molybdenum	74.8			5 "	96.2	ND	78	75-125		20
Nickel	111			5 "	96.2	24.1	91	75-125		20



# United States Environmental Protection Agency Region 9 Laboratory

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Phone: (510) 412-2300 Fax: (510) 412-2302

<b>Project Manager:</b> Zi Zi Searles <b>Project Number:</b> R13S75 <b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>California Site Cleanup Section 1</b> <b>75 Hawthorne Street</b> <b>San Francisco CA, 94105</b>	<b>SDG:</b> 13210B <b>Reported:</b> 08/16/13 15:12
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## Quality Control

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
---------	--------	--------------------------	-----------------------	-------	----------------	------------------	------	----------------	-----	--------------

Batch B13H062 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 08/12/13 Analyzed: 08/13/13

Metals by EPA 6000/7000 Series Methods - Quality Control

**Matrix Spike (B13H062-MS1)**

Source: 1307058-02

Potassium	7,440			500 "	1920	5,380	107	75-125		20
Selenium	372			2 "	385	3.46	96	75-125		20
Silver	19.3			1 "	9.62	9.71	100	75-125		20
Sodium	2,100			50 "	1920	311	93	75-125		20
Thallium	346			5 "	385	ND	90	75-125		20
Vanadium	137			2 "	96.2	45.9	95	75-125		20
Zinc	1,790	Q10		8 "	96.2	1,680	113	75-125		20

**Matrix Spike Dup (B13H062-MSD1)**

Source: 1307058-02

Aluminum	19,600	Q10		100 mg/kg wet	396	16,900	<b>666</b>	75-125	10	20
Antimony	30.8			2 "	99.0	7.8	<b>23</b>	75-125	1	20
Arsenic	806			2 "	396	387	106	75-125	5	20
Barium	609			5 "	396	191	106	75-125	10	20
Beryllium	10.1			0.1 "	9.90	0.556	97	75-125	5	20
Cadmium	16.3			0.5 "	9.90	7.09	93	75-125	3	20
Calcium	12,300	Q10		100 "	1980	9,400	<b>144</b>	75-125	8	20
Chromium	68.8			1 "	39.6	29.6	99	75-125	5	20
Cobalt	107			2 "	99.0	10.2	98	75-125	11	20
Copper	317	Q10		4 "	49.5	238	<b>160</b>	75-125	11	20
Iron	39,900	Q10		100 "	198	36,700	<b>NR</b>	75-125	7	20
Lead	1,510	Q10		3 "	99.0	1,380	<b>134</b>	75-125	2	20
Magnesium	7,940			50 "	1980	5,440	<b>126</b>	75-125	8	20
Manganese	682	Q10		5 "	99.0	551	<b>132</b>	75-125	8	20
Molybdenum	78.8			5 "	99.0	ND	80	75-125	5	20
Nickel	118			5 "	99.0	24.1	95	75-125	6	20
Potassium	7,590			500 "	1980	5,380	111	75-125	2	20
Selenium	392			2 "	396	3.46	98	75-125	5	20
Silver	20.1			1 "	9.90	9.71	105	75-125	4	20
Sodium	2,400			50 "	1980	311	106	75-125	13	20
Thallium	363			5 "	396	ND	92	75-125	5	20
Vanadium	141			2 "	99.0	45.9	96	75-125	3	20
Zinc	1,880	Q10		8 "	99.0	1,680	<b>201</b>	75-125	5	20

**Reference (B13H062-SRM1)**

Aluminum	141			100 mg/kg wet	115		122	47.6-152		
Antimony	65.8			2 "	66.1		100	41.2-158		
Arsenic	307			2 "	253		121	60.9-139		
Barium	ND	U		5 "	1.60			62.5-138		
Beryllium	5.32			0.1 "	4.91		108	61.2-139		
Cadmium	11.3			0.5 "	10.9		103	70.6-128		
Calcium	55,000			100 "	44300		124	68.6-132		
Chromium	29.9			1 "	27.1		110	68.3-132		



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 15:12
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
---------	--------	--------------------------	-----------------------	-------	----------------	------------------	------	----------------	-----	--------------

Batch B13H062 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 08/12/13 Analyzed: 08/13/13  
Metals by EPA 6000/7000 Series Methods - Quality Control

**Reference (B13H062-SRM1)**

Cobalt	41.1		2	"	37.5		110	64.7-135		
Copper	1,770		4	"	1770		100	74.6-126		
Iron	7,530		100	"	6480		116	66.2-134		
Lead	60.5		3	"	57.0		106	72.8-127		
Magnesium	31,800		50	"	29200		109	70.2-130		
Manganese	70.5		5	"	61.1		115	68.2-132		
Nickel	17.3		5	"	16.3		106	55.2-145		
Potassium	ND	U	500	"	39.8			0-215		
Selenium	12.2		2	"	10.0		122	41-159		
Silver	6.24		1	"	5.91		106	45.8-154		
Sodium	ND	U	50	"	72.6			0-298		
Thallium	6.5		5	"	9.51		68	30.5-169		
Vanadium	19.2		2	"	17.6		109	65.9-135		
Zinc	51.7		8	"	47.6		109	43.2-157		



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R13S75

**Project:** Iron King Mine April-May 2013 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 13210B

**Reported:** 08/16/13 15:12

**Qualifiers and Comments**

Q4 The matrix spike and/or matrix spike duplicate associated with this sample did not meet recovery criteria for this analyte (see MS/MSD results for this batch in QC summary)

Q10 The analyte concentration in the unfortified sample is significantly greater than the concentration spiked into the matrix spike and matrix spike duplicate. The reported spike recovery is not a meaningful measure of the dataset's analytical accuracy.

J The reported result for this analyte should be considered an estimated value.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 8/16/2013

**Subject:** Analytical Testing Results - Project R13S75  
SDG: 13210B

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine April-May 2013 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

**Analyses included in this report:**

---

Analysis of In Vitro Gastric Extracts by ICP





**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 16:43
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
13330WellsSt	1307058-01	Soil	07/11/13 15:03	07/26/13 15:50
13336WellsSt	1307058-02	Soil	07/11/13 15:56	07/26/13 15:50
GulchYard	1307058-03	Soil	07/11/13 11:00	07/26/13 15:50

**SDG ID 13210B**

Samples were analyzed using an in-vitro bioaccessibility leaching method. Calculated bio-accessibilities are as follows:

Sample ID	Lab ID	Analyte	In-vitro mg/kg	Total mg/kg	%bio-access.
13330WellsSt	1307058-01	Arsenic	95.9	441	21.7%
13330WellsSt	1307058-01	Lead	122	1440	8.5%
13336WellsSt	1307058-02	Arsenic	84.2	387	21.8%
13336WellsSt	1307058-02	Lead	134	1380	9.7%
GulchYard	1307058-03	Arsenic	43.0	330	13.0%
GulchYard	1307058-03	Lead	10.7	396	2.7%

**Work Order(s)**

**1307058**



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 13210B
<b>Project Number:</b> R13S75	<b>75 Hawthorne Street</b>	<b>Reported:</b> 08/16/13 16:43
<b>Project:</b> Iron King Mine April-May 2013 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1307058-01</b>								<b>Soil - Sampled: 07/11/13 15:03</b>
<b>Sample ID:</b>	<b>13330WellsSt</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		96		2	mg/kg	B13H019	08/13/13	08/13/13	6010C/SOP503
Lead		120		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1307058-02</b>								<b>Soil - Sampled: 07/11/13 15:56</b>
<b>Sample ID:</b>	<b>13336WellsSt</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		84		2	mg/kg	B13H019	08/13/13	08/13/13	6010C/SOP503
Lead		130		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1307058-03</b>								<b>Soil - Sampled: 07/11/13 11:00</b>
<b>Sample ID:</b>	<b>GulchYard</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		43		2	mg/kg	B13H019	08/13/13	08/13/13	6010C/SOP503
Lead		11		3	"	"	"	"	6010C/SOP503

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B13H019 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>										
<b>Prepared &amp; Analyzed: 08/13/13</b>										
<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>										
<b>Blank (B13H019-BLK1)</b>										
Arsenic	ND	U		2 mg/kg						
Lead	ND	U		3 "						
<b>Blank (B13H019-BLK2)</b>										
Arsenic	ND	U		2 mg/kg						
Lead	ND	U		3 "						
<b>LCS (B13H019-BS1)</b>										
Arsenic	402			2 mg/kg	400		101	80-120		200
Lead	98.3			3 "	100		98	80-120		200
<b>Duplicate (B13H019-DUP1) Source: 1307058-01</b>										
Arsenic	94.1			2 mg/kg			95.9		2	20
Lead	127			3 "			122		4	20
<b>Matrix Spike (B13H019-MS1) Source: 1307058-01</b>										
Arsenic	2,010			2 mg/kg	2000		95.9	96	75-125	20
Lead	558			3 "	500		122	87	75-125	20
<b>Reference (B13H019-SRM1)</b>										
Arsenic	57			2 mg/kg	88.7		64	0-200		
Lead	1,060			3 "	1290		82	0-200		
<b>Reference (B13H019-SRM2)</b>										
Arsenic	56.2			2 mg/kg	88.7		63	0-200		
Lead	1,060			3 "	1290		82	0-200		



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**Project Manager:** Zi Zi Searles

**Project Number:** R13S75

**Project:** Iron King Mine April-May 2013 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 13210B

**Reported:** 08/16/13 16:43

**Qualifiers and Comments**

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.

ANALYTICAL REPORT

Prepared by  
Lockheed Martin Information Systems and Global Services/Environmental Services  
Scientific, Engineering, Response and Analytical Services

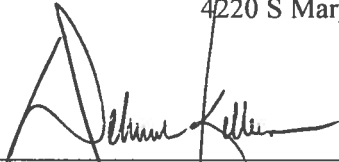
Iron King Mine Site Hydrologic Restoration  
Dewey-Humboldt, Arizona

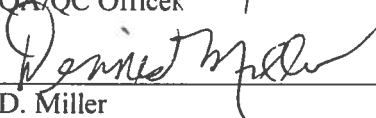
October 2013

EPA Work Assignment No. SERAS-146  
LOCKHEED MARTIN Work Order SER00146  
EPA Contract No. EP-W-09-031

Submitted to  
T. Johnson  
EPA-ERT

4220 S Maryland Parkway, Bldg. D, Suite 800,  
Las Vegas, NV 89119

  
\_\_\_\_\_  
D. Killeen  
QA/QC Officer  
Date 10/1/13

  
\_\_\_\_\_  
D. Miller  
Program Manager  
Date 10/17/13

Analysis by:  
ERT/SERAS

Prepared by:/Validated by:  
Y. Mehra/R. Varsolona

Table of Contents

---

REPORT OF LABORATORY ANALYSIS

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SERAS-146-DAR-101713





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Results of the MS/MSD Analysis for Metals in Soil Table 2.2  
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Section III

Chain of Custody

Appendices

Appendix A Data for Metals in Soil Y 185

Appendix A will be furnished on request.





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TESTING LABORATORIES INFORMATION

Analysis of Metals in Soil by SERAS SOP# 1811, *“Digestion and Analysis of Metals by Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP-AES)”*

ERT/SERAS Laboratory  
2890 Woodbridge Avenue  
Edison, NJ 08837

All analyses were performed according to our NELAP-approved quality assurance program. The test results meet the requirements of the current NELAP standards, where applicable, except as noted in the laboratory case narrative provided. Results are intended to be considered in their entirety and apply only to those analyzed and reported herein.

ERT/SERAS Laboratory is certified by the New Jersey Department of Environmental Protection, NELAP Laboratory Certification ID # 12023 for metals analysis in soil.

---

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Detailed Sample Information

<u>SERAS Sample #</u>	<u>Field Sample #</u>
R309003-01	417
R309003-02	431
R309003-03	442
R309003-04	451
R309003-05	467
R309003-06	477
R309003-07	485
R309003-08	486
R309003-09	513
R309003-10	515
R309003-11	527
R309003-12	621
R309003-13	669
R309003-14	701
R309003-15	820
R309003-16	861
R309003-17	865
R309003-18	873
R309003-19	879





### Introduction

SERAS personnel, in response to WA# SERAS-146, provided analytical support for environmental samples collected from the Iron King Mine Site Hydrologic Restoration Site in Dewey-Humboldt, Arizona as described in the following table. The support also included QA/QC, data review and preparation of an analytical report containing analytical and QA/QC results.

The samples analyzed at SERAS were treated with procedures consistent with those specified in SERAS SOP #1008, *Sample Receiving, Handling and Storage* and SERAS SOP #1009, *Operation of Refrigeration Units*.

Chain of Custody #	Number of Samples	Sampling Date	Date Received	Date Analyzed	Matrix	Analysis/ Method	Laboratory	Data Package
06607	3	04/29/13	09/16/13	09/19/13 and 09/20/13	Soil	Metals/SERAS SOP# 1811	ERT/SERAS	Y 185
	9	04/30/13						
	6	05/01/13						
	1	05/02/13						

### Case Narrative

Sampling was conducted as per the site-specific Quality Assurance Project Plan (QAPP) and analyzed by the analytical methods as stated in the QAPP. The laboratory reported the data to three significant figures. Any other representation of the data is the responsibility of the user. Data were validated using a Stage 4 validation done manually (S4VM) in accordance with the “Guidance for Labeling Externally Validated Data for Superfund Use.” All data validation flags have been inserted into the results tables.

#### Metals in Soil Package Y 185

The initial analysis of samples 431, 451 and 621 for thallium resulted in concentrations with negative values for which the absolute values exceeded the reporting limit (RL) that may be indicative of matrix interference. Data for these samples were reported from diluted analyses resulting in elevated RLs.

The initial analysis of samples 442, 486, 513 and 701 for beryllium resulted in in concentrations with negative values for which the absolute values exceeded the RL that may be indicative of matrix interference. Data for these samples were reported from diluted analyses resulting in elevated RLs.

Copper was above and thallium was below the percent recovery criteria for the MS/MSD of sample 513. Copper (J) and thallium (UJ) results are qualified estimated for samples 417, 431, 442, 451, 467, 477, 485, 486, 513, 515, 527, 621, 669, 701, 820, 861, 865, 873 and 879.

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*The results presented in this report only relate to the samples analyzed. All results are intended to be considered in their entirety. The Environmental Response Team/Scientific, Engineering, Response and Analytical Services laboratory is not responsible for utilization of less than the complete report.*

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**Summary of Abbreviations**

BFB	Bromofluorobenzene
C	Centigrade
CLP	Contract Laboratory Program
COC	Chain of Custody
conc	concentration
cont	continued
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
D	(Surrogate Table) value is from a diluted sample and was not calculated
Dioxin	Polychlorinated dibenzo-p-dioxins (PCDD) and Polychlorinated dibenzofurans (PCDF)
DFTPP	Decafluorotriphenylphosphine
EMPC	Estimated maximum possible concentration
GC/MS	Gas Chromatography/ Mass Spectrometry
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDA	Minimum Detectable Activity
MS (BS)	Matrix Spike (Blank Spike)
MSD (BSD)	Matrix Spike Duplicate (Blank Spike Duplicate)
MW	Molecular Weight
NA	Not Applicable or Not Available
NAD	Normalized Absolute Difference
NC	Not Calculated
NR	Not Requested/Not Reported
NS	Not Spiked
% D	Percent Difference
% REC	Percent Recovery
SOP	Standard Operating Procedure
ppbv	parts per billion by volume
ppm	parts per million
pptv	parts per trillion by volume
PQL	Practical Quantitation Limit
PAL	Performance Acceptance Limit
QA/QC	Quality Assurance/Quality Control
QL	Quantitation Limit
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SERAS	Scientific, Engineering, Response and Analytical Services
SIM	Selected Ion Monitoring
Sur	Surrogate
TIC	Tentatively Identified Compound
TCLP	Toxicity Characteristic Leaching Procedure
VOC	Volatile Organic Compound
*	Value exceeds the acceptable QC limits

m <sup>3</sup>	cubic meter	g	gram	kg	kilogram	L	liter
µg	microgram	µL	microliter	mg	milligram	mL	milliliter
ng	nanogram	pg	picogram	pCi	picocurie	s	sigma

**Data Validation Flags**

J	Value is estimated	R	Value is unusable
J+	Value is estimated high (metals only)	U	Not detected
J-	Value is estimated low (metals only)	UJ	Not detected and RL is estimated
N	Presumptively present (Aroclors only)		

Rev. 1/14/09

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Table 1.1 Results of the Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Method SERAS SOP 1811

Page 1 of 3

SERAS Laboratory Number		NA		R309003-01		R309003-02		R309003-03	
Sample Number		Method Blank-091813		417		431		442	
Sample Location		Lab		N/A		N/A		N/A	
Percent Solids		NA		92		98		82	
Analyte	Result	RL	Result	RL	Result	RL	Result	RL	
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Aluminum	U	8.50	11700	8.46	17800	7.12	2210	9.58	
Antimony	U	1.20	63.9	1.19	6.11	1.01	119	1.35	
Arsenic	U	1.00	2420	0.995	273	0.838	3840	5.64	
Barium	U	0.300	71.7	0.299	182	0.251	43.4	0.338	
Beryllium	U	0.100	U	0.0995	0.338	0.0838	U	0.563	
Cadmium	U	0.200	5.47	0.199	3.53	0.168	0.862	0.225	
Calcium	U	6.00	39800	5.97	17300	5.03	50000	6.76	
Chromium	U	0.400	13.4	0.398	24.2	0.335	3.14	0.451	
Cobalt	U	0.200	6.57	0.199	16.0	0.168	1.62	0.225	
Copper	U	0.500	172	J 0.498	67.4	J 0.419	174	J 0.564	
Iron	U	30.0	76500	29.9	35100	25.1	123000	169	
Lead	U	1.00	3270	0.995	375	0.838	5740	1.13	
Magnesium	U	20.0	8320	19.9	7310	16.8	1070	22.5	
Manganese	U	0.300	646	0.299	781	0.251	34.3	0.338	
Nickel	U	0.500	7.47	0.498	25.8	0.419	U	0.564	
Potassium	U	50.0	2330	49.8	2800	41.9	1430	56.4	
Selenium	U	1.80	47.7	1.79	3.15	1.51	138	2.03	
Silver	U	0.500	16.6	0.498	1.65	0.419	29.6	0.564	
Sodium	U	30.0	887	29.9	99.8	25.1	1550	33.8	
Thallium	U	1.20	U	J 1.19	U	J 5.03	1.65	J 1.35	
Vanadium	U	0.400	50.9	0.398	51.3	0.335	59.7	0.451	
Zinc	U	2.50	2050	2.49	944	2.09	708	2.82	

Table 1.1 (cont) Results of the Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Method SERAS SOP 1811

SERAS Laboratory Number		R309003-04		R309003-05		R309003-06		R309003-07	
Sample Number		451		467		477		485	
Sample Location		N/A		N/A		N/A		N/A	
Percent Solids		95		95		96		95	
Analyte	Result	RL	Result	RL	Result	RL	Result	RL	
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Aluminum	24700	7.53	20100	8.41	18300	7.85	17300	8.10	
Antimony	11.0	1.06	40.2	1.19	61.8	1.11	63.3	1.14	
Arsenic	603	0.886	1350	0.989	3720	4.62	4080	4.77	
Barium	244	0.266	173	0.297	47.2	0.277	63.3	0.286	
Beryllium	0.444	0.0886	U	0.0989	U	0.0923	0.116	0.0953	
Cadmium	4.79	0.177	2.98	0.198	3.18	0.185	19.1	0.191	
Calcium	16100	5.32	5910	5.94	14700	5.54	22000	5.72	
Chromium	28.4	0.354	22.3	0.396	27.3	0.369	30.8	0.381	
Cobalt	19.6	0.177	9.83	0.198	2.94	0.185	15.9	0.191	
Copper	108	J 0.443	154	J 0.495	69.9	J 0.462	188	J 0.477	
Iron	52400	26.6	66100	29.7	121000	139	86000	28.6	
Lead	823	0.886	2850	0.989	3060	0.923	3740	0.953	
Magnesium	10200	17.7	6710	19.8	12500	18.5	14700	19.1	
Manganese	838	0.266	339	0.297	298	0.277	711	0.286	
Nickel	26.3	0.443	14.2	0.495	6.78	0.462	17.8	0.477	
Potassium	3930	44.3	2950	49.5	776	46.2	1420	47.7	
Selenium	6.58	1.59	20.0	1.78	46.8	1.66	53.5	1.72	
Silver	3.60	0.443	13.9	0.495	14.2	0.462	13.3	0.477	
Sodium	195	26.6	275	29.7	305	27.7	571	28.6	
Thallium	U	J 5.32	U	J 1.19	U	J 1.11	U	J 1.14	
Vanadium	63.2	0.354	62.5	0.396	66.6	0.369	69.5	0.381	
Zinc	1240	2.22	1110	2.47	1700	2.31	5410	2.38	

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Table 1.1 (cont) Results of the Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Method SERAS SOP 1811

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SERAS Laboratory Number		R309003-08		R309003-09		R309003-10		R309003-11	
Sample Number		486		513		515		527	
Sample Location		N/A		N/A		N/A		N/A	
Percent Solids		94		98		94		99	
Analyte	Result	RL	Result	RL	Result	RL	Result	RL	
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Aluminum	10600	8.00	12800	8.11	25700	7.74	3570	7.15	
Antimony	63.4	1.13	6.99	1.14	4.24	1.09	86.3	1.01	
Arsenic	1710	0.941	838	0.954	4340	4.55	4920	4.21	
Barium	83.1	0.282	87.7	0.286	147	0.273	1470	0.253	
Beryllium	U	0.471	U	0.477	U	0.0911	0.149	0.0842	
Cadmium	4.76	0.188	2.03	0.191	26.2	0.182	6.19	0.168	
Calcium	22200	5.65	5910	5.72	16400	5.46	1740	5.05	
Chromium	16.5	0.377	28.0	0.381	40.2	0.364	20.1	0.337	
Cobalt	6.86	0.188	9.29	0.191	13.1	0.182	1.87	0.168	
Copper	95.7	J 0.471	114	J 0.477	382	J 0.455	643	J 0.421	
Iron	56000	28.2	62200	28.6	70800	27.3	45400	25.3	
Lead	4100	0.941	249	0.954	226	0.911	6290	0.842	
Magnesium	5390	18.8	4900	19.1	8330	18.2	477	16.8	
Manganese	197	0.282	159	0.286	248	0.273	75.4	0.253	
Nickel	9.00	0.471	14.2	0.477	22.6	0.455	5.69	0.421	
Potassium	1860	47.1	2440	47.7	3300	45.5	2610	42.1	
Selenium	35.9	1.69	22.0	1.72	16.1	1.64	3.11	1.52	
Silver	21.5	0.471	1.13	0.477	1.19	0.455	78.3	0.421	
Sodium	466	28.2	410	28.6	459	27.3	110	25.3	
Thallium	U	J 1.13	U	J 1.14	U	J 1.09	U	J 1.01	
Vanadium	53.3	0.377	66.2	0.381	59.8	0.364	35.8	0.337	
Zinc	1720	2.35	462	2.38	3640	2.28	1360	2.10	

Table 1.1 (cont) Results of the Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Method SERAS SOP 1811

SERAS Laboratory Number		R309003-12		R309003-13		R309003-14		R309003-15	
Sample Number		621		669		701		820	
Sample Location		N/A		N/A		N/A		N/A	
Percent Solids		96		98		99		82	
Analyte	Result	RL	Result	RL	Result	RL	Result	RL	
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Aluminum	27500	8.29	17200	8.03	17600	7.34	19900	7.67	
Antimony	2.38	1.17	3.92	1.13	11.4	1.04	22.5	1.08	
Arsenic	174	0.975	223	0.945	591	0.863	726	0.902	
Barium	536	0.293	95.3	0.283	74.5	0.259	158	0.271	
Beryllium	0.378	0.0975	U	0.0945	U	0.863	U	0.0902	
Cadmium	2.69	0.195	2.06	0.189	4.68	0.173	3.06	0.180	
Calcium	47700	5.85	6910	5.67	6600	5.18	5480	5.41	
Chromium	40.7	0.390	16.8	0.378	20.2	0.345	22.2	0.361	
Cobalt	47.0	0.195	15.7	0.189	14.9	0.173	10.7	0.180	
Copper	1260	J 0.488	78.2	J 0.472	76.6	J 0.432	112	J 0.451	
Iron	48400	29.3	44300	28.3	51900	25.9	57500	27.1	
Lead	102	0.975	147	0.945	363	0.863	1290	0.902	
Magnesium	15600	19.5	6350	18.9	7020	17.3	6550	18.0	
Manganese	876	0.293	453	0.283	407	0.259	359	0.271	
Nickel	108	0.488	14.7	0.472	12.7	0.432	14.8	0.451	
Potassium	3490	48.8	2880	47.2	2630	43.2	3350	45.1	
Selenium	5.14	1.76	U	1.70	4.22	1.55	12.0	1.62	
Silver	2.51	0.488	0.701	0.472	1.82	0.432	7.40	0.451	
Sodium	257	29.3	84.4	28.3	90.9	25.9	156	27.1	
Thallium	U	J 5.85	U	J 1.13	U	J 1.04	U	J 1.08	
Vanadium	61.1	0.390	70.5	0.378	67.0	0.345	63.8	0.361	
Zinc	451	2.44	655	2.36	1480	2.16	978	2.26	

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Table 1.1 (cont) Results of the Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Method SERAS SOP 1811

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SERAS Laboratory Number		R309003-16		R309003-17		R309003-18		R309003-19	
Sample Number		861		865		873		879	
Sample Location		N/A		N/A		N/A		N/A	
Percent Solids		99		98		97		95	
Analyte	Result	RL	Result	RL	Result	RL	Result	RL	
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	
Aluminum	14700	7.53	14400	7.93	19800	8.02	22600	8.30	
Antimony	8.50	1.06	14.6	1.12	11.4	1.13	27.3	1.17	
Arsenic	330	0.886	494	0.933	530	0.944	896	0.976	
Barium	105	0.266	112	0.280	214	0.283	204	0.293	
Beryllium	0.144	0.0886	U	0.0933	0.124	0.0944	0.110	0.0976	
Cadmium	2.71	0.177	2.79	0.187	4.12	0.189	5.17	0.195	
Calcium	4380	5.32	5210	5.60	7520	5.66	8360	5.86	
Chromium	19.8	0.354	18.1	0.373	27.6	0.378	26.3	0.391	
Cobalt	14.1	0.177	13.9	0.187	18.7	0.189	14.0	0.195	
Copper	76.5	J 0.443	87.3	J 0.466	105	J 0.472	141	J 0.488	
Iron	39400	26.6	45500	28.0	44600	28.3	58100	29.3	
Lead	478	0.886	860	0.933	760	0.944	1910	0.976	
Magnesium	6180	17.7	5460	18.7	8650	18.9	7430	19.5	
Manganese	461	0.266	399	0.280	556	0.283	440	0.293	
Nickel	16.7	0.443	14.9	0.466	45.1	0.472	19.8	0.488	
Potassium	2220	44.3	2740	46.6	2600	47.2	3230	48.8	
Selenium	3.76	1.59	6.86	1.68	5.40	1.70	13.2	1.76	
Silver	2.10	0.443	4.16	0.466	3.53	0.472	9.75	0.488	
Sodium	130	26.6	193	28.0	582	28.3	278	29.3	
Thallium	U	J 1.06	U	J 1.12	U	J 1.13	U	J 1.17	
Vanadium	50.4	0.354	56.2	0.373	61.8	0.378	64.8	0.391	
Zinc	949	2.22	844	2.33	1300	2.36	1640	2.44	

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Table 2.1 Results of the LCS Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site

Sample ID: LCS 09/19/13

Analyte	Conc. Recovered mg/kg	Certified Value mg/kg	% Recovery	PALs mg/kg
Aluminum	10300	8840	117	4780 - 12900
Antimony	153	88.2	173	D.L. - 204
Arsenic	97.2	99.6	98	80.5 - 119
Barium	301	310	97	258 - 362
Beryllium	69.2	72.3	96	59.4 - 85.1
Cadmium	183	182	101	149 - 215
Calcium	6510	6790	96	5610 - 7980
Chromium	135	136	99	109 - 164
Cobalt	130	128	102	106 - 149
Copper	96.9	102	95	82.7 - 121
Iron	13600	12600	108	5180 - 19900
Lead	113	115	98	94.1 - 137
Magnesium	2990	3010	99	2320 - 3700
Manganese	316	323	98	266 - 379
Nickel	159	153	104	126 - 180
Potassium	3060	2840	108	2020 - 3670
Selenium	148	150	99	116 - 184
Silver	37.1	40.4	92	30.0 - 50.4
Sodium	2650	2760	96	1960 - 3550
Thallium	142	174	82	137 - 212
Vanadium	98.8	97.6	101	75.2 - 120
Zinc	154	161	96	130 - 192

PAL - Performance Acceptance Limits

Note: Antimony, Total concentration 232 mg/kg and PT-PALs 23.2 - 255 mg/kg

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Table 2.2 Results of the MS/MSD Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine site  
 Results Based on Dry Weight

Sample ID: 513

Page 1 of 1

Analyte	Sample Result mg/kg	MS Spike Added mg/kg	MS Result mg/kg	MS % Recovery	MSD Spike Added mg/kg	MSD Result mg/kg	MSD % Recovery	RPD	QC Limits		
									RPD	%Recovery	
Aluminum	12800	769	12800	NC	741	11800	NC	8	20	75-125	
Antimony	6.99	38.4	39.4	84	37.0	38.3	85	3	20	34-130	
Arsenic	838	38.4	919	NC	37.0	886	NC	4	20	75-125	
Barium	87.7	38.4	127	102	37.0	122	93	4	20	75-125	
Beryllium	U	38.4	37.1	97	37.0	36.4	98	2	20	75-125	
Cadmium	2.03	38.4	40.4	100	37.0	38.8	99	4	20	75-125	
Calcium	5910	769	6240	NC	741	6230	NC	0	20	75-125	
Chromium	28.0	38.4	66.7	101	37.0	63.1	95	6	20	75-125	
Cobalt	9.29	38.4	47.6	100	37.0	45.6	98	4	20	75-125	
Copper	114	38.4	163	128	*	37.0	153	105	6	20	75-125
Iron	62200	769	64900	NC	741	62600	NC	4	20	75-125	
Lead	249	38.4	282	NC	37.0	291	NC	3	20	75-125	
Magnesium	4900	769	5670	NC	741	5400	NC	5	20	75-125	
Manganese	159	38.4	196	NC	37.0	190	NC	3	20	75-125	
Nickel	14.2	38.4	53.7	103	37.0	50.7	99	6	20	75-125	
Potassium	2440	769	3240	104	741	3130	93	3	20	75-125	
Selenium	22.0	38.4	58.6	95	37.0	57.2	95	2	20	75-125	
Silver	1.13	38.4	37.7	95	37.0	36.3	95	4	20	75-125	
Sodium	410	769	1180	100	741	1150	100	3	20	75-125	
Thallium	U	38.4	28.3	74	*	37.0	32.7	88	14	20	75-125
Vanadium	66.2	38.4	108	109	37.0	103	99	5	20	75-125	
Zinc	462	38.4	509	NC	37.0	499	NC	2	20	75-125	





Table 2.3 Results of the Post Digestion Spike (PDS) Analysis for Metals in Soil  
 WA# SERAS-146 Iron King Mine Site

Sample ID: 513

Analyte	Sample Result µg/L	PDS Spike Added µg/L	PDS Result µg/L	PDS % Recovery	Recommended QC Limits %Recovery
Copper	1190	200	1320	NC	80-120
Thallium	U	400	368	92	80-120

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**CHAIN OF CUSTODY RECORD**

Project Name: Iron Kir. Mine  
 Project Number: SERAS-146  
 LM Contact: Dave Aloysius Phone: 732-494-4058

No: **06007**  
 Sheet **01** of **01** (Do not copy)  
 (for addnl. samples use new form)

SERAS  
 WO# 309003

**Sample Identification**

**Analyses Requested**

REAQ#	Sample No	Sampling Location	Matrix	Date Collected	# of Bottles	Container/Preservative	TAL Metals*							
01	417	N/A	Soil	4/30/13	1	4oz Jar / Cdd 4°C	✓	X 9/16/13						
02	431						✓							
03	442						✓							
04	451						✓							
05	467						✓							
06	477						✓							
07	485						✓							
08	486			↓			✓							
09	513			4/29/13			✓							
10	515			↓			✓							
11	527			↓			✓							
12	621			4/30/13			✓							
13	669			5/1/13			✓							
14	701			5/2/13			✓							
15	820			5/1/13			✓							
16	861			↓			✓							
17	865			↓			✓							
18	873			↓			✓							
19	879			↓			✓							

Matrix: \*TAL Metals includes Hg.

Special Instructions:

- A- Air
- AT-Animal Tissue
- DL- Drum Liquids
- DS- Drum Solids
- GW- Groundwater
- O- Oil
- PR-Product
- PT-Plant Tissue
- PW- Potable Water
- S- Soil
- SD- Sediment
- SL- Sludge
- SW- Surface Water
- TX-TCLP Extract
- W- Water
- X- Other

**SAMPLES TRANSFERRED FROM**

**CHAIN OF CUSTODY #:**

9-3066

9-3067

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished by	Date	Received by	Date	Time
All Analysis	<i>[Signature]</i>	9/16/13	<i>[Signature]</i>	9/16/13	15:10						



CHAIN OF CUSTODY RECORD

PROJ. NO.		PROJECT NAME				NO. OF CONTAINERS	REMARKS					
ERAS-146-DAR-101713		Iron King Mine - Humboldt Smelter										
SAMPLERS: (Signature) ZiZi Searles						Wo# 309003						
DATE	TIME	MATRIX S=soil	COMP.	GRAB	SAMPLE IDENTIFICATION							
4/30/13	3:41 PM	S		X	417	1				01	In-Vitro bioaccessibility assay for lead & arsenic SOP EPA 9200.1-86	
4/30/13	4:05 PM	S		X	431	1				02		
4/30/13	4:44 PM	S		X	442	1				03		
4/30/13	5:28 PM	S		X	451	1				04		
4/30/13	6:55 PM	S		X	467	1				05		
4/30/13	1:25 PM	S		X	477	1				06		
4/30/13	1:54 PM	S		X	485	1				07		
4/30/13	1:54 PM	S		X	486	1				08		
4/24/13	2:26 PM	S		X	513	1				09		
4/24/13	2:28 PM	S		X	515	1				10		
4/24/13	3:10 PM	S		X	527	1				11		
4/30/13	3:12 PM	S		X	621	1				12		
5/1/13	1:42	S		X	669	1				13		
5/2/13	6:11 AM	S		X	701	1				14		
5/1/13	8:02	S		X	820	1				15		
Relinquished by: (Signature) ZiZi Searles		Date / Time 9/6/13 4:45 PM		Received by: (Signature) Room Temp 9/16/13 10:00		Relinquished by: (Signature)			Date / Time		Received by: (Signature)	
Relinquished by: (Signature)		Date / Time		Received by: (Signature)		Relinquished by: (Signature)			Date / Time		Received by: (Signature)	
Received for Laboratory by: (Signature) 010		Date / Time		Temp.		Seals Intact (Y/N)		Conditions / Remarks				

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

CHAIN OF CUSTODY RECORD

ERAS-140-DAR-101713	PROJ. NO.		PROJECT NAME				NO. OF CONTAINERS	REMARKS			
			Iron King Mine - Humboldt Smelter								
SAMPLERS: (Signature)							<div style="border: 1px solid black; padding: 5px; transform: rotate(-45deg); display: inline-block;">                 WO#R307003             </div>				
Zizi Searles											
DATE	TIME	MATRIX (S/S/L)	COMP.	GRAB	SAMPLE IDENTIFICATION						
5/1/13	10:32 <sup>AM</sup>	S		X	861		1			16	In-Vitro bioaccessibility
5/1/13	10:40 <sup>AM</sup>	S		X	865		1			17	assay for lead &
5/1/13	10:52 <sup>AM</sup>	S		X	873		1			18	arsenic
5/1/13	11:04 <sup>AM</sup>	S		X	879		1			19	SOP
		<del>S</del>		<del>X</del>							EPA 9200.1-86
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
		<del>S</del>		<del>X</del>							
Relinquished by: (Signature)		Date / Time		Received by: (Signature)		Relinquished by: (Signature)		Date / Time		Received by: (Signature)	
Zizi Searles		5/6/13 4:45 PM		Roxan Temp							
Relinquished by: (Signature)		Date / Time		Received by: (Signature)		Relinquished by: (Signature)		Date / Time		Received by: (Signature)	
Received for Laboratory by: (Signature)		Date / Time		Temp.	Seals Intact (Y/N)		Conditions / Remarks				
011											

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files



**LOCKHEED MARTIN**

Lockheed Martin Information Systems & Global Solutions (IS&GS - Civil)  
Environmental Services SERAS  
2890 Woodbridge Avenue, Building 209 Annex  
Edison, NJ 08837-3679  
Telephone: 732-321-4200, Facsimile: 732-494-4021

DATE: 10/21/2013  
TO: R. Singhvi, ERTC/EPA  
FROM: Misty Barkley, Analytical Support Chemist *MB*  
SUBJECT: Preliminary Results of Project: Iron King WA# 0-146.

Attached please find the preliminary results of the above referenced project for the following samples.

Date Received	chain of custody	No. of Samples	Analyst Requested	Matrix	Lab Job No.	Analysis Date	Report Date
09/18/13	06608	19	IVBA	Soil	L14515	10/03/13	10/03/13

cc: G. Depasquale  
J. Patel  
D. Killeen  
D. Aloysius  
T. Johnson

Lockheed Martin

September 30, 2013

Project ID: 4101015884

ACZ Project ID: L14515

**Sample Receipt**

ACZ Laboratories, Inc. (ACZ) received 19 soil samples from Lockheed Martin on September 18, 2013. The samples were received in good condition. Upon receipt, the sample custodian removed the samples from the cooler, inspected the contents, and logged the samples into ACZ's computerized Laboratory Information Management System (LIMS). The samples were assigned ACZ LIMS project number L14515. The custodian verified the sample information entered into the computer against the chain of custody (COC) forms and sample bottle labels.

**Holding Times**

All analyses were performed within EPA recommended holding times.

**Sample Analysis**

These samples were analyzed for inorganic parameters. The individual methods are referenced on both, the ACZ invoice and the analytical reports.

1. For Arsenic, IVBA values flagged with a "B7", arsenic was detected in the Prep Blank (PBS) above the control limits at 0.00287 mg/Kg. No further action was taken since the sample values were >10 times the concentration detected in the PBS.
2. For Arsenic, IVBA values flagged with an "M3", the MS2 recovery was 130.6%. The control limits are 75-125%. No further action was taken since the analyte concentration in the sample is disproportionate to the spike level. The recovery of the associated control sample (LFB) was acceptable.
3. For Arsenic, Total values flagged with an "M3", the MS/MSD recoveries were 216.2% and 72.3% respectively. The control limits are 75-125%. No further action was taken since the analyte concentration in the sample is disproportionate to the spike level. The recovery of the associated control sample (LCSS) was acceptable.
4. For Lead, Total values flagged with an "M3", the MSD recovery was -229.6%. The control limits are 75-125%. No further action was taken since the analyte concentration in the sample is disproportionate to the spike level. The recovery of the associated control sample (LCSS) was acceptable.
5. For Lead, IVBA values flagged with a "D2", the sample required a dilution since lead exceeded the calibration range.

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 417

ACZ Sample ID: **L14515-01**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	3.690		*	mg/L	0.004	0.02	09/26/13 15:03	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		15			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	20800	2550		*	mg/Kg	4	20	09/26/13 20:40	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.062			mg/L	0.002	0.01	09/26/13 15:03	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.165			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.117			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	20800	3760		*	mg/Kg	2	10	09/26/13 20:40	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	90.9		*	%	0.1	0.5	09/18/13 18:07	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:03	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 11:36 09/24/13 13:00	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:18	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 431

ACZ Sample ID: **L14515-02**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.050		*	mg/L	0.004	0.02	09/26/13 15:10	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		24			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	10300	447		*	mg/Kg	2	10	09/26/13 19:13	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.883			mg/L	0.002	0.01	09/26/13 15:10	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		15			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		14			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	10300	572		*	mg/Kg	1	5	09/26/13 19:13	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	97.9		*	%	0.1	0.5	09/18/13 19:11	spj

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:06	spj
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 12:25	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 15:15	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:22	spj

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 442

ACZ Sample ID: **L14515-03**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.379		*	mg/L	0.004	0.02	09/26/13 15:16	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		1.3			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	21000	2990		*	mg/Kg	4	20	09/26/13 20:56	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.165			mg/L	0.002	0.01	09/26/13 15:16	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.306			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.241			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	21000	5390		*	mg/Kg	2	10	09/26/13 20:56	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	80.8		*	%	0.1	0.5	09/18/13 20:15	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:09	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 12:41	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 16:45	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:26	spl

# ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

## Inorganic Analytical Results

### Lockheed Martin

Project ID: 4101015884  
Sample ID: 451

ACZ Sample ID: L14515-04  
Date Sampled: 04/30/13 00:00  
Date Received: 09/18/13  
Sample Matrix: Soil

#### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.380		*	mg/L	0.004	0.02	09/26/13 15:26	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		24			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	10300	585		*	mg/Kg	2	10	09/26/13 19:26	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.788			mg/L	0.002	0.01	09/26/13 15:26	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		11			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		9.8			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	10300	704		*	mg/Kg	1	5	09/26/13 19:26	pmc

#### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	94.0		*	%	0.1	0.5	09/18/13 21:19	spl

#### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:12	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 12:57	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 17:30	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:30	spl



**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 467

ACZ Sample ID: **L14515-05**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.700		*	mg/L	0.004	0.02	09/26/13 15:29	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		12			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	10200	1480		*	mg/Kg	2	10	09/26/13 19:29	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.019			mg/L	0.002	0.01	09/26/13 15:29	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	10200	2860		*	mg/Kg	1	5	09/26/13 19:29	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	94.9		*	%	0.1	0.5	09/18/13 22:22	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:15	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 13:13 09/24/13 18:15	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:34	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 477

ACZ Sample ID: **L14515-06**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.844		*	mg/L	0.004	0.02	09/26/13 15:32	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		2.4			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	25500	3580		*	mg/Kg	5	30	09/26/13 20:59	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.418			mg/L	0.002	0.01	09/26/13 15:32	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		1.4			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		1.2			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	25500	2960		*	mg/Kg	3	10	09/26/13 20:59	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	95.4		*	%	0.1	0.5	09/18/13 23:26	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:18	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 13:29	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 19:00	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:38	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 485

ACZ Sample ID: **L14515-07**  
 Date Sampled: 04/30/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analysis
Arsenic (IVBA)	M6020 ICP-MS	20	2.670		*	mg/L	0.004	0.02	09/26/13 15:39	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		6.4			%			09/27/13 10:46	calc
Arsenic, total (3050)	M6020 ICP-MS	41200	4180		*	mg/Kg	8	40	09/26/13 21:03	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.074			mg/L	0.002	0.01	09/26/13 15:39	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.172			%			09/27/13 10:46	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.123			%			09/27/13 10:46	calc
Lead, total (3050)	M6020 ICP-MS	41200	4290		*	mg/Kg	4	20	09/26/13 21:03	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analysis
Solids, Percent	CLPSOW390, PART F, D-98	1	93.9		*	%	0.1	0.5	09/19/13 0:30	spj

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analysis
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:22	spj
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 13:46 09/24/13 19:45	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:42	spj

# ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

## Inorganic Analytical Results

### Lockheed Martin

Project ID: 4101015884  
Sample ID: 486

ACZ Sample ID: **L14515-08**  
Date Sampled: 04/30/13 00:00  
Date Received: 09/18/13  
Sample Matrix: Soil

#### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.070		*	mg/L	0.004	0.02	09/26/13 15:42	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		6.1			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10200	1750		*	mg/Kg	2	10	09/26/13 19:39	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.122			mg/L	0.002	0.01	09/26/13 15:42	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.319			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.252			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10200	3820		*	mg/Kg	1	5	09/26/13 19:39	pmc

#### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	93.2		*	%	0.1	0.5	09/19/13 1:34	spl

#### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:25	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 14:02	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 20:30	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:46	spl

# ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

## Inorganic Analytical Results

### Lockheed Martin

Project ID: 4101015884  
Sample ID: 513

ACZ Sample ID: L14515-09  
Date Sampled: 04/29/13 00:00  
Date Received: 09/18/13  
Sample Matrix: Soil

#### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.860		*	mg/L	0.004	0.02	09/26/13 15:45	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		21			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	888		*	mg/Kg	2	10	09/26/13 19:42	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.008	B	*	mg/L	0.002	0.01	09/26/13 15:45	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.364			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.291			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	220		*	mg/Kg	1	5	09/26/13 19:42	pmc

#### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	97.9		*	%	0.1	0.5	09/19/13 2:38	spl

#### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:28	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 14:18	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/24/13 21:15	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:50	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 515

ACZ Sample ID: **L14515-10**  
 Date Sampled: 04/29/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	200	22.70		*	mg/L	0.04	0.2	09/26/13 16:43	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		57			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10500 0	3960		*	mg/Kg	20	100	09/26/13 19:45	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.004	B	*	mg/L	0.002	0.01	09/26/13 15:48	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.182			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.132			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10500 0	220		*	mg/Kg	10	50	09/26/13 19:45	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	93.0		*	%	0.1	0.5	09/19/13 3:41	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:31	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 14:34 09/24/13 22:00	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:54	spl

# ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

## Inorganic Analytical Results

### Lockheed Martin

Project ID: 4101015884

Sample ID: 527

ACZ Sample ID: **L14515-11**

Date Sampled: 04/29/13 00:00

Date Received: 09/18/13

Sample Matrix: Soil

### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	3.120		*	mg/L	0.004	0.02	09/26/13 15:52	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		4.6			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	50500	6730		*	mg/Kg	10	50	09/26/13 21:06	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.047			mg/L	0.002	0.01	09/26/13 15:52	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	50500	9490		*	mg/Kg	5	30	09/26/13 21:06	pmc

### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-08	1	98.5		*	%	0.1	0.5	09/19/13 4:45	spi

### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:34	spi
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 14:50 09/24/13 22:45	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 12:58	spi

# ACZ Laboratories, Inc.

2773 Downhill Drive Steamboat Springs, CO 80487 (800) 334-5493

## Inorganic Analytical Results

### Lockheed Martin

Project ID: 4101015884

Sample ID: 621

ACZ Sample ID: L14515-12

Date Sampled: 04/30/13 00:00

Date Received: 09/18/13

Sample Matrix: Soil

### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.124		*	mg/L	0.004	0.02	09/26/13 16:04	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		4			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10200	310		*	mg/Kg	2	10	09/26/13 19:51	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.219			mg/L	0.002	0.01	09/26/13 16:04	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		18			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		15			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10200	125		*	mg/Kg	1	5	09/26/13 19:51	pmc

### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	95.3		*	%	0.1	0.5	09/19/13 5:49	spl

### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:37	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 15:06	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/25/13 1:00	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:02	spl



**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 669

ACZ Sample ID: **L14515-13**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.314		*	mg/L	0.004	0.02	09/26/13 16:11	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		10			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	305		*	mg/Kg	2	10	09/26/13 19:55	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.262			mg/L	0.002	0.01	09/26/13 16:11	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		14			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		13			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	184		*	mg/Kg	1	5	09/26/13 19:55	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	98.4		*	%	0.1	0.5	09/19/13 6:53	spj

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:40	spj
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 15:23	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/25/13 2:30	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:06	spj

### Lockheed Martin

Project ID: 4101015884  
 Sample ID: 701

ACZ Sample ID: **L14515-14**  
 Date Sampled: 05/02/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

#### Metals Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.250		*	mg/L	0.004	0.02	09/26/13 16:14	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		15			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	841		*	mg/Kg	2	10	09/26/13 20:04	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.172			mg/L	0.002	0.01	09/26/13 16:14	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		3.5			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		3.1			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	490		*	mg/Kg	1	5	09/26/13 20:04	pmc

#### Soil Analysis

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	98.4		*	%	0.1	0.5	09/19/13 7:57	spl

#### Soil Preparation

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:44	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 15:39 09/25/13 3:15	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:10	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 820

ACZ Sample ID: **L14515-15**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.120		*	mg/L	0.004	0.02	09/26/13 16:17	meh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		17			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10200	660		*	mg/Kg	2	10	09/26/13 20:19	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.019			mg/L	0.002	0.01	09/26/13 16:17	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.171			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.122			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10200	1110		*	mg/Kg	1	5	09/26/13 20:19	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	81.8		*	%	0.1	0.5	09/19/13 9:01	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:47	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 15:55 09/25/13 4:00	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:14	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 861

ACZ Sample ID: **L14515-16**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.281		*	mg/L	0.004	0.02	09/26/13 16:21	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		5.7			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	497		*	mg/Kg	2	10	09/26/13 20:24	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.033			mg/L	0.002	0.01	09/26/13 16:21	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.456			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.373			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	723		*	mg/Kg	1	5	09/26/13 20:24	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	97.8		*	%	0.1	0.5	09/18/13 10:04	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:50	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 16:11 09/25/13 4:45	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:18	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 865

ACZ Sample ID: **L14515-17**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.627		*	mg/L	0.004	0.02	09/26/13 16:27	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		9.7			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	649		*	mg/Kg	2	10	09/26/13 20:27	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.016			mg/L	0.002	0.01	09/26/13 16:27	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.155			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.108			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	1030		*	mg/Kg	1	5	09/26/13 20:27	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	98.0		*	%	0.1	0.5	09/19/13 11:08	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:53	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 16:27 09/25/13 5:30	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:22	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 873

ACZ Sample ID: **L14515-18**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	0.774		*	mg/L	0.004	0.02	09/26/13 16:30	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		11			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10100	680		*	mg/Kg	2	10	09/26/13 20:31	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.033			mg/L	0.002	0.01	09/26/13 16:30	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.369			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.296			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10100	894		*	mg/Kg	1	5	09/26/13 20:31	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	97.1		*	%	0.1	0.5	09/19/13 13:16	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:56	spl
Digestion - Hot Plate	M3050B ICP-MS								09/24/13 16:43	mss2
In Vitro Bioaccessibility Assay	EPA 9200.1-86								09/25/13 6:15	brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:26	spl

**Lockheed Martin**

Project ID: 4101015884  
 Sample ID: 879

ACZ Sample ID: **L14515-19**  
 Date Sampled: 05/01/13 00:00  
 Date Received: 09/18/13  
 Sample Matrix: Soil

**Metals Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Arsenic (IVBA)	M6020 ICP-MS	20	1.160		*	mg/L	0.004	0.02	09/26/13 16:34	msh
Arsenic IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		13			%			09/27/13 10:47	calc
Arsenic, total (3050)	M6020 ICP-MS	10200	892		*	mg/Kg	2	10	09/26/13 20:34	pmc
Lead (IVBA)	M6020 ICP-MS	20	0.007	B	*	mg/L	0.002	0.01	09/26/13 16:34	msh
Lead IVBA% (In Vitro RBA)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:47	calc
Lead RBA (In Vivo Drexler & Brattin Estimation)	Calculation (EPA 9200.1-86)		0.0			%			09/27/13 10:47	calc
Lead, total (3050)	M6020 ICP-MS	10200	1630		*	mg/Kg	1	5	09/26/13 20:34	pmc

**Soil Analysis**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Solids, Percent	CLPSOW390, PART F, D-98	1	95.3		*	%	0.1	0.5	09/19/13 14:20	spl

**Soil Preparation**

Parameter	EPA Method	Dilution	Result	Qual	XQ	Units	MDL	PQL	Date	Analyst
Air Dry at 34 Degrees C	USDA No. 1, 1972								09/18/13 16:59	spl
Digestion - Hot Plate In Vitro Bioaccessibility Assay	M3050B ICP-MS EPA 9200.1-86								09/24/13 16:59 09/25/13 7:00	mss2 brd
Sieve-250 um (60 mesh)	ASA No.9, 15-4.2.2								09/19/13 13:30	spl

CHAIN OF CUSTODY RECORD

L14515



Project Name: \_\_\_\_\_  
 Project Number: SERAS-0146  
 LM Contact: Misty Buckley Phone: 732-321-4205

No: 06608  
 Sheet 01 of 01 (Do not copy)  
 (for addnl. samples use new form)

Sample Identification

Analyses Requested

REACH	Sample No	Sampling Location	Matrix	Date Collected	# of Bottles	Container/Preservation	Analyses Requested
	417	N/A	Soil	4/30/13	1	Ziplock Bag / Cold 4°C	IVBA ✓
	431					Cold 4°C	✓
	442						✓
	451						✓
	467						✓
	477						✓
	485						✓
	486						✓
	513			4/29/13			✓
	515						✓
	527						✓
	621			4/30/13			✓
	669			5/1/13			✓
	701			5/2/13			✓
	820			5/1/13			✓
	861						✓
	865						✓
	873						✓
	879						✓

X

JM  
9/17/13

Matrix: \_\_\_\_\_  
 Special Instructions: Payment = Visa Card  
Call Misty Buckley

**SAMPLES TRANSFERRED FROM**  
**CHAIN OF CUSTODY #:**  
9-3066  
9-3067

- A- Air
- AT- Animal Tissue
- DL- Drum Liquids
- DS- Drum Solids
- GW- Groundwater
- O- Oil
- PR- Product
- PT- Plant Tissue
- PW- Potable Water
- S- Soil
- SD- Sediment
- SL- Sludge
- SW- Surface Water
- TX- TCLP Extract
- W- Water
- X- Other

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Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished by	Date	Received by	Date	Time
All Analysis	<i>[Signature]</i>	9/17/13							<i>[Signature]</i>	9-18-13	0:00





**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/4/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14066C

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

---

Metals by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles  
**Project Number:** R14S06  
**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**  
**75 Hawthorne Street**  
**San Francisco CA, 94105**

**SDG:** 14066C  
**Reported:** 04/04/14 10:32

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
GAL-01	1403013-01	Soil	02/28/14 12:05	03/07/14 13:08
GAL-02	1403013-02	Soil	02/28/14 12:30	03/07/14 13:08
GAL-03	1403013-03	Soil	02/28/14 13:20	03/07/14 13:08
GAL-04	1403013-04	Soil	02/28/14 13:30	03/07/14 13:08
GAL-05	1403013-05	Soil	02/28/14 13:45	03/07/14 13:08
GAL-06	1403013-06	Soil	02/28/14 13:30	03/07/14 13:08
MTP-01	1403013-07	Soil	02/27/14 15:30	03/07/14 13:08
MTP-02	1403013-08	Soil	02/27/14 15:45	03/07/14 13:08
MTP-03	1403013-09	Soil	02/27/14 15:45	03/07/14 13:08
MTP-04	1403013-10	Soil	02/27/14 16:10	03/07/14 13:08
MTP-05	1403013-11	Soil	02/27/14 16:15	03/07/14 13:08
MTP-06	1403013-12	Soil	02/27/14 16:25	03/07/14 13:08
MTP-07	1403013-13	Soil	02/27/14 16:50	03/07/14 13:08
MTP-08	1403013-14	Soil	02/27/14 17:00	03/07/14 13:08
MTP-09	1403013-15	Soil	02/27/14 17:05	03/07/14 13:08
MTP-10	1403013-16	Soil	02/27/14 17:10	03/07/14 13:08

**SDG ID 14066C**

All samples were dried and sieved prior to preparation for analysis.

Samples were digested per EPA method SW 3050.

**Work Order(s)**

**1403013**



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:32
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403013-01</b>								<b>Soil - Sampled: 02/28/14 12:05</b>
<b>Sample ID:</b>	<b>GAL-01</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,300		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,700		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-02</b>								<b>Soil - Sampled: 02/28/14 12:30</b>
<b>Sample ID:</b>	<b>GAL-02</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		170		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		180		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-03</b>								<b>Soil - Sampled: 02/28/14 13:20</b>
<b>Sample ID:</b>	<b>GAL-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		710		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,900		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-04</b>								<b>Soil - Sampled: 02/28/14 13:30</b>
<b>Sample ID:</b>	<b>GAL-04</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		2,700		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		510		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-05</b>								<b>Soil - Sampled: 02/28/14 13:45</b>
<b>Sample ID:</b>	<b>GAL-05</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		650		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		810		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-06</b>								<b>Soil - Sampled: 02/28/14 13:30</b>
<b>Sample ID:</b>	<b>GAL-06</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		2,500		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		480		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-07</b>								<b>Soil - Sampled: 02/27/14 15:30</b>
<b>Sample ID:</b>	<b>MTP-01</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		5,100		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		2,500		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-08</b>								<b>Soil - Sampled: 02/27/14 15:45</b>
<b>Sample ID:</b>	<b>MTP-02</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		4,300		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		3,000		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-09</b>								<b>Soil - Sampled: 02/27/14 15:45</b>
<b>Sample ID:</b>	<b>MTP-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		310		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		770		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-10</b>								<b>Soil - Sampled: 02/27/14 16:10</b>
<b>Sample ID:</b>	<b>MTP-04</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,800		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,900	J, Q4	3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-11</b>								<b>Soil - Sampled: 02/27/14 16:15</b>



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:32
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403013-11</b>								<b>Soil - Sampled: 02/27/14 16:15</b>
<b>Sample ID:</b>	<b>MTP-05</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,300		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,200		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-12</b>								<b>Soil - Sampled: 02/27/14 16:25</b>
<b>Sample ID:</b>	<b>MTP-06</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		2,100		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,300		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-13</b>								<b>Soil - Sampled: 02/27/14 16:50</b>
<b>Sample ID:</b>	<b>MTP-07</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,000		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		970		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-14</b>								<b>Soil - Sampled: 02/27/14 17:00</b>
<b>Sample ID:</b>	<b>MTP-08</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,500		2	mg/kg	B14C037	03/19/14	03/25/14	6010C/SOP503
Lead		1,500		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-15</b>								<b>Soil - Sampled: 02/27/14 17:05</b>
<b>Sample ID:</b>	<b>MTP-09</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		2,700		2	mg/kg	B14C037	03/19/14	03/26/14	6010C/SOP503
Lead		1,300		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-16</b>								<b>Soil - Sampled: 02/27/14 17:10</b>
<b>Sample ID:</b>	<b>MTP-10</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		890		2	mg/kg	B14C037	03/19/14	03/26/14	6010C/SOP503
Lead		580		3	"	"	"	"	6010C/SOP503



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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:32
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
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Batch B14C037 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 03/19/14 Analyzed: 03/25/14  
Metals by EPA 6000/7000 Series Methods - Quality Control

**Blank (B14C037-BLK1)**

Arsenic	ND	U		2 mg/kg						
Lead	ND	U		3 "						

**Matrix Spike (B14C037-MS1)**

Source: 1403013-10

Arsenic	2,200			2 mg/kg	392	1,830	94	75-125		20
Lead	2,000	Q10		3 "	98.0	1,940	55	75-125		20

**Matrix Spike Dup (B14C037-MSD1)**

Source: 1403013-10

Arsenic	2,220			2 mg/kg	388	1,830	101	75-125	1	20
Lead	2,000	Q10		3 "	97.1	1,940	55	75-125	0.01	20

**Reference (B14C037-SRM1)**

Arsenic	286			2 mg/kg	252		113	60.9-139		
Lead	54.9			3 "	56.7		97	72.8-127		



United States Environmental Protection Agency  
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**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14066C

**Reported:** 04/04/14 10:32

**Qualifiers and Comments**

Q4 The matrix spike and/or matrix spike duplicate associated with this sample did not meet recovery criteria for this analyte (see MS/MSD results for this batch in QC summary)

Q10 The analyte concentration in the unfortified sample is significantly greater than the concentration spiked into the matrix spike and matrix spike duplicate. The reported spike recovery is not a meaningful measure of the dataset's analytical accuracy.

J The reported result for this analyte should be considered an estimated value.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/4/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14066C

**From:** Brenda Bettencourt, Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

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Analysis of In Vitro Gastric Extracts by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14066C

**Reported:** 04/04/14 10:39

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
GAL-01	1403013-01	Soil	02/28/14 12:05	03/07/14 13:08
GAL-02	1403013-02	Soil	02/28/14 12:30	03/07/14 13:08
GAL-03	1403013-03	Soil	02/28/14 13:20	03/07/14 13:08
GAL-04	1403013-04	Soil	02/28/14 13:30	03/07/14 13:08
GAL-05	1403013-05	Soil	02/28/14 13:45	03/07/14 13:08
GAL-06	1403013-06	Soil	02/28/14 13:30	03/07/14 13:08
MTP-01	1403013-07	Soil	02/27/14 15:30	03/07/14 13:08
MTP-02	1403013-08	Soil	02/27/14 15:45	03/07/14 13:08
MTP-03	1403013-09	Soil	02/27/14 15:45	03/07/14 13:08
MTP-04	1403013-10	Soil	02/27/14 16:10	03/07/14 13:08
MTP-05	1403013-11	Soil	02/27/14 16:15	03/07/14 13:08
MTP-06	1403013-12	Soil	02/27/14 16:25	03/07/14 13:08
MTP-07	1403013-13	Soil	02/27/14 16:50	03/07/14 13:08
MTP-08	1403013-14	Soil	02/27/14 17:00	03/07/14 13:08
MTP-09	1403013-15	Soil	02/27/14 17:05	03/07/14 13:08
MTP-10	1403013-16	Soil	02/27/14 17:10	03/07/14 13:08

**SDG ID 14066C**

All samples were dried and sieved prior to preparation for analysis.

Samples were subjected to an in-vitro extraction prior to digestion and analysis. Results reported are the available metal in the soil after extraction.

**Work Order(s)**

**1403013**





**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:39
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403013-01</b>								<b>Soil - Sampled: 02/28/14 12:05</b>
<b>Sample ID:</b>	<b>GAL-01</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		41		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		58		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-02</b>								<b>Soil - Sampled: 02/28/14 12:30</b>
<b>Sample ID:</b>	<b>GAL-02</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		7.1		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		46		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-03</b>								<b>Soil - Sampled: 02/28/14 13:20</b>
<b>Sample ID:</b>	<b>GAL-03</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		24		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		470		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-04</b>								<b>Soil - Sampled: 02/28/14 13:30</b>
<b>Sample ID:</b>	<b>GAL-04</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		930		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		8.8		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-05</b>								<b>Soil - Sampled: 02/28/14 13:45</b>
<b>Sample ID:</b>	<b>GAL-05</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		66		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		32		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-06</b>								<b>Soil - Sampled: 02/28/14 13:30</b>
<b>Sample ID:</b>	<b>GAL-06</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		920		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		11		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-07</b>								<b>Soil - Sampled: 02/27/14 15:30</b>
<b>Sample ID:</b>	<b>MTP-01</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		170		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		22		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-08</b>								<b>Soil - Sampled: 02/27/14 15:45</b>
<b>Sample ID:</b>	<b>MTP-02</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		400		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		2,100		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-09</b>								<b>Soil - Sampled: 02/27/14 15:45</b>
<b>Sample ID:</b>	<b>MTP-03</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		59		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		120		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-10</b>								<b>Soil - Sampled: 02/27/14 16:10</b>
<b>Sample ID:</b>	<b>MTP-04</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		340		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		20		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-11</b>								<b>Soil - Sampled: 02/27/14 16:15</b>



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:39
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403013-11</b>								<b>Soil - Sampled: 02/27/14 16:15</b>
<b>Sample ID:</b>	<b>MTP-05</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		390		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		27		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-12</b>								<b>Soil - Sampled: 02/27/14 16:25</b>
<b>Sample ID:</b>	<b>MTP-06</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		260		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		27		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-13</b>								<b>Soil - Sampled: 02/27/14 16:50</b>
<b>Sample ID:</b>	<b>MTP-07</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		230		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		12		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-14</b>								<b>Soil - Sampled: 02/27/14 17:00</b>
<b>Sample ID:</b>	<b>MTP-08</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		360		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		28		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-15</b>								<b>Soil - Sampled: 02/27/14 17:05</b>
<b>Sample ID:</b>	<b>MTP-09</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		650		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		13		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403013-16</b>								<b>Soil - Sampled: 02/27/14 17:10</b>
<b>Sample ID:</b>	<b>MTP-10</b>								<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic		110		2	mg/kg	B14C099	03/21/14	03/25/14	6010C/SOP503
Lead		23		3	"	"	"	"	6010C/SOP503



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14066C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/04/14 10:39
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B14C099 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>										
<b>Prepared: 03/21/14 Analyzed: 03/25/14</b>										
<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>										
<b>Blank (B14C099-BLK1)</b>										
Arsenic	1.3	C1, J		2 mg/kg						
Lead	ND	U		3 "						
<b>Blank (B14C099-BLK2)</b>										
Arsenic	1.3	C1, J		2 mg/kg						
Lead	ND	U		3 "						
<b>LCS (B14C099-BS1)</b>										
Arsenic	390			2 mg/kg	400		97	80-120		200
Lead	92.5			3 "	100		93	80-120		200
<b>Duplicate (B14C099-DUP1) Source: 1403013-10</b>										
Arsenic	328			2 mg/kg		335			2	20
Lead	18			3 "		20.2			12	20
<b>Matrix Spike (B14C099-MS1) Source: 1403013-10</b>										
Arsenic	4,260			2 mg/kg	4000	335	98	75-125		20
Lead	919			3 "	1000	20.2	90	75-125		20
<b>Reference (B14C099-SRM1)</b>										
Arsenic	56.7			2 mg/kg	89.0		64	0-200		
Lead	1,020			3 "	1300		78	0-200		



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**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14066C

**Reported:** 04/04/14 10:39

**Qualifiers and Comments**

J The reported result for this analyte should be considered an estimated value.

C1 The reported concentration for this analyte is below the quantitation limit.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/30/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14087B

**From:** Duane James, Acting Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

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Metals by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles  
**Project Number:** R14S06  
**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**  
**75 Hawthorne Street**  
**San Francisco CA, 94105**

**SDG:** 14087B  
**Reported:** 04/30/14 16:59

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
2216-02	1403073-01	Soil	03/05/14 16:24	03/28/14 11:15
106-04	1403073-02	Soil	02/26/14 15:00	03/28/14 11:15
108-03	1403073-03	Soil	02/24/14 09:40	03/28/14 11:15
109-11	1403073-04	Soil	02/19/14 10:56	03/28/14 11:15
126-14	1403073-05	Soil	02/27/14 16:42	03/28/14 11:15
2014-08	1403073-06	Soil	01/31/14 10:59	03/28/14 11:15
2324-03	1403073-07	Soil	02/05/14 14:41	03/28/14 11:15
2328-02	1403073-08	Soil	02/05/14 10:18	03/28/14 11:15
2408-01	1403073-09	Soil	03/10/14 10:45	03/28/14 11:15
2410-03	1403073-10	Soil	03/10/14 16:13	03/28/14 11:15
2426-09	1403073-11	Soil	02/05/14 15:46	03/28/14 11:15
2519-10	1403073-12	Soil	03/10/14 12:00	03/28/14 11:15
2523-05	1403073-13	Soil	02/19/14 15:35	03/28/14 11:15
2602-09	1403073-14	Soil	02/13/14 14:16	03/28/14 11:15
2615-03	1403073-15	Soil	02/20/14 10:53	03/28/14 11:15
2743D-11	1403073-16	Soil	02/24/14 15:27	03/28/14 11:15
2755-07	1403073-17	Soil	02/22/14 13:40	03/28/14 11:15
2808-15	1403073-18	Soil	02/21/14 14:35	03/28/14 11:15
2901-06	1403073-19	Soil	02/26/14 10:02	03/28/14 11:15
3004-08	1403073-20	Soil	03/03/14 14:50	03/28/14 11:15

**SDG ID 14087B**

All samples were dried and sieved prior to preparation for analysis.

Samples were digested per EPA method SW 3050B.

**Work Order(s)**

**1403073**



**United States Environmental Protection Agency  
Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 16:59
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403073-01</b>								<b>Soil - Sampled: 03/05/14 16:24</b>
<b>Sample ID:</b>	<b>2216-02</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		280		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		350		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-02</b>								<b>Soil - Sampled: 02/26/14 15:00</b>
<b>Sample ID:</b>	<b>106-04</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		250		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		21		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-03</b>								<b>Soil - Sampled: 02/24/14 09:40</b>
<b>Sample ID:</b>	<b>108-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		420		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		770		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-04</b>								<b>Soil - Sampled: 02/19/14 10:56</b>
<b>Sample ID:</b>	<b>109-11</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		170		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		230		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-05</b>								<b>Soil - Sampled: 02/27/14 16:42</b>
<b>Sample ID:</b>	<b>126-14</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		180		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		22		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-06</b>								<b>Soil - Sampled: 01/31/14 10:59</b>
<b>Sample ID:</b>	<b>2014-08</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		310		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		300		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-07</b>								<b>Soil - Sampled: 02/05/14 14:41</b>
<b>Sample ID:</b>	<b>2324-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		230		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		170		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-08</b>								<b>Soil - Sampled: 02/05/14 10:18</b>
<b>Sample ID:</b>	<b>2328-02</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		780		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		520		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-09</b>								<b>Soil - Sampled: 03/10/14 10:45</b>
<b>Sample ID:</b>	<b>2408-01</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		220		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		840		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-10</b>								<b>Soil - Sampled: 03/10/14 16:13</b>
<b>Sample ID:</b>	<b>2410-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		290		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		2,000		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-11</b>								<b>Soil - Sampled: 02/05/14 15:46</b>



**United States Environmental Protection Agency  
Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 16:59
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403073-11</b>								<b>Soil - Sampled: 02/05/14 15:46</b>
<b>Sample ID:</b>	<b>2426-09</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		340		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		65		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-12</b>								<b>Soil - Sampled: 03/10/14 12:00</b>
<b>Sample ID:</b>	<b>2519-10</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		160		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		47		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-13</b>								<b>Soil - Sampled: 02/19/14 15:35</b>
<b>Sample ID:</b>	<b>2523-05</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		170		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		220		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-14</b>								<b>Soil - Sampled: 02/13/14 14:16</b>
<b>Sample ID:</b>	<b>2602-09</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		140		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead	RE1	15,000		15	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-15</b>								<b>Soil - Sampled: 02/20/14 10:53</b>
<b>Sample ID:</b>	<b>2615-03</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		1,200		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		16		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-16</b>								<b>Soil - Sampled: 02/24/14 15:27</b>
<b>Sample ID:</b>	<b>2743D-11</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		650		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		9.5		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-17</b>								<b>Soil - Sampled: 02/22/14 13:40</b>
<b>Sample ID:</b>	<b>2755-07</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		150		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		34		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-18</b>								<b>Soil - Sampled: 02/21/14 14:35</b>
<b>Sample ID:</b>	<b>2808-15</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		410		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		18		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-19</b>								<b>Soil - Sampled: 02/26/14 10:02</b>
<b>Sample ID:</b>	<b>2901-06</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		160		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		14		3	"	"	"	"	6010C/SOP503
<b>Lab ID:</b>	<b>1403073-20</b>								<b>Soil - Sampled: 03/03/14 14:50</b>
<b>Sample ID:</b>	<b>3004-08</b>								<b>Metals by EPA 6000/7000 Series Methods</b>
Arsenic		260		2	mg/kg	B14D031	04/07/14	04/22/14	6010C/SOP503
Lead		650		3	"	"	"	"	6010C/SOP503





**United States Environmental Protection Agency**  
**Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 16:59
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
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Batch B14D031 - 3050B Sld Acid Dig - Metals by 6010

Prepared: 04/07/14 Analyzed: 04/22/14  
 Metals by EPA 6000/7000 Series Methods - Quality Control

**Blank (B14D031-BLK1)**

Arsenic	ND	U		2 mg/kg						
Lead	ND	U		3 "						

**Matrix Spike (B14D031-MS1)**

Source: 1403073-01

Arsenic	695			2 mg/kg	400	280	104	75-125		20
Lead	449			3 "	100	347	101	75-125		20

**Matrix Spike (B14D031-MS2)**

Source: 1403073-02

Arsenic	661			2 mg/kg	396	253	103	75-125		20
Lead	110			3 "	99.0	20.9	90	75-125		20

**Matrix Spike Dup (B14D031-MSD1)**

Source: 1403073-01

Arsenic	712			2 mg/kg	400	280	108	75-125		2
Lead	447			3 "	100	347	99	75-125	0.4	20

**Matrix Spike Dup (B14D031-MSD2)**

Source: 1403073-02

Arsenic	667			2 mg/kg	396	253	105	75-125		1
Lead	110			3 "	99.0	20.9	90	75-125	0.04	20

**Reference (B14D031-SRM1)**

Arsenic	268			2 mg/kg	254		105	60.9-139		
Lead	52.6			3 "	57.2		92	72.8-127		



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**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087B

**Reported:** 04/30/14 16:59

**Qualifiers and Comments**

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/30/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14087B

**From:** Duane James, Acting Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

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Analysis of In Vitro Gastric Extracts by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles  
**Project Number:** R14S06  
**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**  
**75 Hawthorne Street**  
**San Francisco CA, 94105**

**SDG:** 14087B  
**Reported:** 04/30/14 17:04

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
2216-02	1403073-01	Soil	03/05/14 16:24	03/28/14 11:15
106-04	1403073-02	Soil	02/26/14 15:00	03/28/14 11:15
108-03	1403073-03	Soil	02/24/14 09:40	03/28/14 11:15
109-11	1403073-04	Soil	02/19/14 10:56	03/28/14 11:15
126-14	1403073-05	Soil	02/27/14 16:42	03/28/14 11:15
2014-08	1403073-06	Soil	01/31/14 10:59	03/28/14 11:15
2324-03	1403073-07	Soil	02/05/14 14:41	03/28/14 11:15
2328-02	1403073-08	Soil	02/05/14 10:18	03/28/14 11:15
2408-01	1403073-09	Soil	03/10/14 10:45	03/28/14 11:15
2410-03	1403073-10	Soil	03/10/14 16:13	03/28/14 11:15
2426-09	1403073-11	Soil	02/05/14 15:46	03/28/14 11:15
2519-10	1403073-12	Soil	03/10/14 12:00	03/28/14 11:15
2523-05	1403073-13	Soil	02/19/14 15:35	03/28/14 11:15
2602-09	1403073-14	Soil	02/13/14 14:16	03/28/14 11:15
2615-03	1403073-15	Soil	02/20/14 10:53	03/28/14 11:15
2743D-11	1403073-16	Soil	02/24/14 15:27	03/28/14 11:15
2755-07	1403073-17	Soil	02/22/14 13:40	03/28/14 11:15
2808-15	1403073-18	Soil	02/21/14 14:35	03/28/14 11:15
2901-06	1403073-19	Soil	02/26/14 10:02	03/28/14 11:15
3004-08	1403073-20	Soil	03/03/14 14:50	03/28/14 11:15

**SDG ID 14087B**

All samples were dried and sieved prior to preparation for analysis.

Samples were subjected to an in-vitro extraction prior to digestion and analysis. Results reported are the available metal in the soil after extraction.

**Work Order(s)**

**1403073**



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 17:04
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b> 1403073-01									<b>Soil - Sampled: 03/05/14 16:24</b>
<b>Sample ID:</b> 2216-02									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	17		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		190		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-02									<b>Soil - Sampled: 02/26/14 15:00</b>
<b>Sample ID:</b> 106-04									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	23		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		6.0		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-03									<b>Soil - Sampled: 02/24/14 09:40</b>
<b>Sample ID:</b> 108-03									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	110		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		280		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-04									<b>Soil - Sampled: 02/19/14 10:56</b>
<b>Sample ID:</b> 109-11									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	47		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		140		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-05									<b>Soil - Sampled: 02/27/14 16:42</b>
<b>Sample ID:</b> 126-14									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	13		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		7.8		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-06									<b>Soil - Sampled: 01/31/14 10:59</b>
<b>Sample ID:</b> 2014-08									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	45		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		19		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-07									<b>Soil - Sampled: 02/05/14 14:41</b>
<b>Sample ID:</b> 2324-03									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	42		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		59		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-08									<b>Soil - Sampled: 02/05/14 10:18</b>
<b>Sample ID:</b> 2328-02									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	130		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		200		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-09									<b>Soil - Sampled: 03/10/14 10:45</b>
<b>Sample ID:</b> 2408-01									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	24		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		69		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-10									<b>Soil - Sampled: 03/10/14 16:13</b>
<b>Sample ID:</b> 2410-03									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	28		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		160		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-11									<b>Soil - Sampled: 02/05/14 15:46</b>



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Region 9 Laboratory**

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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 17:04
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b> 1403073-11									<b>Soil - Sampled: 02/05/14 15:46</b>
<b>Sample ID:</b> 2426-09									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	18		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		26		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-12									<b>Soil - Sampled: 03/10/14 12:00</b>
<b>Sample ID:</b> 2519-10									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	20		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		24		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-13									<b>Soil - Sampled: 02/19/14 15:35</b>
<b>Sample ID:</b> 2523-05									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	31		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		77		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-14									<b>Soil - Sampled: 02/13/14 14:16</b>
<b>Sample ID:</b> 2602-09									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	24		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead	RE2	12,000		30	"	"	"	04/26/14	6010C/SOP503
<b>Lab ID:</b> 1403073-15									<b>Soil - Sampled: 02/20/14 10:53</b>
<b>Sample ID:</b> 2615-03									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	49		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		2.9	C1, J	3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-16									<b>Soil - Sampled: 02/24/14 15:27</b>
<b>Sample ID:</b> 2743D-11									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	39		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		ND	U	3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-17									<b>Soil - Sampled: 02/22/14 13:40</b>
<b>Sample ID:</b> 2755-07									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	11		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		16		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-18									<b>Soil - Sampled: 02/21/14 14:35</b>
<b>Sample ID:</b> 2808-15									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	15		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		5.7		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-19									<b>Soil - Sampled: 02/26/14 10:02</b>
<b>Sample ID:</b> 2901-06									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	15		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		5.5		3	"	"	"	04/23/14	6010C/SOP503
<b>Lab ID:</b> 1403073-20									<b>Soil - Sampled: 03/03/14 14:50</b>
<b>Sample ID:</b> 3004-08									<b>Analysis of In Vitro Gastric Digestion Extracts</b>
Arsenic	RE1	51		2	mg/kg	B14D040	04/10/14	04/25/14	6010C/SOP503
Lead		480		3	"	"	"	04/24/14	6010C/SOP503



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<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087B
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 17:04
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B14D040 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>										
<b>Prepared: 04/10/14 Analyzed: 04/23/14</b>										
<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>										
<b>Blank (B14D040-BLK1)</b>										
Lead	ND	U		3 mg/kg						
<b>Blank (B14D040-BLK2)</b>										
Lead	ND	U		3 mg/kg						
<b>Blank (B14D040-BLK3)</b>										
Arsenic	1.2	C1, J		2 mg/kg						
<b>Blank (B14D040-BLK4)</b>										
Arsenic	1.4	C1, J		2 mg/kg						
<b>LCS (B14D040-BS2)</b>										
Arsenic	2,060			2 mg/kg	2000		103	80-120		200
Lead	529			3 "	500		106	80-120		200
<b>Duplicate (B14D040-DUP1)</b>										
<b>Source: 1403073-01</b>										
Lead	191			3 mg/kg		186			2	20
<b>Duplicate (B14D040-DUP2)</b>										
<b>Source: 1403073-02</b>										
Lead	5.48			3 mg/kg		6.04			10	20
<b>Duplicate (B14D040-DUP3)</b>										
<b>Source: 1403073-01RE1</b>										
Arsenic	17.6			2 mg/kg		17.1			3	20
<b>Duplicate (B14D040-DUP4)</b>										
<b>Source: 1403073-02RE1</b>										
Arsenic	21.2			2 mg/kg		22.9			8	20
<b>Matrix Spike (B14D040-MS1)</b>										
<b>Source: 1403073-01</b>										
Lead	1,120			3 mg/kg	990	186	94	75-125		20
<b>Matrix Spike (B14D040-MS2)</b>										
<b>Source: 1403073-02</b>										
Lead	918			3 mg/kg	1000	6.04	91	75-125		20
<b>Matrix Spike (B14D040-MS3)</b>										
<b>Source: 1403073-01RE1</b>										
Arsenic	4,280			2 mg/kg	3960	17.1	108	75-125		20
<b>Matrix Spike (B14D040-MS4)</b>										
<b>Source: 1403073-02RE1</b>										
Arsenic	4,370			2 mg/kg	4000	22.9	109	75-125		20
<b>Reference (B14D040-SRM2)</b>										
Lead	1,050			3 mg/kg	1290		82	0-200		
<b>Reference (B14D040-SRM3)</b>										
Arsenic	63.1			2 mg/kg	89.0		71	0-200		
Lead	1,190			3 "	1300		91	0-200		
<b>Reference (B14D040-SRM4)</b>										
Arsenic	61.6			2 mg/kg	88.1		70	0-200		



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**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087B

**Reported:** 04/30/14 17:04

**Qualifiers and Comments**

J The reported result for this analyte should be considered an estimated value.

C1 The reported concentration for this analyte is below the quantitation limit.

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.





**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/30/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14087C

**From:** Duane James, Acting Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

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Metals by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087C

**Reported:** 04/30/14 17:14

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
3005-18	1403074-01	Soil	03/04/14 16:41	03/28/14 11:15

**SDG ID 14087C**

All samples were dried and sieved prior to preparation for analysis.

Samples were digested per EPA method SW 3050B.

**Work Order(s)**

**1403074**



**United States Environmental Protection Agency**  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
 Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 17:14
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403074-01</b>							<b>Soil - Sampled:</b>	<b>03/04/14 16:41</b>
<b>Sample ID:</b>	<b>3005-18</b>							<b>Metals by EPA 6000/7000 Series Methods</b>	
Arsenic	RE2	230		2	mg/kg	B14D140	04/25/14	04/26/14	6010C/SOP503
Lead	RE2	450		3	"	"	"	"	6010C/SOP503

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B14D140 - 3050B Sld Acid Dig - Metals by 6010</b>						<b>Prepared: 04/25/14 Analyzed: 04/26/14</b>				
						<b>Metals by EPA 6000/7000 Series Methods - Quality Control</b>				
<b>Blank (B14D140-BLK1)</b>										
Arsenic	ND	U		2	mg/kg					
Lead	ND	U		3	"					
<b>Matrix Spike (B14D140-MS1) Source: 1403074-01RE2</b>										
Arsenic	666			2	mg/kg	388	226	113	75-125	20
Lead	543			3	"	97.1	449	97	75-125	20
<b>Matrix Spike Dup (B14D140-MSD1) Source: 1403074-01RE2</b>										
Arsenic	662			2	mg/kg	396	226	110	75-125	0.6
Lead	532			3	"	99.0	449	84	75-125	2
<b>Reference (B14D140-SRM1)</b>										
Arsenic	255			2	mg/kg	252		101	60.9-139	
Lead	53.1			3	"	56.6		94	72.8-127	



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**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087C

**Reported:** 04/30/14 17:14

**Qualifiers and Comments**

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.



**United States Environmental Protection Agency  
Region 9 Laboratory**

**1337 S. 46th Street Building 201  
Richmond, CA 94804**

**Date:** 4/30/2014

**Subject:** Analytical Testing Results - Project R14S06  
SDG: 14087C

**From:** Duane James, Acting Director  
EPA Region 9 Laboratory  
MTS-2

**To:** Zi Zi Searles  
California Site Cleanup Section 1  
SFD-7-1

Attached are the results from the analysis of samples from the **Iron King Mine 2013/2014 Sampling** project. These data have been reviewed in accordance with EPA Region 9 Laboratory policy.

A full documentation package for these data, including raw data and sample custody documentation, is on file at the EPA Region 9 Laboratory. If you would like to request additional review and/or validation of the data, please contact Eugenia McNaughton at the Region 9 Quality Assurance Office.

If you have any questions, please ask for Richard Bauer, the Lab Project Manager at (510)412-2300.

Electronic CC: David Aloysius, SERAS  
Donna Getty, SERAS  
Jeff Dhont, EPA Region 9

**Analyses included in this report:**

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Analysis of In Vitro Gastric Extracts by ICP



United States Environmental Protection Agency  
**Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087C

**Reported:** 04/30/14 17:08

**ANALYTICAL REPORT FOR SAMPLES**

Sample ID	Laboratory ID	Matrix	Date Collected	Date Received
3005-18	1403074-01	Soil	03/04/14 16:41	03/28/14 11:15

**SDG ID 14087C**

All samples were dried and sieved prior to preparation for analysis.

Samples were subjected to an in-vitro extraction prior to digestion and analysis. Results reported are the available metal in the soil after extraction.

**Work Order(s)**

**1403074**



**United States Environmental Protection Agency  
Region 9 Laboratory**

1337 S. 46th Street, Building 201, Richmond, CA 94804  
Phone:(510) 412-2300 Fax:(510) 412-2302

<b>Project Manager:</b> Zi Zi Searles	<b>California Site Cleanup Section 1</b>	<b>SDG:</b> 14087C
<b>Project Number:</b> R14S06	<b>75 Hawthorne Street</b>	<b>Reported:</b> 04/30/14 17:08
<b>Project:</b> Iron King Mine 2013/2014 Sampling	<b>San Francisco CA, 94105</b>	

**Sample Results**

Analyte	Reanalysis / Extract	Result	Qualifiers / Comments	Quantitation Limit	Units	Batch	Prepared	Analyzed	Method
<b>Lab ID:</b>	<b>1403074-01</b>							<b>Soil - Sampled: 03/04/14 16:41</b>	
<b>Sample ID:</b>	<b>3005-18</b>							<b>Analysis of In Vitro Gastric Digestion Extracts</b>	
Arsenic	RE1	52		2	mg/kg	B14D046	04/10/14	04/23/14	6010C/SOP503
Lead		310		3	"	"	"	04/22/14	6010C/SOP503

**Quality Control**

Analyte	Result	Qualifiers / Comments	Quantitation Limit	Units	Spike Level	Source Result	%REC	%REC Limits	RPD	RPD Limit
<b>Batch B14D046 - In Vitro Gastric Extraction - In Vitro Extraction, ICP, Sample Basis</b>						<b>Prepared: 04/10/14 Analyzed: 04/22/14</b>				
<b>Blank (B14D046-BLK1)</b>						<b>Analysis of In Vitro Gastric Digestion Extracts - Quality Control</b>				
Lead	ND	U		3 mg/kg						
<b>Blank (B14D046-BLK2)</b>										
Lead	ND	U		3 mg/kg						
<b>Blank (B14D046-BLK3)</b>										
Arsenic	3.1			2 mg/kg						
<b>Blank (B14D046-BLK4)</b>										
Arsenic	3.3			2 mg/kg						
<b>LCS (B14D046-BS1)</b>										
Lead	537			3 mg/kg	500		107	80-120		200
<b>LCS (B14D046-BS2)</b>										
Arsenic	1,950			2 mg/kg	2000		97	80-120		200
<b>Duplicate (B14D046-DUP1)</b>						<b>Source: 1403074-01</b>				
Lead	313			3 mg/kg		312			0.3	20
<b>Duplicate (B14D046-DUP2)</b>						<b>Source: 1403074-01RE1</b>				
Arsenic	50.4			2 mg/kg		51.7			2	20
<b>Matrix Spike (B14D046-MS1)</b>						<b>Source: 1403074-01</b>				
Lead	1,350			3 mg/kg	990	312	105	75-125		20
<b>Matrix Spike (B14D046-MS2)</b>						<b>Source: 1403074-01RE1</b>				
Arsenic	3,910			2 mg/kg	3960	51.7	97	75-125		20
<b>Reference (B14D046-SRM1)</b>										
Lead	1,180			3 mg/kg	1260		93	0-200		
<b>Reference (B14D046-SRM2)</b>										
Arsenic	64.5			2 mg/kg	86.4		75	0-200		



United States Environmental Protection Agency  
**Region 9 Laboratory**

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**Project Manager:** Zi Zi Searles

**Project Number:** R14S06

**Project:** Iron King Mine 2013/2014 Sampling

**California Site Cleanup Section 1**

**75 Hawthorne Street**

**San Francisco CA, 94105**

**SDG:** 14087C

**Reported:** 04/30/14 17:08

**Qualifiers and Comments**

U Not Detected

NR Not Reported

RE1, RE2, etc: Result is from a sample re-analysis.