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**APPENDIX G**

Summary of the Phase I Sludge Drying Bed Pilot Test  
Aspen Seep Bioreactor

# Atlantic Richfield Company

**Anthony R. Brown**  
Project Manager Mining

4 Centerpointe Drive  
La Palma, CA 90623-1066  
Office: (714) 228-6770  
Fax: (714) 228-6749  
E-mail: Anthony.Brown@bp.com

October 19, 2010

Mr. Gary Riley  
SFD-7-2  
US EPA Region 9  
75 Hawthorne Street  
San Francisco, CA 94105

**RE: Summary of the Phase I Sludge Drying Bed Pilot Test  
Aspen Seep Bioreactor  
Leviathan Mine  
Alpine County, California**

Dear Mr. Riley:

This letter presents the results Phase I of the Aspen Seep Bioreactor Sludge Drying Bed Pilot Test (SDB Pilot) conducted by Atlantic Richfield Company (Atlantic Richfield). The SDB Pilot was conducted in accordance with Amendment #2 – Pilot-Scale Sludge Drying Bed Test, Aspen Seep Bioreactor (dated September 16, 2009; approved by EPA on September 21, 2009) to Atlantic Richfield's 2009 Removal Action Work Plan (RAWP) dated March 1, 2009 prepared for the Leviathan Mine Site (site).

The Aspen Seep Bioreactor (ASB) requires periodic sludge removal for effective operation. The SDB Pilot test was planned to evaluate the effectiveness and potential cost savings of the drying bed technology for dewatering ASB sludge when compared to belt press and centrifuge dewatering technologies that have been previously used at the site. This memorandum summarizes the rationale, the design, monitoring, results and further recommendations as a result of completion of the SDB Pilot test. Phase II of the SDB Pilot test is currently under evaluation and may be scheduled for the 2011 Atlantic Richfield Work Season (ARWS). Conceptually Phase II of the SDB Pilot test will include implementation of sludge drying beds on a larger scale as described in Atlantic Richfield's 2010 RAWP dated May 11, 2010 prepared for the site.

The SDB Pilot was operated from October 26, 2009 through July 20, 2010. The pilot was decommissioned and dewatered sludge was removed from the site on July 28, 2010 for disposal at US Ecology in Beatty, Nevada.



### **Rationale for Sludge Drying Bed Pilot Test**

The ASB removes metals from influent water by raising the pH and allowing aluminum hydroxides to precipitate and generating metal sulfides via sulfate reduction. Metal-sulfide and hydroxide solids along with spent biomass accumulate as sludge within the treatment system. This sludge requires periodic removal to maintain efficient operation of the ASB. To facilitate offsite disposal, the low solids content sludge requires dewatering. Although mechanical dewatering technologies employed in the past have worked effectively, they require seasonal mobilization and operation of the dewatering equipment and have also resulted in relatively high sludge management costs. Passive sludge dewatering has also been attempted using filter bags. The drying bed technology is being evaluated to determine if it requires less labor while providing greater reliability and fewer safety considerations in comparison to other dewatering alternatives.

This pilot test satisfied in part, the requirements under Paragraph 50(j) of the Administrative Settlement Agreement and Order on Consent for Removal Action, CERCLA Docket No. 2008-29. In addition to the goals of optimizing current treatment operations, information gained during this pilot test may be used in the Remedial Investigation/Feasibility Study (RIFS) to support an evaluation of the long-term operation of the ASB.

### **Sludge Drying Bed Pilot Design**

The SDB Pilot test was designed to answer the following questions:

- Will the sludge dewater over the duration of this pilot test?
- What is the impact on the sludge moisture content when the sludge bed is exposed to rain/snow and freezing temperatures?
- What is the chemical and physical composition and stability of the sludge?
- What effect does an underlying drainage layer have upon the rate of dewatering?

The SDB Pilot was designed to test two different drying bed configurations. One configuration included dewatering by filtration, decanting and evaporation. The second configuration did not include drainage and decanting and tested dewatering by evaporation only. The design of the first configuration is shown in Figure 1 – SDB Pilot Design, Trial 1. The design of the second configuration is shown in Figure 2 – SDB Pilot Design, Trial 2. Both trials included the following features:

- The SDB Pilot test was constructed in the area to the south of the ASB where the previous centrifuge and belt press dewatering operations have been located. This area is shown on Figure 3 – Phase I SDB Pilot Site Layout.
- The tests were implemented in two roll-off style filter bins. These filter bins have a metal grate support basket that allows collection of liquids that pass through the bed matrix to collect in the bottom of the bin and gravity drain through a 4-inch outlet located on the side of the bin, near the bottom.
- The existing sludge conveyance and liquid return lines were used to load and drain the bins.
- The bins were each supported with steel I-beams in two corners to create an approximate 1.5% drainage gradient to direct decanted and filtered water to the filter bin outlet piping located on the base of the bin.
- Soil from the site was piled around the bins to better simulate in-ground conditions of a full-scale drying bed and to mitigate wall effects. This soil covering also provided a walkway between the bins for personnel access.

#### Trial 1 - Filtration and Decanting:

- The filtration system was constructed of a coarse gravel layer approximately 9-inches deep overlain by a filter sand layer approximately 9-inches thick. A non-woven geotextile was installed between the two drainage material layers to prevent the sand from migrating into the gravel layer. This system allowed free draining water to drain through the filtration system.
- A decanting system was created by installing the geotextile along the sides of the bin and to the full height to allow separated/clarified water or meteoric precipitation to decant through the geotextile. A 60-mil high density polyethylene (HDPE) liner was installed over the geotextile layer along the sidewalls and was keyed into the sand layer to prevent loss of sludge during loading through direct drainage. V-shaped notches were cut into the HDPE liner after sludge loading to allow decanting of separated water.
- One of the existing HDPE sludge pipelines, installed for previous sludge dewatering activities, was used as a drainage collection pipe and was connected to the filter bin outlet to allow gravity drainage of filtered and decanted water back to Pond 3 of the ASB.
- A sump (a 25-gallon poly tank) was installed in this drainage collection pipe approximately 1.5 feet from the filter bin outlet with a sample collection port to allow sampling of the filtered and decanted water.

#### Trial 2 - Without Filtration or Decanting:

- Trial 2 did not include drainage or decanting.
- Fill material was used to create a support layer of the same height as the filtration system in Trial 1.
- An HDPE liner (60 mil) was used to line the bin and prevent sludge or water from draining from the applied sludge layer.
- Trial 2 did not discharge water.

### **Sludge Loading**

Construction of the SDB Pilot was completed and sludge loading was conducted on October 26, 2009. A photo log from the construction is included as Appendix 1.

The SDB Pilot sludge loading depth was designed according to optimum sludge loading capacity ranges as specified in the Water Environment Federation Manual of Practice 8 (WEF and ASCE, 1998). This recommended loading is specified for municipal biological sludge; however, it served as a design basis for the SDB Pilot test. Based on previous site data, the sludge solids content was assumed at 4% indicating that a 13-inch sludge loading depth was optimum.

The ASB Pond 4 water level was lowered to the sludge water interface prior to sludge removal. The remaining sludge in Pond 4 was then mixed in the pond by circulating the sludge with a diesel powered sludge pump located near the Pond 4 berm for approximately four hours. This circulation loop was connected to the HDPE sludge line. A valve was installed in the HDPE sludge line to control the flow rate of sludge loaded to the trial bins. Excess sludge beyond the desired flow rate was allowed to return to Pond 4 during the loading process.

### **Monitoring Program**

Monitoring and sampling was conducted immediately following setup. Thereafter, sampling was conducted every two weeks when the site was accessible or monthly during regular limited access season (LAS) visits. Full monitoring and sample collection were not conducted when snow and ice impeded access to the sludge surface, or if no liquid filtrate was available in the filtrate collection sump. However, portions of the prescribed monitoring and sampling were performed whenever possible.

During each monitoring event digital photos were taken and the average sludge depth and the overlying water layer were measured. Sludge samples were collected and analyzed by Sierra Environmental Monitoring located in Reno, Nevada for the following:

- Percent solids and Percent moisture (by weight);
- Density of wet solids;
- Density of dried solids (sample was dried at 105 degrees Celsius and crushed);
- Paste pH;
- Volatile organics or total organic carbon (TOC). TOC replaced the volatile organics measurement because the sulfur compounds are also lost during the combustion step and therefore TOC was determined to be a more effective measurement of the organic or biological component of the sample; and
- Average depth of sludge, depth of standing water, and other field observations were recorded.

Sample collection of the Trial 1 filtrate from the collection sump was analyzed for the following:

- Field measurement of pH, oxidation reduction potential (ORP), dissolved oxygen (DO), temperature and specific conductance;
- Lab measurement of total metals, dissolved metals, acidity, alkalinity, sulfate, total suspended solids (TSS), and total dissolved solids (TDS) (analysis performed at TestAmerica Lab in Irvine, California); and
- In one instance the field pH of the clarified water in Trial 2 was measured.

Additional sludge samples were collected for further sludge characterization. Initial samples were collected for the following analyses:

- Microprobe analysis to determine surface chemistry and mineralogy, performed at Hazen Research Laboratories in Golden, Colorado;
- Elemental analysis performed at Huffman Laboratories in Golden, Colorado; and
- Globe® particle density determination, performed at MSE Technology Applications, Inc. in Butte, Montana.

Dewatered sludge was collected near the end of the SDB pilot operation for the following analyses:

- Acid-base accounting, performed at ACZ Laboratories in Steamboat, Colorado;
- Meteoric Water Mobility Leaching Procedure (barrel roll method), performed at ACZ Laboratories in Steamboat, Colorado;
- Waste characterization extraction and analysis appropriate for RCRA and California Standards, including Toxicity Characteristic Leaching Procedure (TCLP), Total Threshold Limit Concentration (TTLC), Soluble Threshold Limit Concentration (STLC), and Synthetic Precipitate Leaching Procedure (SPLP) were performed at

TestAmerica Labs in Irvine, California. These samples were collected on April 26, 2010.

- Additional samples for the Modified DI-WET extraction procedure were collected on July 20, 2010 and sent to TestAmerica Labs, in Irvine, California.

## Results

Selected digital images are proved as a photo log in Appendix 2. Results from periodic sampling of the sludge in the trials are presented in Table 1. Trial 1 filtrate field monitoring and laboratory analytical data is presented in Table 2.

### *Trial 1 (filtration and decanting), periodic sampling:*

Trial 1 showed significant dewatering and sludge volume reduction within 24 days of test initiation. Percent solids increased from 5.4 to 20.8% in this time frame with a subsequent volume reduction of 63% based on sludge depth. Vertical cracks were observed in the sludge layer and dry fine sand like material was observed at the top of the sludge bed. The SDB bins were covered with ice and snow for the majority of the winter.

Sludge sampling began again in April 2010, and a general decline in solids content and volume was observed. Very little sludge volume remained at the end of the pilot test. The final sludge volume based on depth measurements on June 22, 2010 was approximately 11% of the starting volume.

Wet based sludge density increased over time. Dried density had a general decline with some variability. The variability in this measurement may be due to inherent variability associated with crushing the sample to determine the dried density. Total organic carbon made up approximately 3 to 10% of the sample, again variability in the measurement was observed. Paste pH demonstrated a slight but steady decline from an initial value of 7.9 on October 26, 2009 to a slightly acidic pH of 6.6 on June 22, 2010.

Filtrate collected from Trial 1 was initially slightly basic for all sampling points in the fall of 2009. Liquid filtrate was not available during the December and January site visits. Sampling on February 2, 2010 demonstrated acidic filtrate (pH 3.9) and constituent concentrations above the discharge criteria for several metals. This trend continued through completion of the pilot.

### *Trial 2 (without filtration or decanting), periodic sampling:*

During the first 24 days of operation Trial 2 had only a minor reduction in volume. Separation of clarified water was observed at 17 days of operation. The clarified water layer remained frozen during all site visits from December through March. Water was observed during the April 26<sup>th</sup>,

2010 site visit. At this time the water layer was measured at a greater thickness than the visible sludge layer. Evaporation of the water occurred slowly over the next month and a half. On June 8, 2010 no water was observed above the sludge. Qualitative field observations indicated that rain events added water to the bin.

Little change in solids content was observed during the fall sampling, but once the water layer evaporated a significant increase in sludge solids was measured coupled with a pronounced reduction in sludge volume. As shown in Table 1 the clarified water layer above the sludge evaporated between May 25 and June 8, 2010. The sludge began to dry significantly during this time period, based on visual observations, but drying did not appear to be consistent throughout the bed area as portions of the bed appeared wetter than other areas. Ponding of water likely occurred in various portions of the bed during evaporation. Deep cracks formed through the entire thickness of the sludge.

Final sludge volume on June 22, 2010 was approximately 29% of the initial sludge volume. Duplicate samples of sludge from Trial 2 were collected on July 20, 2010 to corroborate high percent solids results (51%) from the June 22, 2010 sampling event. The July 20, 2010 percent solids sampling results were anomalously high (97-98% solids).

Only minor changes in wet sludge density were observed throughout the pilot until the final sludge analysis on July 20, 2010. Total organic carbon made up approximately 3 to 10% of the sample, again variability of the measurement was observed. Paste pH was variable between pH 7.9 and 6.8.

#### *Initial Sludge Characterization*

The surface mineralogy results from Hazen Laboratories are included as Appendix 3. Microprobe analysis of sludge loaded to the SDB trials at the initiation of the SDB pilot determined the presence of pyrite in the sludge. The occurrence of pyrite as very small cubic crystals indicates that pyrite likely formed within the bioreactor system. If the pyrite had been carried into the system with the seep water, it is anticipated that weathered (irregular shaped) pyrite particles would have been observed instead of well formed crystals. The redox conditions, microbial community, and water chemistry support the formation of biogenic pyrite in the bioreactor system.

Iron, aluminum, silica, calcium and sulfur were reported with the greatest frequency. Calcite particles were dispersed through the sample sections. A scan was conducted to determine the presence of other metals; this analysis was not exhaustive but copper, nickel and zinc were found associated with some pyrite crystals in very low percentages.

Results of elemental analysis of the sludge collected at the beginning of the pilot are presented in Table 3. Similar to the surface iron, sulfur, aluminum, and calcium were reported at higher concentrations than the other metals.

Globe particle density analysis is a method that measures the density of a material excluding porosity associated with the bulk material. Samples were collected on November 19, 2009 and results indicate a particle density of 3.42 g/mL for Trial 1 and 3.21 g/mL for Trial 2.

#### *Final Sludge Characterization*

Acid-base accounting indicated that the sludge material is acid generating. Results are presented in Table 4. However, waste characterization sampling analytical results, presented in Table 5, indicate that dewatered sludge from both trials meets the numeric limits for classification as a nonhazardous sludge by both Resource Conservation and Recovery Act (RCRA) and California regulations.

As suggested by Region 9 EPA in the Approval and Direction to Implement Leviathan Mine Site Work Plan Amendment, 2010 Removal Action Work Plan (April 12, 2010) and confirmed in the Response to April 12, 2010 Comments to the 2010 Removal Action Work Plan (May 11, 2010), the modified California waste extraction test was also conducted on the sludge using deionized water as the extraction fluid as part of the waste characterization sampling analysis. Results of this extraction and analysis are also presented in Table 5.

Meteoric Water Mobility Leaching Procedure testing was performed by the barrel-roll method. The barrel-roll method was selected over the column percolation method because laboratory experience indicated that the size and consistency of the solids delivered were expected to induce flow complications in the column setup. The barrel-roll method was identified as a more appropriate method for the sludge solids. This test has been used by the Nevada Department of Environmental Protection to evaluate the potential for dissolution and mobility of certain constituents from a mine rock sample by meteoric water. Results are presented in Table 6. Since there are no applicable numeric threshold limits for the site for this particular test, the results are presented in comparison with the site discharge criteria.

#### **Discussion**

Trial 1 dewatered rapidly under conditions that were not favorable for evaporation alone. No significant dewatering occurred in the Trial 2 bin during the fall and winter seasons. Significant drying of the Trial 2 sludge only occurred under optimal, warm and dry weather conditions. Qualitative field observations indicated longer retention of meteoric water in the Trial 2 bin than

in the Trial 1 bin after rain events. However, due to the limited number of sampling events during the LAS, limited conclusions have been made regarding the impact of individual snow/rain events and freezing conditions on the sludge moisture content.

The very high percent solids results obtained toward the end of the pilot in Trial 2 were unexpected. The sludge in Trial 2 developed very hard rock-like clumps, while the Trial 1 sludge was described as presenting an uppermost very fine silt-like very dry layer. Under this layer the sludge was more consolidated and formed hard clumps of material (see Figures 15 through 18 in Appendix B). The percent solids analysis is done with the sample as received. No crushing or other manipulation is done to the rock like sample portions before drying at 105 degrees Celsius to determine weight (moisture) loss on drying. It is possible that, due to the nature of rock-like sludge aggregation, only the surface sludge is exposed to drying and the inner material may hold moisture content. Another alternative is that chemical reactions within the sludge may have driven-off water associated with the sludge while drying in the sun. It is unclear at present why this would have been more significant in Trial 2 than Trial 1. Whether or not the percent solids values measured are precise or not, the sludge appears to have undergone significant drying during the dry and warm weather period.

The acidic filtrate observed from Trial 1 is consistent with the detection of pyrite in the sludge composition. Oxygenated meteoric water began to oxidize pyrite and leach metals once the sludge solids dewatered. Organic carbon was still measured in the sludge toward the end of the pilot, however, it appears that insufficient organic material was present to mitigate iron and sulfur oxidation reactions with iron and sulfate reduction reactions.

The acid-base accounting results indicate that the dewatered sludge is acid producing which is consistent with the identification of pyrite in the ASB sludge. Although the sludge sample collected was acid generating, waste characterization sampling procedures indicate that the sludge passes criteria to be considered a non-hazardous waste. These test procedures simulate possible conditions in municipal landfills, where acidic but generally reducing conditions exist. The sludge is anticipated to be appropriate for disposal in reducing conditions, where oxidation is limited. However, under oxidizing conditions, either in the presence of oxygen or soluble ferric iron, this sludge is anticipated to produce an acidic metal containing leachate.

### **Conclusions and Recommendations**

The results from Phase I of the SDB Pilot test indicate that dewatering of the ASB treatment generated solids with an onsite sludge drying bed are effective and have the potential to decrease the annual operating costs required for sludge dewatering and removal over cost

expenditures required for mobilization of centrifuge or belt press dewatering. This pilot test demonstrated that a bed with filtration and decanting dewatered the ASB sludge to similar solids content as previous belt press and centrifuge dewatering technologies within 24 days during conditions not optimal for evaporative dewatering. The design with filtration and decanting allowed more rapid removal of collected water from meteoric precipitation events compared to the bin without filtration (Trial 2). Collection of meteoric water may impact the dewatering schedule if a precipitation event occurs just prior to planned sludge removal. Although very high percent solids were measured in Trial 2, without filtration and decanting, at the end of the Pilot, the average final sludge volume was greater in Trial 2 than in the Trial 1 bin, with filtration and decanting.

Based on data collected thus far, limited conclusions can be made regarding the impact of individual snow/rain events and freezing conditions on the sludge moisture content. However, during the LAS when these conditions are most common, it is anticipated that dewatering operations would be discontinued or dewatering filtrate would be directed back to the treatment system.

Since only one sludge loading depth was evaluated in the Phase I SDB Pilot, flexibility for testing greater depths or layered application is recommended in the design of the Phase II SDB Pilot. This will allow optimization of usage and operations costs. If sludge dewatering is managed with relatively short dewatering intervals it is anticipated that production of acidic filtrate can be avoided. However, maintenance of sludge in the drying bed for longer periods can be done successfully with collection and treatment of the filtrate in the bioreactor system.

Further consideration is required to determine the appropriate location for installation of a large scale SDB during the Phase II SDB Pilot. Relatively level surface locations are limited in the vicinity of the ASB. The location south of the ASB where mobile sludge dewatering equipment has been staged in the past facilitates loading of the bed, access to the bed for removal and disposal operations, and collection and conveyance of filtrate back to the ASB by gravity flow. However, this area of the site is within the upper portion of the Leviathan Creek Basin Landslide, and there may be geotechnical risks associated with utilizing this location for a SDB. Since some other locations may require greater efforts for bed loading and filtrate collection, design requirements, and construction and operations costs must be reevaluated for other proposed locations.

Sincerely,



Tony Brown  
Project Manager Mining

Attachment:

Table 1 – Sludge Sampling Results  
Table 2 – Filtrate Sample Results  
Table 3 – Elemental Analysis Results  
Table 4 – Dewatered Sludge Acid Base Accounting Results  
Table 5 – Waste Characterization Analytical Results  
Table 6 – Meteoric Water Mobility Leaching Procedure Results

Figure 1 – SDB Pilot Design, Trial 1  
Figure 2 – SDB Pilot Design, Trial 2  
Figure 3 – Phase I SDB Pilot Site Layout

Appendix A – Phase I Sludge Drying Bed Pilot Construction Photo Log  
Appendix B – Phase I Sludge Drying Bed Pilot Monitoring Photo Log  
Appendix C – Surface Chemistry Analysis Results Report, Hazen Laboratories

cc: Kevin Meyer, Region 9 EPA – via electronic  
Chuck Curtis, Lahontan Regional Water Quality Control Board – via electronic  
Nathan Block, Esq., BP America Inc. – via electronic  
Adam Cohen, Esq., Davis Graham & Stubbs LLP – via electronic  
Joe Niland, AMEC Geomatrix – via electronic  
Sandy Riese, EnSci – via electronic  
Dave McCarthy, Copper Environmental – via electronic  
Bruce Wielinga, AMEC Geomatrix – via electronic  
Bill Malyk, AMEC Geomatrix – via electronic  
Marc Lombardi, AMEC Geomatrix – via electronic

## **TABLES**

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**TABLE 1**  
**Sludge Sampling Results**  
**ASPEN SEEP BIOREACTOR SLUDGE DRYING BED PHASE 1**  
 Provisional Data

Parameter	Date	Average Sludge Depth (in)	% Solids (weight basis)	% Moisture Content (weight basis)	Density (g/cm <sup>3</sup> wet basis)	Density (g/cm <sup>3</sup> dried basis)	% Volatile Solids (dry weight basis)	TOC (mg/kg)	TOC (as %)	Paste pH	Depth of Water Above Sludge (inches)	pH of Water Above Sludge
Bulk Sludge <sup>a</sup>	10/26/2009	---	5.5	94.5	1.04	1.18	17.1	62000	6.2	7.9	---	---
Trial 1	10/26/09 <sup>b</sup>	13.4	5.4	94.6	1.04	1.26	17.8	69000	6.9	7.9	0	---
	11/2/2009	6.6	9.3	90.7	1.06	1.34	21.5	---	---	---	0	---
	11/11/2009	5	17.9	82.1	1.08	1.17	16	---	---	---	0	---
	11/17 & 11/19/09 <sup>c</sup>	5	20.8	79.2	1.21	1.23	---	---	---	7.9	0	---
	4/26/2010	2.7	29	71	1.21	1.17	---	100000	10	6.8	0	---
	5/13/2010	1.2	47	53	1.19	1.05	---	44000	4.4	7.2	0	---
	5/25/2010	1.4	32	68	1.26	0.99	---	49000	4.9	6.9	0	---
	6/8/2010	1.3	35	65	1.2	1.13	---	33000	3.3	6.7	0	---
6/22/2010	1.5	32	68	1.17	1.32	---	80000	8	6.6	0	---	
Trial 2	10/26/09 <sup>b</sup>	13.5	5.6	94.4	1.04	1.27	17.9	74000	7.4	7.9	0	---
	11/2/2009	12.3	5.8	94.2	1.05	1.28	16.4	---	---	---	0	---
	11/11/2009	10.8	5.8	94.2	1.06	1.32	11.8	---	---	---	0.5	---
	11/17 & 11/19/09 <sup>c</sup>	10.9	7.2	92.8	1.07	1.28	---	---	---	8.0	1	---
	4/26/2010	4.6	13	87	1.13	1.35	---	93000	9.3	7.8	6.5	7.8
	5/13/2010	5.3	14	86	1.08	1.28	---	58000	5.8	8.0	4.6	---
	5/25/2010	5.3	13	87	1.09	1.24	---	44000	4.4	6.8	2.3	---
	6/8/2010	4.7	26	74	1.16	1.32	---	56000	5.6	7.8	0	---
	6/22/2010	3.9	51	49	1.09	1.32	---	89000	8.9	7.7	0	---
	7/20/2010	---	97.3	2.7	1.39	1.32	---	100000	10	7.1	0	---
7/20/2010-duplicate	---	97.9	2.1	1.23	1.3	---	95000	9.5	7.2	0	---	

Notes

TOC = Total Organic Carbon analysis      NA = Not available      --- = Parameter was not measured

a = Bulk sludge sampled during installation

b = Laboratory data differs in the 11-9-09 memo due to a misinterpretation of the sample labels. Data presented here is corrected for each Trial.

c = Photos and depths collected on November 17, sludge samples for laboratory analysis collected on November 19, 2009

**TABLE 2**  
**TRIAL 1 FILTRATE SAMPLE RESULTS**  
**ASPEN SEEP BIOREACTOR SLUDGE DRYING BED PHASE 1**  
 Provisional Data

Parameter	Basis	October 28 2009 089ASPSUMP320 Sump mg/L	November 2 2009 091ASPSUMP323 Sump mg/L	November 11 2009 092ASPSUMP324 Sump mg/L	November 19 2009 097ASPSUMP338 Sump mg/L	February 2 2010 103ASPSUMP348 Sump mg/L	March 9 2010 105ASPSUMP353 Sump mg/L	April 26 2010 107ASPSUMP356 Sump mg/L	May 13 2010 111ASPSUMP363 Sump mg/L	May 25 2010 113ASPSUMP366 Sump mg/L	June 8 2010 117ASPSUMP376 Sump mg/L	June 22 2010 120ASPSUMP381 Sump mg/L	ASB Discharge Criteria	
													Maximum Discharge Criteria mg/L	Average Discharge Criteria mg/L
pH <sup>1</sup>	Field	8.2	8.4	8.6	8.5	<b>3.9</b>	<b>3.7</b>	<b>2.8</b>	<b>3.7</b>	<b>3.0</b>	<b>2.8</b>	<b>2.6</b>	---	6.0 - 9.0 <sup>2</sup>
Al	Dissolved	<0.08	<0.04	<0.04	<0.04	<b>22</b>	<b>27</b>	<b>9.8</b>	<b>17</b>	<b>19</b>	<b>18</b>	<b>19</b>	4	2
As	Dissolved	0.0017	0.0012	0.0012	<0.0045	<0.0045	<0.018	<0.0009	<0.009	0.0033	0.0045	0.010	0.34	0.15
Cd	Dissolved	<0.0001	<0.0001	<0.0001	<0.0005	<b>0.0063</b>	<b>0.0091J</b>	<0.0001	0.0029J	0.0029	0.003	0.0031	0.009	0.004
Ca	Dissolved	154	107	102	109	370	320	217	321	353	327	370	--	--
Cr	Dissolved	0.0017J	0.00096J	0.0014J	<0.0045	0.025	0.018	<0.0009	<0.009	0.012	0.011	0.016	0.97	0.31
Cu	Dissolved	0.0040	0.0036	0.005	0.0033J	<b>0.14</b>	<b>0.23</b>	0.0021	<b>0.17</b>	<b>0.14</b>	<b>0.12</b>	<b>0.11</b>	0.026	0.016
Hardness	Dissolved	750	700	810	800	2600	2600	1200	1700	1800	1700	1900	--	--
Fe	Dissolved	<0.03	<0.015	<0.015	0.0259J	<b>12.9</b>	<b>13.0</b>	<b>8.56</b>	<b>5.8</b>	<b>15.5</b>	<b>19.3</b>	<b>27</b>	2	1
Pb	Dissolved	<0.0002	<0.0002	<0.0002	<0.001	<b>0.0064</b>	<b>0.0067J</b>	0.00022J	0.0043J	0.0042	0.0042	<b>0.0084</b>	0.136	0.005
Mg	Dissolved	88	110	130	130	420	430	150	210	230	210	240	--	--
Ni	Dissolved	0.013	0.01	0.01	0.011	<b>0.71</b>	<b>0.87</b>	0.0036	<b>0.28</b>	<b>0.31</b>	<b>0.31</b>	<b>0.32</b>	0.84	0.094
Se	Dissolved	0.0021	0.0015J	0.0018J	0.0035J	0.0042J	<0.010	--	--	--	--	--	--	--
Zn	Dissolved	<0.005	<0.005	<0.005	<0.025	<b>0.69</b>	<b>0.97</b>	0.21	<b>0.32</b>	<b>0.62</b>	<b>0.63</b>	<b>0.70</b>	0.21	0.21
Al	Total	<0.08	<0.04	<0.04	--	--	27	9.5	20	20	19	20	--	--
As	Total	0.0029	<0.0009	<0.0009	--	--	<0.018	0.0029	<0.009	0.0034	0.0051	0.0095	--	--
Cd	Total	0.00011J	<0.0001	<0.0001	--	--	0.0088J	0.0019	0.0031J	0.0027	0.003	0.0028	--	--
Ca	Total	154	100	98.3	--	--	320	207	348	364	359	352	--	--
Cr	Total	0.0013J	<0.0009	0.0017J	--	--	<0.018	0.0064	0.010J	0.011	0.011	0.014	--	--
Cu	Total	0.0041	0.0038	0.007	--	--	0.24	0.16	0.19	0.16	0.13	0.11	--	--
Fe	Total	0.119	0.0359J	0.0539	--	--	13.7	7.36	6.68	16.3	21	25.5	--	--
Pb	Total	<0.0002	<0.0002	0.00023J	--	--	0.0073J	0.0022	0.0065J	0.0045	0.0042	0.0068	--	--
Mg	Total	87	97	130	--	--	430	150	240	230	230	240	--	--
Ni	Total	0.013	0.0099	0.01	--	--	0.88	0.14	0.30	0.32	0.29	0.29	--	--
Se	Total	0.0016J	0.0012J	0.002	0.0031J	<b>0.0062</b>	<0.010	0.0021	<b>0.0082J</b>	0.0031	0.0032	0.0024	NP	0.005
Zn	Total	0.010J	0.017J	0.0063J	--	--	0.99	0.22	0.33	0.64	0.67	0.63	--	--
% Solids	Total	0.10%	0.10%	0.10%	0.20%	--	0.50%	0.20%	0.33%	0.40%	0.35%	0.36%	--	--
Cl	Total	--	--	--	14	7	6.6	0.88J	7	1.6	1.3	0.91J	3	--
TSS	Total	--	--	--	4.0	18	17.0	10	10	8J	9J	12	--	--
TDS	Total	--	--	--	1930	5020	4920	1940	2880	3210	3220	3480	--	--
Acidity	Total	--	--	--	<2	1400	1400	220	700	800	820	590	--	--
Alkalinity (Total)	Total	--	--	--	284	<2.0	<2.0	2.0	2.0	<2.0	<2.0	<2.0	--	--
Alkalinity (Bicarbonate)	Total	--	--	--	346	<2.4	<2.4	2.4	2.4	<2.40	<2.40	<2.40	--	--
Alkalinity (Carbonate)	Total	--	--	--	<1.2	<1.2	<1.2	1.2	1.2	<1.20	<1.20	<1.20	--	--
Alkalinity (Hydroxide)	Total	--	--	--	<0.7	<0.7	<0.7	0.70	0.70	<0.70	<0.70	<0.70	--	--
Sulfate	Lab Filtered	--	--	--	964	2890	2870	1310	1960	2140	2070	2460	--	--
ORP (mV)	Field	116	73	6.3	6.3	344	447	438	157	298	394	289	--	--
DO (mg/L)	Field	7.1	--	5.3	5.3	--	3.6	2.4	11.9	12.2	9.4	10.16	--	--
Temp (C)	Field	4.8	18.4	7.5	7.5	5.3	4.5	14.4	12.2	12.4	16.4	15.0	--	--
SpC (uS/cm)	Field	1284	1018	1522	1522	4909	2823	1738	1977	1870	2925	2051	--	--
Appearance of Solids	Field	none observed	none observed	none observed	none observed	none observed	none observed	none observed	none observed	none observed	none observed	none observed	--	--

**Notes**

pH value was collected in field; pH is in standard units.  
 < = Analyte NOT DETECTED at or above the method detection limit.  
 J = Estimated value. Analyte detected at a level less than the Reporting Limit and greater than or equal to the Method Detection Limit. The user of this data should be aware that this data is of limited reliability.

Values in bold are effluent concentrations greater than the maximum or average discharge criteria.  
 Filtrate water was not generated during the initial Trial 1 set up; no water was available for sampling on 10/26/09.

**Abbreviations**

ORP (mV) = oxidation reduction potential in millivolts  
 DO (mg/L) = dissolved oxygen in milligrams per liter  
 NP = Not Promulgated

Temp (C) = temperature in Celsius  
 SpC (uS/cm) = specific conductance in microSiemens per centimeter  
 NA = Not Available

--- Parameter was not measured

**TABLE 3**  
**RESULTS of ELEMENTAL ANALYSIS OF SLUDGE**  
**PHASE I ASPEN SEEP BIOREACTOR SLUDGE DRYING BED PILOT**  
 Provisional Data

<b>Parameter</b>	<b>Basis</b>	<b>October 26 2009 Trial 1 %</b>	<b>October 26 2009 Trial 2 %</b>
Loss on Drying	Wet Weight	94.56	94.46
Carbon	Dry Weight	5.23	5.06
Hydrogen	Dry Weight	1.56	1.58
Nitrogen	Dry Weight	0.42	0.38
Oxygen	Dry Weight	23.9	23.8
Aluminum	Dry Weight	7.58	7.78
Calcium	Dry Weight	5.42	5.71
Chromium	Dry Weight	<0.002	<0.002
Copper	Dry Weight	0.18	0.18
Iron	Dry Weight	19.14	18.92
Magnesium	Dry Weight	1.42	1.46
Manganese	Dry Weight	1.13	1.17
Nickel	Dry Weight	0.07	0.07
Potassium	Dry Weight	0.1	0.1
Selenium	Dry Weight	<0.01	<0.01
Sodium	Dry Weight	0.51	0.53
Sulfur	Dry Weight	13.94	13.6
Zinc	Dry Weight	0.12	0.12

**TABLE 4**  
**DEWATERED SLUDGE ACID BASE ACCOUNTING RESULTS**  
**PHASE I ASPEN SEEP BIOREACTOR SLUDGE DRYING BED PILOT**  
 Provisional Data

Parameter	Method	Units	June 22	June 22
			2010	2010
			122TRIAL1BIN384	122TRIAL2BIN385
			Trial 1	Trial 2
Acid Generation Potential <sup>1</sup>	M600/2-78-054 1.3	t CaCO <sub>3</sub> /Kt	400	422
Acid Neutralization Potential <sup>2</sup>	M600/2-78-054 1.3	t CaCO <sub>3</sub> /Kt	182	117
Acid-Base Potential <sup>3</sup>	M600/2-78-054 1.3	t CaCO <sub>3</sub> /Kt	-218	-305
Neutralization Potential as CaCO <sub>3</sub>	M600/2-78-054 3.2.3	%	18.2	11.7
Sulfur HCl Residue	M600/2-78-054 3.2.4-MOD	%	7.84	8.23
Sulfur HNO <sub>3</sub> Residue	M600/2-78-054 3.2.4-MOD	%	1.33	2.33
Sulfur Organic Residual Mod	M600/2-78-054 3.2.4-MOD	%	1.33	2.33
Sulfur Pyritic Sulfide	M600/2-78-054 3.2.4-MOD	%	6.51	5.9
Sulfur Sulfate	M600/2-78-054 3.2.4-MOD	%	4.92	5.27
Sulfur Total	M600/2-78-054 3.2.4-MOD	%	12.8	13.5
Total Sulfur minus Sulfate	M600/2-78-054 3.2.4-MOD	%	7.84	8.23

Notes

<sup>1</sup> Calculation on sulfur total

<sup>2</sup> Calculation

<sup>3</sup> Calculation on total sulfur

**TABLE 5**  
**WASTE CHARACTERIZATION ANALYTICAL RESULTS**  
**PHASE I ASPEN SEEP BIOREACTOR SLUDGE DRYING BED**  
 Provisional Data

Parameter	Analytical Results										Regulatory Threshold <sup>c</sup>		
	Total Metals (mg/kg)	STLC (mg/L)	TCLP (mg/L)	SPLP (mg/L)	DI-WET (mg/L)	Total Metals (mg/kg)	STLC (mg/L)	TCLP (mg/L)	SPLP (mg/L)	DI-WET (mg/L)	TTLIC (Regulatory Limits for Total Metals) (mg/kg)	STLC (Regulatory Limits) (mg/L)	TCLP (Regulatory Limits) (mg/L)
	Sample Date: 26 April 2010 Sample ID: 109Trial1Bin359 - Trial 1				22 June 2010 127Trial1bin403	Sample Date: 26 April 2010 Sample ID: 109Trail2Bin360 - Trial 2				22 June 2010 127Trial2bin404			
Aluminum	19,000	1,000	1.3	<0.40	<0.050	10,000	690	10	<0.40	<0.050	NA	NA	NA
Antimony	<4.4	<0.14	<0.20	<0.070	<0.0020	<0.88	<0.14	<0.20	<0.070	<0.0020	500	15	NA
Arsenic	6.3 J	<0.13	<0.20	<0.070	0.0016	3.2	<0.13	<0.20	<0.070	<0.0010	500	5.0	5.0
Barium	14.0	0.56	0.22	<0.060	0.041	6.9	0.42	<0.20	<0.060	0.058	10,000	100	100
Beryllium	3.8	0.29	<0.080	<0.010	<0.00050	2.1	0.16	<0.080	<0.010	<0.00050	75	0.75	NA
Cadmium	2.3 J	0.072 J	<0.10	<0.020	0.00200	1.2	<0.040	<0.10	<0.020	<0.0010	100	1.0	1.0
Chromium	<1.5	0.11	<0.10	<0.020	<0.0020	1.3	0.089 J	<0.10	<0.020	<0.0020	500 <sup>a</sup>	5 (560) <sup>b</sup>	5.0
Cobalt	110	2.7	0.65	<0.020	0.12	57	0.44	<0.20	<0.020	0.023	8,000	80	NA
Copper	460	1.8	<0.20	<0.030	0.18	250	<0.060	<0.20	<0.030	0.16	2,500	25	NA
Iron	46,000	1,500	50	<0.15	<0.0400	22,000	1,200	210	0.15 J	<0.0400	NA	NA	NA
Lead	<2.5	<0.080	<0.10	<0.040	<0.0010	<0.50	<0.080	<0.10	<0.040	<0.0010	1,000	5.0	5.0
Mercury	0.053	<0.001	<0.002	<0.001	0.00022	0.00023	<0.001	<0.002	<0.001	<0.00020	20	0.2	0.2
Molybdenum	<0.99	<0.040	<0.40	<0.020	<0.0020	<0.20	<0.040	<0.40	<0.020	<0.0020	3,500	350	NA
Nickel	160	4.5	1.1	<0.020	0.14	85	1.5	<0.20	<0.020	0.029	2,000	20	NA
Selenium	<5.0	<0.16	<0.10	<0.080	0.0025	<1.0	<0.16	<0.10	<0.080	0.0029	100	1.0	1.0
Silver	<4.0	0.15 J	<0.20	<0.060	<0.0010	<0.80	0.14 J	<0.20	<0.060	<0.0010	500	5.0	5.0
Thallium	<4.0	<0.14	<0.10	<0.070	<0.0010	1.8 J	<0.14	<0.10	<0.070	<0.0010	700	7.0	NA
Vanadium	2.0 J	0.11 J	<0.20	<0.030	<0.0010	0.95 J	0.070 J	<0.20	<0.030	<0.0010	2,400	24	NA
Zinc	290	15	2.2	<0.060	0.04300	150	4.4	<0.40	<0.060	0.021	5,000	250	NA
pH (standard units)	7.63				NA	8.05				NA	Corrosivity criteria for pH = ≤ 2.0 or ≥ 12.5 <sup>c</sup>		
Soil Moisture (% by weight)	NA				NA	NA				NA			

**Notes**

<sup>a</sup> Concentration limit for total chromium and/or chromium (III) is 2,500 mg/L and limit for chromium (VI) is 500 mg/L.

<sup>b</sup> The federal hazardous waste level for soluble chromium is 5mg/L. California has a Waste Extraction Test (WET) soluble level for chromium (III) (560 mg/L) and chromium (VI) (5 mg/L). To use the 560 mg/L regulatory threshold, it must be demonstrated first that the waste is not a Resource Conservation Recovery Act (RCRA) waste.

<sup>c</sup> Title 22 California Code of Regulations, Section 66261.24 (a)(2): Samples were tested for waste extraction test, solubility, and total concentrations. If the results of the STLC or TTLIC equal or exceed their respective regulatory thresholds, the waste is a hazardous waste.

**Abbreviations**

"<" = Constituent not detected at or above the reporting limit or the method detection limit listed

J = The associated value is an estimated quantity

LTS = Lime Treatment System

mg/kg = milligrams per kilograms

mg/L = milligrams per liter

NA = Not applicable

SPLP = Synthetic precipitation leaching procedure

STLC = Soluble threshold limit concentration

TCLP = Toxicity characteristics leaching procedure

TTLIC = Total threshold limit concentration UNR

DI-WET= Modified Waste Extraction Test with deionized water

**TABLE 6**  
**METEORIC WATER MOBILITY LEACHING PROCEDURE RESULTS -**  
**BARREL ROLL METHOD**  
**PHASE I ASPEN SEEP BIOREACTOR SLUDGE DRYING BED**  
 Provisional Data

Parameter	Method	June 29 2010 123TRIAL1BIN386 Bulk Sludge mg/L	June 29 2010 123TRIAL2BIN387 Bulk Sludge mg/L	ASB Discharge Criteria	
				Maximum Discharge Criteria mg/L	Average Discharge Criteria mg/L
Aluminum	M6010B ICP	0.04 J	0.15 J	4	2
Antimony	M6010B ICP-MS	<0.0004	<0.0004	---	---
Arsenic	M6010B ICP-MS	0.002 J	0.0024	0.34	0.15
Barium	M6010B ICP	0.084	0.181	---	---
Beryllium	M6010B ICP	0.002 J	0.003 J	--	--
Cadmium	M6010B ICP-MS	0.0035	0.0037	0.009	0.004
Chromium	M6010B ICP	<0.01	<0.01	0.97	0.31
Cobalt	M6010B ICP	0.11	0.04 J	--	--
Copper	M6010B ICP	0.29	1.02	0.026	0.016
Iron	M6010B ICP	<0.02	0.15	2	1
Lead	M6010B ICP-MS	0.0002 J	0.0008	0.136	0.005
Mercury	M7470 CVAA	<0.0002	<0.001	---	---
Molybdenum	M6010B ICP	0.01 J	0.01 J	--	--
Nickel	M6010B ICP	0.19	0.09	0.84	0.094
Selenium	M6010B ICP-MS	0.0071	0.0099	--	0.005
Silver	M6010B ICP-MS	<0.00005	<0.00005	--	--
Thallium	M6010B ICP-MS	0.0003 J	0.0003 J	--	--
Vanadium	M6010B ICP	0.01 J	0.012 J	--	--
Zinc	M6010B ICP	0.05 J	0.03 J	0.21	0.21

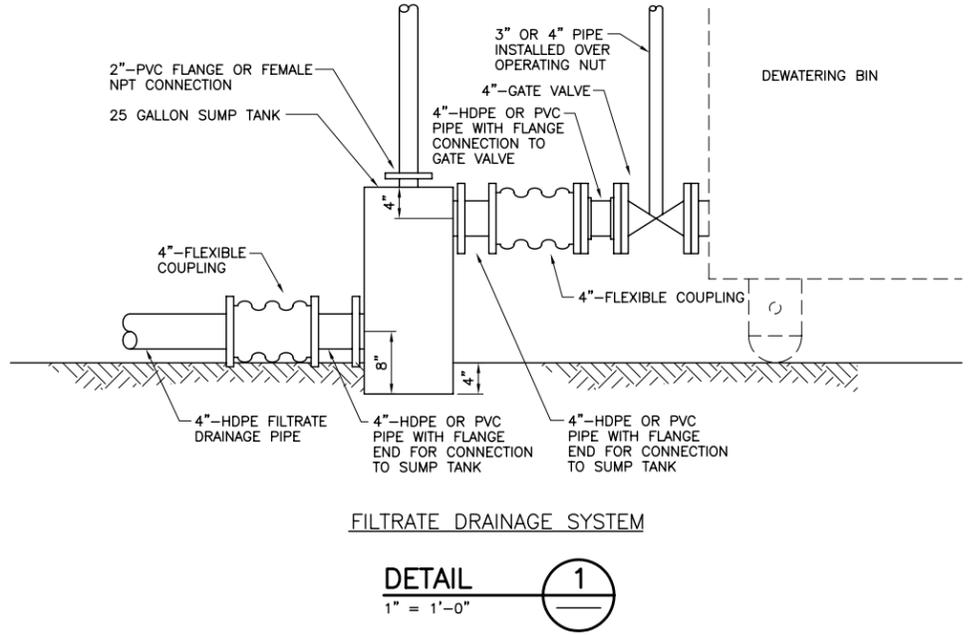
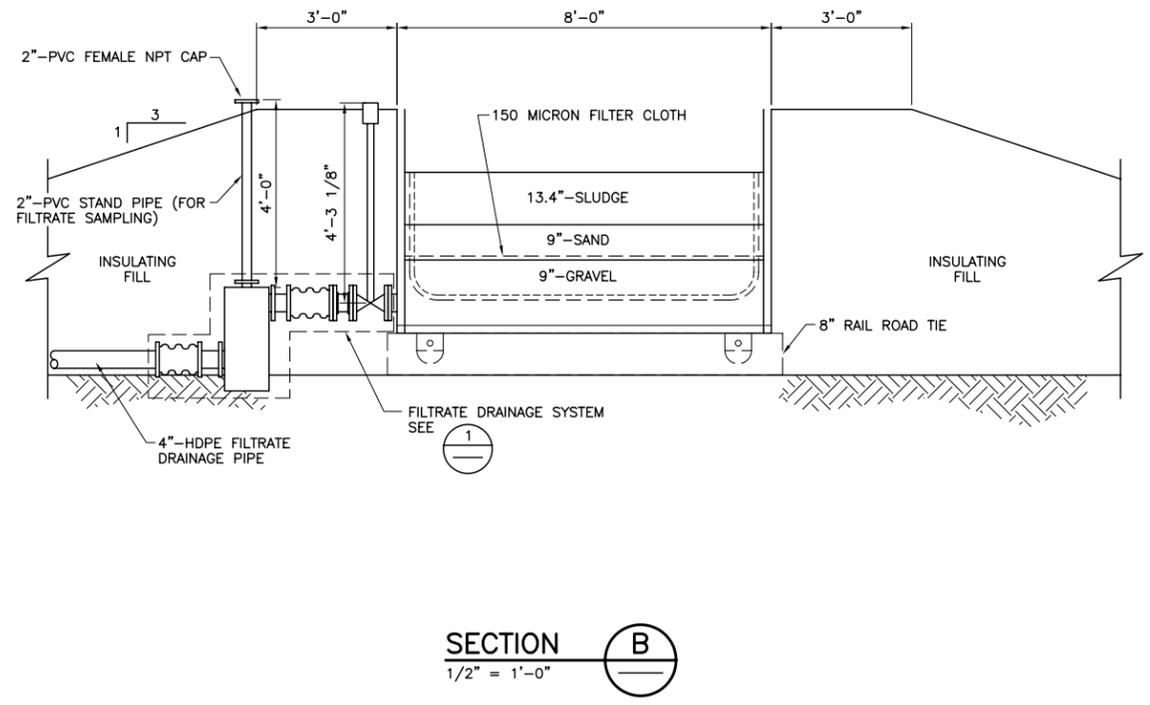
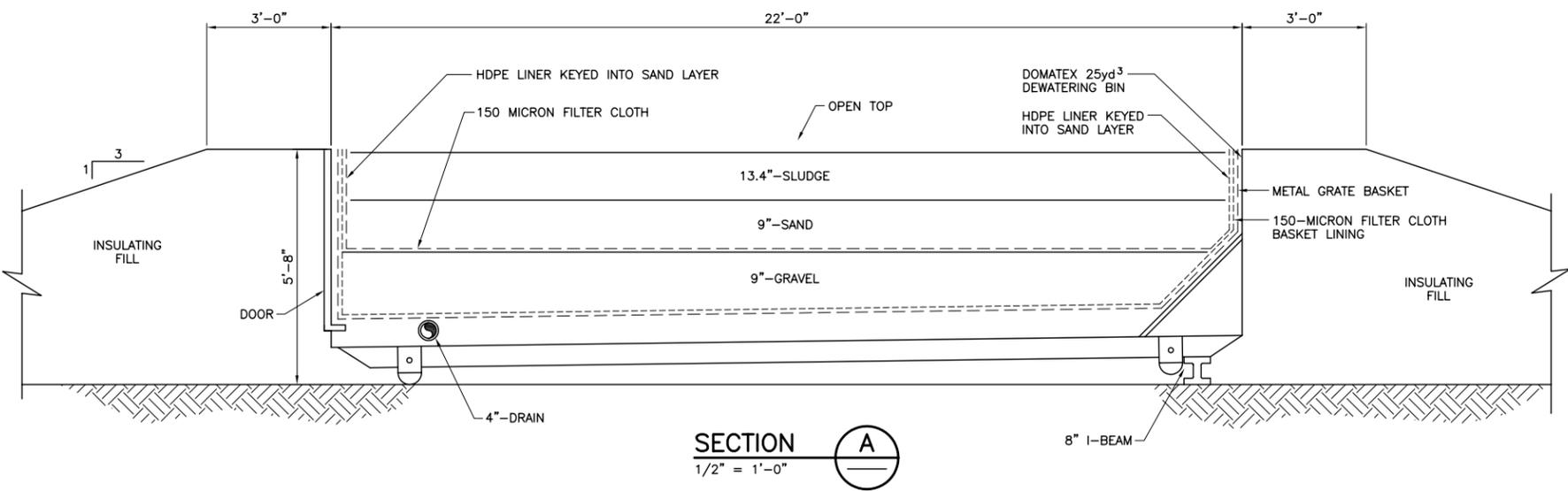
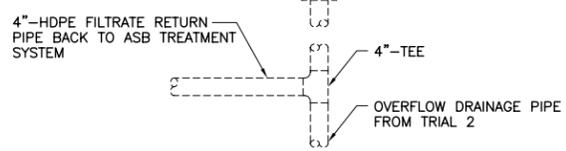
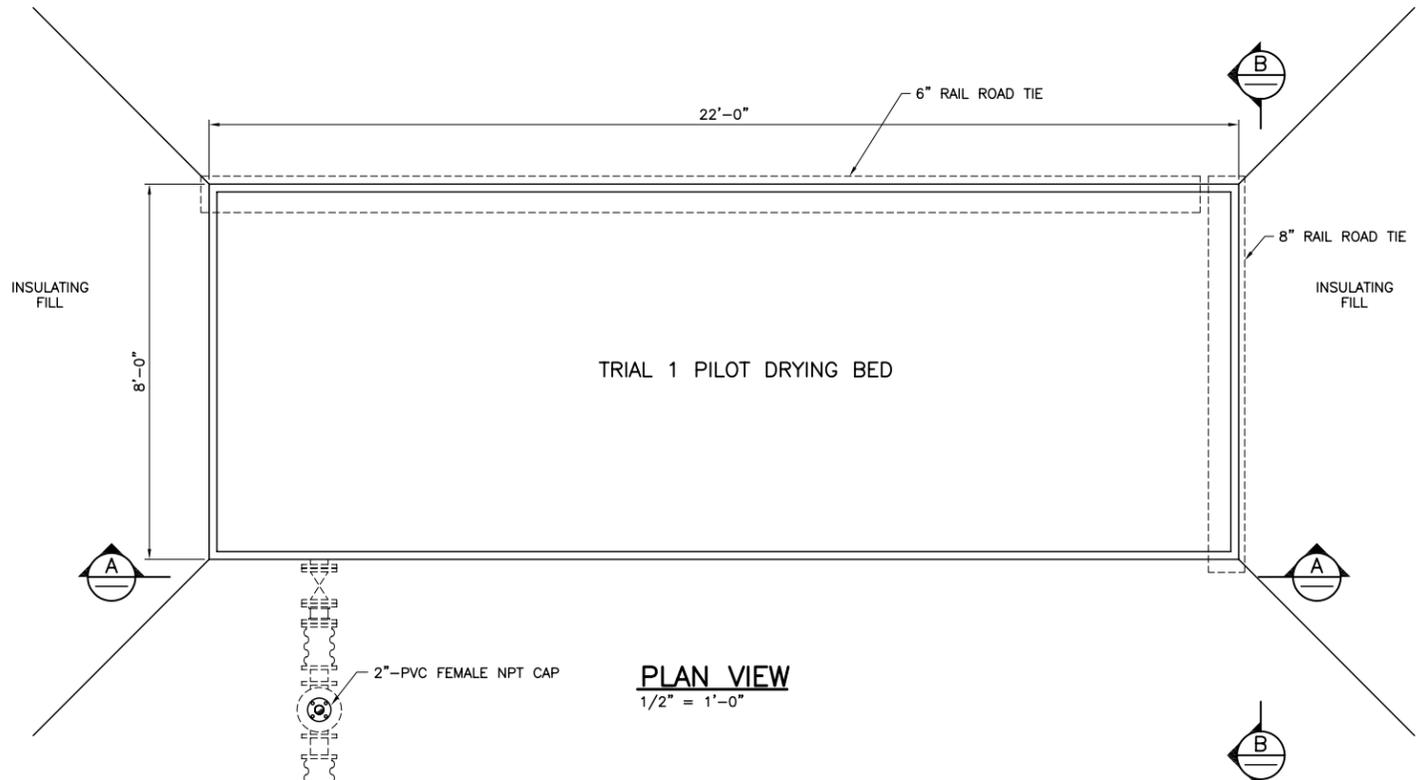
**Notes**

< = Analyte NOT DETECTED at or above the method detection limit.

J = Estimated value. Analyte detected at a level less than the Reporting Limit and greater than or equal to the Method Detection Limit. The user of this data should be aware that this data is of limited reliability.

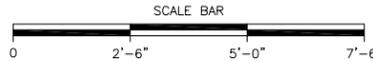
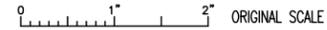
## **FIGURES**

---



Plot Date: 08/06/10 - 4:04pm. Plotted by: AET  
 Drawing Path: T:\Pete\Geomatrix\2009\0829\4-K Aspen Seep Leviathan Mine (13091B). Drawing Name: 13091B-PFD AUG-2010.dwg

CAUTION: THIS PLAN MAY BE REDUCED



REFERENCES:	NO.	REVISION	DATE	APRVD
PLANS				
DATUM				

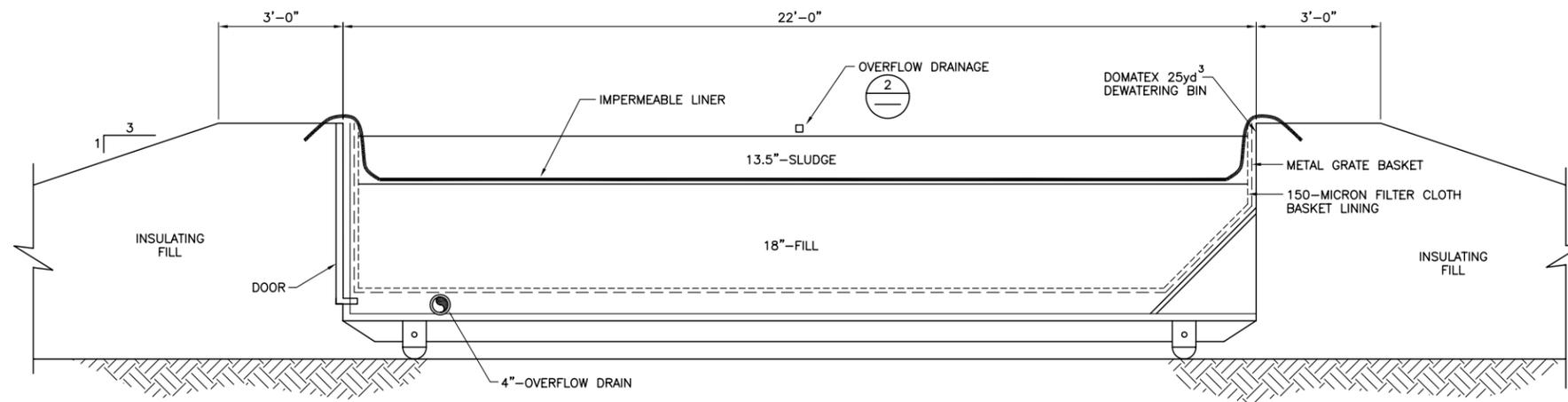
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DESIGNED	_____
CHECKED	_____
REVIEWED	_____

**ASPEN SEEP  
LEVIATHAN MINE  
ALPINE COUNTY, CALIFORNIA**

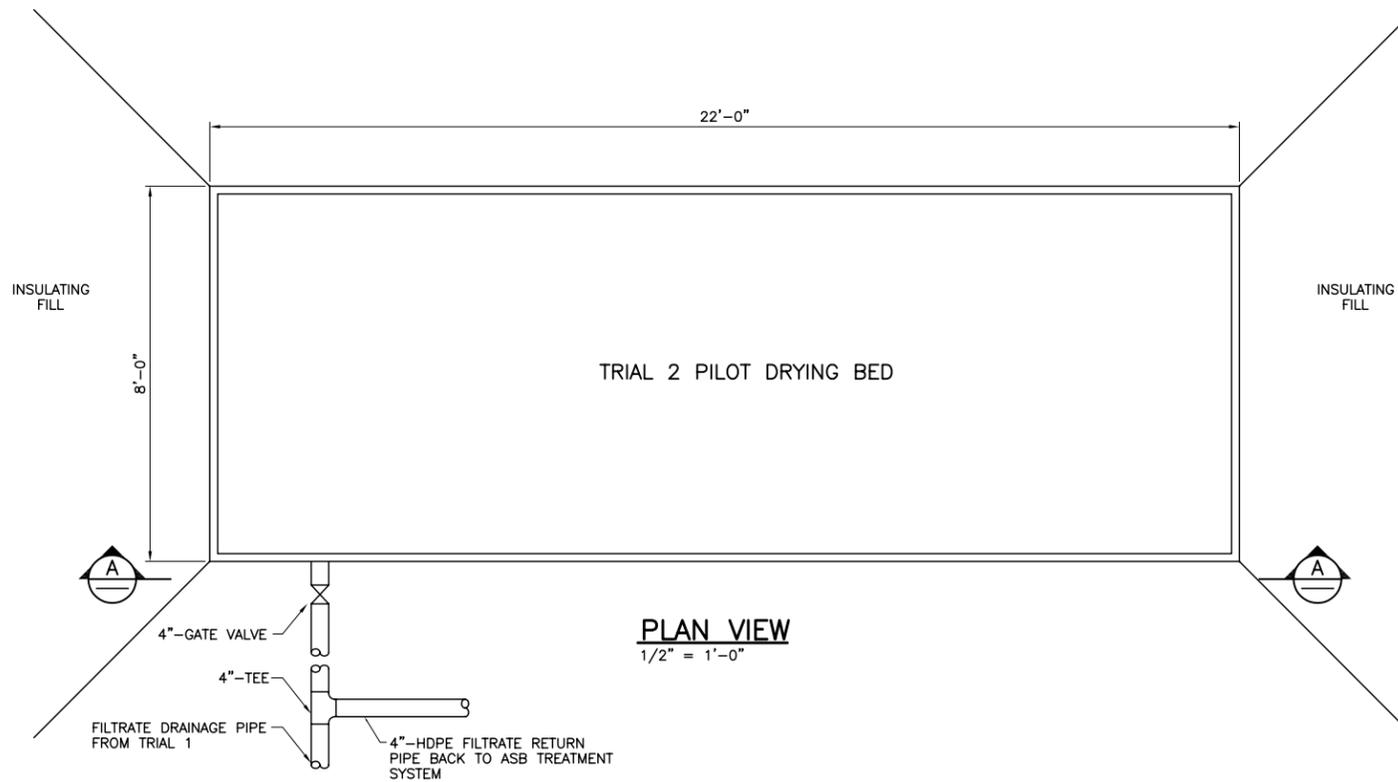
**AMEC Geomatrix**  
 AMEC Geomatrix Limited  
 420 Weber Street, North, Unit G  
 Waterloo, Ontario N2L 4E7  
 (519) 886-7500

ASPEN SEEP LEVIATHAN MINE  
 ALPINE COUNTY, CALIFORNIA  
  
 PHASE 1 SLUDGE DRYING BED PILOT  
 TRIAL 1 DESIGN

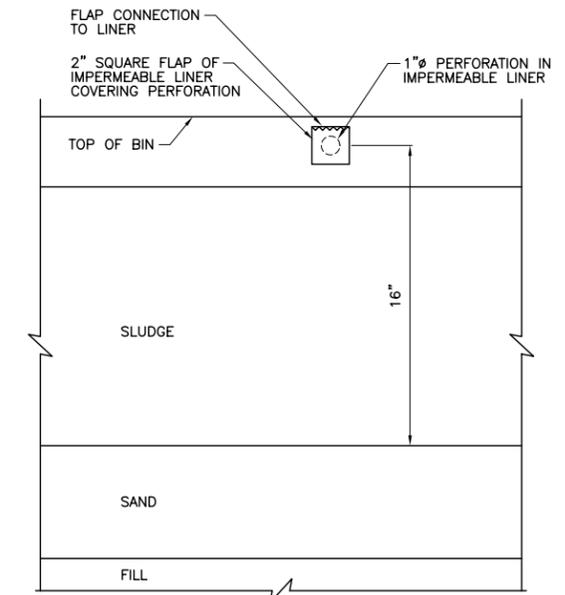
DATE:	AUGUST 2010
SCALE:	1/2" = 1'-0"
SHEET:	1 OF 1 SHEETS
PROJ No:	13091B1104.407C
FIG-01	



**SECTION A**  
1/2" = 1'-0"

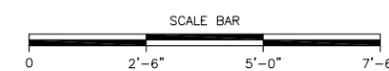


**PLAN VIEW**  
1/2" = 1'-0"



**OVERFLOW DRAINAGE**

**DETAIL 2**  
N.T.S.



CAUTION: THIS PLAN MAY BE REDUCED

NO.	REVISION	DATE	APRVD

DRAWN \_\_\_\_\_  
DESIGNED \_\_\_\_\_  
CHECKED \_\_\_\_\_  
REVIEWED \_\_\_\_\_

**ASPEN SEEP  
LEVIATHAN MINE  
ALPINE COUNTY, CALIFORNIA**

**AMEC Geomatrix**  
AMEC Geomatrix Limited  
420 Weber Street, North, Unit G  
Waterloo, Ontario N2L 4E7  
(519) 886-7500

**ASPEN SEEP LEVIATHAN MINE  
ALPINE COUNTY, CALIFORNIA**

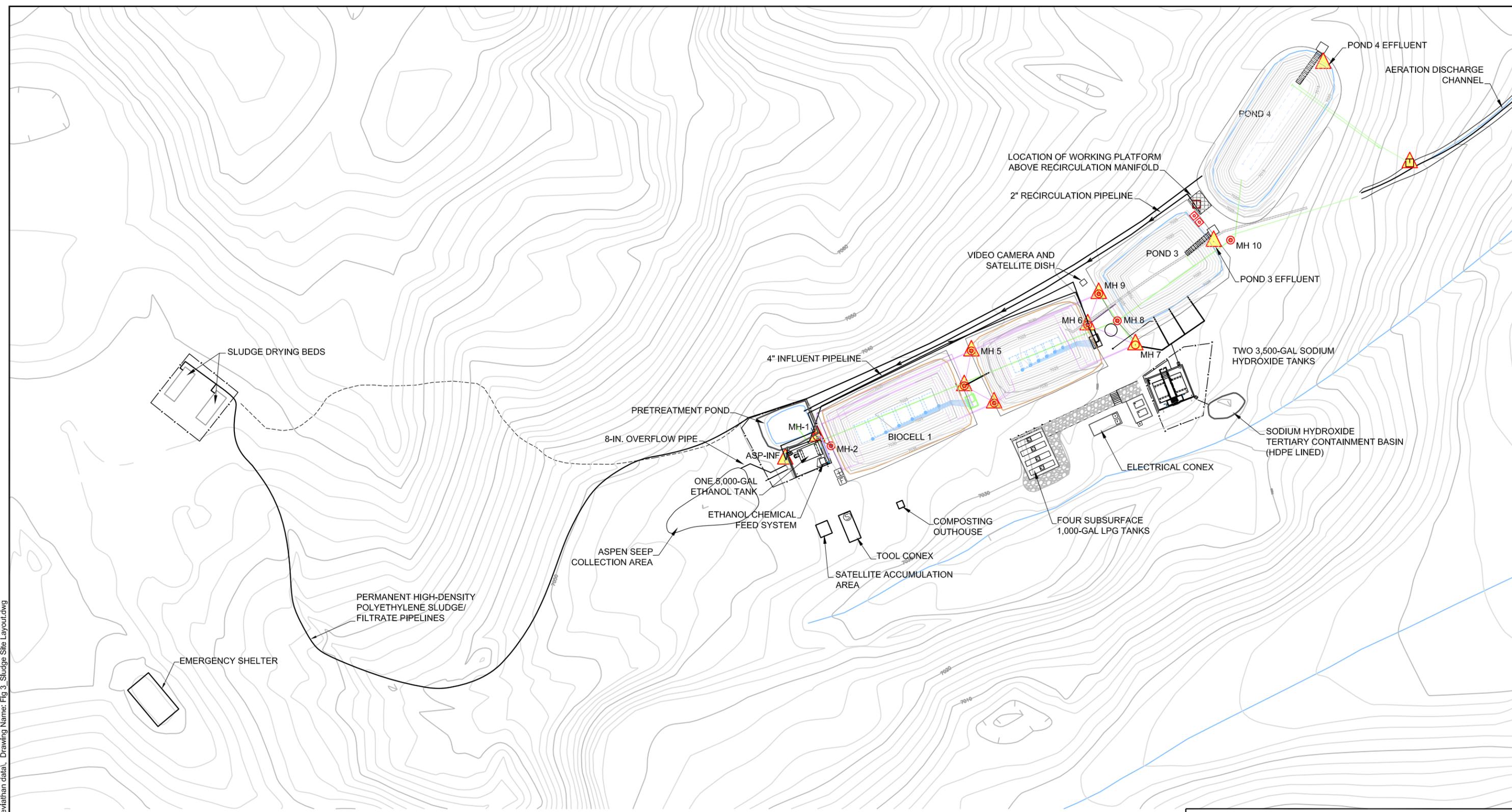
**PHASE 1 SLUDGE DRYING BED PILOT  
TRIAL 2 DESIGN**

DATE: AUGUST 2010  
SCALE: 1/2" = 1'-0"  
SHEET: 1 OF 1 SHEETS

PROJ No: 13091B1104.407C  
FIG-02

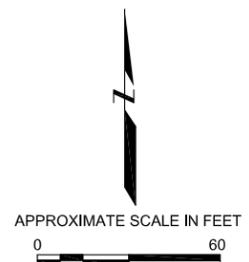
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Plot Date: 08/06/10 - 10:45am. Plotted by: donna.velasek  
 Drawing Path: C:\Donna\Donna Work\Leviathan data\ Drawing Name: Fig 3 - Sludge Site Layout.dwg



**Explanation**

	Bioreactor sample point		Spill kit
	Manhole		Fire extinguisher
	Rock slope		
	Submersible pump		
	Flowmeter/totalizer		
	First aid		



<p><b>SITE LAYOUT</b>          Phase I Sludge Drying Bed Pilot          Leviathan Mine Site          Alpine County, California</p>		
By: dpv	Date: 08/06/10	Project No. 13091.110
<b>AMEC Geomatrix</b>		Figure 3

**APPENDIX A**

---

Phase I Sludge Drying Bed Pilot Construction Photo Log

## Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Filtrate sump for Trial 1



Completed insulating fill placed around Sludge Drying beds, prior to initiation of filling sludge.

## Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Drain rock placed into Trial 1. Depth of drain rock is 9-inches



Initiation of placing filter sand in Trial 1. Final depth of filter sand is 9-inches.

# Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Trial Bin 1 prior to sludge placement



Trial bin 2 prior to sludge placement

## Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



ASB Pond 4 prior to mixing operations.



ASB Pond 4 during mixing operations. Note the visible sludge flow lines going toward the hose intake. Mixing operation occurred for approximately 4 hours to ensure sludge consistency.

## Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



ASB Pond 4 immediately prior to filling sludge beds



Consistency of sludge prior to filling sludge bins

# Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Trial Bin 2 during filling



Trial Bin 2 during filling.

# Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Trial Bin 1 during filling



Trial Bin 1 during filling. Note the slight wicking effect of sludge into the sand layer

# Site Photographs - ASB Sludge Drying Beds

Leviathan Mine Site  
Alpine County, California



Trial 1 at completion



Trial 2 at completion

**APPENDIX B**

---

Phase I Sludge Drying Bed Pilot Monitoring Photo Log



Figure 1. November 2, 2009 Trial 1 bin (left) overview, (right) sludge close up view. No free water was observed on top of the sludge layer.



Figure 2. November 2, 2009 Trial 2 bin (left) overview, (right) sludge close up view. The sludge appears settled with approximately one inch of clarified water above the bulk sludge.



Figure 3. Photos from November 11, 2009, 16 days from start. Trial 1 bin (left) overview, (right top) sludge close up view, (right bottom) view of orange-brown coloration on portion of sludge and sand in the corner of bin (sand was added to fill a gap that existed after filling the bin with sludge initially). Surface of dried sludge described as powdery.



Figure 4. Photos from November 17, 2009, 22 days from start. Trial 1 bin (left) overview, (right) sludge close up view.

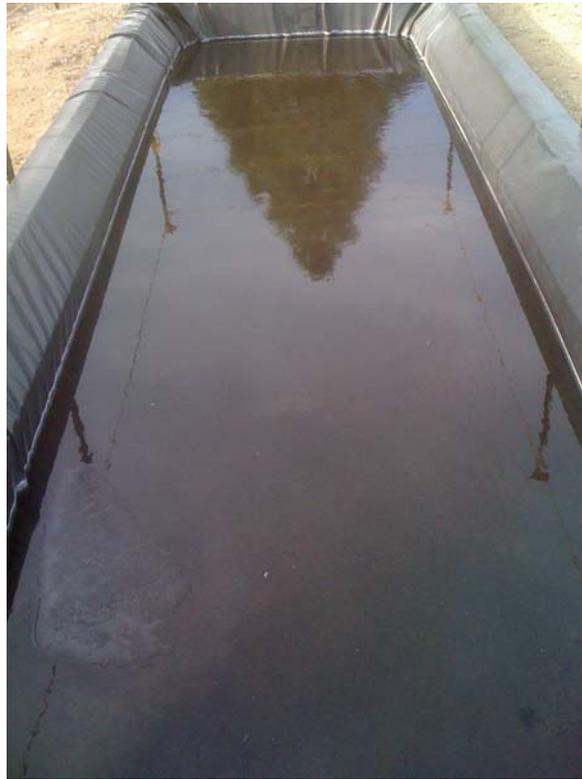


Figure 5. Photo from November 11, 2009, 16 days from start. Trial 2 bin. The sludge appears settled with approximately a half inch of clarified water above the bulk sludge. Sludge observed by field crew to be very “soupy”. Depth of water reduced from November 2, observation of 1” of clarified water. Note ice floating in clarified water.



Figure 6. Photos from November 17, 2009, 22 days from start. Trial 2 bin (left) overview-looking southeast, (right) sludge close up view. Approximately 1” of clarified water observed on top of settled sludge. Sludge described as very light and unconsolidated and could be easily mixed into the water layer. Ice was observed along the edge of the bin.



Figure 7. December 17, 2009, (left) Trial 1 overview (right) Trial 2 close up of ice under snow.



Figure 8. January 14, 2010, snow cover on SDB bins.



Figure 9. February 2, 2010, (left) Trial 1 close-up of snow to sludge surface (sludge was frozen and deemed unfeasible to sample). Trial 2 (right) close-up of snow to ice surface (no sludge samples were collected).



Figure 10. March 9, 2009 filtrate sample collection.



Figure 11. Trial 1 May 13, 2010.

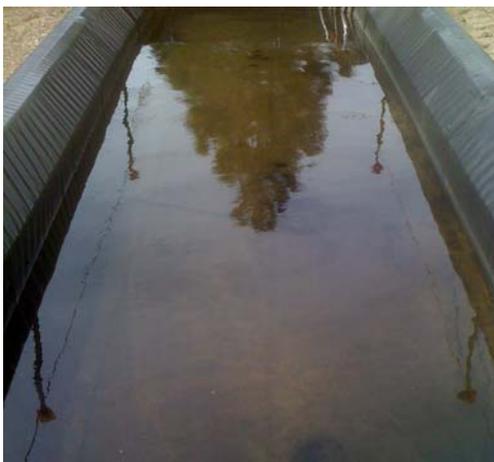


Figure 12. Trial 2 May 13, 2010 (left) overview, (right) close up of cracks developing in settled sludge under the clarified water.



Figure 13. Trial 1 June 8, 2010 (left) overview of bin, fine powdery material top layer mostly on the right side of bin, (right) close-up of a grab sample in gloved hand (rocks in background are the base material of the walkway between the bins).



Figure 14. Trial 2 June 8, 2010 (left) overview of bin, deep dry cracks observed on far end of bin, wet sludge observed at close end of the bin, (middle) wet sludge sampled from close end of the bin, (right) visually drier sludge sampled from far end of the bin.



Figure 15. Trial 1 June 22, 2010 (left) overview of bin, (right) close up of grab sample in gloved hand.



Figure 16. Trial 2 June 22, 2010 (left) overview of bin, very dry with deep crevasses, (right) close up of crevasses in bin with depth measurements.



Figure 17. Trial 1 July 20, 2010 (left) overview of bin, (right) close up of bin.



Figure 18. Trial 2 July 20, 2010 (left) overview of bin, (right) close up of bin.

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**APPENDIX C**

Surface Chemistry Analysis Results Report, Hazen Laboratories

January 20, 2010

**E-mail and Mail Delivery**

Joy D. Jenkins, PhD  
Project Engineer  
AMEC Geomatrix, Inc.  
1401 17<sup>th</sup> Street, Suite 600  
Denver, CO 80202-1485

E-mail: [joy.jenkins@amec.com](mailto:joy.jenkins@amec.com)

Subject: Characterization of Reducing Bioreactor Products  
Hazen Project 11061

Dear Dr. Jenkins:

This report presents the characterization results of sludge samples from a sulfate-reducing bioreactor. Three slurry samples were delivered to Hazen Research, Inc. on November 23, 2009, for analysis by electron microprobe and x-ray diffraction (XRD) to determine the form(s) of iron present. The possible presence of any FeS is of particular interest. The samples were received, chilled, and have been kept refrigerated in storage to minimize oxidation of the sulfides. The sample identifications and approximate weights are shown in Table 1.

**Table 1. Sample Identification**

Sample	Client Sample Identification	Weight, g
1	087ASP1BIN314 10/26/09	350
2	087ASP2BIN315 10/26/09	350
3	078P3ASPSLG295 8/6/09	550

Note: For brevity, the sample number is used to identify the sample throughout this report

All three samples were analyzed by XRD, but microprobe analysis was performed only on Samples 2 and 3 because Samples 1 and 2 are duplicate samples from the same process stream. Preliminary reports were e-mailed as data were obtained.

1. XRD analysis on all three samples: 12/4/2009
2. Microprobe analysis on Sample 2: 12/4/2009 and 12/7/2009
3. Microprobe analysis on Sample 3: 1/5/2010

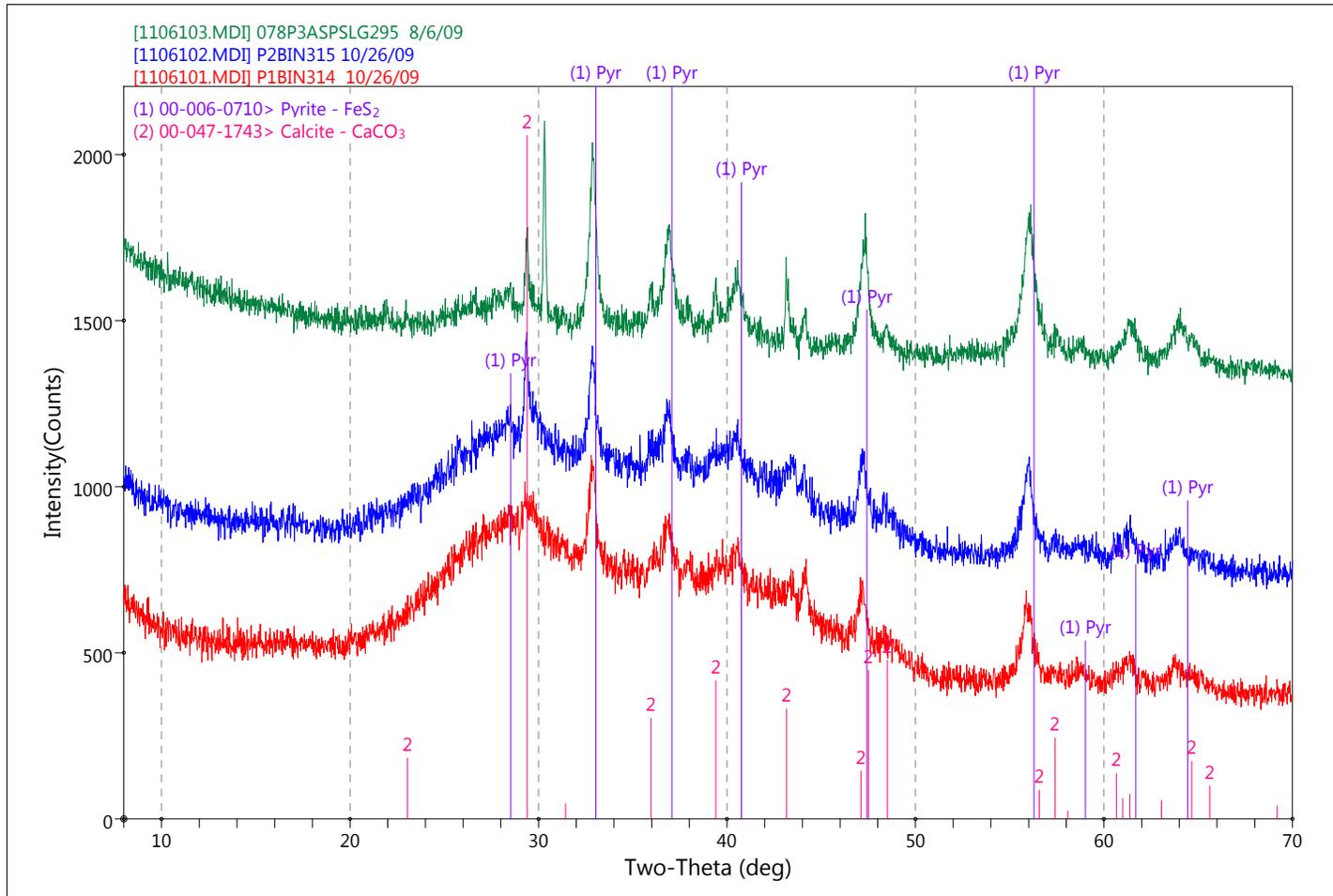
### XRD ANALYSIS

The samples were filtered and analyzed as moist filter cakes to avoid dehydration and analyzed as dry solids. The dry solids were obtained by washing a portion of the moist filter cake in the filter with acetone and evaporating the acetone. The XRD patterns of all three as-received samples show pyrite as the only sulfide, with the highest concentration in Sample 3. The XRD patterns of the moist Samples 1 and 2 show a large broad peak, indicating a major amount of amorphous material, but the broad peak was much smaller in Sample 3 (see Figure 1). In the acetone-washed samples, the broad peak disappeared. To determine whether something had been washed out by the acetone, another moist portion of Sample 1 was dried at 60°C in an atmosphere of argon and analyzed. The broad peak was not present in this sample, indicating that just drying the sample caused the peak to disappear (see Figure 2). While preparing the samples, it was noted that Samples 1 and 2 filtered much slower than Sample 3 and that the moist filter cakes seemed gelatinous. Sample 3 is more granular. The individual patterns of the acetone-dried samples are shown in Figures 3–5. A summary of the XRD results is shown in Table 2.

**Table 2. Summary of XRD Results**

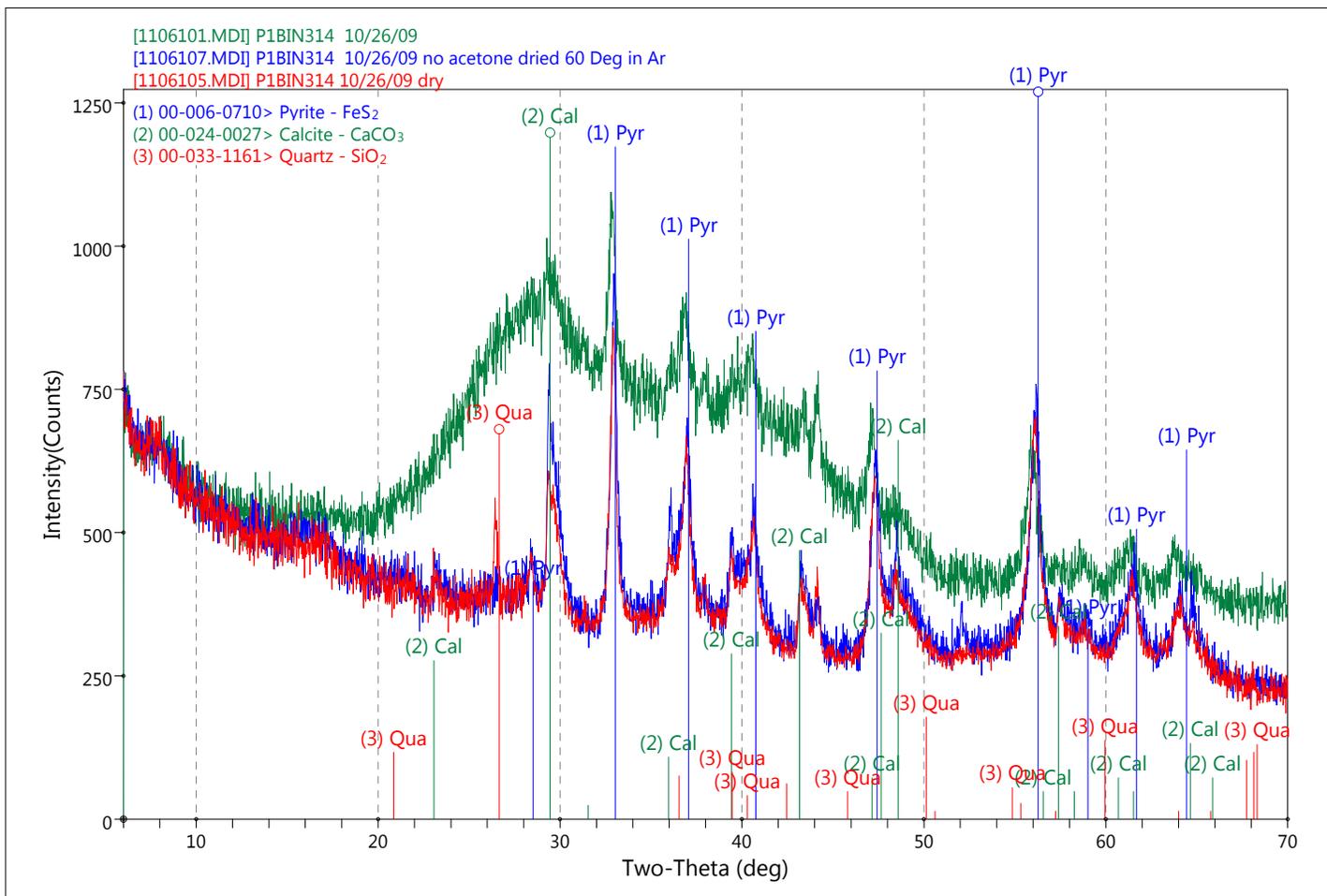
Sample	1	2	3
Amorphous	major	major	minor?
Pyrite, FeS <sub>2</sub>	subordinate	subordinate	major
Troilite, FeS	not detected	not detected	possible trace
Other Fe sulfides	not detected	not detected	not detected
Calcite	not detected	trace	trace

Other sulfides specifically searched for but not detected include mackinawite (FeS), smythite (Fe<sub>9</sub>S<sub>11</sub>), and Fe<sub>9</sub>S<sub>10</sub>.



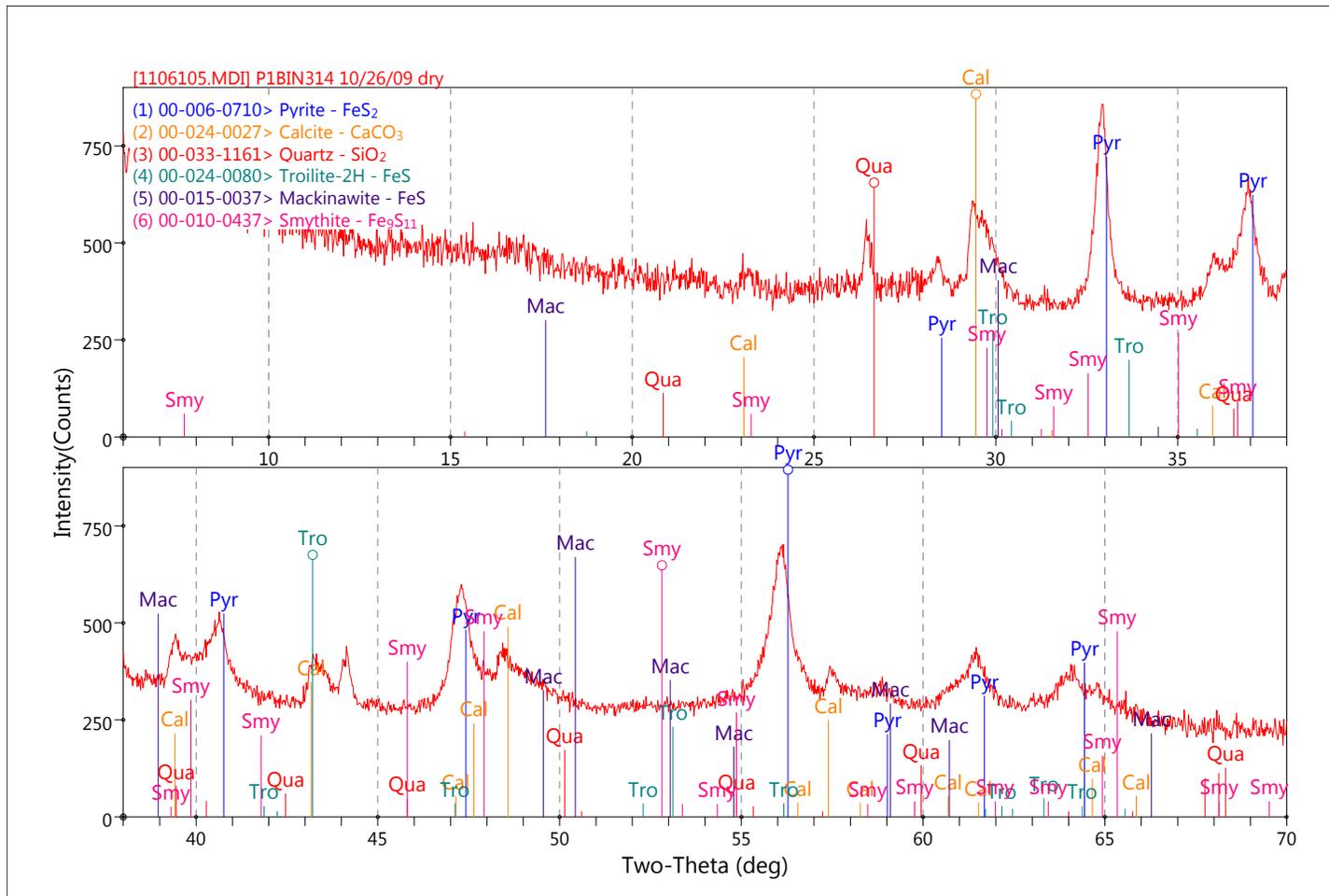
**Figure 1. XRD Patterns of Samples 1–3, Moist Filter Cakes**

This figure shows XRD patterns obtained from the moist filter cakes of all three samples and illustrates the broad peak present in Samples 1 (red) and 2 (blue), which is apparently due to an amorphous, gelatinous aluminum hydroxide silica gel. These are elements in the matrix of the sample as analyzed with the microprobe. There may be a minor amount present in Sample 3 (green). The peak at 30.2° two-theta in Sample 3 could not be identified. It is not present in the dried sample (Figure 5). It may be due to an electronic noise spike because it is so sharp.



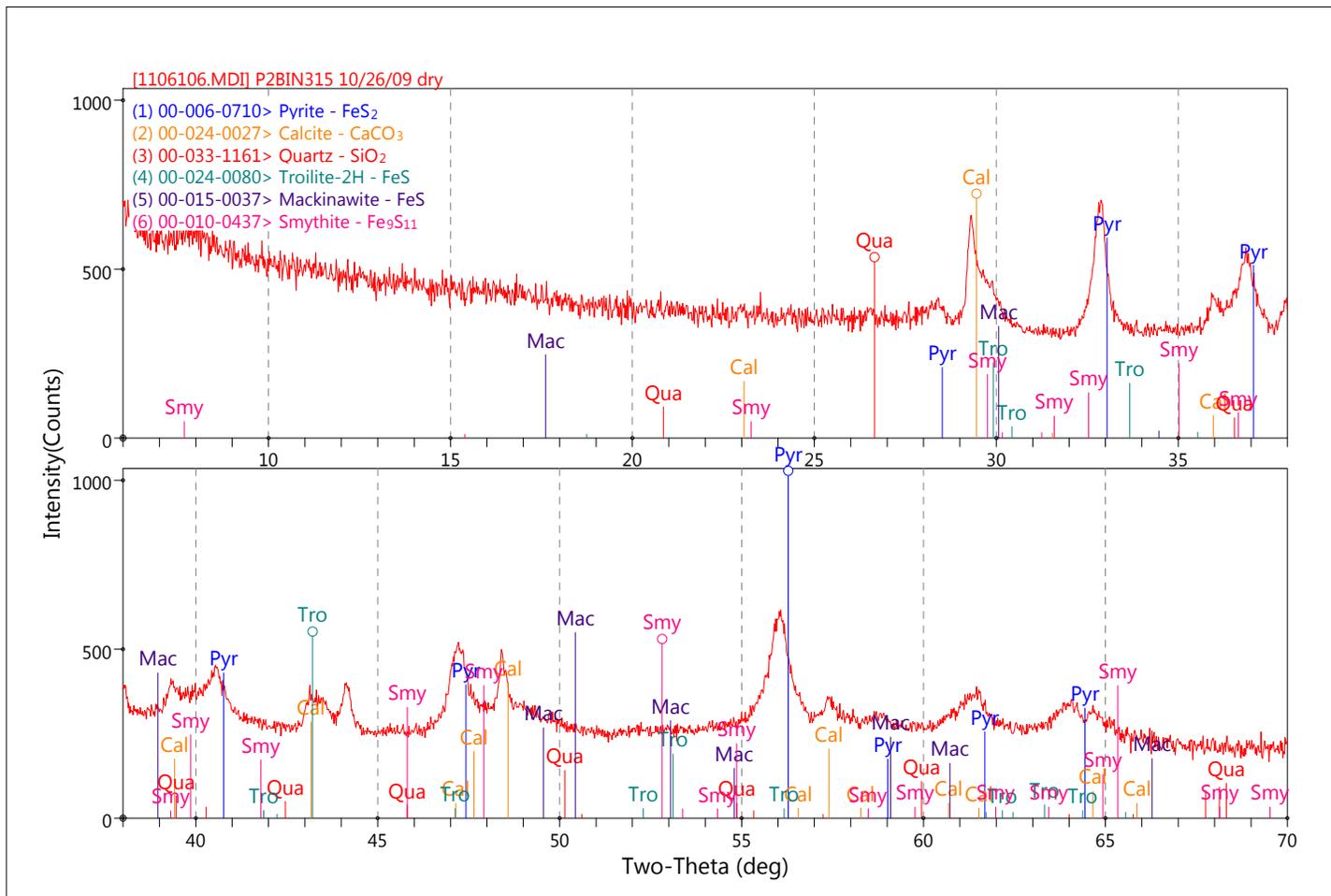
**Figure 2. XRD Patterns of Moist, Acetone-Dried, and Dried Sample 1**

This figure shows XRD patterns of Sample 1 obtained from a moist sample (green), an acetone-dried sample (red), and a sample dried at 60°C in argon (blue). The dried samples are essentially identical.



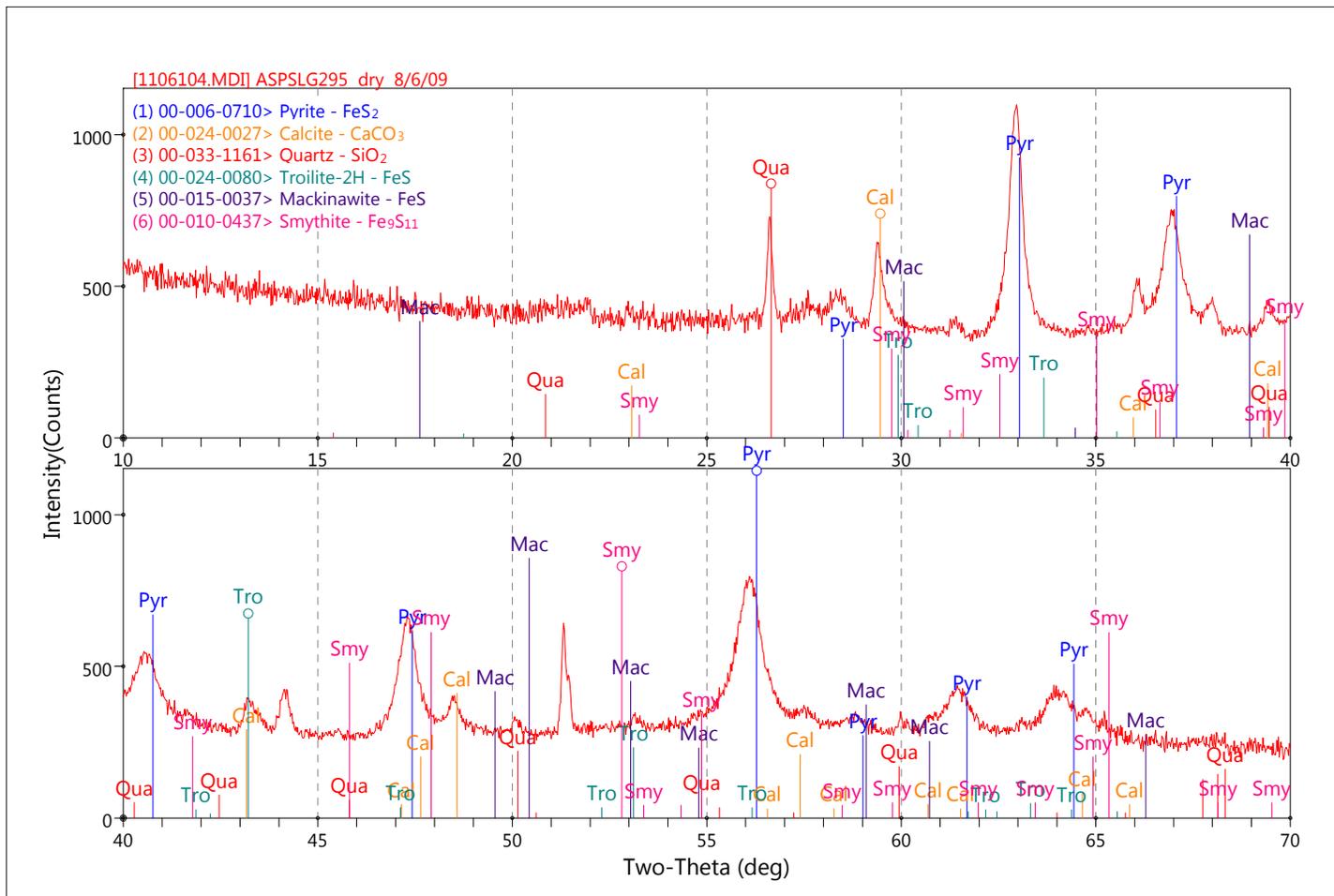
**Figure 3. XRD Pattern of Dried Sample 1**

This figure shows the XRD pattern of the dried Sample 1. There is no evidence of any troilite, mackinawite, or smythite, but pyrite is present. The peak at 43.2° two-theta is due to calcite, not troilite. The next strongest peak of troilite is at 53.1° two-theta, but none is evident.



**Figure 4. XRD Pattern of Dried Sample 2**

This figure shows the XRD pattern of the dried Sample 2. There is no evidence of any troilite, mackinawite, or smythite. The peak at 43.2° two-theta is due to calcite, not troilite. The next strongest peak of troilite is at 53.1° two-theta, but none is evident.



**Figure 5. XRD Pattern of Dried Sample 3**

This figure shows the XRD pattern of the dried Sample 3. There is no evidence of any mackinawite or smythite. The peak at 43.2° two-theta is due to calcite, not troilite. The next strongest peak of troilite is at 53.1°, and a trace is evident in this sample.

## MICROPROBE ANALYSIS

Polished sections of the acetone-dried samples were prepared for microprobe analysis. The polished sections were prepared soon after drying and were kept in a N<sub>2</sub> atmosphere after preparation. Energy dispersive x-ray microanalysis was performed on selected spots to identify the elements present, but all quantitative analyses were performed by wavelength dispersive analysis. A 15-kV accelerating voltage was used to minimize the x-ray excitation volume in the sample, and the beam current was about 25 nA. The very fine-grained heterogeneous nature of the samples often leads to errors in analysis because x-rays are generated from a volume of sample that is sometimes larger than the particles being analyzed, which makes it difficult to determine which elements are associated with each other. In some areas, wavelength dispersive x-ray line scans were performed in an effort to indicate elemental associations in small particles.

The analyses, along with microprobe images and x-ray line scans, are shown in Figures 6–20.

### SAMPLE 2

The sample consists of heterogeneous agglomerations that have numerous inclusions of pyrite that range in size from about 0.3 to 2 μm in diameter and 5–30-μm calcite particles dispersed throughout. The identification of the pyrite is based on the XRD analysis and the cubic morphology, as well as the 2:1 atomic ratio of sulfur to iron in the microprobe analysis. The pyrite sometimes occurs in relatively dense clusters of submicron particles, but no framboidal pyrite was observed.

The composition of the agglomerate matrix is fairly uniform with about 13–15% Fe, 5–7% Al, 1–2% Si, about 1% Ca, and 7–10% S. A significant, if not major, portion of the iron and sulfur is in the matrix, and the sulfur-to-iron atomic ratio is about 1:1, but no FeS or iron sulfate is evident in the XRD patterns. A portion of the sample was acidified with 6 N HCl as a test for FeS, and only a trace of H<sub>2</sub>S could be detected with lead acetate paper. The sulfur is present as sulfide as determined by the energy shift of the sulfur x-rays generated, which is about 1.4 eV lower than x-rays generated from sulfate. This energy difference can be measured with a wavelength dispersive x-ray scan of the sulfur K-α peak, which has an energy of 2.31 keV, using a PET analyzing crystal. X-ray line scans of iron, aluminum, and sulfur obtained for the matrix illustrate its nearly uniform composition and a lower sulfur-to-iron ratio than that of the pyrite particles traversed in the scan. Examples of this are shown in Figures 9 and 11. The exact nature of the iron–sulfur occurrence in the matrix is not understood.

Shrinkage cracks are present in some of the agglomerations, as shown in Figure 7. The electron beam of the microprobe also caused shrinkage cracks to develop because of the heating effects, as shown in Figure 12. The gelatinous nature of the moist filter cakes is probably due to an amorphous aluminum hydroxide–silica gel because no aluminum–silicon compounds were detected in the XRD pattern.

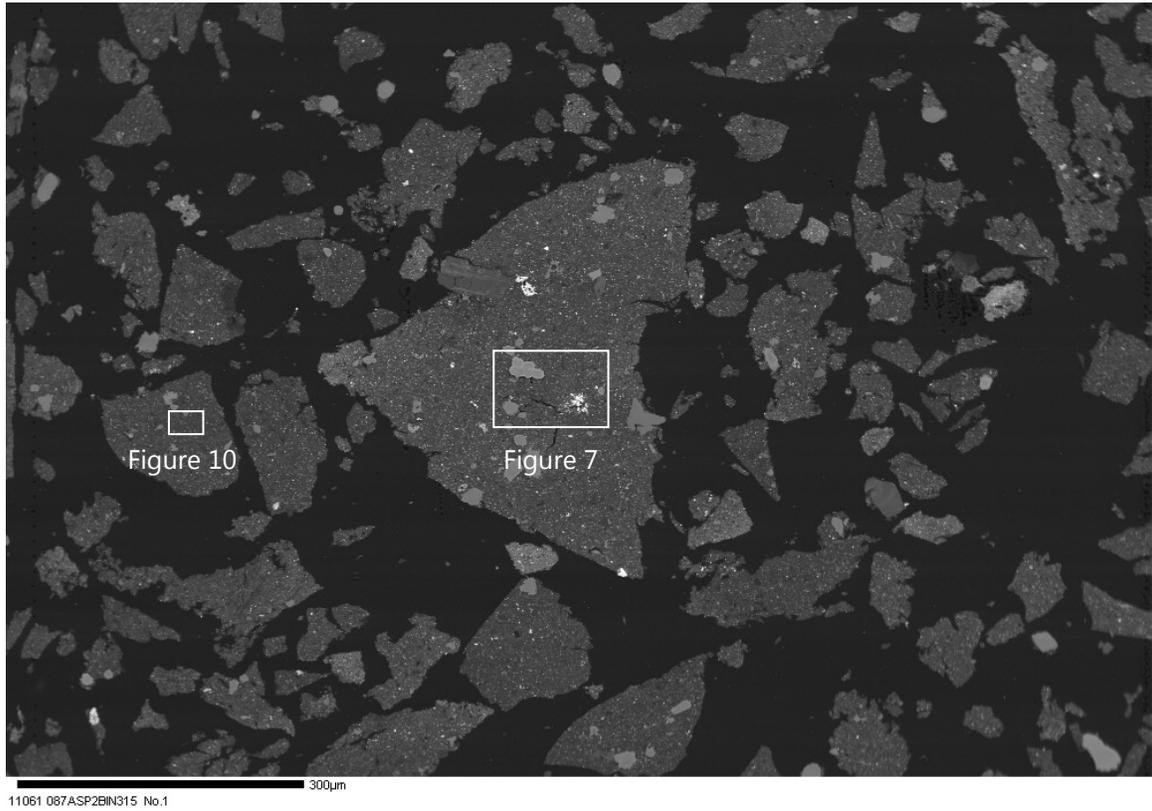
The samples also contain low concentrations of copper, nickel, and zinc, so a brief survey was done to look for those elements. Ten pyrite particles were analyzed, and the concentrations were found to be

quite variable. Some had detectable amounts of all three elements, and some had only one or two. The average concentrations were 0.14% Cu, 0.29% Ni, and 0.06% Zn. The highest concentrations encountered were 0.8% Cu, 0.8% Ni, and 0.4% Zn. A thorough search was not made, but pyrite was the only material in which these elements were detected. There may be other sources, but possibly with non-detectable concentrations. Under the conditions used, the detection limit for all three elements is about 0.03%. The counting time was 20 s, and the backgrounds were measured close to the peaks (1 mm on each side instead of the normal 4 mm).

### **SAMPLE 3**

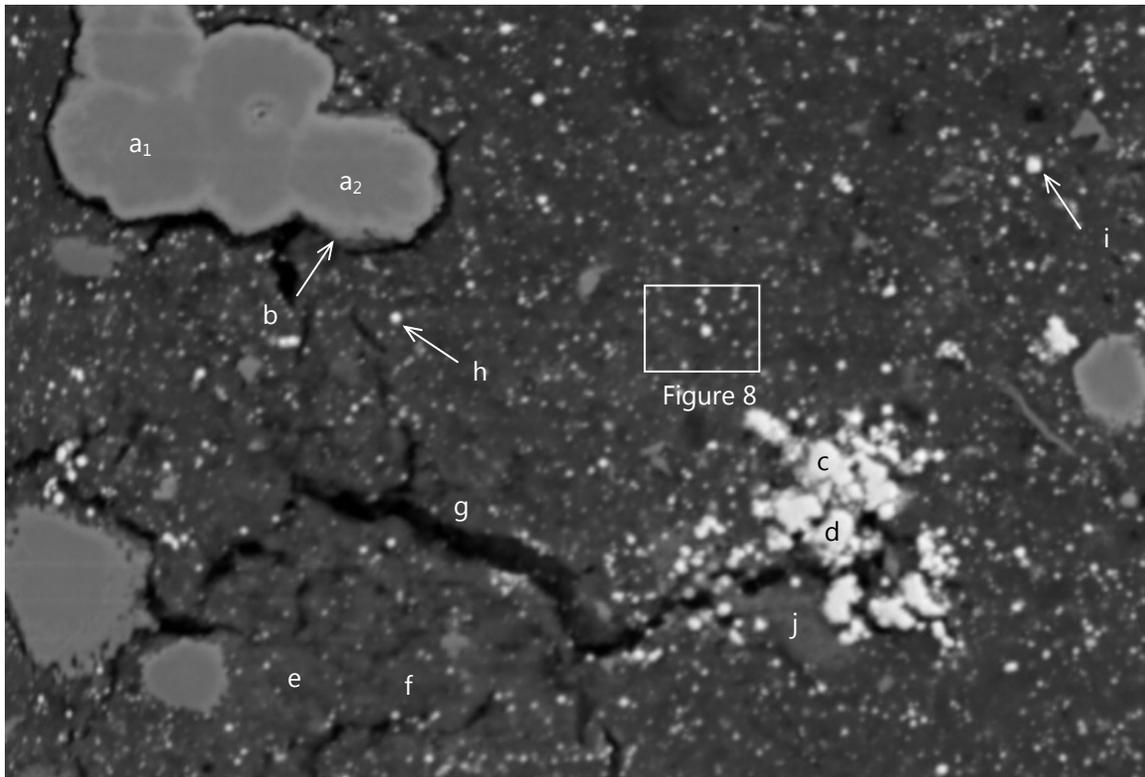
This sample also consists of agglomerations similar to those of Sample 2, but nearly all of the agglomerations have prominent shrinkage cracks (see Figure 15), and no further shrinkage was produced by the electron beam, indicating this as-received sample was more dehydrated than the others, as indicated by the lack of a broad peak in the XRD pattern.

The matrix composition of the agglomerations seems to be more erratic compared with that of Sample 2. The sulfur-to-iron atomic ratio is commonly about 0.5:1 in this sample. The concentrations of iron and aluminum are about twice as high, and the sulfur concentration is about half that of the Sample 2. The higher concentrations in this sample are apparently due to the concentrating effect of more advanced dehydration. This occurrence of pyrite in this sample is similar to that of Sample 2.



**Figure 6. Sample 2, Example 1**

This figure shows typical particles in the sample consisting of heterogeneous agglomerated fines. The boxed areas are shown enlarged in Figures 7 and 10.

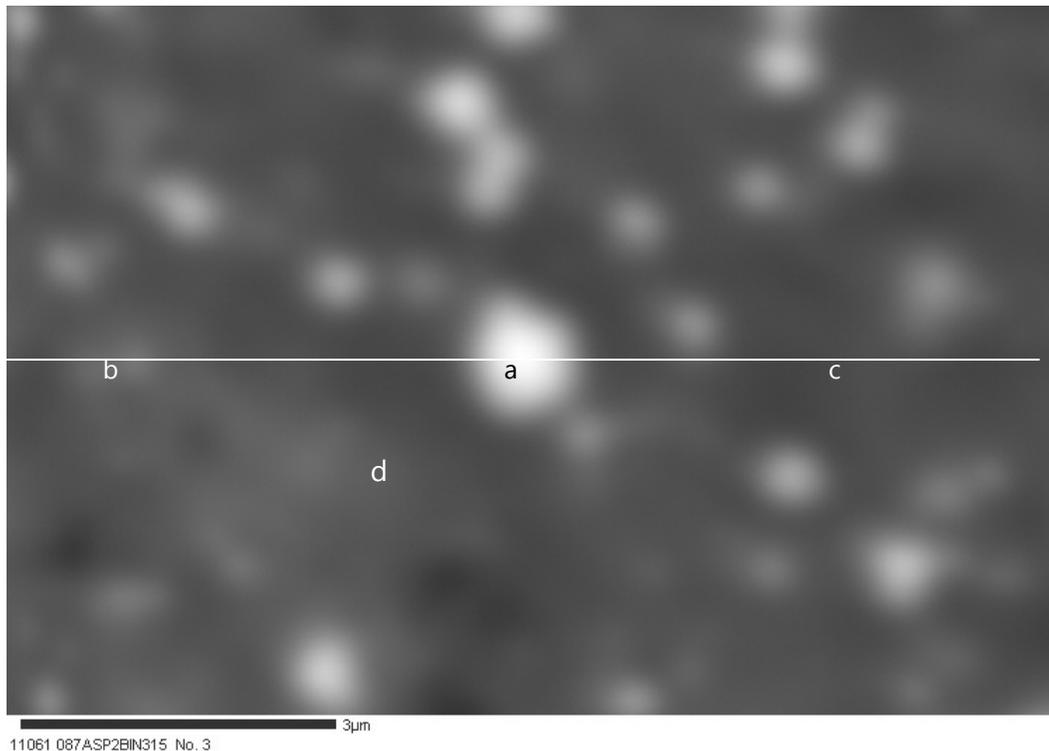


11061 087.ASP2BIN315 No. 2

**Figure 7. Sample 2, Example 2**

This figure shows an enlarged view of the boxed area indicated in Figure 6. Particle a is a manganoan calcite. All of the white spots are apparently pyrite. A few of the larger ones were analyzed and have the proper sulfur/iron atomic ratio. The dark matrix has a surprisingly high concentration of iron and sulfur and the sulfur/iron atomic ratio is about 1:1, but there was no indication of any FeS compound in the XRD pattern. The composition of the matrix is relatively uniform. The boxed area is shown enlarged in Figure 8.

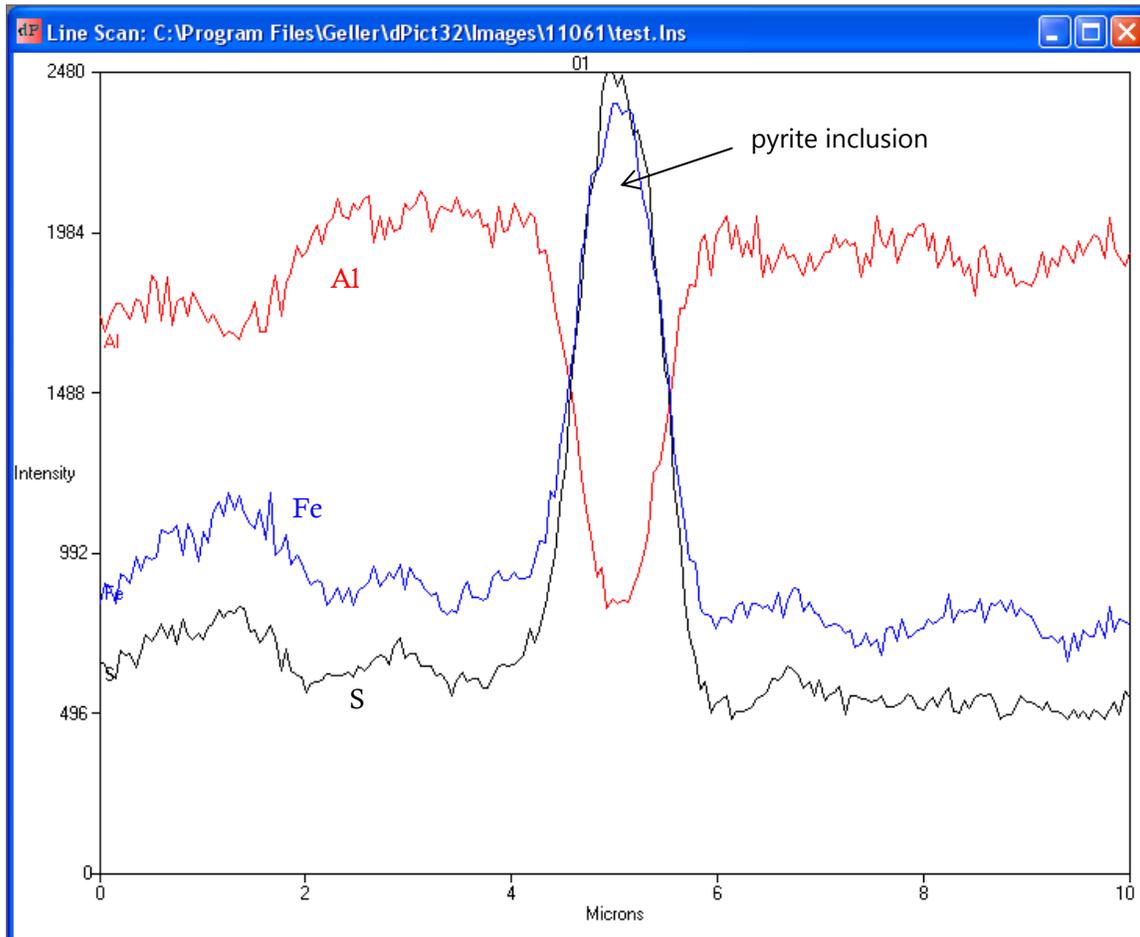
Spot	Analysis, wt%						S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	Mn	
a <sub>1</sub>	0.8	24.0				10.6	
a <sub>2</sub>	0.6	23.4				11.2	
b	6.0	8.1				27	
c	43					53	2.14
d	44					51	2.05
e	14.2	1.1	6.6	1.9	9.6		1.18
f	13.6	1.0	6.5	2.0	6.7		0.86
g	15.1	0.8	5.8	1.8	7.9		0.91
h	34		2.0	0.6	36		1.87
i	39		0.9	0.3	47		2.11
j	19	0.6	5.1	1.9	9.2		0.85



**Figure 8. Sample 2, Example 3**

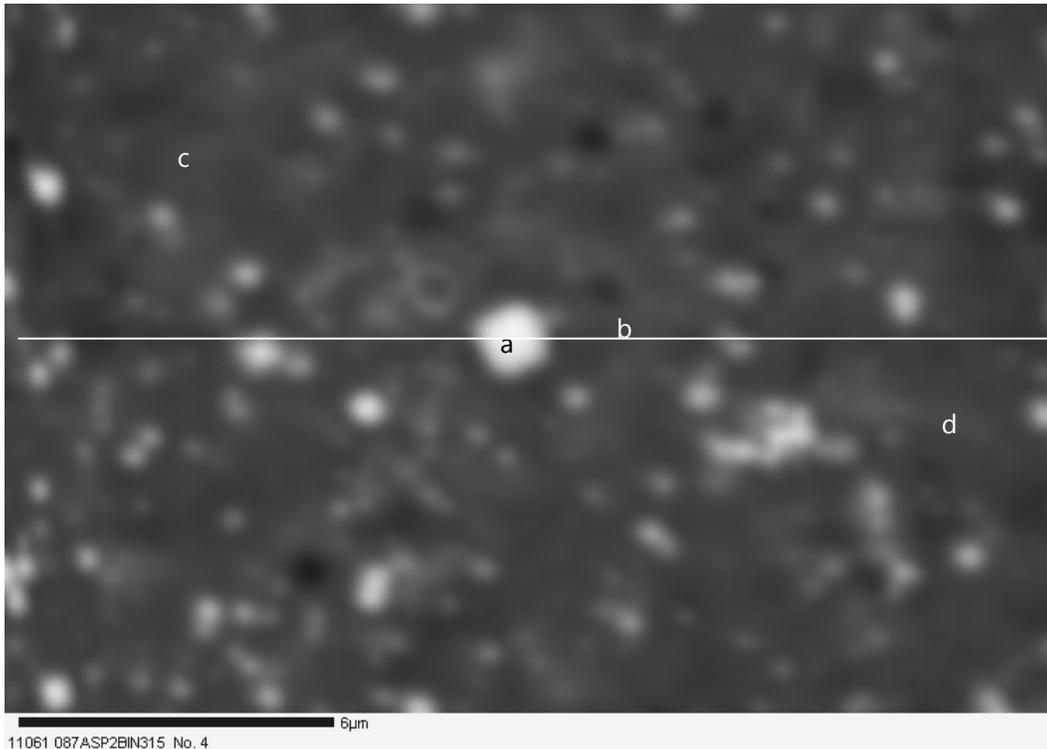
This figure shows an enlarged view of the boxed area in Figure 7. Particle a is apparently pyrite, and the gray matrix has a sulfur/iron atomic ratio of about 1:1. An x-ray line scan along the white line is shown in Figure 9. The form of the iron and sulfur in the matrix is not clear; FeS is not evident in the XRD pattern, but it could be amorphous.

Spot	Analysis, wt%					S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	
a	35	nd	2.3	0.6	36	1.78
b	15.0	0.7	5.4	1.7	10.6	1.23
c	14.8	0.7	5.9	1.9	9.3	1.09
d	14.0	0.9	7.0	2.4	9.3	1.16



**Figure 9. Sample 2, X-ray Line Scan From Figure 8**

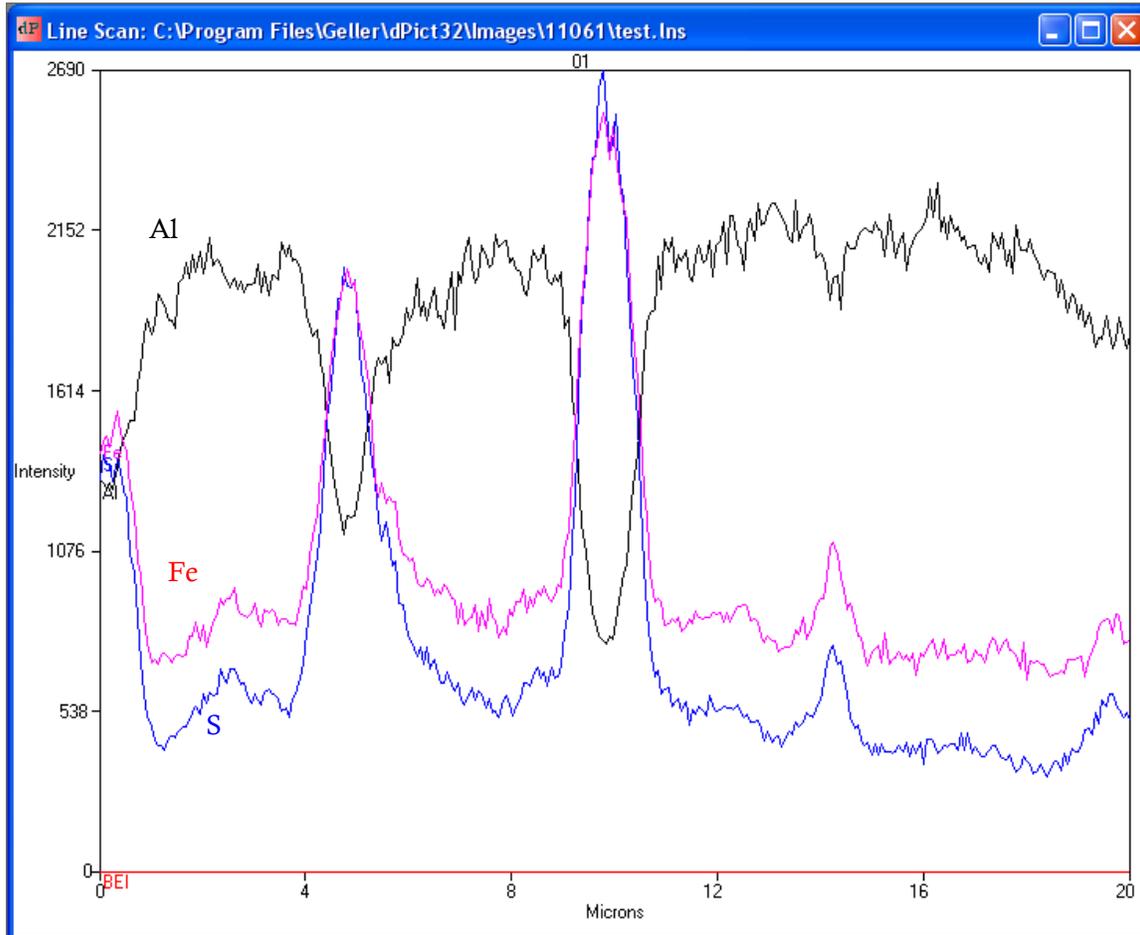
This figure shows an x-ray line scan along the white line in Figure 8. Note the difference in the sulfur/iron ratio between the pyrite inclusion and the gray matrix. The composition of the matrix is relatively uniform.



**Figure 10. Sample 2, Example 4**

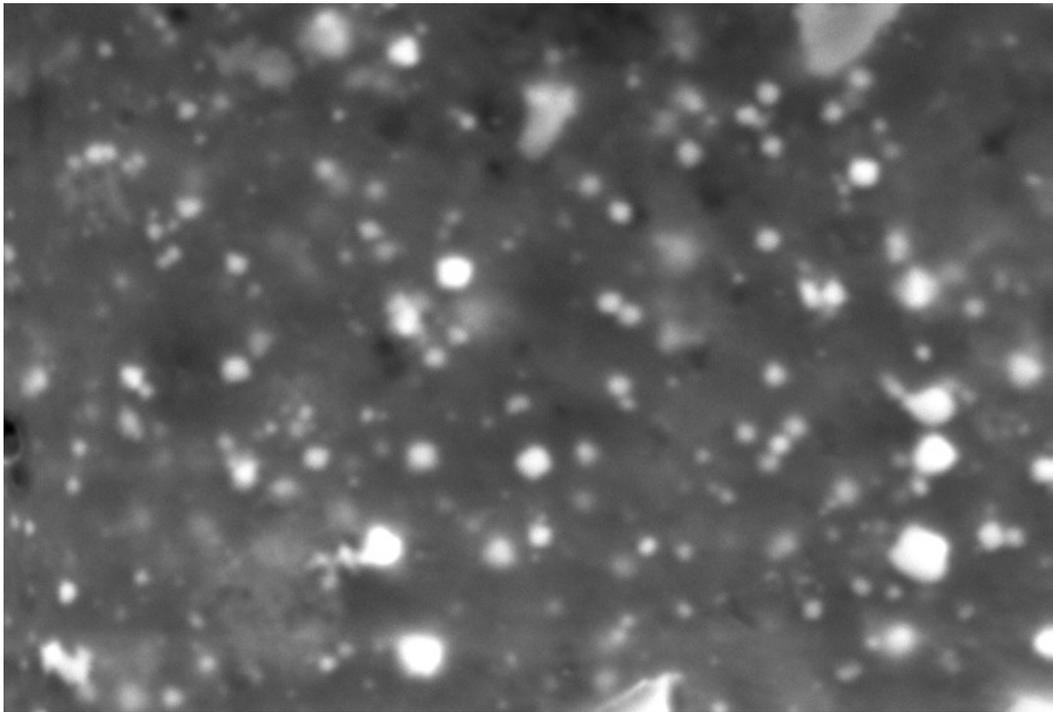
This figure shows an enlarged view of the smaller boxed area in Figure 6. An x-ray line scan along the white line is shown in Figure 11.

Spot	Analysis, wt%					S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	
a	38		1.9	0.5	41	1.87
b	12.8	0.8	6.4	2.2	7.9	1.07
c	12.6	1.0	5.1	1.5	8.3	1.15
d	11.9	0.9	6.4	2.2	6.8	0.99

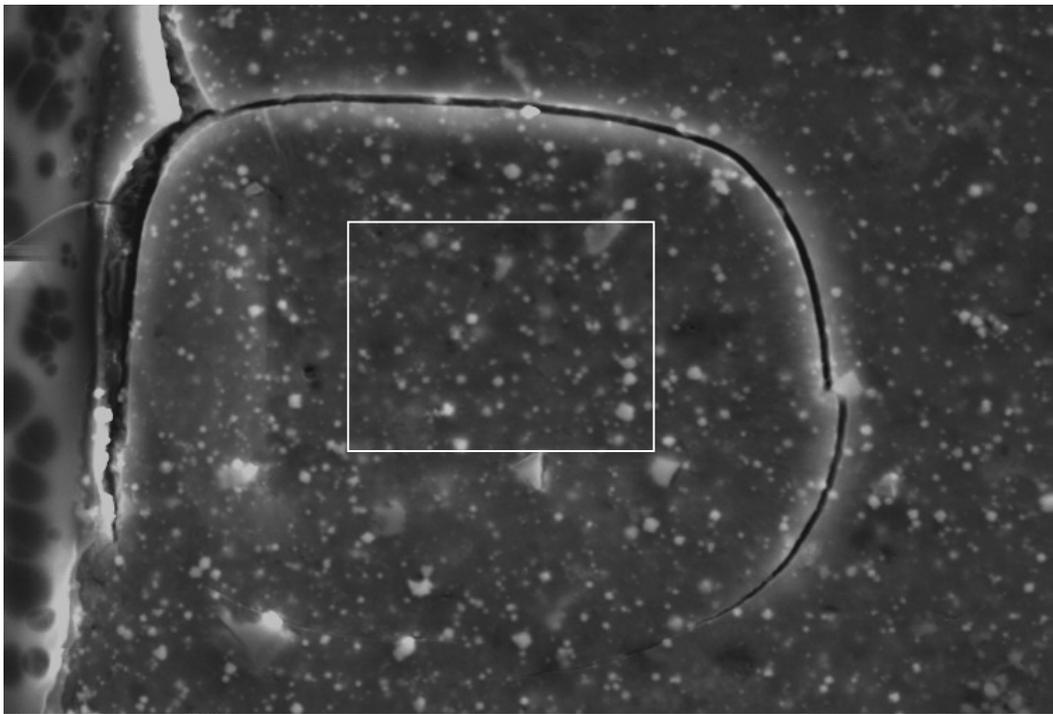


**Figure 11. Sample 2, X-ray Line Scan from Figure 10**

This figure shows an x-ray line scan along the white line in Figure 10. It also illustrates the sulfur/iron ratio difference between the pyrite inclusions and the matrix.



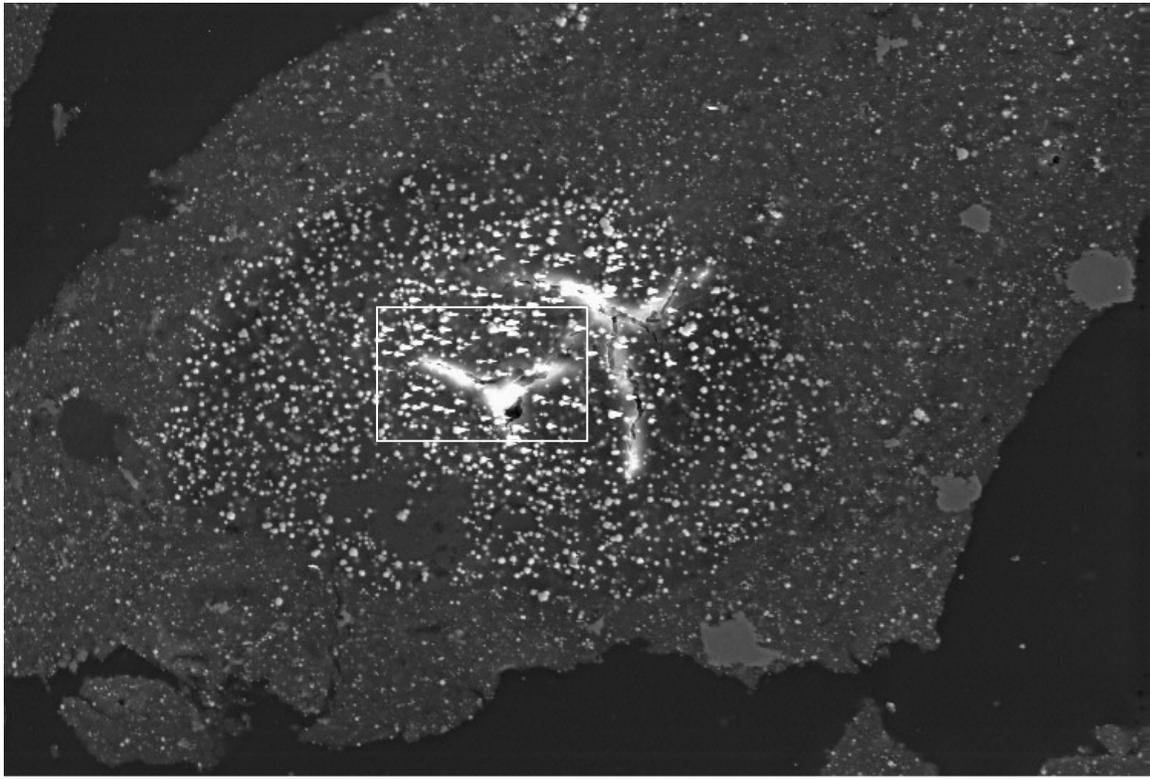
11061 087ASP2BIN315 No. 5 SEI



11061 087ASP2IN315 No. 5b

**Figure 12. Sample 2, Example 5**

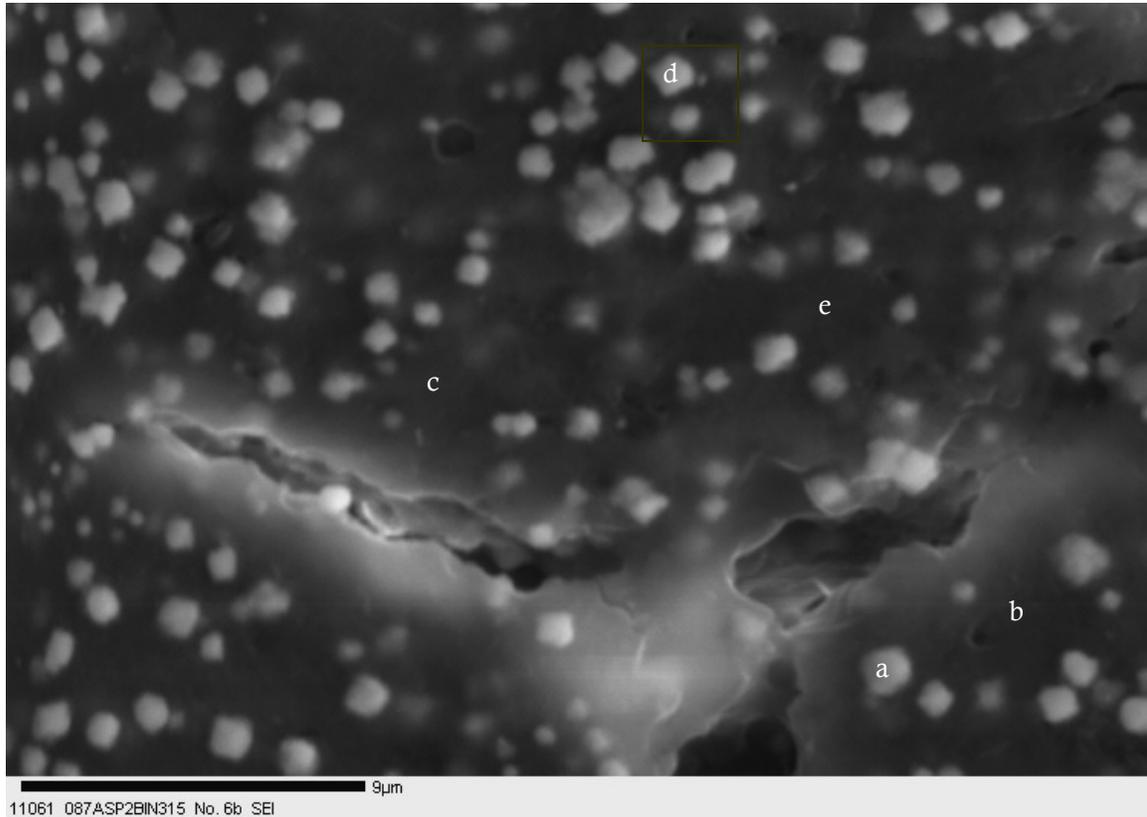
This figure shows the shrinkage crack produced in the gray matrix by electron-beam heating in the boxed area while the upper photo was being obtained. The white particles are pyrite.



11061 087ASP2BIN315 No. 6 SEI

**Figure 13. Sample 2, Example 6**

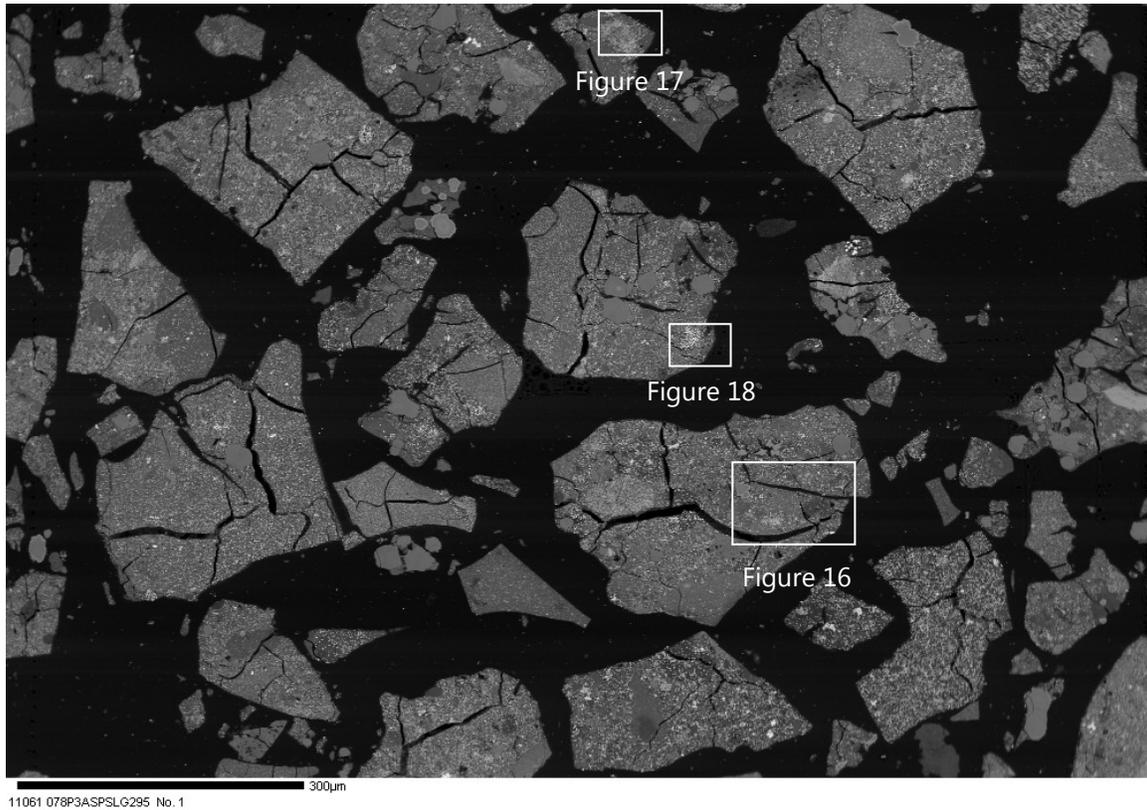
This particle has an unusually high number of pyrite inclusions in the core area. An enlarged view of the boxed area is shown in Figure 14, which illustrates the fine particle size and morphology of the pyrite.



**Figure 14. Sample 2, Example 7**

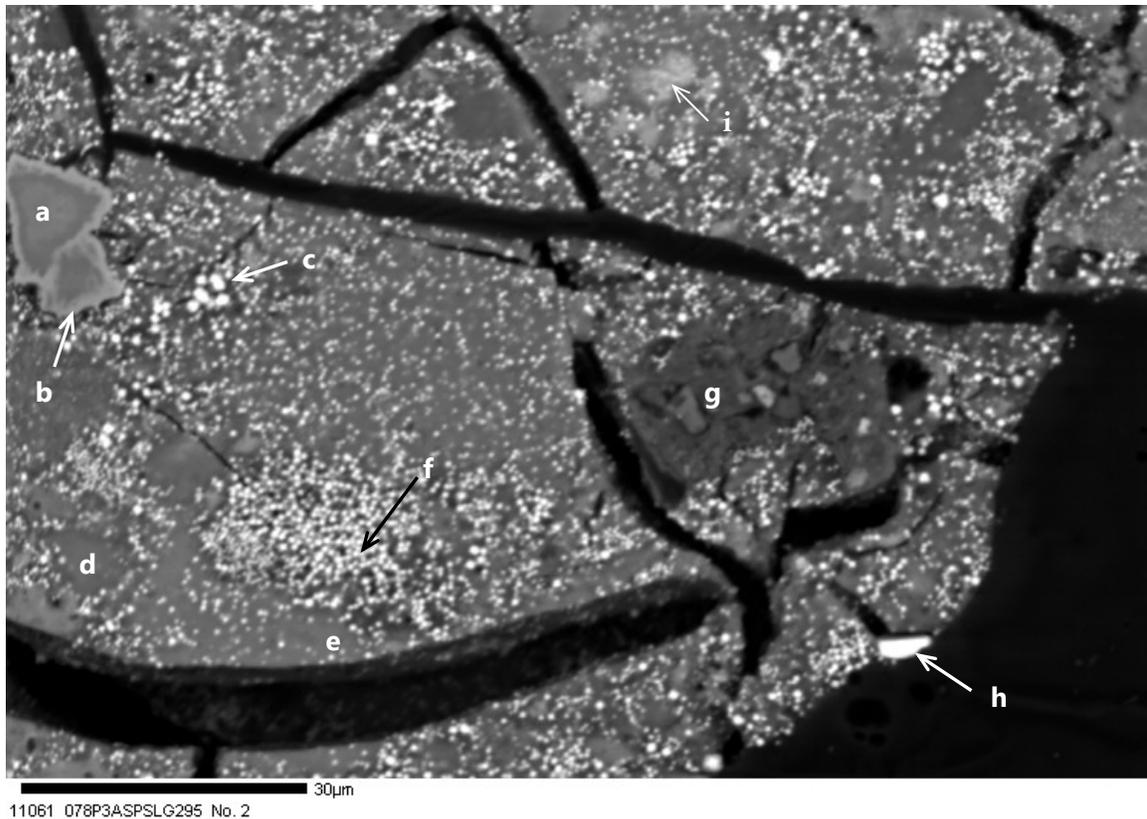
This figure shows a secondary electron image of the boxed area shown in Figure 13 and illustrates the uniform size and cubic morphology of the pyrite. The pyrite was apparently precipitated in the bioreactor rather than being transported as suspended particulate pyrite in the mine water.

Spot	Analysis, wt%					S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	
a	39		2.1	0.7	44	1.99
b	10.6	0.8	12.4	4.2	10.5	1.73
c	5.0	0.9	12.8	5.3	4.0	1.37
d	35	0.3	3.9	1.4	37	1.89
e	11.2	0.7	9.7	3.8	12.3	1.92



**Figure 15. Sample 3, Example 1**

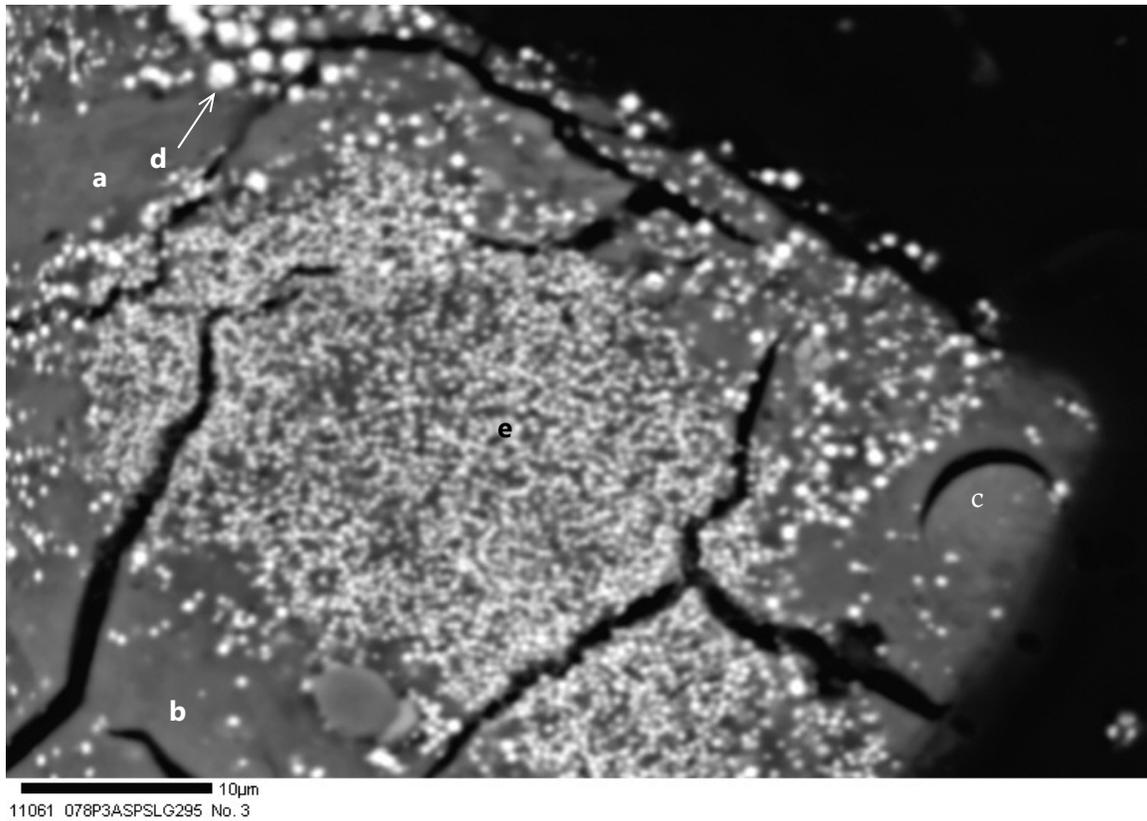
This figure shows typical particles in the sample. Nearly all of the agglomerations in this sample have shrinkage cracks, indicating that they may have been dehydrated in the biomass reactor process. This would cause them to be granular, resulting in fast filtration compared with the gelatinous nature of the other sample. Also, additional shrinkage cracks were not produced by the electron beam of the microprobe. This is consistent with dehydration and the lack of an amorphous peak in the XRD pattern of the moist filter cake for this sample. The boxed areas are shown enlarged in Figures 16, 17, and 18.



**Figure 16. Sample 3, Example 2**

This is a closeup of the lower boxed area shown in Figure 15. The small white inclusions are pyrite that are in a gray matrix bearing iron, aluminum, sulfur, and oxygen. There is no other sulfide identified in the XRD pattern or any other compound that accounts for the iron or the aluminum. Particle a is a manganoc calcite. Particle h is a manganese sulfide, MnS.

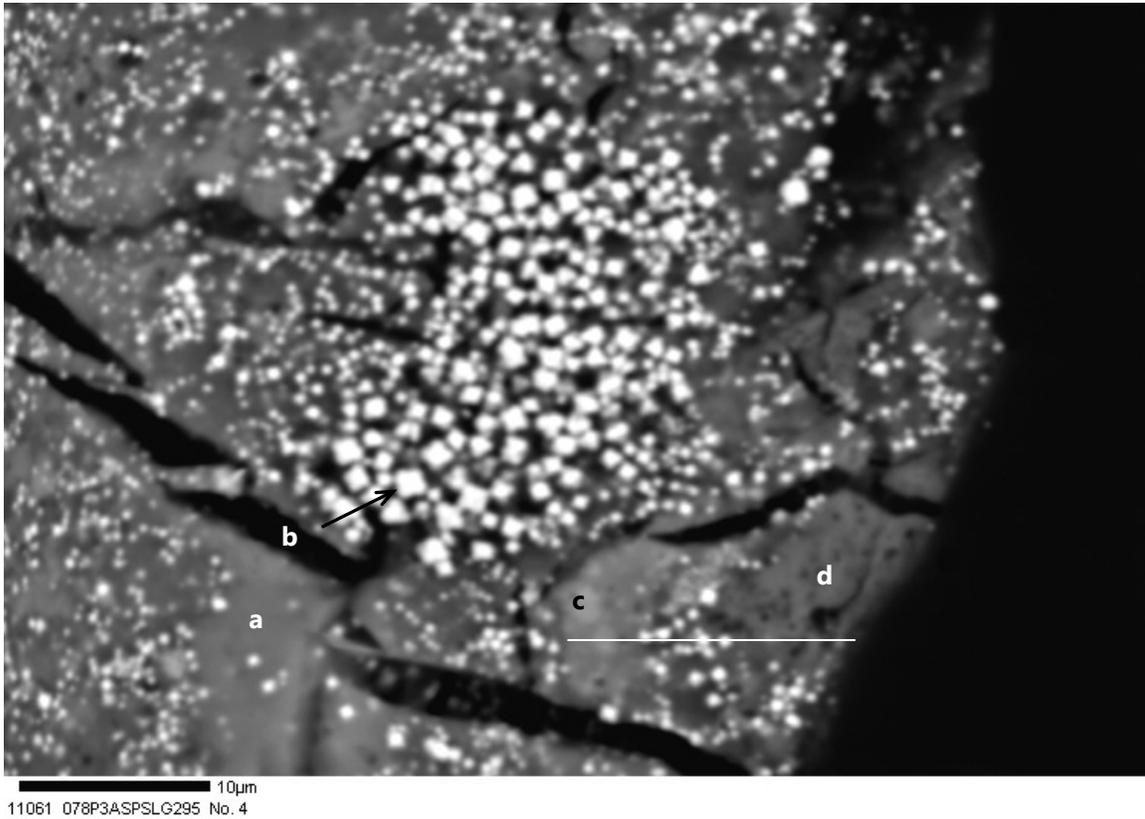
Spot	Analysis, wt%						S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	Mn	
a	0.7	29				11	
b	26	4.9	3.2	0.9	4.1	7.9	0.27
c	42		1.8	0.4	50		2.10
d	29	2.2	15	3.6	10.7		0.64
e	34	2.0	10	2.8	5.9		0.30
f	38		4.5	1.4	44		2.02
g	6.1	0.5	14	19	6.3		1.77
h	1.2				36	58	
i	44	0.9	7.5	1.1	19		0.76



**Figure 17. Sample 3, Example 3**

This is a closeup of the top boxed area shown in Figure 15. The white inclusions are pyrite. The matrix is similar to that of Figure 15 but has more aluminum. The sulfur/iron ratio in the matrix of this sample is generally lower than that of Sample 2, where it is about 1;1.

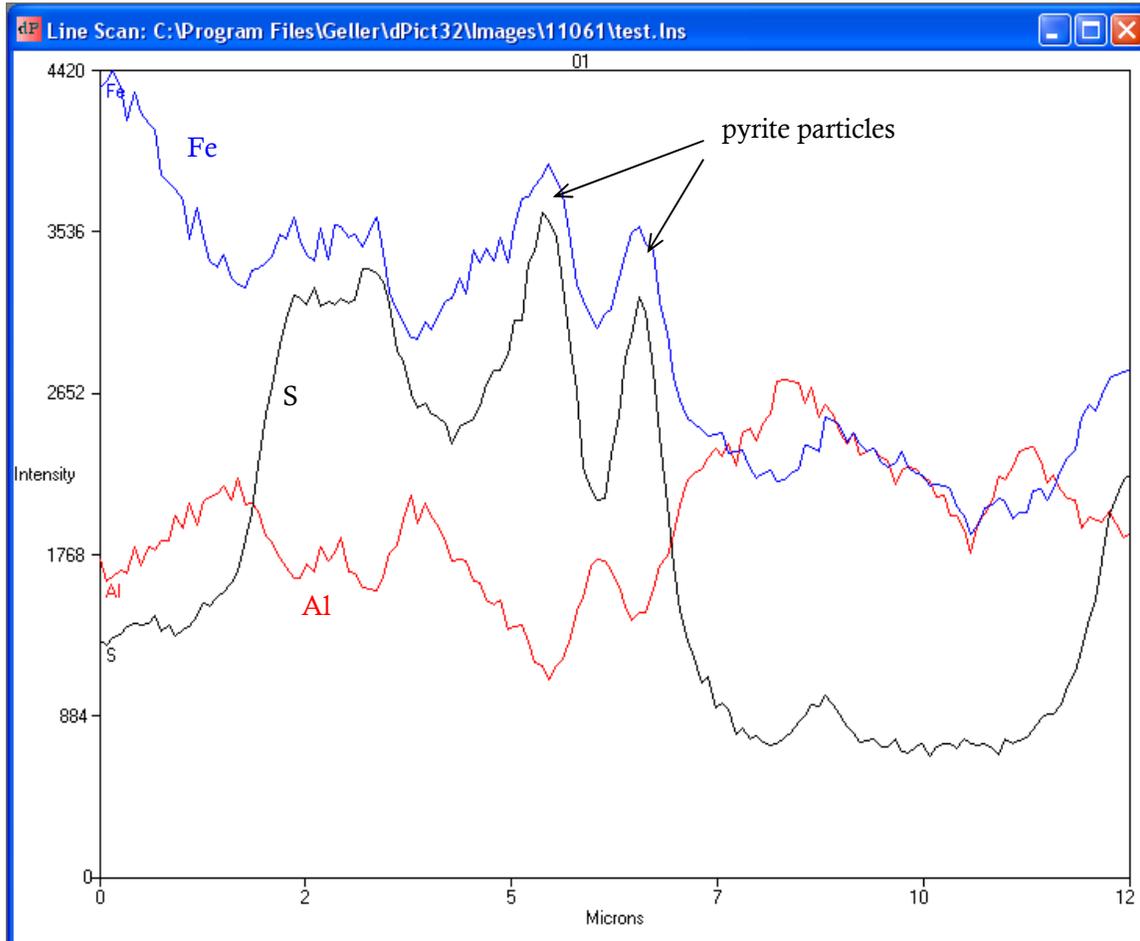
Spot	Analysis, wt%					S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	
a	24	2.2	18	3.8	5.4	0.39
b	26	2.2	19	4.2	4.3	0.29
c	23	2.8	19	4.4	6.2	0.47
d	41				47	1.99
e	32		8.5	3.0	38	2.06



**Figure 18. Sample 3, Example 4**

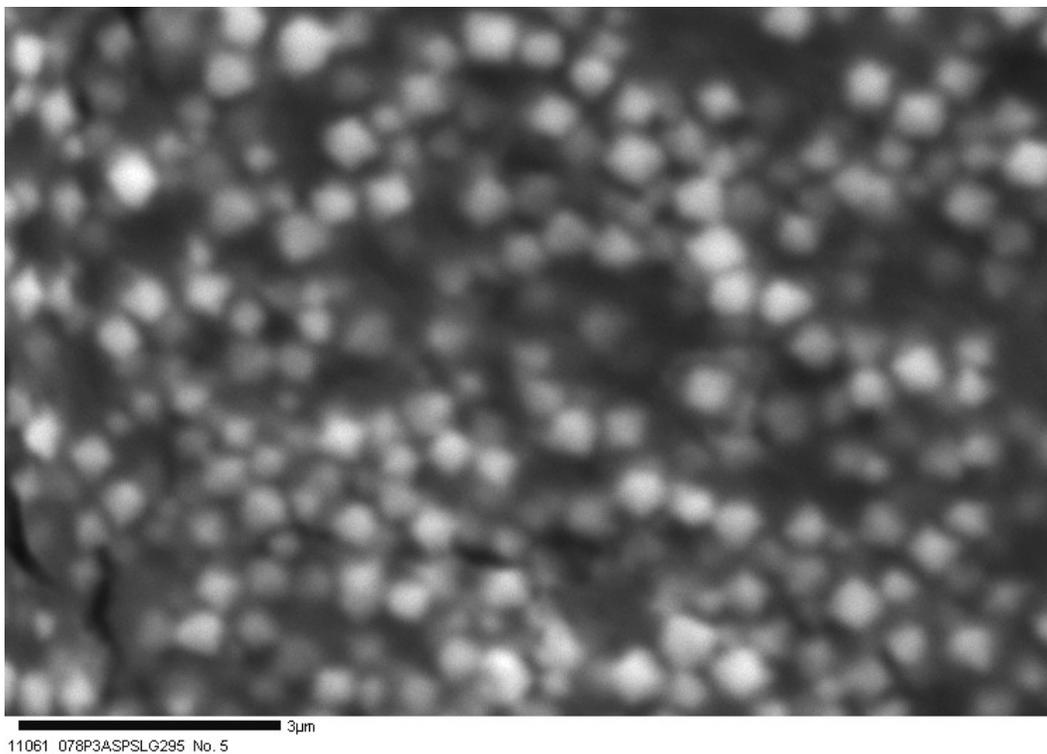
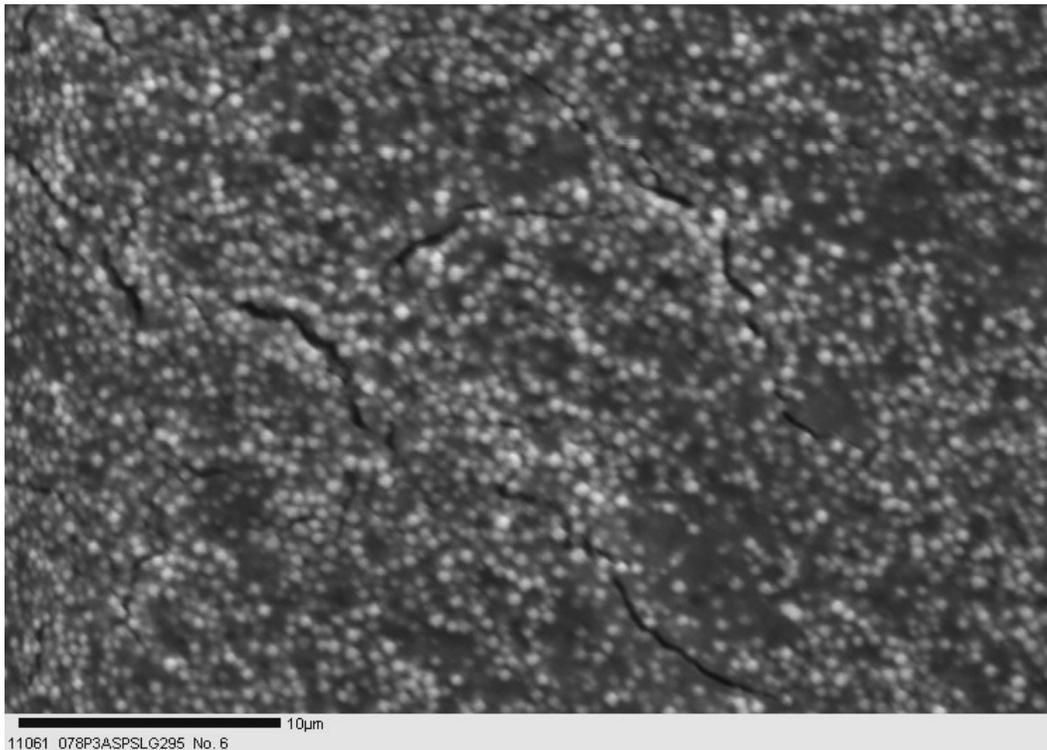
This is a closeup of the middle boxed area in Figure 15 showing a cluster of relatively large pyrite particles in a void. An x-ray line scan along the white line in the photo is shown in Figure 19.

Spot	Analysis, wt%					S/Fe Atomic Ratio
	Fe	Ca	Al	Si	S	
a	38	2.4	11	2.3	5.3	0.25
b	42		0.5	0.1	52	2.13
c	37	1.5	12	3.0	11	0.52
d	21	2.6	13	3.8	4.7	0.40



**Figure 19. Sample 3, X-ray Line Scan from Figure 18**

This figure shows an x-ray line scan along the white line shown in Figure 18. It illustrates the difference in the sulfur/iron ratios between the aluminum-bearing gray matrix material and the pyrite particles.



**Figure 20. Sample 3, Example 5**

This figure shows two secondary-electron images of gray matrix material illustrating the texture at different magnifications. It shows that even the smaller pyrite particles found in the gray, aluminum-rich matrix are cubic. They are very uniform in size at about 0.3–0.4 µm in diameter.

If you have any questions about this report, please contact me.

Sincerely,



Robert K. Johnson  
Metallurgist

RKJ/wcf

xc: Roland Schmidt, Hazen Research, Inc.

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**APPENDIX H**

2010 Quality Assurance/ Quality Control Assessment Reports