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Date: August 11, 2006
Refer to: EP2006-0442

Mr. James Bearzi
NMED-Hazardous Waste Bureau
2905 Rodeo Park Drive East, Building 1
Santa Fe, NM 87505-6303

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AUG 2006
NMED HWB
Waste Bureau

SUBJECT: UPDATE TO THE ANALYSIS OF SOLIDS RELATING TO TURBIDITY IN REGIONAL WELL R-34

Dear Mr. Bearzi:

Enclosed please find two hard copies of the report entitled "Update to the Analysis of Solids Relating to Turbidity in Regional Well R-34." This update includes analyses of the second sample of groundwater obtained from regional well R-34 in March of this year. The analytical results remain largely inconclusive as to whether the solids in the groundwater can be attributed to the native formation or to annular fill material.

Analytical results from the latest sampling round (January 2006) from R-34 indicate that groundwater sampled from the aquifer is representative of ambient conditions. Iron, manganese, sodium, and sulfate are within the range of background values; dissolved oxygen measured with a flow-through cell was 4 mg/L and turbidity was 10 NTUs, which is at the US Geological Survey's recommended threshold. R-34 was ranked "good" in the Well Screen Analysis Report (LA-UR-05-8615) and the analytical results indicate that water quality is not impacted by residual drilling fluids or well construction.

If you would like to discuss the results of this report or have questions, please contact Jean Dewart at (505) 665-0239 (dewart@lanl.gov) or Mat Johansen at (505) 665-5046 (mjohansen@doeal.gov).

Sincerely,

Andrew Phelps,
Associate Director,
Environmental Programs
Los Alamos National Laboratory

Sincerely,

David Gregory,
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Enclosure: Update to the Analysis of Solids Relating to Turbidity in Regional Well R-34

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LA-UR-06-3477
June 2006
ER2006-0419

Analyses of Solids Relating to Turbidity in Regional Well R-34

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Prepared by Environmental and Remediation Support Services

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LA-UR-06-3477
ER2006-0419

Analyses of Solids Relating to Turbidity in Regional Well R-34

June 2006

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Acronyms and Abbreviations

DOE	Department of Energy (U.S.)
EAPD	Environmental Programs Associate Director
EES	Earth and Environmental Science (a LANL division)
EP	Environmental Programs (a LANL directorate)
HWB	Hazardous Waste Bureau
LANL	Los Alamos National Laboratory
LASO	Los Alamos Site Office (DOE)
NMED	New Mexico Environment Department
NMED-OB	NMED's Oversight Bureau
NTU	nephelometric turbidity unit
SOP	standard operating procedure
WSP	Water Steward Project
XRD	x-ray diffraction

1.0 INTRODUCTION AND PURPOSE

This report is provided in response to a letter received on February 23, 2006, from the New Mexico Environment Department's Hazardous Waste Bureau (NMED-HWB). The purpose of this report is to address NMED's concerns that the measured turbidity in samples collected from regional well R-34 over the last few sampling rounds is attributable to the presence of annular-fill bentonite (a rock with abundant swelling smectite content) in the screened interval. Turbidity values measured at the time of sampling during the last three sampling events range from 8.37 to 17.4 nephelometric turbidity units (NTUs). Well R-34 was drilled using water, air, foam (QUIK-FOAM), and a polymer (EZ-MUD). The potential sources of smectite within the screened interval would be either from the bentonite chips used as annular-fill material or from the natural formation. Annular-fill bentonite would only be expected to be present in the screened interval if the annular-fill material were in contact with the screen. At R-34, the well casing is known to have inadvertently shifted 3.4 ft upward during the removal of the drill casing.

The screened interval of R-34 (883.7 ft to 906.6 ft) is believed to be situated in the pumiceous subunit that generally underlies the fanglomerates within the Puye Formation, but the exact definition of the surrounding lithology is uncertain because of a large interval (from 845 ft to 1000 ft) where no cuttings were returned.

The NMED-HWB letter requested that x-ray diffraction (XRD) analyses be performed at an independent, external laboratory on solids obtained from turbid groundwater collected during purging of R-34 before sampling. However, Los Alamos National Laboratory (LANL or the Laboratory) received concurrence from NMED staff to perform the analyses at the Laboratory's Earth and Environmental Science (EES) laboratory because no off-site laboratory was identified that could perform the analyses within the time frame requested. The XRD analyses were performed in accordance with the quality assurance-certified full-pattern software method developed for the Yucca Mountain Project and the following standard operating procedures (SOPs) provided in Attachment 1 (Standard Operating Procedures Pertaining to XRD Analyses): SOP-09.03, "Operation of Siemens D-500 X-Ray Diffractometers," SOP-09.04, "Calibration and Alignment of the Siemens Diffractometers," and SOP-09.05, "Clay Mineral and Zeolite Separation."

As requested by NMED's Oversight Bureau (NMED-OB), LANL has archived quantities of each of the collected samples if NMED should later choose to have them reanalyzed.

The NMED-HWB letter also requested the evaluation of other wells exhibiting elevated turbidity. However, no other intermediate or regional wells constructed using drilling fluids have exhibited elevated turbidity to date. Therefore, LANL has not evaluated other wells.

2.0 SCOPE

LANL developed a three-tiered approach to evaluate the source of the turbidity solids in groundwater at regional well R-34:

- Stage 1 - Determine the mineral composition and abundance within each of the following samples by quantitative XRD analysis:
    - Two groundwater samples collected at maximum turbidity during purging at R-34 on two separate dates
    - The cuttings returned from the 815-ft- to 845-ft-depth interval during the drilling of R-34

    - The bentonite chips used as annular fill at R-34

- Stage 2 - If differences in mineralogy determined in Stage 1 are not definitive, further differentiate the clay mineral components by analyzing all clay-containing samples using glycolation and NEWMOD structural analysis (calculation of layer components such as illite/smectite interstratification and kaolinite/smectite layering that can help to differentiate between different clays).
- Stage 3 - If mineral differences as determined in Stage 2 are still inconclusive, seek chemical and mineralogical distinctions between the clay-rich materials used in the well construction and native clay-bearing materials by instrumental neutron activation analysis.

3.0 STAGE 1 ACTIVITIES

3.1 Analyses

Initially, quantitative XRD analyses were to be performed on the solids obtained from two groundwater samples collected from R-34 on November 29, 2005, and on March 2, 2006; drill cuttings from the 815-ft- to 845-ft-depth interval at R-34; and bentonite chips used as annular fill at R-34. Because no cuttings were returned from the 845-ft- to 1000-ft-depth interval at R-34 (see Attachment 2, Excerpts from the R-34 Well Completion Report), no cuttings were available from the screened interval in question (883.7-ft to 906.6-ft depth). However, sparse cuttings were returned from shallower depths within the same pumiceous unit. These cuttings contained vitric, relatively unaltered pumice and very fine siltstone. For more complete coverage, LANL collected three samples from these shallower depths (815 ft to 820 ft, 820 ft to 825 ft, and 840 ft to 845 ft) for XRD analysis. In addition, at the request of NMED-OB, LANL collected a sample of the pumiceous subunit (at a depth interval of 1063 ft to 1068 ft) at nearby regional well R-13, where cuttings returned during drilling were more abundant. The R-13 samples provide a basis for determining whether the sparse cuttings from R-34 are adequately representative of the pumiceous host rock.

In addition, the following three different samples of the bentonite chips used in the annular space of R-34 were also analyzed: 3/8-in. chips used above the 402-ft depth, 1/2-in. chips used from 562 ft to 578 ft, and 1/2-in. chips used from 717 ft to 875 ft and deeper than 935 ft.

The following samples were separated by size to assist in determining the source of the turbidity:

- Particulate material from R-34 groundwater collected on November 29, 2005—the sample with the highest turbidity (164 NTUs)
- Particulate material from R-34 groundwater collected on March 2, 2006—the sample with the highest turbidity (302 NTUs)
- The 1/2-in. bentonite chips used in the annulus of R-34 (from the 717-ft to 875-ft or below-935-ft-depth intervals)
- Pumice from the 1063-ft- to 1068-ft-depth interval in R-13
- Pumice from the 840-ft- to 845-ft-depth interval in R-34 (separated into greater than, and less than, 1-µm fractions).

In summary, LANL analyzed a total of 11 samples using quantitative XRD:

- Two samples of solids concentrated from turbid R-34 groundwater

- Pumiceous samples from three different depth intervals in R-34
- One size-separated sample of R-34 pumiceous sediment from the 840-ft- to 845-ft-depth interval
- One R-13 pumice sample from the same subunit as that in the screened interval in R-34
- Three different samples of bentonite chips used in the annular space of R-34
- One sample of size-separated 1/2-in. bentonite chips representing annular-fill material used above and below the screen at R-34

3.2 Results

The results of the XRD analyses are summarized in Figures 3.2-1 through 3.2-7 and Table 3.2-1. Key features of these data are the presence of smectite, kaolinite, and clinoptilolite in all samples (turbid groundwater, native formation, and annular fill). Because the rock encountered at the screened interval in R-34 is a slightly altered volcanic ash/tuff, and because the bentonite chips used as annular fill are similarly formed from the alteration of volcanic ash layers, the bulk chemical composition and alteration processes for these materials are similar and result in a similar mineralogy (Table 3.2-1).

Several samples were fractionated by size to assist in determining the source of the turbidity, but no distinctive minerals were observed in the size fractions that would uniquely differentiate one sample from another. Hematite, a mineral not detected in the bentonite chip samples, was present in both the pumiceous host rock from R-34 and in the second groundwater sample (March 2006). Although its presence may be a specific indicator of a particulate contribution from the host rock, the abundances are too small (less than, or equal to, 1.3 wt%) for use as a quantitative tracer.

In general, the same minor minerals (such as kaolinite and clinoptilolite) that occur in both the native formation (pumiceous sediment; Figure 3.2-7) and the bentonite chips used in the annular-fill material (Figure 3.2-6) are observed in the solids collected from the turbid groundwater of R-34 (Figures 3.2-1 through 3.2-5; compilation in Table 3.2-1). Particulates in the two groundwater samples are almost identical in mineral composition and abundance (Table 3.2-1). However, the March 2006 groundwater sample has a coarser particle size distribution (predominantly greater than 5 μm rather than between 0.5 μm and 1.5 μm). Regression analysis between the two groundwater samples, comparing mineral abundances between the bulk particulates and between each of the five size fractions, yields regression coefficients to the second power (R^2) of greater than, or equal to, 0.99.

The bentonite chips in the annular fill have a strong tendency to retain significant amounts of quartz even in the fine-size separates. This indicates that very small authigenic quartz grains are intergrown with the clay. Because the particulates in the groundwater contain significant amounts of smectite and very little quartz, they may be derived from the native formation (pumiceous sediment) and not from the bentonite chips (Table 3.2-1).

Elevated clinoptilolite content in both the R-34 turbidity particulates and the bentonite chips used in the annular-fill material suggest that some solids may be derived from the bentonite chips and not from the native formation (pumiceous sediment) (Table 3.2-1).

The greatest mass of material in the bentonite chips from the annular-fill material is in the less-than-0.1-μm fraction. This is inconsistent with the size fractions obtained from the R-34 particulates and the native formation (pumiceous unit), where the greatest mass is in particles greater than 1 μm, and the less-than-0.1-μm fraction is minimal or essentially absent (Table 3.2-1). These differences in size population suggest that fine clay particles from the annular fill are not arriving at the screened interval.

Figures 3.2-1 through 3.2-3 present the raw data from which mineralogical abundances for the corresponding size fractions in Table 3.2-1 are derived. The figures illustrate the prominent peaks of minerals within the various R-34 turbidity size fractions from the November 29, 2005, and March 2, 2006, groundwater samples.

Using glycolation causes a fixed interlayer expansion; Figure 3.2-5 shows the results of glycolation and provides a basis for comparing mineralogy for the 0.25-µm to 0.1-µm size fractions (the purest clay separates) without the effects of relative hydration.

Figure 3.2-6 demonstrates that the mineralogy of two batches of 1/2-in. bentonite chips used in the fill material for two separate depth intervals at R-34 appear similar and have a significant quartz component. However, these same 1/2-in. bentonite chips differ from the 3/8-in. bentonite chips, which tend to be dominated by amorphous silica with disordered cristobalite and tridymite structural components (opal-CT), plus cristobalite (not quartz) as the dominant silica phases. Hence, the 3/8-in. and 1/2-in. bentonite chips are either from different sources or from different alteration zones of the same mined bentonite deposit.

Figure 3.2-7 presents the raw data from which the mineralogical abundances for native formation materials in Table 3.2-1 are derived. The large, broad humps in Figure 3.2-7 from approximately $16^{o}2\theta$ to approximately $35^{o}2\theta$ are the patterns produced by volcanic glass in pumice. The broad peak at approximately $6^{o}2\theta$ is evidence of smectite. The phase abundances from Figure 3.2-7 are reported in Table 3.2-1.

4.0 CONCLUSIONS

The Stage 1 analyses revealed that similar major, minor, and trace minerals were present in both the native formation and the bentonite chips, and that size separation did not clearly identify any specific and indicative mineral presence or absence in sufficient abundance to distinguish clearly between the native pumiceous formation and the bentonite chips. In addition, the analyses also showed the following:

- Turbid water samples collected approximately three months apart have almost identical mineral composition in bulk and size fractions. However, the later sample has a coarser size distribution and a higher clay/clinoptilolite ratio in the coarsest size fraction.
- Composition and mineralogy of the native formation and bentonite chips represent similar materials, both derived by alteration of silicic, volcanic tephra. However, the alteration of the bentonite sources used as annular fill is essentially complete, with no remnant volcanic glass, whereas alteration is only incipient and glass remains abundant in all size fractions of the native pumiceous formation at R-34 and R-13. Since glass is not observed in the R-34 turbidity sample, this is a possible indication that turbidity solids are derived from the annular fill.
- Paucity of fine-grained quartz associated with smectite in the R-34 turbidity sample (less than 5 wt% for both the November 2005 and the March 2006 samples) suggests that most turbidity particulates originate from the native formation, where quartz is predominantly coarse (several micrometers in size). This contrasts with the 1/2-in. bentonite chips used near the screened interval at R-34, in which fine-grained quartz occurs in amounts up to approximately 50 wt% in particles of a size range comparable with the most abundant turbidity materials pumped from R-34.
- Presence of clinoptilolite at concentrations greater than 1 wt% in both the R-34 turbidity sample and the bentonite chips suggests that the turbidity sample contains a component derived from the annular-fill bentonite.

- Dominance of particulates greater than 1 µm in both the R-34 turbidity sample and the native formation is consistent, suggesting that the particles are derived from the native formation rather than from the annular-fill bentonite, in which more than 40% of the particulate mass is less than 0.25 µm.

In Table 4.0-1, LANL synthesizes the above conclusions and the implications for evaluating whether the turbidity sample from R-34 represents the natural formation or annular-fill materials.

In summary, the quantitative XRD mineralogy analyses are not definitive for determining whether the turbidity sample is derived from the annular-fill bentonite or the host pumiceous formation.

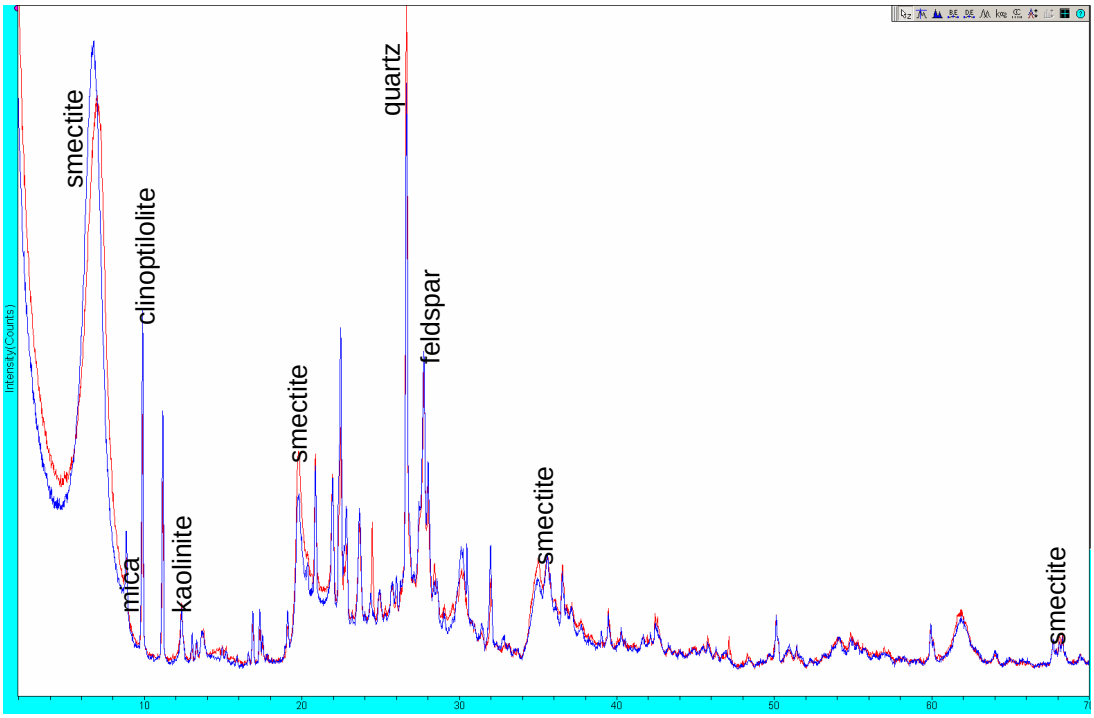


Figure 3.2-1. XRD patterns for the greater-than-5-µm size fraction for the November 29, 2005 (blue), sample and the March 2, 2006 (red), R-34 groundwater samples

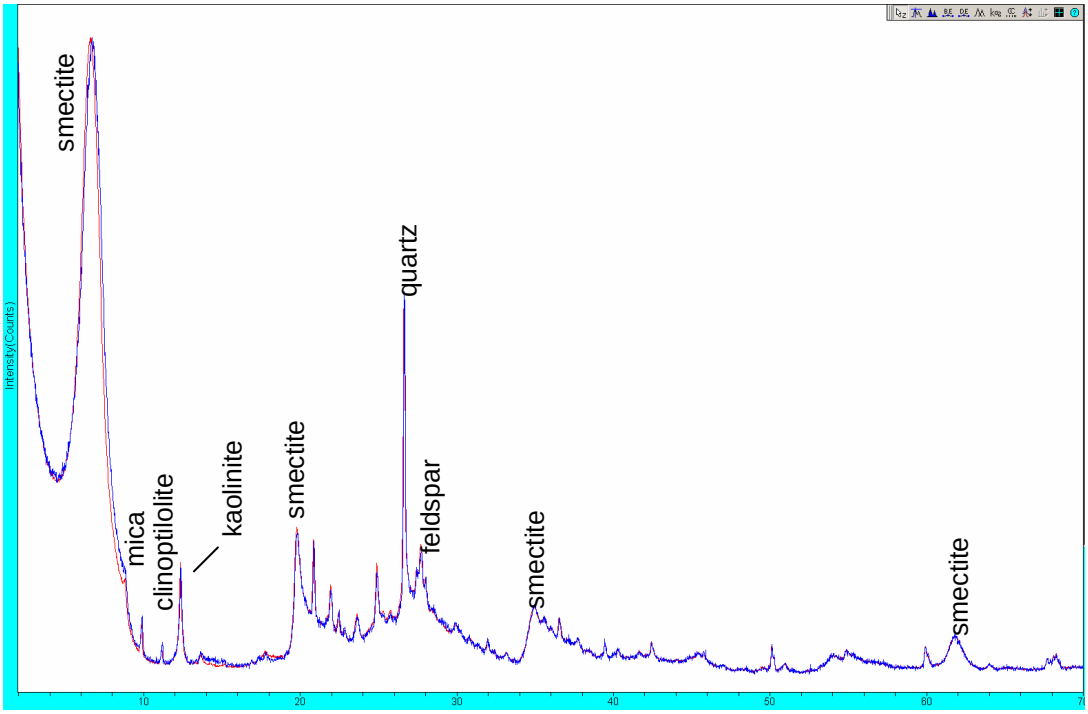


Figure 3.2-2. XRD patterns for the 5-µm to 1.5-µm size fractions of the November 29, 2005 (blue), sample and the March 2, 2006 (red), R-34 groundwater samples

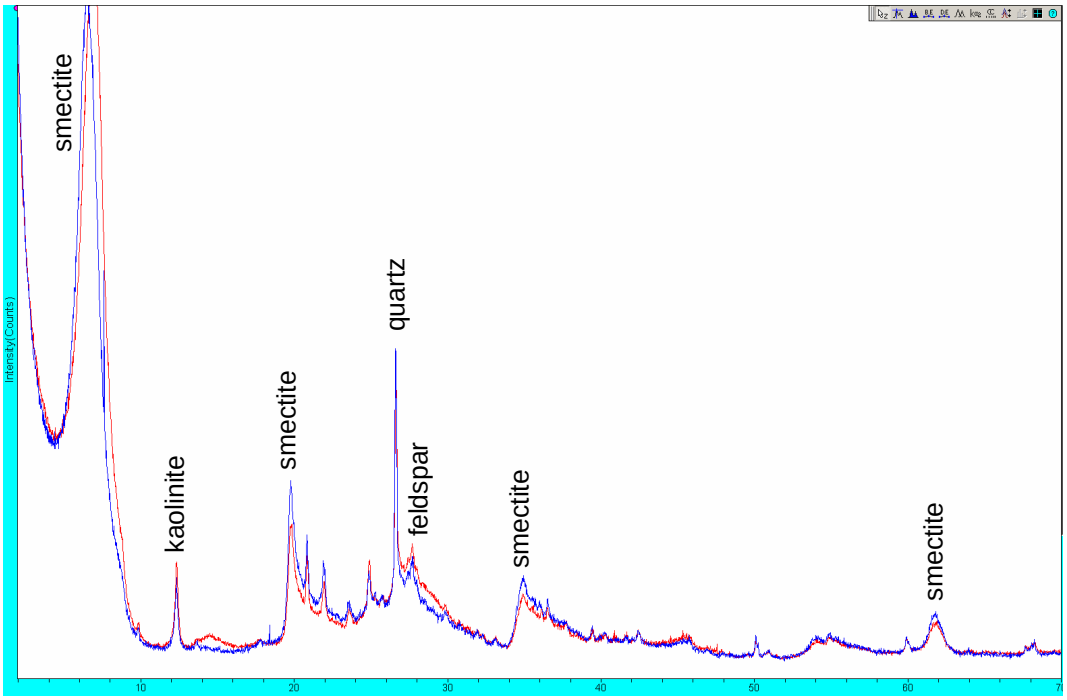


Figure 3.2-3. XRD patterns from the 1.5-µm to 0.25-µm size fractions of the November 29, 2005 (blue), sample and the March 2, 2006 (red), R-34 groundwater samples

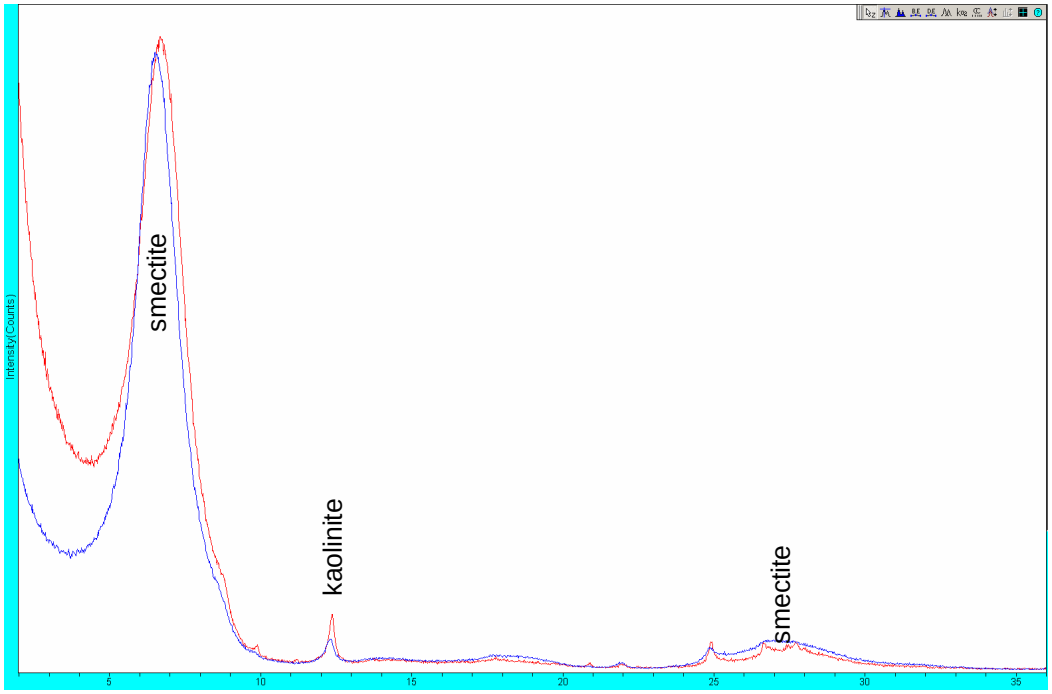


Figure 3.2-4. XRD patterns for the 0.25-µm to 0.1-µm size fractions of the November 29, 2005 (blue), sample and the March 2, 2006 (red), R-34 groundwater samples

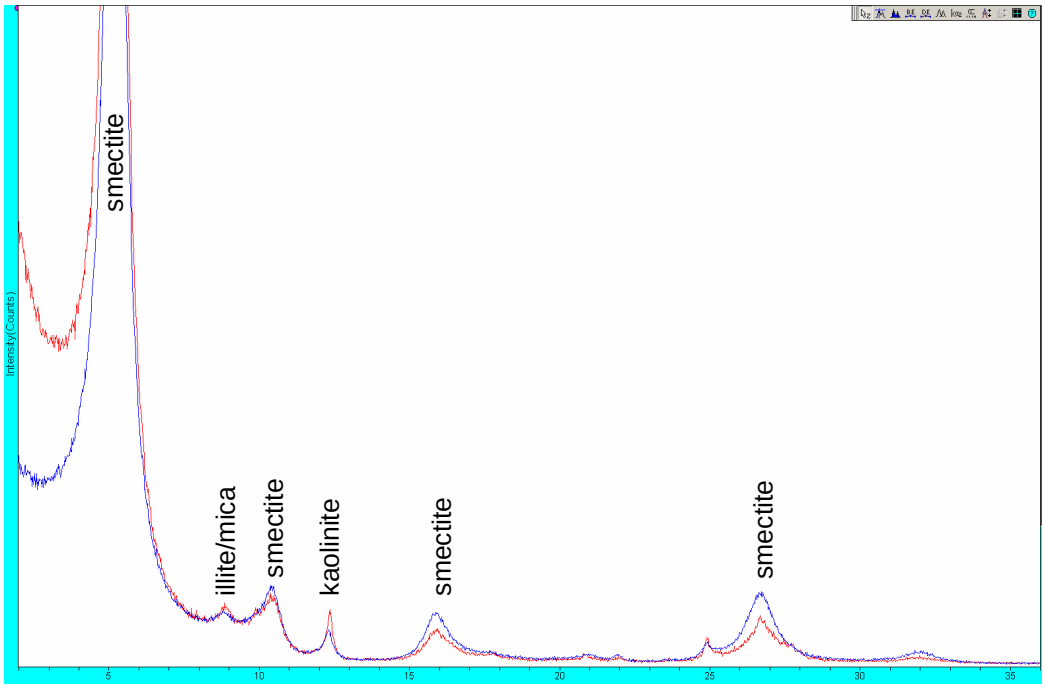
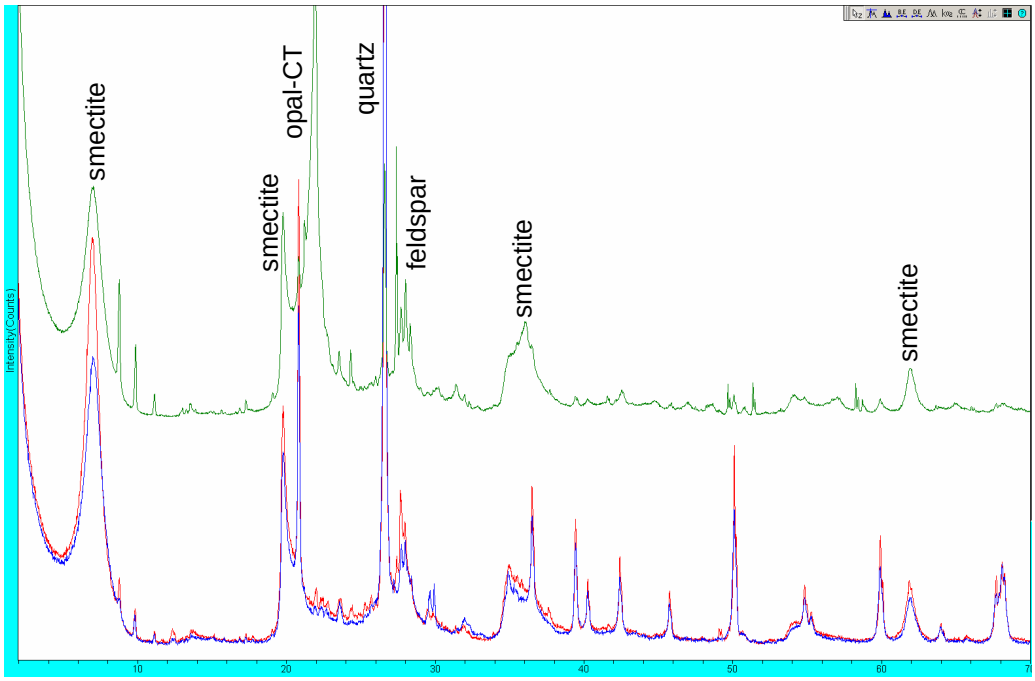
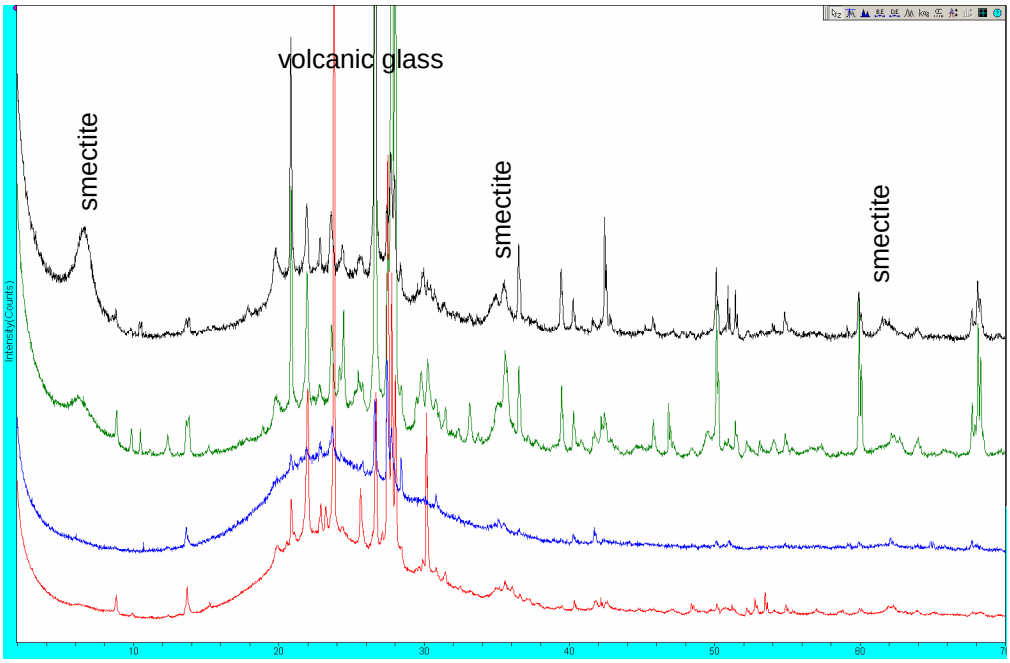


Figure 3.2-5. XRD patterns for glycolated-oriented mounts on off-axis quartz plates for the 0.25-µm to 0.1-µm size fractions of the November 29, 2005 (blue), sample and the March 2, 2006 (red), R-34 groundwater samples



Note: Red pattern represents the 1/2-in. chips from the 562-ft- to 578-ft-depth interval; blue pattern represents the 1/2-in. chips from the 717-ft to 875-ft and below-975-ft-depth intervals; green pattern represents the 3/8-in. chips from the above-402-ft-depth interval.

Figure 3.2-6. XRD patterns of bentonite chips from R-34



Note: Red pattern represents the R-34 pumice sample from the 815-ft- to 820-ft-depth interval; blue pattern represents the R-34 pumice sample from the 820-ft- to 825-ft-depth interval; green pattern represents the R-34 pumice sample from the 840-ft- to 845-ft-depth interval; black pattern represents the R-13 pumice sample from the 1063-ft- to 1068-ft-depth interval.

Figure 3.2-7. XRD patterns of bulk samples of various native pumiceous samples

Table 3.2-1
Results of Quantitative X-Ray Diffraction Analyses

Sample	Size/Range	Grams Sample	Smectite	Kaolinite	Clinoptilolite	Cristobalite	Quartz	Alkali-Feldspar	Plagioclase	Glass	Hornblende	Mica	Other
R-34 Solids from Turbid Groundwater (11/29/05, 10:53 AM)													
Bulk analysis[a]	n/a[b]	n/a	73.2	4.4	6.9	0.3	3.8	2.3	5.7	—	—	3.5	Hematite 0.2
1-hour settled fraction	>5 µm	1.103	59.7	2.1	15.8	0.5	4.3	4.6	9.6	—	—	3.4	Hematite 0.2
Overnight settled fraction	5–1.5 µm	2.220	77.7	5.4	3.7	0.1	3.8	1.3	4.3	—	—	3.8	Hematite 0.2
5000 rpm/5-minute fraction	1.5–0.25 µm	0.315	81.9	4.8	1.0	0.7	2.7	2.4	3.5	—	—	3.1	Hematite 0.2
8000 rpm/40-minute fraction	0.25–0.1 µm	[c]	~95%	~5%	—	—	—	—	—	—	—	Trace	—
Evaporated supernatant	<0.1 µm	0.039	~5%	—	—	—	—	—	—	—	—	—	Precipitates; see below[d]
R-34 Solids from Turbid Groundwater (3/2/06, 12:15 PM)													
Bulk analysis[a]	n/a[b]	n/a	70.9	3.3	7.7	0.4	4.3	3.0	6.8	—	—	3.8	Hematite 0.2
1-hour settled fraction	>5 µm	3.866	66.8	2.3	9.8	0.5	4.8	4.0	8.1	—	—	3.8	Hematite 0.2
Overnight settled fraction	5–1.5 µm	1.618	78.8	5.4	3.7	0.2	3.4	0.7	4.1	—	—	3.6	Hematite 0.2
5000 rpm/5-minute fraction	1.5–0.25 µm	0.286	80.7	5.0	1.0	—	2.7	2.8	3.6	—	—	4.0	Hematite 0.2
8000 rpm/40-minute fraction	0.25–0.1 µm	[c]	~92%	~6%	~1%	—	—	—	—	—	—	~1%	—
Evaporated supernatant	<0.1 µm	0.125	~2%	—	—	—	—	—	—	—	—	—	Precipitates; see below[d]
R-34 Pumice Samples													
815-ft- to 820-ft-depth interval	n/a	0.155	1.6	0.2	0.4	1.5	1.8	6.1	5.6	80.7	—	2.0	Hematite 0.1
820-ft- to 825-ft-depth interval	n/a	0.328	1.3	—	—	—	1.0	3.8	2.6	91.2	—	—	—
840-ft- to 845-ft-depth interval	n/a	0.844	15.2	0.9	1.2	0.1	12.7	7.4	23.4	36.2	0.1	2.1	Hematite 0.8
Size Separation of R-34 Pumice Sample from 840-ft- to 845-ft-Depth Interval[e]													
Greater than 1 µm	>1 µm	0.708	6.2	0.5	0.5	0.4	18.9	5.1	20.5	44.4	0.1	1.9	Hematite 1.3
Less than 1 µm	<1 µm	0.045	58.6	2.3	—	0.2	1.0	2.0	1.9	32.4	—	0.7	Hematite 0.2, calcite 0.9

Table 3.2-1 (continued)

Sample	Size/Range	Grams Sample	Smectite	Kaolinite	Clinoptilolite	Cristobalite	Quartz	Alkali-Feldspar	Plagioclase	Glass	Hornblende	Mica	Other
R-13 Pumice Sample from the 1063-ft- to 1068-ft-Depth Interval[e]													
Bulk pumice sample	n/a	—	23.1	0.1	0.7	—	9.3	3.8	9.8	52.3	0.1	0.7	—
5-minute settled fraction	>18 µm	1.002	13.8	0.1	0.1	1.0	14.9	2.9	8.0	58.7	—	0.6	—
Overnight settled fraction	18–1.5 µm	0.156	33.1	0.5	0.2	0.1	4.4	3.3	6.0	50.9	0.1	1.3	—
8000 rpm/40-minute fraction	1.5–0.1 µm	0.033	76.0	1.7	0.6	—	1.7	2.0	3.6	12.1	—	2.3	—
Bentonite Chips Used in the R-34 Well Construction													
3/8-in. chips above 402 ft	n/a	~350	40.9	—	4.5	10.6	2.7	2.2	5.5	—	—	7.7	Opal-CT 25.8
1/2-in. chips from 562 ft to 578 ft	n/a	~350	60.8	0.7	1.4	—	21.9	3.1	6.6	—	—	5.0	Opal-CT 0.3
1/2-in. chips from 717 ft to 875 ft or below 935 ft	n/a	~350	60.2	0.6	2.2	—	23.9	3.3	6.0	—	—	3.7	—
Size Separation of 1/2-in. Bentonite Chips from the 717-ft to 875-ft or below-935-ft-Depth Intervals													
5-minute settled fraction	>18 µm	1.543	18.0	0.6	5.6	—	52.3	7.3	13.4	—	—	2.8	—
5-hour settled fraction	18–2 µm	1.524	16.5	2.7	6.1	0.3	53.9	7.4	7.3	—	—	5.8	—
Overnight settled fraction	2–1.5 µm	0.386	32.1	2.4	3.0	0.4	45.6	5.7	4.6	—	—	6.3	—
5000 rpm/5-minute fraction	2.5–0.25 µm	0.532	63.4	1.0	3.1	—	23.7	2.8	5.1	—	—	0.8	—
8000 rpm/40-minute fraction	0.25–0.1 µm	0.503	97.8	0.4	—	—	2.2	—	—	—	—	—	—
Evaporated supernatant	<0.1 µm	2.390	100	—	—	—	—	—	—	—	—	—	—

[a] Bulk analysis for this sample was reconstructed by normalizing the abundances from the individual size fractions and weights obtained.

[b] n/a = Not applicable.

[c] Tiny amount of sample, left in suspension to make oriented clay mounts for possible Stage 2 analyses.

[d] Evaporated <0.1-µm fraction precipitate from the 11/29/05 groundwater sample: Amorphous (likely Opal-A) 66 wt%, Calcite 25 wt%, Halite 4 wt%. Evaporated <0.1-µm fraction precipitate from the 3/2/06 groundwater sample: Amorphous (likely Opal-A) 76 wt%, Calcite 20 wt%, Halite 2 wt%. The precipitates in these samples represent dissolved or extremely fine components in the R-34 water.

[e] The R-34 depth interval of 840 ft to 845 ft and the R-13 depth interval of 1063 ft to 1068 ft were chosen for quantitative XRD analyses because they were the most clay-rich and had the most material available for sampling.

Table 4.0-1
Properties of the R-34 Turbidity Samples

Similarities Specific to the Natural Formation	Similarities Specific to the Annular Bentonite Fill
Paucity of fine-grained quartz	Lack of volcanic glass particulates
Very fine particulates (less than 0.25 μm) rare	Clinoptilolite present at greater than 1 wt%

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Attachment 1

Standard Operating Procedures Pertaining to XRD Analyses

Identifier:	Revision:	Effective Date:	Review Date:
SOP-09.03	**1**	**04/11/01**	**12/16/2003**

ER Document Catalog Number: **ER2001-0126**

Author: David Vaniman

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Operation of Siemens D-500 X-ray Diffractometers

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

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Revision Log

Revision No.	***Effective Date***	***Prepared By***	***Description of Changes***	***Affected Pages***
R0	03/16/92	David Bish	New procedure	all
R1	04/11/01	David Vaniman	Revised to update records management and to fit new format for SOPs.	all
Review	12/16/2003	Mark Thacker	Deemed adequate.	All

Operation of Siemens D-500 X-ray Diffractometers

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Operation of Siemens D-500 X-ray Diffractometers

1.0 PURPOSE

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation used to obtain X-ray powder diffraction data from the Siemens D500 X-ray powder diffractometers at the Los Alamos National Laboratory (Laboratory) ER Project.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when collecting X-ray diffraction data for the ER Project using the LANL EES Siemens D-500 diffractometers.

3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure, and the training is documented in accordance with QP-2.2.

3.2 The **Geology Task Leader** will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

4.1 *Machine Custodian* — The person who is in charge of an instrument and is responsible for the maintenance and safety of the instrument. This person's name shall be posted on the front of the diffractometers.

4.2 *Group Environment, Safety, and Health Plan* — A health and safety plan that is specific to the research Group activities and has been approved by the appropriate Division-level health and safety representative.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved Group environment, safety, and health plan.

5.1 Powder X-ray diffraction is a method by which investigators can identify the minerals present in a rock and obtain quantitative information on their abundance's and physical properties.

5.2 Malfunctions of the equipment are readily apparent. For the diffractometers, one either obtains counts or no counts.

5.3 The diffractometers produce ionizing radiation using high voltage sources. However, the diffractometers are interlocked such that if the panels are all in place, risk to the operator is almost non-existent.

5.4 Only two possibilities (rare) for data corruption exist: 1) loss of X-ray flux; 2) occurrence of noise spikes. The first problem can be identified by the lack of any diffracted intensity, i.e., counts = 0. Such data should be discarded. The occurrence of the second possibility can be noted by “peaks” only one 2T step wide. Such data are generally usable and only need to be discarded when a noise spike overlaps on an important peak.

5.5 The person requesting XRD analyses will record of sample submittals and analysis results in their research notebook.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Team Leader

6.3 Quality Program Project Leader

6.4 Geology Task Leader

6.5 ER Project personnel

7.0 EQUIPMENT

7.1 Siemens D500 X-Ray Powder Diffractometers

7.2 Materials Data Inc. Jade X-Ray Data Package

7.3 Materials Data Inc. DataScan Instrument Control Package

8.0 PROCEDURE

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

Note: The machine custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers. The geology task leader (TL) has the responsibility to assure correct implementation of this procedure for ER work. The TL may delegate performance of the procedure to any properly trained and certified individual.

8.1 Calibration

For Alignment and calibration, see SOP-9.04, Calibration and Alignment of the Siemens Diffractometers.

8.2 Control of Samples

Samples will be tracked, stored, shipped, and handled in accordance with SOP-9.01. Care shall be exercised to label all X-ray runs with correct sample name, checking sample output against names on the sample bottles.

8.3 Diffractometer Operation

6.3.1 Turn on diffractometer as outlined in the Siemens D500/501 Operating Instructions.

6.3.2 Insert sample and turn on X-rays either by pressing the shutter-open button or by placing shutter in automatic mode.

6.3.3 The instrument is operated and data analyses conducted using the MDI software packages.

8.4 System Shut-Down

The diffractometer is shut down in accordance with the instruction manual or the instructions listed on the front of the diffractometer.

8.5 Data Analysis

6.5.1 The data are regressed and displayed using the MDI software package.

6.5.2 Crystalline phases are identified by comparing their patterns with patterns of pure standards, patterns from the ICDD files, or with calculated patterns.

6.5.3 Quantitative X-ray diffraction analysis is conducted in accordance with the methods of FULLPAT (ref. Chipera and Bish, 2001).

8.6 Procedural Deviations

Deviations from this procedure and the effects it may have on the resulting work shall be documented as described in QP-4.2.

8.7 Documentation

6.7.1 All raw X-ray data stored on magnetic or optical media shall periodically be backed up onto magnetic tape and stored in a fireproof safe.

6.7.2 Records that are readily regenerated from the raw data such as hard copy plots and peak search data sheets may be placed in labeled three-ring binders.

8.8 Lessons Learned

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure.

Chipera S. J. and D. L. Bish, “FULLPAT: A Full Pattern Quantitative Analysis Program for X-ray Powder Diffraction.” User Manual, LANL XRD laboratory, 2001.

MDI (Materials Data Incorporated), DataScan 3.2, An Automated Control and Data Acquisition System for X-ray Diffractometers, 1995-1999.

MDI (Materials Data Incorporated), Jade 5, XRD Pattern Processing, 1991-1999.

Siemens D500/501 Operating Instructions, C72000-B3463-A42, Siemens Corporation, Cherry Hill, New Jersey.

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

SOP-9.10, Sample Control and Field Documentation

SOP-9.04, Calibration and Alignment of the Siemens Diffractometers

10.0 RECORDS

The Geology Task Leader (TL) is responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

Notebook records of the sample handling and results of analysis relevant to production of X-ray diffraction data.

Data submittals for the ER electronic database.

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

Identifier:	Revision:	Effective Date:	Review Date:
SOP-09.04	**1**	**04/11/01**	**12/16/2003**

ER Document Catalog Number: **ER2001-0124**

Author: David Vaniman

environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Calibration and Alignment of the Siemens Diffractometers

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36.

Revision Log

Revision No.	***Effective Date***	***Prepared By***	***Description of Changes***	***Affected Pages***
R0	03/16/92	David Bish	New procedure	all
R1	04/11/01	David Vaniman	Updated to reflect new format	all
Review	12/16/2003	Mark Thacker	Deemed process adequate.	All

CALIBRATION AND ALIGNMENT OF THE SIEMENS DIFFRACTOMETERS

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CALIBRATION AND ALIGNMENT OF THE SIEMENS DIFFRACTOMETERS

1.0 PURPOSE

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation required to align and calibrate the Siemens D500 X-ray powder diffractometers at the Los Alamos National Laboratory (Laboratory) ER Project.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when aligning and calibrating the Siemens D500 X-ray powder diffractometers for the ER Project

3.0 TRAINING

3.1 All users of this SOP are trained by self-study, and the training is documented in accordance with QP-2.2.

3.2 The Geology Task Leader (TL) will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

3.3 Due to safety considerations, the machine custodian is solely responsible for performing machine alignment. The ER user and the machine custodian performing calibration have the responsibility to adhere to this procedure.

4.0 DEFINITIONS

4.1 *Machine Custodian* — The person who is in charge of an analytical X-ray instrument and who is responsible for the maintenance and safety of the instrument.

4.2 *Shorting Plug or Key* — The plug (θ-2θ machine) or key (θ-θ machine) used to bypass the safety circuits to allow alignment. To be used only by the machine custodian.

4.3 *NIST* — National Institute of Science and Technology.

4.4 *Site-Specific Health and Safety Plan (SSHASP)*—A health and safety plan that is specific to a site or ER-related field activity that has been approved by an ER health and safety representative. This document contains information specific to the project including scope of work, relevant history, descriptions

of hazards by activity associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment [PPE]) and hazard mitigation.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved Group environment, safety, and health plan.

Calibration and, if necessary, alignment of the Siemens X-ray diffraction equipment must be performed on a periodic basis in order to assure that all data collected on the instruments yield accurate d-spacings. The calibration described here involves only d-spacings. Calibration may use any well-characterized d-spacing standard that is traceable to NIST, such as their standard reference materials. It is not necessary that the diffractometer have no error in 2θ. Calibration is performed to determine the magnitude and direction of any error in 2θ so that appropriate calibration curves may be used when obtaining d-spacings. Alignment is performed only when calibration data reveal a significant error in alignment, usually after modification of the instrument (e.g., installation of new X-ray tube, installation of alternate sample holders, installation of the TTK).

5.1 Certain aspects of the alignment procedure require that one bypass the interlocks to work on the instrument with the X-ray beam on. The use of extreme caution and safety equipment such as lead-lined gloves is mandatory.

5.2 Highly accurate d-spacing measurements are not crucial to qualitative phase identification or quantitative phase analysis. However, it is useful and good laboratory practice to have a well- aligned diffractometer. In general, errors less than 0.02° 2θ are desirable, but errors up to 0.10° 2θ are acceptable in many cases.

5.3 There are few sources for error that would not be readily detected. A potential situation where minor error could occur is when the linear absorption coefficient or transparency of a sample being run is not similar to that of the standard used to calibrate the instrument. In this case, if accurate d-spacings were desired, the instrument should be calibrated with a standard of appropriate linear absorption coefficient or an analytical correction should be performed to correct for transparency effects.

5.4 The calibration sticker paced on the instrument shall constitute evidence that the procedure has been implemented and satisfactorily accomplished.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Team Leader

6.3 Quality Program Project Leader

6.4 Geology Task Leader

6.5 ER Project personnel

7.0 EQUIPMENT

7.1 NIST SRM 640a or 640b Si Powder d-spacing Standard.

7.2 Granite surface plate or precision flat plate.

7.3 Height Gauge.

7.4 Dial Indicator Gauge.

7.5 Precision Right-Angle Block.

7.6 Assorted Slits.

7.7 Strip Chart Recorder.

7.8 Assorted Screw Drivers and Wrenches.

7.9 Shorting Plug or Key.

7.10 MDI software packages (MDI DataScan 3.2 and MDI Jade 5).

7.11 LCLSQ, lattice parameter refinement program.

8.0 PROCEDURE

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

Note: Only NIST traceable standards such as NIST SRM 640b shall be used to calibrate or check the calibration of the Siemens X-ray powder

diffractometers. Deviations from this procedure shall be documented as required in QP-4.2.

8.1 Calibration of Diffractometers

As required, either by time elapsed since last annual calibration check or by a change in the instrument or sample holder geometry, the instrument shall be calibrated against a certified standard reference material (SRM), such as NIST SRM 640a or 640b (Si powder), or against a secondary reference standard traceable to an NIST standard.

8.1.1 The calibration standard may be mounted either in a cavity or as a thin film to minimize specimen transparency effects.

8.1.2 The calibration standard shall be examined on the instrument over a 2θ range practical for the instrument and standard. A recommended procedure is to examine NIST SRM 640a or 640b from ~18° 2θ to~150° 2θ using 0.01-0.02°-2θ steps and counting for at least 1.0 sec per step.

8.1.3 Determine peak positions for all diffraction maxima using the profile fitting routine in the Jade software package, typically using the Pearson VII profile. The results of the profile refinement may be compared with the certified values for the standard to assess the magnitude of the 2θ error.

8.2 Alignment of the Diffractometer

If a calibration check reveals significant errors in alignment (2θ errors generally greater than 0.02° 2θ), an alignment of the diffractometer should be performed. If only some aspects of the instrument have been modified, only an abbreviated alignment will be required. For example, if a new X-ray tube has been installed, only the tube height will require alignment. When the TTK stage is installed, both sample height (6.2.3) and θ zero point (6.2.5) must be checked and adjusted if necessary.

6.2.1 Specimen holder pin height. This step is performed only if the specimen stop-screws have been modified. Thus, in practice it is seldom if ever performed. 6.2.1 is not applicable to the TTK stage.

8.2.1.1 Check the three specimen stop-screws on the specimen holder for the same height. The two outer pins should be coplanar with the inner reference pin. Tools Needed are as follows:

- Granite surface plate or precision flat plate;
- Height gauge;
- Dial indicator gauge; and,

- Precision right-angle block.

8.2.1.2 Set the level of the specimen holder using the bar by the inner stop-screw.

8.2.1.3 Set the dial indicator height using the bar or inner screw.

8.2.1.4 Before adjusting stop-screws, the locking screws should be loosened. There is one for each stop-screw.

8.2.1.5 When the height of the three stop-screws is set, retighten the locking screws.

8.2.2 Install the specimen holder on the specimen carrier using the push rod that is built into the carrier. Use the flat surface of the rod and try to make the holder parallel to the rod. This step is performed only if the specimen holder has been removed from the specimen carrier.

8.2.3 (With shorting plug or key in). The axis of rotation of the goniometer is on the specimen surface when the maximum intensities appear at θ = 0° (front side) and 180° (back side). Note that the maximum intensities are approximately the same. This step is done only with the θ-2θ diffractometer and only if the specimen height has been modified. In practice, this is seldom if ever performed.

8.2.3.1 Slit positions: I II III IV Slit: 1°1° --- .018° (These are final slits - can start with wider slits, particularly #IV.)

8.2.3.2 Install glass alignment slit.

8.2.3.3 Insert an absorber plate into the direct beam to limit counts to below ~40,000 counts/sec. Set tube power to minimum (~7kV and ~3mA).

8.2.3.4 Find the maximum intensities by adjusting θ and 2θ independently and alternately on front (θ = θ°). Record 2θ and full scale counts.

8.2.3.5 Find the maximum intensities as above on back side (θ at 180°). This maximum intensity should be about the same value as the front side (θ = θ°). If intensity is different, move holder up and down - if you can't get it, the three pins are not in the same plane and 6.2.1 should be performed.

8.2.3.6 It is not important at this time that the θ and 2θ counting mechanisms read zero, but note only the 2θ values.

8.2.3.7 Ignore the θ value alltogether. The 2θ values only need to be to be the same, for θ front and back.

8.2.3.8 Compare the 2θ numbers found on front and back sides. The difference should be no greater than 0.02°. Do a θ-2θ scan on both front and back to insure that you are aligning on the main peak.

8.2.3.9 If the difference is greater than 0.02°, the specimen holder has to be moved closer or farther away from the push rod. If the front 2θ value is larger than the back 2θ value, the holder needs to moved towards the pin, i.e., down.

8.2.3.10 If the intensities (front and back sides) are not within ~10% of each other, then step 6.2.3.8 should be rechecked.

8.2.4 Adjust tube height for maximum intensities when 2θ is reading zero. Use only the 0.018 slit in position IV and the glass slit in the sample position. Use no other slits.

8.2.4.1 Loosen the nut at top of tube slightly with small wrench in the D500 accessory kit. Loosen the allen screw at the lower back of the tube.

8.2.4.2 Move θ to front side.

8.2.4.3 Adjust 2θ off of maximum intensities, slightly toward zero.

8.2.4.4 Adjust tube height to get back toward maximum intensity.

8.2.4.5 Adjust θ so maximum intensity is restored.

8.2.4.6 Repeat above steps until you get zero 2θ with maximum intensity.

8.2.4.7 Tighten the nut and screw loosened in 6.2.4.1 above.

8.2.4.8 You should not have to set the height again unless a new tube is installed.

8.2.4.9 If the Kevex Psi detector is installed, steps 6.2.4.1 through 6.2.4.8 cannot be performed. Instead, tube-height alignment is performed using a high-angle reflection of a NIST standard such as Si. The tube height is incrementally adjusted until the high-angle reflection is located at the correct 2θ value obtained using profile refinement. Turning the allen screw on top of the tube clockwise (from above) lowers the observed peak positions.

8.2.5 With the glass slit in the sample position, adjust θ for zero with maximum intensity by means of the eccentric screw (Item 183 in Fig. 20 of reference B). As in previous 2θ adjustment, move toward zero slowly. Peak the eccentric in small steps until you get to zero. Recheck after adjusting slits (slits 0.1, 3, --, 0.018 in slit positions I, II,

III, and IV, respectively). This alignment cannot be performed with the Kevex Psi detector installed, and the scintillation detector must be installed. This step needs to be performed only if the specimen carrier has been removed.

8.2.6 Adjust slit holder for slit positions I and II. Put in the smallest slits. If there is no intensity change, the slits are properly aligned. Do the same for slit positions III and IV.

8.2.6.1 Insert the following slits into the slit holder: Slit position: I II III IV Slit: 0.1°3°--- 0.018°.

8.2.6.2 Remove the sheet metal shield of the slit holder and, if necessary, the Soller slits.

8.2.6.3 Loosen the locking screws.

8.2.6.4 Reposition the sheet metal shield (radiation protection).

8.2.6.5 Displace the slit holder by means of the two adjusting screws until the ratemeter or chart recorder indicates maximum intensity. In order to turn one adjusting screw in, it is necessary to loosen the opposite screw because these are pressure screws.

8.2.6.6 Insert smallest (0.1°) slit in position II, and you should get the same intensity or you must readjust.

8.2.6.7 Loosen two bottom screws for slit III and IV holder (allen screws are right behind slit III in white painted metal). Adjust slit holder for III and IV with the smallest slits in positions III and IV.

8.2.7 Run a scan on the glass slit through zero.

8.2.7.1 Set up for scan: Slit position: I II III IV Slit: 1°1°1°0.018°

8.2.7.2 Set generator to minimum power (~7kV/3mA for FK60-04 Cu tube), ratemeter to ~$2x10^4$ counts/sec full scale, time constant = ~0.2, absorber in direct beam, diffractometer set to step at 1/5°/min upscale, the chart recorder set to feed at 2 cm/min, have θ and 2θ coupled.

8.2.7.3 Start scan at -999.500° 2θ (scan range is dependent on tube type).

8.2.7.4 This scan must be run automatically on the θ-θ machine, using the same conditions, starting 2θ position, and a 0.01° 2θ step size.

8.2.7.5 A good alignment will fulfill the following conditions:

- The maximum of the primary beam must be within 0.006° of 0°.
- The amplitudes of the two side maxima should not differ from one another by more than ~30%.
- The amplitude of the main maximum should be about
- three times as high as those of the side maximum.

8.2.8 Optimization of 2θ calibration. This is done using a certified d-spacing standard such as the NIST SRM 640 Si series and compensates for systematic errors such as sample transparency and 2θ offset.

8.2.8.1 Mount the calibration standard as in 6.1.1.

8.2.8.2 With θ and 2θ synchronized, drive to high angles (typically over 100° 2θ) and locate a peak of appreciable intensity (one that can be easily detected). Set the X-ray tube kV and mA to typical operational values (45 kV, 35 mA).

8.2.8.3 Perform a quick automatic scan over the range of the peak and determine the observed peak position using the profile fitting routine in the MDI Jade software.

8.2.8.4 If the position of maximum intensity is not at the certified value, adjust the peak position by adjusting the tube height as in 6.2.4. When this step is complete, the position of maximum intensity should be at the certified value.

8.2.8.5 Refine the adjustments made in 6.2.5 and 6.2.6.

8.2.8.6 For a quick check of the quality of alignment, one or two high-angle reflections may be measured as in 6.1. After determining that alignment is sufficient, perform complete calibration as in 6.1.

8.3 Optimization and calibration of the solid-state Si detectors.

8.3.1 Perform detector energy calibration as described in Kevex 4911 user's manual.

8.3.2 Set the detector bias to the appropriate value listed on the front of each diffractometer.

8.4 Data Analysis

The MDI Jade software package is used to regress all data produced or required by this procedure. The program LCLSQ used to determine specimen displacement errors.

8.5 **Lessons Learned**

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure.

MDI (Materials Data Incorporated), DataScan 3.2, An Automated Control and Data Acquisition System for X-ray Diffractometers, 1995-1999.

MDI (Materials Data Incorporated), Jade 5, XRD Pattern Processing, 1991-1999.

Kevex 4911 Single Channel Spectrum Analyzer User's Manual, #7257-4911.

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

10.0 RECORDS

Calibration records shall include date of calibration, standard used, signature of the person performing calibration, the peak positions observed for the standard and the necessary corrections to 2θ. Alignment records shall include the date of alignment, the procedures performed, and the signature of the machine custodian performing the alignment. A new calibration sticker shall be placed on the front of the diffractometer following each calibration identifying when the instrument was calibrated, when the next calibration is due, and the name of the person who did the calibration.

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

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environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Clay Mineral and Zeolite Separation

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

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R0	3/16/92	David Bish	New procedure	all
R1	04/11/01	David Vaniman	Updated to incorporate processes that had been in SOP 9.06 (Zeolite Purification and Separation)	all
Review	12/16/2003	Mark Thacker	Deemed process adequate.	All

Clay Mineral and Zeolite Separation

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Clay Mineral and Zeolite Separation

1.0 PURPOSE

This SOP describes the process for clay mineral and zeolite separation from geological samples at the Los Alamos National Laboratory (Laboratory) ER Project. The separation process is based on sedimentation.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when performing clay mineral or zeolite separations for the ER Project

3.0 TRAINING

3.1 All users of this SOP are trained by self-study, and the training is documented in accordance with QP-2.2.

3.2 The Geology Task Leader (TL) will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

Note: A glossary of definitions can be located on the ER Project internal homepage *http://erinternal.lanl.gov.*

4.1 *Clay Mineral*—One of a complex and loosely defined group of finely-crystalline hydrous silicates. Common clay minerals at the LANL site include illite-smectites with a basic structure of two tetrahedral sheets on either side of an octahedral sheet, or kaolinite with one tetrahedral sheet to each octahedral sheet.

4.2 *Zeolite*—One of a group of silicate minerals analogous in composition to feldspars but with an open structure that permits cation exchange. The zeolite mineral clinoptilolite has been found at the LANL site.

5.0 BACKGROUND AND PRECAUTIONS

This procedure is based on Stokes Law settling of particles in solution. Clay- or zeolite-rich samples can be purified or extracted from a sample by means of sedimentation in water. This method relies on differences in particle sizes and densities of mineral components in a sample.

Precautions in the use of this procedure include the use of only de-ionized or distilled water to wash and disaggregate the samples, in order to avoid cation-exchange effects in the concentrated clays or zeolites. For safety purposes, the ultrasonic probe used in disaggregation is operated in a sound-dampening box to protect the hearing of the operator.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Geology Task Leader

6.3 Quality Program Project Leader

6.4 Author

6.5 ER Project personnel

7.0 EQUIPMENT

The equipment used in this procedure is listed below.

7.1 *Shatterbox* — A shatterbox or comparable equipment (e.g., ballmill) can be used to powder solid samples before ultrasonic disaggregation.

7.2 *Ultrasonic Probe* — An ultrasonic probe capable of operation at 200 W is used to separate mineral grains.

7.3 *Centrifuge* — A centrifuge capable of operation at 8000 RPM is used to sediment fine particles.

8.0 PROCEDURE

Note: Subcontractors performing work under the ER Project's quality program may follow this standard operating procedure (SOP) for clay-minera and zeolite separation or may use their own procedure(s) as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and have been approved by the ER Project's Quality Program Project Leader (QPPL) before the commencement of the activitie(s).

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-

5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

8.1 Samples of loose soil or fine, uncemented sediment may be processed without grinding in a shatterbox. Most samples, however, must be crushed before individual minerals can be separated by sedimentation.

8.2 Samples that require processing in a shatterbox or other crusing device should be ground for at about 1-2 minutes to break the sample down to a particle size comparable to that of its constituent phases. Sample size will depend on the abundance of clays or zeolites in the original material. For enriched materials a few grams may suffice; clay- or zeolite-poor materials may require several tens of grams of the parent material.

8.3 The powdered sample or the original fine-grained material is placed in ~700-800 ml of distilled or deionized water in a 1000 ml plastic beaker. This sample is disaggregated for ~10-20 minutes using an ultrasonic probe operated at ~200 W. Longer times may be used if the sample is difficult to disaggregate. Smaller samples can be processed using appropriately scaled beakers.

8.4 Remove and cover the beaker. Place the covered beaker on a vibrationally stable surface without cooling it (the ultrasonic probe generates heat in the suspension) and allow the suspension to settle for 30 to 60 seconds. The sediment is composed of the coarse fraction (> ~20-30 μm) which includes most of the quart, feldspar, and other unwanted minerals, as well as any larger aggregates that were not crushed to component mineral size. The sediment may be discarded or saved for other analyses.

8.5 Decant or syphon the supernatant into a second beaker and allow it to rest on the vibrationally stable surface for ~1 hour, to settle out the ~20-3 μm size fraction. In zeolite-rich samples, this will generally be a fairly pure zeolite fraction.

8.6 Decant or syphon the remaining supernatant into a third beaker and allow it to rest on the vibrationally stable surface overnight (15-20 hours) to settle out the ~3-1 μm size fraction. This fraction may also be a rather pure zeolite sediment if the host rock is zeolite-rich.

8.7 The remaining supernatant will contain most of the clay fraction. Place this supernatant in centrifuge tubes and centrifuge at ~5000 RPM for ~5 minutes to separate the ~2-0.35 μm size fraction. This sediment may be analyzed by XRD to determine the purity of clay separation. If further processing is indicated for clay separation, proceed to step 8.8.

8.8 Decant or syphon the supernatant from the first centrifugation, place in clean centrifuge tubes, and centrifuge at ~8000 RPM for 40 to 60 minutes. The sediment collected after this process will consist of the ~0.35-0.1 μm size

fraction. This sediment may be analyzed by XRD to determine the purity of clay separation. If further processing is indicated for clay separation, proceed to step 8.9.

8.9 Further processing may consist of placing the residual supernatant on a low-temperature hotplate to evaporate the liquid, or further centrifuging the sample at ~15000 RPM for ~1 hour.

8.10 If a clay sample is collected, an oriented mount for XRD analysis can be prepared by suspending the sample in ~10-20 ml of deionized water, placing some of this suspension on a dry glass or quartz slide, and setting the slide on a warm surface to evaporate the suspension.

8.11 Lessons Learned

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure:

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

10.0 RECORDS

The Geology Task Leader (TL) is responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

10.1 Data submittals for the ER electronic database.

10.2 Notebook records of the sample handling and results of analysis relevant to production of X-ray diffraction data.

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

Attachment 2

Excerpts from the R-34 Well Completion Report

FINAL
COMPLETION REPORT
CHARACTERIZATION WELL R-34
LOS ALAMOS NATIONAL LABORATORY
LOS ALAMOS, NEW MEXICO
PROJECT NO. 37151

Prepared for:

The United States Department of Energy and the
National Nuclear Security Administration through the
United States Army Corps of Engineers
Sacramento District

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8.5-1	Geodetic Data

Table 5.2-1 summarizes the logging conducted in the R-34 borehole by Schlumberger, KA, and LANL. Schlumberger's report is presented in Appendix C along with the interpretive logging report and the geophysical logs, compiled as a montage (provided on the CD attached).

6.0 LITHOLOGY AND HYDROGEOLOGY

A preliminary description of the hydrogeologic features encountered during drilling operations at R-34 is presented below. Included are summary descriptions of geologic units identified during characterization of the cuttings sampled and a review of geophysical logs. LANL EES-6 staff provided preliminary interpretation of geologic contact zones. Several of the geologic units encountered during drilling were relatively unstable, resulting in significant periods of lost circulation and producing sections of poor or intermixed cuttings and limited intervals for geophysical logging. Stratigraphic contacts are based on available information. Groundwater occurrences are interpreted from drilling evidence, open-hole video logging, geophysical logging, and water-level measurements.

6.1 Stratigraphy and Lithology

Rock units and stratigraphic relationships are interpreted based on visual examination of the borehole drill cutting samples, as well as preliminary interpretations of the geophysical data, and are briefly discussed in order of younger to older occurrence. The drill cuttings from R-34 commonly are mixtures of cuttings derived from one or more geologic units. In addition, poor circulation of drilling fluids resulted in long intervals in which no cuttings were returned to the surface. Because of these problems, the borehole geophysical logs were particularly useful for identifying geologic contacts. The interpretations presented below are preliminary and may be revised upon additional analysis of petrographic, geochemical, mineralogical, and geophysical logging data. A lithologic log of the borehole containing detailed descriptions that identify the texture and composition of sample intervals is presented in Appendix D.

Alluvium, Qal (0 to 18 ft bgs)

Cuttings collected from the borehole indicate that unconsolidated alluvium exists from the ground surface to approximately 18 ft bgs. The position of the contact between the alluvium and the Tshirege Member of the Bandelier Tuff is uncertain, and additional geologic characterization is underway to identify this contact. Samples collected from 0 to 18 ft bgs consisted of silt with sands and gravels typically composed of volcanic lithics, tuff fragments, and quartz crystals. The composition is comparable to that of the Bandelier Tuff, from which these sediments are likely derived.

Tshirege Member, Bandelier Tuff, Qbt1g (18 to 96 ft bgs)

The lower vitric unit of the Tshirege Member of the Bandelier Tuff was encountered in the borehole between 18 and 96 ft bgs. The tuff is grayish to pale orange, poorly welded, and pumice-rich. The coarse fraction (i.e., the >#10 sieve size) of most cuttings samples in this interval is made up of vitric, fibrous pumices. The fine fraction (i.e., the >#35 sieve size) is made up of pumices and tuff fragments, quartz and sanidine crystals, and a small percentage of lithics.

Otowi Member, Bandelier Tuff, Qbo (96 ft to 134 ft bgs)

Rhyolitic ash-flow tuff representing the Otowi Member of the Bandelier Tuff was intersected in the borehole from 96 to 134 ft bgs. Cuttings showed that the Otowi Member is locally pumiceous, lithic-bearing, and poorly welded. The coarse fraction of most cuttings samples from this interval is made up of predominantly volcanic lithic fragments of intermediate volcanic

composition including iron-stained dacite, vitric pumices, and a very small fraction of crystals. Fine-fraction samples are made up predominantly of quartz and sanidine crystals, with subordinate amounts of volcanic lithics and pumice.

Guaje Pumice Bed, Bandelier Tuff, Qbog (134 ft to 146 ft bgs)

The Guaje Pumice Bed, encountered from 134 to 146 ft bgs, is made up of pumice-fall deposits that form the basal subunit of the Otowi Member of the Bandelier Tuff. Cuttings from the interval were severely milled in the process of drilling, to the extent that there was very little recovery of the coarse fraction, indicating the friable nature of this unit. The fine fraction is predominantly (i.e., more than 50 percent by volume) vitric pumice with subordinate amounts of quartz and sanidine crystals, as well as lithic fragments of intermediate volcanic composition.

Cerros del Rio Basalt, Tb (146 to 678 ft bgs)

The Cerros del Rio basalt, comprising basaltic lavas and intercalated scoria deposits, was encountered in the borehole from 146 to 678 ft bgs. Cuttings indicated that the upper portion of this section, from 148 to about 298 ft bgs, consists of medium to dark gray, massive to vesicular basaltic lava that contains occasional phenocrysts in an aphanitic groundmass. The groundmass exhibits minor weathering and/or alteration. Below 298 ft bgs, distinctive reddish intervals of oxidized scoriaceous basaltic cinders occur to 610 ft bgs. The interval again changed back to medium blue-gray massive basaltic lava from 610 to 678 ft bgs.

Older Alluvium, Ta (678 to 725 ft bgs)

Volcaniclastic sediments consisting of silty gravel and broken to rounded gravel clasts were encountered from 678 to 725 ft bgs in the borehole. The clasts were composed of basalt, scoria, and various intermediate volcanic composition clastic rocks and quartzite that were subrounded to rounded.

Puye Formation, Pumiceous subunit, Tpp (725 to 1,050 ft bgs)

The Puye Formation provided sporadic sample returns of pumiceous volcaniclastic constituents including abundant pumices with minor amounts of detrital clasts of intermediate volcanic composition. Large intervals of poor recovery of cuttings were encountered in this unit. Geophysical logs suggest that the upper part of the unit, from 725 – 790 ft bgs, may be Puye fanglomerate similar to that found below Cerros del Rio basalt in deep wells in nearby Mortandad Canyon.

Puye Formation, Totavi Lentil subunit, Tpt (1,050 to 1,065 ft bgs)

Due to lack of geophysical logs and poor cutting returns, the Totavi Lentil interval thickness is estimated. Available cuttings collected by circulation at TD indicate the presence of Precambrian granitic clasts, quartzite, and varied intermediate volcanic composition clasts that are rounded to subrounded. Sample chips also include scoria and pumice fragments obtained from overlying formations.

6.2 Groundwater Occurrence and Characteristics

The SAP indicated that there was a possibility regional groundwater would be encountered in the Puye Formation at approximately 915 ft bgs.

During borehole drilling, one aqueous sample (GW34-04-53519) was collected from a depth between 641.4 and 664 ft bgs to determine if perched groundwater was present. A groundwater

sample (Sample ID GW34-04053521) was collected near the end of well development from the screened interval (883.7 to 906.6 ft bgs).

The processed geophysical logs indicate a significant increase in water saturation below 794 ft bgs, which is consistent with the water level in the well at the time of the logging. The estimated pore volume water saturation (fraction of the total pore volume containing water) computed from the ELAN (Elemental Analysis) is high (above 75 percent) from 794 ft bgs to the bottom of the logged interval (933 ft bgs), compared with 40 to 65 percent in the interval directly above 794 ft bgs. The estimate is even higher when computed directly from bulk density and ELAN water-filled porosity for a grain density range of 2.45 to 2.65 grams per cubic centimeter (g/cc), ranging from 80 percent (2.65 g/cc) to consistently 90 to100 percent (2.45 g/cc). These results suggest that the static water level for the regional aquifer occurs at 794 ft bgs. After completion of drilling on August 10, 2004, the DTW was again measured in the open borehole and was found to be at 796.17 ft bgs. Prior to aquifer testing, on September 11, 2004 the static water level was measured at 796 ft bgs.

KBr was added to the drilling fluid as a tracer to aid in determining perched and regional groundwater. The KBr tracer was introduced using a bromide injection system. Results of sampling and analysis for KBr indicate that the initial injection concentration was too low. Bromide concentrations began to decrease at approximately 270 ft bgs, as shown in Figure 6.2-1. Due to the low concentration and lost circulation, samples were not taken between about 340 to 600 ft bgs. The problem with the injection system was repaired, and tracer was again added to the system from a depth of about 600 to 700 ft bgs. The final results of sampling and analysis for the KBr injection tracer do not indicate groundwater over the depth interval evaluated.

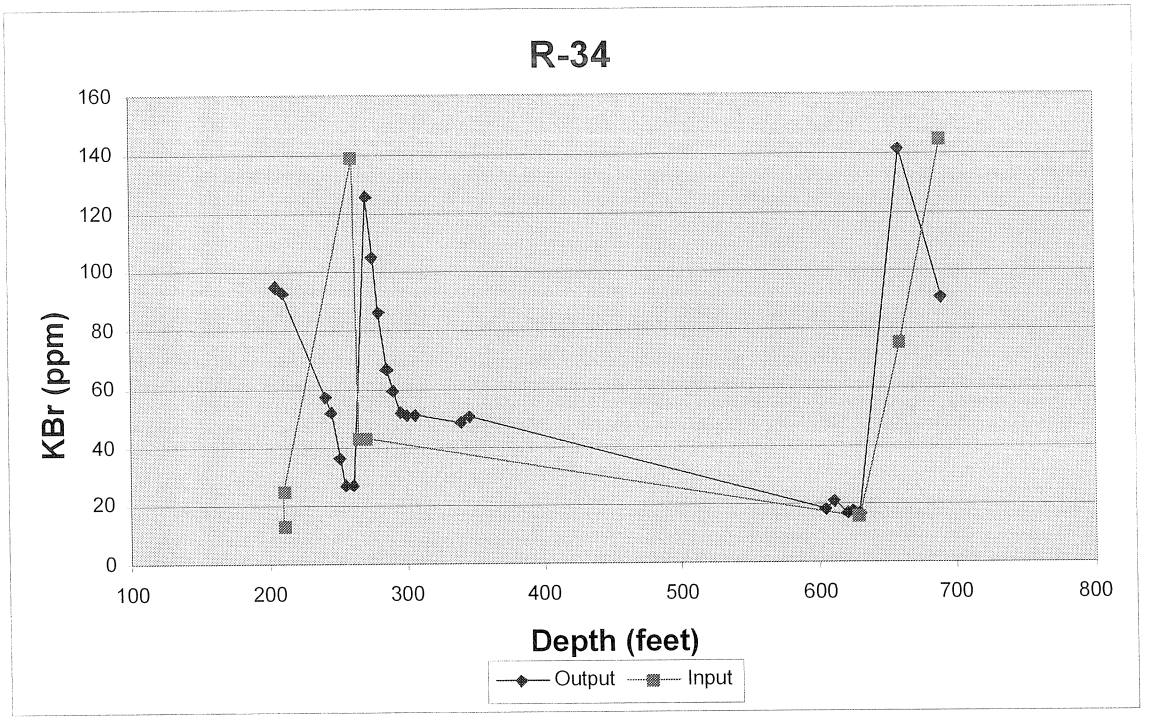

Figure 6.2-1. KBr Concentrations in Borehole During Drilling

7.0 WELL DESIGN AND CONSTRUCTION

R-34 was installed as a hydrogeologic characterization and groundwater monitoring well. Following approval of the well design by DOE, LANL and the New Mexico Environment Department (NMED), KA received the final construction specifications for R-34 on August 11, 2004. Well installation activities were performed from August 13 to September 20, 2004.

7.1 Well Design

Data from geophysical logs and borehole cuttings, as well as field water level measurements and field observations, were evaluated to determine the screen placement interval for the well. Design of the well was performed in accordance with LANL ER SOP-05.01, Revision 3 and an approved well design was provided to KA by DOE and LANL. The well was designed with a single screen interval to monitor potential contaminants and groundwater chemistry in the uppermost productive zone of the regional aquifer in the Puye Formation.

7.2 Well Construction

R-34 was constructed of 4.5-inch inner diameter (ID)/5.0-inch OD, type A304, stainless-steel casing fabricated to American Society for Testing and Materials (ASTM) A312 standards. Two nominal 12-ft lengths of 5-in OD compatible, 0.020-inch rod-based wire-wrapped well screens were also used. The casing and screens were factory cleaned before shipment and delivery to the site. Additional decontamination of the stainless-steel components was performed on site prior to well construction using a high-pressure steam cleaner. The screened interval chosen for the well was 883.7 to 906.6 ft bgs. Stainless steel casing was placed below the screen to construct a 14.1 ft-deep sump. External couplings, also of type A304 stainless steel fabricated to ASTM A312 standards, were used to connect individual casing and screen joints. The 9⅝-inch drill casing remained in the borehole during well construction of the screened interval to maintain hole stability. Centralizers were not able to pass through the drill casing and thus were not used in the well. Figure 7.2-1 is an as-built schematic of the completed well.

Placement of annular fill into the borehole consisted of using a 2.5-inch OD steel tremie pipe to deliver various materials to specified backfill intervals. It was determined based on sounding that approximately 90 ft of formation material had sloughed into the borehole from the TD of 1,065 up to 975 ft bgs. From 975 up to 935 ft bgs, a mixture of 8/20 silica sand and bentonite was placed as the foundation for the filter pack above. Well casing and screens were lowered in the borehole. A primary filter pack consisting of 8/20 silica sand was placed across the screened interval from 935 up to 877 ft bgs. A secondary filter pack of 20/40 silica sand was placed above the primary filter pack from 877 up to 875 ft bgs. Prior to placement, filter pack materials were mixed with municipal water to form a slurry. The filter pack was then swabbed to induce settling prior to bentonite placement. Next, a seal consisting of a mixture of 8/20 sand and bentonite chips was placed above the secondary filter pack in the annular space between 875 and 717 ft bgs. The 9⅝-inch drill casing was pulled, resulting in a shift upward of the well casing of approximately 3.4 feet (final well component depths are reflected on Figure 7.2-1). During retraction of the drill casing, another formation collapse occurred, filling in the annular space from 717 up to 667 ft bgs. Between 667 and 82 ft bgs, the annular space was filled with a mixture of 70 percent bentonite and 30 percent 8/20 sand. The bentonite mixture was hydrated in approximately 50-ft lifts. After removing the 13⅜-in conductor casing, concrete backfill, consisting of 2,500 pounds per square inch (psi) concrete, was placed from 82 ft bgs to ground surface. Quantities of annular fill materials used are presented in Table 7.2-1.