

## **ATTACHMENT H**

### **Kd DISTRIBUTION COEFFICIENTS TEST RESULTS**

#### **CONSISTS OF:**

**Report by Savannah River National Laboratory**

**WSRC-STI-2007-00244**

**Complete with Appendices A through L (by WSRC)**

**MACTEC and STL Data Appended as Appendix M**

**Volume 1 of 1**

**KEY WORDS:**

**Distribution Coefficients,  
Cobalt, Cesium  
Strontium**

**RETENTION:**

**Permanent**

**DISTRIBUTION COEFFICIENTS FOR THE COMBINED LICENSE  
(COL) APPLICATION OF THE VC SUMMER NUCLEAR STATION  
SITE**

**Daniel I. Kaplan**

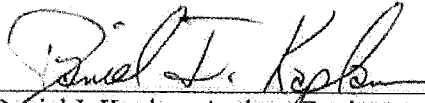
**AUGUST 21, 2007**

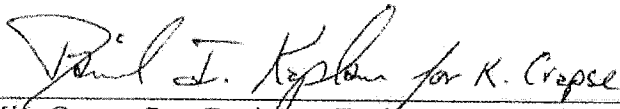
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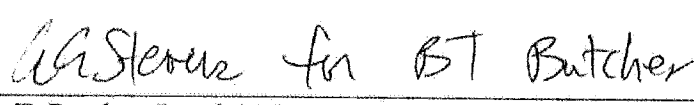
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## LIST OF ACRONYMS

avg	average
bgs	below ground surface
COL	Combined License
$K_d$	distribution coefficient
MWR	moderately weathered rock
PWR	partially weathered rock
XRD	X-ray diffraction analysis
XRF	X-ray fluorescence analysis
SRNL	Savannah River National Laboratory
stdev	standard deviation

## ABSTRACT

Rock and soil distribution coefficient,  $K_d$  values, measurements (the ratio of radionuclide concentration sorbed to the concentration in the aqueous phase) were conducted in support of Combined License (COL) application at the V.C. Summer Nuclear Power site in Fairfield County, South Carolina. Fifteen rock and five soil and four representative groundwater samples from the V.C. Summer site were provided for the Savannah River National Laboratory to conduct site-specific cobalt (Co), cesium (Cs), and strontium (Sr)  $K_d$  measurements. Mineralogical (X-ray diffraction and X-ray fluorescence) and surface area information was also measured for selected samples. Comparisons of  $K_d$  measurements were made: 1) as a function of four ground rock sizes (approximately 10-, 100-, 1000-, and 10,000- $\mu\text{m}$ ) 2) more widely between finely ( $\sim 10 \mu\text{m}$ ) and coarse ( $\sim 10,000 \mu\text{m}$ ) ground rock samples, 3) between rock samples collected along fractures and from the general matrix rock, and 4) between five soil samples. The intent for some of the comparisons was to understand how these radionuclides may sorb in a fractured media. At issue, is that there is not a standardized test for measuring sorption to fractured media; instead there is only the standardized test for measuring sorption to porous media (ASTM 4646:  $K_d$  test). In almost all samples, Co  $K_d$  values (average across all treatments = 513.6 mL/g) > Cs  $K_d$  values (average = 268.5 mL/g) > Sr  $K_d$  values (average = 36.1 mL/g). A comparison of the effect of particle size of one rock sample on  $K_d$  values showed that there was little difference in  $K_d$  values between the 10- and 100- $\mu\text{m}$  particles, but that the Co, Cs, and Sr  $K_d$  values consistently decreased by about an order of magnitude as the particle sizes increased from 100 to 10,000- $\mu\text{m}$  particles. As expected, the finest ground samples (10  $\mu\text{m}$ ) always had appreciably greater  $K_d$  values than their coarse ground (10,000  $\mu\text{m}$ ) counterparts; the 10 $\mu\text{m}$ :10,000 $\mu\text{m}$  ratios of the  $K_d$  values for Co = 16.6, Cs = 12.1, and Sr = 7. In comparisons between fractured and matrix rock samples, the  $K_d$  values were always greater in the 10 $\mu\text{m}$  fracture samples than in the comparable matrix samples. For similar comparisons with the coarse ground samples (10,000 $\mu\text{m}$ ), there was either no difference (Co and Sr  $K_d$  values) or the fracture samples were larger (Cs  $K_d$  values) than the matrix samples. Finally, soil sample  $K_d$  values were generally comparable in magnitude with the rock 10- $\mu\text{m}$  particle  $K_d$  values and followed the same trends, namely Co > Cs > Sr  $K_d$  values. In general, the  $K_d$  values reported here followed expected trends based on geochemical principles and will provide the necessary sorption data for assessing the risk associated with subsurface movement of Co, Cs, and Sr at the V.C. Summer site.

## 1.0 INTRODUCTION

Distribution coefficients,  $K_d$  values, when used in reactive transport codes are conditional on the sediment type and groundwater (Krupka et al 1999a). For this reason it is ideal to use  $K_d$  values derived from site-specific materials when possible. The objective of this project was to provide site-specific cobalt (Co), cesium (Cs), and strontium (Sr)  $K_d$  values for the V.C Summer COL application. Some additional objectives were to compare  $K_d$  values:

- 1) as a function of four ground rock sizes (approximately 10-, 100-, 1000-, and 10,000- $\mu\text{m}$ );
- 2) between finely ( $\sim 10 \mu\text{m}$ ) and coarse ( $\sim 10,000 \mu\text{m}$ ) ground rock samples;
- 3) between rock samples collected along fractures and from the general matrix rock; and
- 4) between five soil samples.

Additionally, X-ray Diffraction (XRD), X-ray Fluorescence (XRF) and BET surface area analyses were conducted on select samples to provide insight into the solid phase mineralogy. Fifteen rock, five soil, and four representative groundwater samples from the V.C. Summer site were provided by MACTEC for use in this study.

## 2.0 MATERIALS AND METHODS

### 2.1 MATERIALS

The Materials and Methods are described in detail in Appendix A. The following is a brief description to permit understanding of the results. The representative rock, soil, and groundwater samples from the V.C. Summer site are described in Table 1. The radionuclides used in the  $K_d$  measurements were gamma emitters purchased from Analytix, Inc. (Atlanta, GA; <http://www.analytixinc.com/prod01.htm>). The solids/groundwater suspensions were spiked with the following isotopes:

- $^{60}\text{Co}$ : the sediments suspension was initially spiked at a concentration of  $6.89\text{e-}7$  Ci/L.
- $^{134}\text{Cs}$ : the sediments suspension was initially spiked at a concentration of  $1.14\text{e-}5$  Ci/L.
- $^{85}\text{Sr}$ : the sediments suspension was initially spiked at a concentration of  $1.19\text{e-}5$  Ci/L.

The magnitude of sorption to geological materials does not vary with isotope. So even if the COL application is interested in making transport calculations with different isotopes of Co, Cs, and Sr than those used in these tests, the  $K_d$  values will remain the same. At these very low concentrations, competition for sorption sites as a result of introducing more than one radionuclide at a time is essentially non-existent. The test matrix is presented in Table 2 and the lithology of each sample is presented in Table 3.



**Table 1.** Sample description as provided by MACTEC.

<b>K<sub>d</sub> Sample Locations for Soil, Rock and Groundwater</b>					<b>Comments</b>
<b>Boring</b>	<b>K<sub>d</sub> Sample Depth (feet below ground surface (bgs))</b>	<b>K<sub>d</sub> Sample location description</b>	<b>Lithology of K<sub>d</sub> Sample</b>	<b>Water Sample Collection Location</b>	<b>Sample Selection</b>
B-205	98-100	Choose a fracture zone from which to extract 2 samples for K <sub>d</sub> analyses (1 sample for the 10µm sample and the other for the ~1cm sample) - Sample should be a minimum of 6 inches in length.	Bedrock	OW-205a	Fracture zone 1 = 98.6', or Fracture zone 2 = 99.4'
B-212/212a	Sample #14 (48.5-50.0)	If this is not enough sample, mix with Sample 13. Both are Saprolite (SM)	Saprolite	OW-212	Jar Sample
B-212/212a	Sample # 18 (58.5-60.0)	PWR - choose representative 6 inch sample	PWR <sup>1</sup>	OW-212	Jar Sample
B-212/212a	86-96	Complete total of 6 K <sub>d</sub> tests on one 18" core sample. 4 samples from the rock matrix and 2 samples from along a fracture surface - Sample should be a minimum of 18 inches in length.	Bedrock	OW-205a	Fracture zones = 87.1' and 88.0', Matrix samples = 87.1'-87.65' or 87.65'-88' or 88'-88.65' or 88.65'- 89.2'
B-305	64-64.5	Choose a fracture zone from which to extract 2 samples for K <sub>d</sub> analyses (1 at approximately 10 microns and 1 at approximately 1 cm) - Sample should be a minimum of 6 inches in length.	Bedrock	OW-305a	Fractured zone = 64.2'-64.6'
B-305	59-60	Choose 6 inch sample from fracture zone logged from 57.5 to 60.5'	MWR <sup>1</sup>	OW-327	Core fractured zone = 59.4'-59.9'

B-327	56-59	Choose 6" sample of good water bearing fractures	MWR <sup>1</sup>	OW-327	Core fractured zone = 58.7'-59.1'
B-333	63-65	Choose 6" sample of good water bearing fractures (Highly Weathered to Completely Weathered)	PWR <sup>1</sup>	OW-327	Core weathered zone = 63.2'-65'. If possible, hand crush highly weathered material for testing and use fine material for testing.
B-620	Sample #12 (38.5-40.0)	If sample is not enough, combine with Sample #11	Saprolite	OW-212	Jar Sample
B-627	66.5 to 76.5	Choose a fracture zone from which to extract 2 samples for K <sub>d</sub> analyses (1 for the 10µm sample and the other for the 1cm sample) - Sample should be a minimum of 6 inches in length.	Bedrock	OW-305a	Weathered fracture zones = ~59.5' I Could not identify any decent fractures at the requested depth. Best fractures are at 59.5' near top of rock.
B-627	Sample #9 (23.5-25.0)	If sample is not enough, combine with sample #10	Saprolite	OW-327	Jar Sample
B-627	Sample #14 (47.0-48.5) and Sample #15 (53.5-55.0)	If enough material, combine these 2 into 1 sample for K <sub>d</sub> analysis	PWR	OW-327	Jar Sample
<p>Notes:</p> <p><sup>1</sup> MWR = moderately weathered rock, PWR = partially weathered rock. These samples are not technically considered "bedrock", however, these weathered rock samples are still relatively intact and will require crushing before K<sub>d</sub> analyses can be run. Samples should be crushed to 10 microns and tests run as appropriate for bedrock samples.</p>					

**Table 2.** K<sub>d</sub> test metrics.

Boring	Kd Sample Depth (feet bgs)	Jar Sample (Soil Material)	Rock Fracture Surface 10 μm <sup>(1)</sup>	Rock Fracture Surface 10,000 μm <sup>(1)</sup>	Rock (Matrix) 10 μm <sup>(1)</sup>	Rock (Matrix) 100 μm <sup>(1)</sup>	Rock (Matrix) 1000 μm <sup>(1)</sup>	Rock (Matrix) 10,000 μm <sup>(1)</sup>
B-205	98-100		1	1				
B-212/212a	Sample #14 (48.5-50.0)	1						
B-212/212a	Sample # 18 (58.5-60.0)	1						
B-212/212a	86-96		1	1	1	1	1	1
B-305	64-64.5		1	1				
B-305	59-60		1					
B-327	56-59		1					
B-333	63-65		1					
B-620	Sample #12 (38.5-40.0)	1						
B-627	66.5 to 76.5		1	1				
B-627	Sample #9 (23.5-25.0)	1						
B-627	Sample #14 (47.0-48.5) and Sample #15 (53.5-55.0)	1						
<sup>(1)</sup> Samples sizes are approximate. They may be revised to meet the limitations of the laboratory sampling equipment.								

**Table 3.** Number of each type of geological material included in  $K_d$  tests.

<b>Material Type</b>	<b>Number of Samples</b>
Saprolite	3
PWR <sup>a</sup>	3
MWR <sup>a</sup>	2
Bedrock	12

<sup>a</sup> PWR = partially weathered rock; MWR = moderately weathered rock

**Table 4.** Number of  $K_d$  test versus solid phase type.

<b>Solid Phase</b>	<b>Total Number of <math>K_d</math> Tests/Type</b>
Jar Sample (Soil Material)	5 <sup>a</sup>
Rock Fracture Surface 10 $\mu$ m	7
Rock Fracture Surface 10,000 $\mu$ m	4
Rock (Matrix) 10 $\mu$ m	1
Rock (Matrix) 100 $\mu$ m	1
Rock (Matrix) 1000 $\mu$ m	1
Rock (Matrix) 10,000 $\mu$ m	1
<b>TOTAL <math>K_d</math> TESTS</b>	<b>20</b>

<sup>a</sup> Includes three Saprolite and two PWR samples.

The source solution was carefully selected so that the concentrations of Co, Cs, and Sr would not exceed the linear sorption range. In the linear range, the  $K_d$  construct is appropriate to use. At higher concentrations, it is often necessary to use more complicated models, such as the Freundlich model, and yet at higher concentrations, precipitation of the radionuclide of interest may occur. If precipitation occurs, the measurement would incorrectly attribute reversible sorption to the  $K_d$  values, where in fact “irreversible” precipitation occurred. (The latter should be described with a solubility constant and not a  $K_d$  value.) The tests were conducted at several orders of magnitude lower concentrations than would be necessary for precipitation of Co, Cs, or Sr. Regarding non-linear sorption, Krupka et al. (1999b) reported that non-linear sorption of Cs occurs at concentrations greater than about  $10^{-5}$  M (0.12 Ci/L  $^{137}\text{Cs}$ ) and Sr at about  $10^{-7}$  M (1.2e-3 Ci/L  $^{90}\text{Sr}$ ). Clearly, the concentrations used in these  $K_d$  tests are several orders of magnitude lower than where non-linear sorption occurs.

## 2.2 METHODS

### 2.2.1 Distribution Coefficients

A detailed description of the methods used to measure  $K_d$  values is presented in Appendix A. This procedure follows ASTM D 4646-03: Standard test method for 24-hr batch-type measurement of contaminant sorption by soils and sediments (Appendix G). Briefly, the rock samples came in 5-cm diameter cores. Using a Westward 6-ton hydraulic press, ~0.5-cm thin slices were cut from the various core samples as described in Table 2, Table 3, and Table 4. These slices were then ground by various techniques to obtain the other size particles.

- To create the *1-cm sized particles*, a chisel and hammer were used (See photos in Appendix D). About a dozen pieces were made from the slice, from which one piece, with the dimensions closest to 1-cm, was selected for the  $K_d$  test.
- To create the *10- $\mu\text{m}$  sized particles*, a chisel and hammer were used to break up the thin sections created from the hydraulic press to ~1-cm pieces (photos in Appendix D). Approximately 20 g of these 1-cm pieces were placed in a Spex 8510 Shatter box for 5 minutes, ground, and passed through #400 (38 $\mu\text{m}$ ) and #500 (25 $\mu\text{m}$ ) sieves. The <25 $\mu\text{m}$  fractions were saved.
- To create the *100- and 1000- $\mu\text{m}^2$  sized particles*, a chisel and hammer was used to break up the thin sections made from the hydraulic press to particles that were ~1-cm (10,000 $\mu\text{m}$ ) in diameter. Then approximately 100 g of these particles were placed in a jaw crusher (Retsch Jaw Crusher Type BB51 with tungsten carbide plates) for 10 minutes. The jaw crusher sample were then passed through a 1.4-mm sieve (#14 sieve) and the less than fraction was placed in a shatter box (Spex 8510 Shatterbox) for 10 seconds. The shatter box sample were then pass particles through a series of sieves (1400, 1180, 1000, 850, 710, 150, 125, 106, 90, and 75  $\mu\text{m}$ ) and shaken on an AS200 Analytical Sieve Shaker (Type AS200 Digit). The <75- $\mu\text{m}$  (#200 sieve) material was then placed in a Spex 8000 mixer/mill for 10 seconds and then in a Spex 8510 Shatter box for 5 min. This material was then passed through a 25- $\mu\text{m}$  (#500 sieve). The “100- $\mu\text{m}$ ” was the >90 $\mu\text{m}$ / $<106\mu\text{m}$  fraction. The “1000- $\mu\text{m}$ ” fractions was a combination of two fractions (to provide enough sample) >1000 $\mu\text{m}$ / $<1180\mu\text{m}$  and the >850 $\mu\text{m}$ / $<1000\mu\text{m}$  fractions.

For the five soil samples, the sample was first thoroughly mixed and then approximately 100 g sub-samples were dried at 85 °C in a drying oven. The dried samples were passed through a 2-mm sieve. The entire soil sample passed through the 2-mm sieve.

Thus our best estimate of the particle sizes for each category is:

- “10,000 $\mu\text{m}$ ” = 10,000 $\mu\text{m}$
- “1000 $\mu\text{m}$ ” = >850 $\mu\text{m}$  <1180 $\mu\text{m}$
- “100 $\mu\text{m}$ ” = >90 $\mu\text{m}$  <106 $\mu\text{m}$
- “10 $\mu\text{m}$ ” = 13 $\mu\text{m}$  (or more specifically, <25 $\mu\text{m}$ )
- “soil” = <2mm.

Throughout the text, each fraction will be identified by their target size, rather than our best estimate of the actual size.

0.5-g of dried solid phase (or 1 particle in the case of the 10,000 $\mu\text{m}$  particle size tests (generally about 1.2 g)) was added to a centrifuge tube, followed by 12 mL of groundwater. The suspension was mixed overnight to pre-equilibrate the sediments with the groundwater chemistry prior to adding the radionuclide spike. To separate the phases, the tubes were centrifuged and the aqueous phase was decanted and disposed.

Following pre-equilibrating the sediment sample, 10 mL groundwater was added to the tubes followed by 80  $\mu\text{L}$  of a gamma spike solution. The spike activities introduced to each suspension were:

- 2,340 dpm/mL  $^{60}\text{Co}$  ( $6.89 \times 10^{-13}$  Ci/L),
- 26,400 dpm/mL  $^{85}\text{Sr}$  ( $1.19 \times 10^{-11}$  Ci/L), and
- 25,400 dpm/mL  $^{134}\text{Cs}$  ( $1.14 \times 10^{-11}$  Ci/L),

as shown in Table 15 for the “No soil Control” data.

Because the background solution of the radionuclide spikes was very acidic (~0.1 M HCl), it was necessary to adjust the pH by adding base to each suspensions until the pH returned to approximately that of background, pH 6. The actual amount of base added was recorded; it was in the order of 90  $\mu\text{L}$  of 0.1 M NaOH. It is important to maintain sediment pH because pH can greatly influence the extent that radionuclides sorb to sediments. The suspensions were periodically shaken over a 7 day contact period. At the end of the contact period, the samples were permitted to settle and the aqueous phase was passed through a 0.1 $\mu\text{m}$  filter and analyzed by standard gamma spectroscopy. All  $K_d$  measurements were conducted in duplicate. Three positive controls (spikes only in groundwater, no solid phase added) and eight negative controls (each of the 4 groundwater in duplicate, but no soil or spike) were included. Therefore a total of:

(2 reps x 20 sediments) + 3 positive controls + 8 negative controls = 51 test tube measurements were made.

The three positive controls were used to provide estimates of radionuclide concentrations in the absence of sediment and accounted for any sorption to the labware that may have occurred as an experimental artifact. It was these sample results that were used to establish the initial solute concentration used in the  $K_d$  calculation. The negative controls provided a measure of how much radioactivity is in the native groundwater; they all came back indicating that there was  $<1$  dpm/mL ( $<0.45$  pCi/mL) of  $^{60}\text{Co}$ ,  $^{85}\text{Sr}$ , and  $^{134}\text{Cs}$ . The latter control is not used in the calculation of  $K_d$  values.

### 2.2.2 Solid Phase Characterization

XRF, XRD, and BET analyses were conducted on seven samples selected by Bechtel. The samples were (Boring/depth below ground surface [ft]): B-205(98.6-99.8 bgs), B-212/212a(86-96 bgs), B-305(64-64.5 bgs), B-305(59-60 bgs), B-327(56-59 bgs), B-333(63-65 bgs), and B-627(66.5-76.5 bgs). These tests were conducted with finely ground samples ( $\sim 10\mu\text{m}$ ). The procedures followed were from the Savannah River National Laboratory's standard operating procedures (which have been transmitted to MACTEC under separate cover):

XRD: "Panalytical X'pert Pro 3040, X-ray Diffraction System." Manual L.16.1, Procedure ADS-1120, Rev. 0.

BET: "Procedure for Operating the Micromeritics ASAP 2010 Surface Area and Pore Volume Analyzer." Manual L16.1, Procedure: ADS-1.114, Rev 2.

XRF: "Rigaku ZSX 101E, Wavelength Dispersive X-ray Fluorescence System." Manual L.16.1, Procedure ADS-115, Rev. 2.

Another important reference for the XRD work, useful for interpreting the results is Whittig and Allardice (1986).

The simple statistics used in this report (averages standard deviations, and regression) were generated by EXCEL (Microsoft Inc).

The lab notebook used to record the activity of this work was WSRC-NB-2007-00029, pages 5 - 33. This unclassified notebook will be kept "permanently" by the Department of Energy.

### 2.3 ANALYTICAL STANDARDS ANALYZED WITH SAMPLES

Internal (SRNL) QA standards of nine isotopes that span the gamma energy range of interest were measured each morning prior to analyzing any samples. The results from these analyses are presented in Appendix E. A mark in the QA results in the "Deviation/Flags" column of "In" means the measured value needs to be "investigated", but the instrument is within specifications of the standard. A "Deviation/Flags" comment of "Ac" means the measured value is outside the acceptable range and "action" is required. The "action" flag indicates that the analyses results are unacceptable. During the five days that the samples were analyzed, there were a total of 90 standards that were measured, of which nine

“investigate” flags and zero “action” flags were identified. This indicates that all measurement QA standards were within specification.

USGS standards, USGS Diabase W1 Standard and USGS Syenite STM-1 Standard ([http://minerals.cr.usgs.gov/geo\\_chem\\_stand/](http://minerals.cr.usgs.gov/geo_chem_stand/)) were used to check the accuracy of the XRF (See Appendix E). These standards were run at the start of analyzing the study’s samples. All measured elemental concentrations of the USGS standard samples are presented in Appendix E and indicate the instrument was operating correctly.

One standard was run at the start of the XRD analyses, which were all completed in one day. The standard was provided by the manufacturer of the XRD instrument, PANalytical, Lelyweg 1, 7602 EA ALMELO, The Netherlands; <http://www.panalytical.com/>. Two parameters are checked, the peak location ( $26.640^\circ$ ) and the peak intensity. Peak intensity does not have a critical value; instead this value is compared to other values previously measured from this sample. When it is ~20% of the original value (when the radiation source used in the XRD was new) than it indicates that the radiation source needs to be replaced. In this case the peak intensity denoted in Figure 2 of Appendix E indicated <5% reduction, indicating that the gamma source did not need replacing.

There are no certified standards for the BET surface area measurements. Instead there are reference materials. The reference material used to calibrate the Micromeritics ASAP 2010 was purchased from the vendor, Micromeritics Instrument Corporation (Norcross GA) and had a BET surface area of  $2.85 \pm 0.09 \text{ m}^2/\text{g}$ . The reference material was run at the start and end of the study sample analyzes. The reference material had a measured surface area of  $2.84 \text{ m}^2/\text{g}$  at the beginning and at the end of the sample measurements, indicating the instrument was calibrated correctly.

## 3.0 RESULTS & DISCUSSIONS

### 3.1 ALL $K_d$ DATA

The  $K_d$  values for the 20 samples are presented in Table 5 and associated descriptive statistics are presented in Table 6. A ranking of the radionuclides across all the various treatments and lithologies in order of their mean  $K_d$  values is: Co ( $>513.6 \text{ mL/g}$ ) < Cs ( $268.5 \text{ mL/g}$ ) < Sr ( $36.1 \text{ mL/g}$ ). This ranking of the radionuclides is consistent with general geochemical principles and are consistent with the ranking reported in Thibault et al. 1990 and Krupka et al. 1999b.



**Table 5.** Co, Cs, and Sr  $K_d$  values (average and standard deviation of two values).

SRNL ID #	Boring	Lithology	Particle Size ( $\mu\text{m}$ )	Solid Phase	Co $K_d$ (mL/g)	Sr $K_d$ (mL/g)	Cs $K_d$ (mL/g)
401	B-205 (98-100 bgs)	Bedrock	10	Fracture	493.9 $\pm$ 298.7	13.8 $\pm$ 1.3	106.4 $\pm$ 26.8
402		Bedrock	10000	Fracture	74.1 $\pm$ 15.1	1.9 $\pm$ 1.5	3.0 $\pm$ 0.8
403	B-212/212a (86-96 bgs)	Bedrock	10	Fracture	735.6 $\pm$ 162.5	34.4 $\pm$ 7.3	110.2 $\pm$ 25.3
404		Bedrock	10000	Fracture	24.6 $\pm$ 3.8	0.9 $\pm$ 0.1	2.1 $\pm$ 0.1
405		Bedrock	10	Matrix	300.3 $\pm$ 0.0	8.3 $\pm$ 0.0	8.5 $\pm$ 0.3
406		Bedrock	100	Matrix	359.5 $\pm$ 45.0	7.6 $\pm$ 0.8	11.2 $\pm$ 0.1
407		Bedrock	1000	Matrix	252.6 $\pm$ 111.7	2.3 $\pm$ 0.9	3.0 $\pm$ 0.2
408		Bedrock	10000	Matrix	26.1 $\pm$ 2.2	0.7 $\pm$ 0.4	1.0 $\pm$ 0.3
409	B-305 (64-64.5 bgs)	Bedrock	10	Fracture	549.5 $\pm$ 53.4	14.2 $\pm$ 2.5	24.7 $\pm$ 2.8
410		Bedrock	10000	Fracture	19.2 $\pm$ 0.0	8.2 $\pm$ 7.2	6.5 $\pm$ 0.6
411	B-305 (59-60 bgs)	MWR <sup>(a)</sup>	10	Fracture	788.7 $\pm$ 399.6	83.5 $\pm$ 24.8	140.2 $\pm$ 91.9
412	B-327 (56-59 bgs)	MWR	10	Fracture	658.1 $\pm$ 323.4	86.8 $\pm$ 52.4	99.7 $\pm$ 89.4
413	B-333 (63-65 bgs)	PWR	10	Fracture	>1433.4 $\pm$ 682.2	134.8 $\pm$ 31.7	730.4 $\pm$ 8.9
414	B-627 (66.5 -76.5 bgs)	Bedrock	10	Fracture	387.7 $\pm$ 381.6	33.9 $\pm$ 6.6	1035.1 $\pm$ 374.6
415		Bedrock	10000	Fracture	4.4 $\pm$ 2.6	13.4 $\pm$ 3.4	93.8 $\pm$ 34.4
416	B-212/212a (#14 (48.5-50 bgs)	Saprolite	<2000	Soil	676.1 $\pm$ 63.7	62.0 $\pm$ 0.3	89.3 $\pm$ 0.2
417	B-212/212a (#18 (58.5-60 bgs)	PWR	<2000	Soil	390.8 $\pm$ 370.5	45.0 $\pm$ 22.6	81.5 $\pm$ 2.9
418	B-620 #12 (38.5-40 bgs)	Saprolite	<2000	Soil	415.9 $\pm$ 188.5	38.5 $\pm$ 2.5	71.0 $\pm$ 7.4
419	B-627 #9 (23.5-25 bgs)	Saprolite	<2000	Soil	1576.4 $\pm$ 727.0	67.9 $\pm$ 25.6	512.6 $\pm$ 283.3
420	B-627 #14 (47-48.5bgs); #15 (53.5-55 bgs)	PWR	<2000	Soil	1105.3 $\pm$ 891.1	63.9 $\pm$ 0.0	2240.3 $\pm$ 942.7

<sup>(a)</sup> MWP = moderately weathered rock; PWR = partially weathered rock.

**Table 6.** Descriptive statistics of the  $K_d$  Values for all samples (mL/g).

	Co $K_d$ <sup>(a)</sup>	Sr $K_d$	Cs $K_d$
Mean	513.6	36.1	268.5
Standard Error	100.7	8.2	120.6
Median	403.4	24.0	85.4
Standard Deviation	450.2	36.8	539.4
Range	1572.0	134.1	2239.4
Minimum	4.4	0.7	1.0
Maximum	1576.4	134.8	2240.3

<sup>(a)</sup> Co  $K_d$  measurements include a greater-than-value, therefore complete statistics were not possible to conduct.

### 3.2 $K_d$ VALUES AS A FUNCTION OF PARTICLE SIZE

One sample, B-212/212a (86-96 bgs), was ground to varying sizes and the  $K_d$  value of each size fraction was measured (Table 7).

**Table 7.**  $K_d$  values as a function of particle size (averages of two replicates).

Boring	Part. Size (microns)	Solid Phase	Co		Sr		Cs	
			$K_d$ (mL/g)	stdev	$K_d$ (mL/g)	stdev	$K_d$ (mL/g)	stdev
B-212/212a (86-96 bgs)	10	Matrix	300.3	0.0	8.3	0.0	8.5	0.3
B-212/212a (86-96 bgs)	100	Matrix	359.5	45.0	7.6	0.8	11.2	0.1
B-212/212a (86-96 bgs)	1000	Matrix	252.6	111.7	2.3	0.9	3.0	0.2
B-212/212a (86-96 bgs)	10000	Matrix	26.1	2.2	0.7	0.4	1.0	0.3

The key findings from these samples are:

- There was not a significant difference between the  $K_d$  values of the 10 $\mu$ m and 100 $\mu$ m samples.
- For particles in the range of 100 to 10,000  $\mu$ m, there was the expected inverse relationship between size and  $K_d$  values. Regression analysis between  $K_d$  values and particles in the 100 to 10,000  $\mu$ m size range indicated that there was a significant relationship between particle size and Co  $K_d$  ( $R^2 = 0.84$ ;  $n = 6$ ;  $P \leq 0.01$ ), but not with Cs  $K_d$  ( $R^2 = 0.50$ ;  $n = 6$ ;  $P \leq 0.011$ ) or Sr  $K_d$  ( $R^2 = 0.53$ ;  $n = 6$ ;  $P \leq 0.10$ ). Regression correlations became significant for Sr and Cs when the  $K_d$  values were logarithmically transformed:  $\log(\text{Sr-}K_d)$ :  $R^2 = 0.97$ ,  $n = 6$ ,  $P \leq 0.0004$ ; and  $\log(\text{Cs-}K_d)$ :  $R^2 = 0.88$ ,  $n = 6$ ,  $P \leq 0.02$ . Additional descriptive statistics, including the regression equations, are included in Appendix B.

### 3.3 K<sub>d</sub> VALUES OF FINELY- VERSUS COARSE-GROUND SAMPLES

There were five paired samples of finely ground (10µm) and coarse ground (10,000 µm) rock material for which K<sub>d</sub> values were measured (Table 8).

**Table 8.** K<sub>d</sub> values of finely- versus coarse-ground samples (averages of two replicates).

Boring	Part. Size (µm)	Solid Phase	Co		Sr		Cs	
			K <sub>d</sub> (mL/g)	stdev	K <sub>d</sub> (mL/g)	stdev	K <sub>d</sub> (mL/g)	stdev
B-205, 98-100 bgs	10	Fracture	493.9	298.7	13.8	1.3	106.4	26.8
B-205, 98-100 bgs	10000	Fracture	74.1	15.1	1.9	1.5	3.0	0.8
B-305 (64-64.5 bgs)	10	Fracture	549.5	53.4	14.2	2.5	24.7	2.8
B-305 (64-64.5 bgs)	10000	Fracture	19.2	0.0	8.2	7.2	6.5	0.6
B-627 (66.5 -76.5 bgs)	10	Fracture	387.7	381.6	33.9	6.6	1035.1	374.6
B-627 (66.5 -76.5 bgs)	10000	Fracture	4.4	2.6	13.4	3.4	93.8	34.4
B-212/212a (86-96 bgs)	10	Fracture	735.6	162.5	34.4	7.3	110.2	25.3
B-212/212a (86-96 bgs)	10000	Fracture	24.6	3.8	0.9	0.1	2.1	0.1
B-212/212a (86-96 bgs)	10	Matrix	300.3	0.0	8.3	0.0	8.5	0.3
B-212/212a (86-96 bgs)	10000	Matrix	26.1	2.2	0.7	0.4	1.0	0.3
			<b>Mean avg</b>	<b>Mean stdev</b>	<b>Mean avg</b>	<b>Mean stdev</b>	<b>Mean avg</b>	<b>Mean stdev</b>
<b>Average</b>	10		493.4	166.0	20.9	12.3	257.0	437.4
	10000		29.7	26.3	5.0	5.6	21.3	40.6
<b>10µm/10000µm Ratio</b>			16.6		4.2		12.1	

The key findings from these samples are:

- As expected, the 10µm fraction K<sub>d</sub> values were always much greater than the corresponding 10,000µm fraction K<sub>d</sub> values. The 10µm/10,000µm ratios were 16.6 for Co, 4.2 for Sr, and 12.1 for Cs. This ranking is the same as the ranking of overall K<sub>d</sub> values.
- Ranking of overall K<sub>d</sub> values for both fine and coarse samples are: Co > Cs > Sr; except B-627 (66.5 to 76.5 bgs), which was Cs > Co > Sr. Regarding the exception, both the 10µm and 10,000µm samples followed this trend, providing added credence that this exception [B-627 (66.5 to 76.5 bgs)] was real and not some experimental artifact. Such unique sorption behavior may be attributed to the mineralogy of the B-627(66.5 to 76.5 bgs) sample compared to the other rock samples. Effects of mineralogy are discussed in Section 3.6.

Differences between matrix and fracture K<sub>d</sub> values will be discussed in the next section.

**3.4 K<sub>d</sub> VALUES OF SAMPLES RECOVERED FROM FRACTURES VERSUS FROM THE MATRIX**

Samples were collected from one rock sample at two locations, along a naturally formed fracture and within the rock matrix (away from fractures, within the bulk portion of the rock sample (Table 9).

**Table 9.** K<sub>d</sub> values of fracture vs. matrix samples (averages of two replicates).

Boring	Part. Size (μm)	Solid Phase	Co		Sr		Cs	
			K <sub>d</sub> (mL/g)	stdev	K <sub>d</sub> (mL/g)	stdev	K <sub>d</sub> (mL/g)	stdev
B-212/212a (86-96 bgs)	10	Fracture	735.6	162.5	34.4	7.3	110.2	25.3
B-212/212a (86-96 bgs)	10000	Fracture	24.6	3.8	0.9	0.1	2.1	0.1
B-212/212a (86-96 bgs)	10	Matrix	300.3	0.0	8.3	0.0	8.5	0.3
B-212/212a (86-96 bgs)	10000	Matrix	26.1	2.2	0.7	0.4	1.0	0.3

The key findings from these samples are:

- Co, Sr, and Cs K<sub>d</sub> values of finely ground (10μm) rock samples were greater in samples collected along the fracture than in comparable bulk matrix samples.
- Co and Sr K<sub>d</sub> values for coarse-ground (10,000μm) were similar between fracture and matrix materials. Cs K<sub>d</sub> values of the coarse materials were >2x greater in the fracture compared to the matrix rock.

**3.5 K<sub>d</sub> VALUES OF THE SOIL SAMPLES AND THE 10μm ROCK SAMPLES FROM THREE ROCK LITHOLOGIES**

The 10μm fraction was the most common rock size fraction analyzed. There were three rock lithologies sampled (Table 3). The only unusual sample was B-333(63-66 bgs), classified as a partially weathered rock (PWR). It was not a solid rock like the other samples. Instead it could be easily crushed by hand. This sample was crushed by hand and then the fines were ground to ~10μm, as requested in the work instructions.

**Table 10.**  $K_d$  values of 10 $\mu$ m rock samples (averages of two replicates).

Boring	Part. Size	Lithology	Solid Phase	Co		Sr		Cs	
				$K_d$ (mL/g)	stdev	$K_d$ (mL/g)	stdev	$K_d$ (mL/g)	stdev
B-305 (64-64.5 bgs)	10 $\mu$ m	Bedrock	Fracture	549.5	53.4	14.2	2.5	24.7	2.8
B-627 (66.5 -76.5 bgs)	10 $\mu$ m	Bedrock	Fracture	387.7	381.6	33.9	6.6	1035.1	374.6
B-205 (98-100 bgs)	10 $\mu$ m	Bedrock	Fracture	493.9	298.7	13.8	1.3	106.4	26.8
B-212/212a (86-96 bgs)	10 $\mu$ m	Bedrock	Fracture	735.6	162.5	34.4	7.3	110.2	25.3
B-305 (59-60 bgs)	10 $\mu$ m	MWR <sup>(a)</sup>	Fracture	788.7	399.6	83.5	24.8	140.2	91.9
B-327 (56-59 bgs)	10 $\mu$ m	MWR	Fracture	658.1	323.4	86.8	52.4	99.7	89.4
B-333 (63-65 bgs)	10 $\mu$ m	PWR	Fracture	>1433.4	682.2	134.8	31.7	730.4	8.9
B-212/212a (#18 (58.5-60 bgs)	<2mm	PWR	Soil	390.8	370.5	45.0	22.6	81.5	2.9
B-627 #14 (47-48.5bgs); #15 (53.5-55 bgs)	<2mm	PWR	Soil	1105.3	891.1	63.9	0	2240.3	942.7
B-620 #12 (38.5-40 bgs)	<2mm	Saprolite	Soil	415.9	188.5	38.5	2.5	71.0	7.4
B-627 #9 (23.5-25 bgs)	<2mm	Saprolite	Soil	1576.4	727.0	67.9	25.6	512.6	283.3
B-212/212a (#14 (48.5-50 bgs)	<2mm	Saprolite	Soil	676.1	63.7	62.0	0.3	89.3	0.2
				mean	mean	mean	mean	mean	mean
				avg	stdev	avg	stdev	avg	stdev
Soil Average	<2mm	Saprolite	Soil	889.5	609.0	56.1	15.5	224.3	249.9
Bedrock Average	10 $\mu$ m	Bedrock	Fracture	541.7	145.7	24.1	11.6	319.1	479.0
MWR Average	10 $\mu$ m	MWR	Fracture	723.4	92.4	85.2	2.3	120.0	28.6
PWR Average	10 $\mu$ m	PWR	/ Soil	>976.5	533.1	81.3	47.3	1017.4	1107
Overall Average	All	All	All	>708.6	438.7	52.2	36.5	403.2	635.5

(a) MWR = moderately weathered rock; PWR = partially weathered rock.

The key findings from this data set are:

- Lithology had a significant effect on  $K_d$  values. The ranking of the lithologies by their Co  $K_d$  values are: PWR > Saprolite > MWR > Bedrock. For Sr  $K_d$  values the ranking is: MWR = PWR > Saprolite > Bedrock. The variability for the Cs  $K_d$  values was especially large, making such a ranking inappropriate.
- Ranking elements by their average  $K_d$  values: Co > Cs > Sr. Again, difference may be attributed to unique mineralogy of the latter. As will be presented in the following section, the mineralogy varied moderately between the samples, with the most unique mineralogy belonging to the easily crushed sample, B-333(63-66 bgs) (Table 11). A discussion of how mineralogy may have influenced  $K_d$  values is presented in Section 3.6.

### 3.6 ROCK CHARACTERIZATION BY X-RAY DIFFRACTION, X-RAY FLUORESCENCE AND BET SURFACE AREA

X-ray diffraction (XRD; Table 11) X-ray fluorescence (XRF; Table 12), and BET surface area analyses (Table 13) were conducted on seven samples. XRD provides information about the mineralogy of crystalline minerals. XRF provides element concentration data of solids. BET surface area provides specific surface area data.

The actual XRD spectra are presented in Appendix B and the QA standard is presented in Appendix E (Figure 2). The minerals identified in the samples by XRD are consistent with the type of rock material known to exist at the site (Table 11). The one exception was for sample B-205 (98-100 bgs), where hematite and kaolinite were detected. These minerals are not common in diorite, but perhaps reflect the presence of some weathering products.

The mica minerals, biotite and muscovite, are present in all samples and are well known to be strong sorbing minerals of Cs (Krupka et al. 1999b). Cs sorbs strongly to these minerals because its ionic radius (as well as hydrated radius) fits very well into the interlayers of the mineral structure (especially of frayed edge sites common in the micaceous mineral, illite). Of the two samples that had the greatest  $K_d$  values, “B-627 (66.5-76.5)” and “B-627 #14 (47-48.5bgs) #15 (53.5-55 bgs),” only the former had XRD analysis conducted on it, and it contained biotite. Comparing the Cs  $K_d$  values of the 10 $\mu$ m rock samples (Table 10), the lowest value was 24.7 mL/g measured in sample B-305 (64-64.5 bgs). This sample also had some biotite, albeit less than B-627 (66.5-76.5) (See spectra in Appendix B), and also quartz, ferropargasite, and anorthite, all minerals that are not expected to have a high affinity for Cs.

Strontium is well known to sorb via cation exchange. Therefore, mineral surface charge and surface area are especially important. Comparing the Sr  $K_d$  values of the 10 $\mu$ m rock samples (Table 10), the lowest values were 14.2 and 13.8 mL/g measured in samples B-305 (64-64.5 bgs) and B-205 (98-100 bgs), respectively. The largest Sr  $K_d$  value was measured in sample B-333(63-66). XRD was performed on all three samples. It is not clear which specific mineral(s) were likely responsible for these differences. (As will be noted in the following discussions regarding surface area (Table 13), this sample had the greatest surface area, and thus this attribute, rather than the specific mineralogy, is likely responsible for the high Sr sorption by this sample.)

Cobalt also sorbs to mineral surfaces by cation exchange and via inner sphere sorption. The highest Co  $K_d$  values were measured on samples B-333 (63-65 bgs) (which also sorbed large amounts of Sr) and B-627 #9 (23.5-25 bgs). XRD was conducted on the former, but not the latter sample. Apparently the assembly of dickite, biotite, microcline and albite are strong sorbing agents of cations. Quartz has little charge or surface area and is well known to sorb solutes weakly.

The QA standards ran at the start and end of the sample XRF samples were within tolerances (Appendix E). The elemental concentrations were consistent between both replicates (Table

12). The high  $\text{Fe}_2\text{O}_3$  and  $\text{SiO}_2$  concentrations are expected for this suite of minerals identified by XRD (Table 11). The highest  $\text{Fe}_2\text{O}_3$  concentration was measured in sample B-212/212a (86-96 bgs), consistent with the mineral identification by XRD of the Fe-containing minerals, ferrosilite and biotite. The highest  $\text{K}_2\text{O}$  concentration was measured in sample B-333 (63-65 bgs), consistent with the mineral identification by XRD of the K-containing minerals, microcline and biotite,

BET sample and reference material results are presented in Table 13. It is important to note that the surface area of the samples is controlled by the mineralogy, and perhaps more importantly by how and to what extent they were ground. As such, it is important to note that the surface areas are all quite similar, i.e., within an order of magnitude of each other. This is important because it indicates that the grinding processes did not create any experimental artifacts for the  $K_d$  measurement (i.e., sorption would be expected to increase with increases in surface area, to the extent the samples were ground). The surface areas did not correlate with the lithology. The sample B-333(63-66 bgs) was the unique sample that was easy to crush by hand, much like a cemented sand sample. This sample had the highest Sr and Co  $K_d$  values, and quite high Cs  $K_d$  values (Table 10).

**Table 11. XRD of seven rock samples (10µm).**

SRNL #	Sample	Lithology	Rock type <sup>(b)</sup>	Minerals	Comments
401	B-205 (98-100 bgs)	Bedrock	Quartz diorite with quartz dikes	quartz, albite, hematite, kaolinite, biotite, hornblende	Surprised to see hematite and kaolinite in a diorite, perhaps weathering products.
403	B-212/212a (86-96 bgs)	Bedrock	Hornblend gneiss	quartz, plagioclase, magnesiohornblend, ferrosilite, anthophyllite, kaolinite, biotite	Anthophyllite is a questionable match. Ferrosilite is the best match for the pyroxene phase; Ferrohornblende for the amphibole phase
409	B-305 (64-64.5 bgs)	Bedrock	Amphibolite Schist	quartz, ferropargasite, anorthite, biotite	
411	B-305 (59-60 bgs)	MWR <sup>(a)</sup>	Granodiorite	quartz, microcline, albite, biotite, ferrohornblende	
412	B-327 (56-59 bgs)	MWR	Quartz, biotite, hornblend plagioclase gneiss	quartz, plagioclase, muscovite, kaolinite	
413	B-333 (63-65 bgs)	PWR <sup>(a)</sup>	Granodiorite	quartz, dickite, biotite, microcline, albite	Dickite is a slightly better match to the identified peaks than kaolinite; dickite exists in Graves Mt, GA.
414	B-627 (66.5 -76.5 bgs)	Bedrock	Granodiorite	quartz, clinochlore, microcline, biotite, albite	

<sup>(a)</sup> MWR = moderately weather rock ; PWR = partially weathered rock

<sup>(b)</sup> Rock type provided by Clay Sams (MACTEC, Charlotte, NC).



**Table 12.** XRF of seven rock samples (10 $\mu$ m) conducted in duplicate.

SRNL #	Sample	Lithology	Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	LOI <sup>(b)</sup>
401-a	B-205, 98-100 bgs	Bedrock	Fracture	60.01	15.48	7.23	0.114	2.27	4.44	4.29	2.07	1.21	0.624	2.19
401-b				60.10	15.52	7.23	0.112	2.26	4.44	4.28	2.06	1.22	0.576	2.21
403-a	B-212/212a (86-96 bgs)	Bedrock	Fracture	48.25	17.74	11.53	0.192	8.29	9.98	1.24	0.49	0.88	0.184	1.24
403-b				48.25	17.71	11.54	0.192	8.31	10.00	1.24	0.49	0.88	0.182	1.22
409-a	B-305 (64-64.5 bgs)	Bedrock	Fracture	55.47	15.82	9.13	0.179	4.35	9.46	3.46	0.83	0.68	0.136	0.50
409-b				55.49	15.81	9.15	0.178	4.36	9.46	3.45	0.82	0.68	0.108	0.49
411-a	B-305 (59-60 bgs)	MWR	Fracture	63.66	16.27	5.12	0.119	1.28	2.98	3.50	4.54	0.90	0.295	1.35
411-b				63.71	16.30	5.12	0.118	1.28	2.97	3.50	4.52	0.90	0.240	1.34
412-a	B-327 (56-59 bgs)	MWR	Fracture	70.75	14.98	3.01	0.087	0.95	2.41	3.03	1.95	0.53	0.049	2.25
412-b				71.00	14.92	2.96	0.087	0.91	2.39	3.01	1.92	0.52	0.091	2.21
413-a	B-333 (63-65 bgs)	PWR	Fracture	67.05	15.21	5.69	0.16	0.96	0.45	0.71	4.38	0.91	0.093	4.40
413-b				66.94	15.28	5.71	0.16	0.96	0.45	0.72	4.39	0.92	0.162	4.33
414-a	B-627 (66.5 -76.5 bgs)	Bedrock	Fracture	60.72	16.24	7.52	0.086	1.85	2.03	3.73	3.24	1.26	0.395	2.93
414-b				60.63	16.25	7.55	0.087	1.86	2.06	3.75	3.27	1.27	0.357	2.90

<sup>(a)</sup> Samples prepared by fusing 1.5 g sample, 7.5 g lithium tetraborate, 0.45 g lithium fluoride and 2 drops of 100  $\mu$ L of a 44 wt% lithium bromide solution ground in an agate mortar and pestle, and fired at 1000C for 15 min. The disks were polished to 1  $\mu$ m before running XRF.

<sup>(b)</sup> LOI = loss on ignition; LOI was done by heating a 3 g sample two times at 1000 C for 1 hr.

**Table 13.** Surface area of seven rock samples (10 $\mu$ m).

Description	Lithology	Surface Area (m <sup>2</sup> /g)
SRNL standard. Target = 2.85 $\pm$ 0.09 m <sup>2</sup> /g		2.84
B-205, 98-100 bgs	Bedrock	7.4
B-212/212a (86-96 bgs)	Bedrock	6.2
B-305 (64-64.5 bgs)	Bedrock	2.2
B-305 (59-60 bgs)	MWR	6.0
B-327 (56-59 bgs)	MWR	3.2
B-333 (63-65 bgs)	PWR	11.3
B-627 (66.5 -76.5 bgs)	Bedrock	8.8
SRNL standard. Target = 2.85 $\pm$ 0.09 m <sup>2</sup> /g		2.84

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**APPENDIX A. Detailed Description of the Materials and Methods**

## R&D Directions: K<sub>d</sub> Measurements of VC Summer Site Rock/Sediment Samples

### Objective

Measure Co, Cs, and Sr K<sub>d</sub> values of 20 rock/sediment samples collected from the VC Summer, in South Carolina. Because these K<sub>d</sub> values will be used in a reactive transport code as part of a Early Site Permit Project, all attempts will be made to make the experimental conditions of the tests representative of an accidental release. These measurements will include site-specific sediments, site-specific groundwater, and radionuclides concentrations that are in the linear sorption range. The linear sorption range is the lower range in an adsorption isotherm (x-axis: aqueous solute concentration, y-axis: sorbed concentration) before non-linearity occurs (the result of limitations of sorption sites with respect to aqueous radionuclide concentrations).<sup>1</sup> Furthermore, the sediments were pre-equilibrated with the groundwater prior to spiking with radionuclides, again to minimize “shocking the chemistry” and to provide a measure of steady state conditions, as implied by the K<sub>d</sub> construct.

The following method is taken from ASTM D 4646: Standard test method for 24-hr batch-type measurement of contaminant sorption by soils and sediments.

### Materials

1. 20 rock/sediment samples from the VC Summer site (Table 14)
2. four VC Summer site groundwater samples
3. 3 μCi source of Radionuclide Mixture Option 4 from Analytix, Inc. (<http://www.analytixinc.com/prod01.htm>)
4. 15-mL centrifuge tubes
5. sandwich baggies
6. large weighing boats
7. 0.1-μm syringe filters
8. 4-M NaOH
9. Sieves: #500 (25-μm), #400 (38-μm), #200 (75-μm), #170 (90-μm), #140 (106-μm), #120 (125-μm), #100 (150-μm), #25 (710-μm), #20 (850-μm), #18 (1000-μm), #16 (1180-μm), #14 (1400-μm).

### Methods

1. **Preparing crush rock samples:** The rock will come in two-inch (5-cm) diameter cores and will need to be crushed into 6 different sizes.

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<sup>1</sup> From a risk assessment point of view, it is important not to promote precipitation of the radionuclide by introducing too high a spike concentration into the sediment-groundwater system. This may be construed by regulators and reviewers of the ESP as a **non-conservative** estimate of the sorption capacity of the sediment for the radionuclide. Additionally, this may result in the precipitation of radionuclide to glassware, an experimental artifact that would be difficult (costly) to identify.

- a. Using the Westward 6-ton hydraulic press, cut ~0.5-cm thin slices from the various core samples as described in the file associated with the Statement of Work “SCEG Kd Sample Location 092106rev\_mfc.xls”. These slices will then be used to create all the other size particles.
  - b. *1-cm<sup>2</sup> sized particles*: Using a chisel and hammer, break up thin sections from Step #1a to approximately 1-cm<sup>2</sup>. Make a dozen pieces. We will select only 1 or 2 pieces per sorption test, depending on weight. (Samples 402, 404, 410, 415, 408)
  - c. *10- $\mu$ m<sup>2</sup> sized particles*: Using a chisel and hammer, break up thin sections from Step #1a to ~1-cm<sup>2</sup> pieces. Place approximately 20 g in Spex 8510 Shatter box for 5 minutes, grind, and pass through #400 (38- $\mu$ m) and #500 (25- $\mu$ m) sieves. Save the 25 to 38  $\mu$ m fraction. (Samples 401, 403, 409, 411, 412, 414).
  - d. *100- and 1000- $\mu$ m<sup>2</sup> sized particles*. Using a chisel and hammer, break up thin sections from Step #1a to ~1-cm<sup>2</sup> pieces. Place ~100-g of these pieces in a jaw crusher (Retsch Jaw Crusher Type BB51 with tungsten carbide plates) for 10 minutes, then take the <1.4-mm fraction (#14 sieve) and place them in a shatter box (Spex 8510 Shatterbox) for 10 seconds. Pass particles through sieves (1400, 1180, 1000, 850, 710, 150, 125, 106, 90, and 75  $\mu$ m) and shake on an AS200 Analytical Sieve Shaker (Type AS200 Digit). Take the <75- $\mu$ m (#200 sieve) material and put in a Spex 8000 mixer/mill for 10 seconds and then in a Spex 8510 Shatter box for 5 min. Then pass this material through a 25- $\mu$ m (#500 sieve). Depending on the percentages of materials in each size fraction, we will combine fractions to define the “100- $\mu$ m<sup>2</sup>” and the “1000- $\mu$ m<sup>2</sup>” fractions. (Samples #406 and #407).
2. **Preparing sediment (jar) samples**: Mix field-moist sediments in jars. Transfer to a labeled aluminum weighing boats and place in oven at 85°C until no weight change is noted. (Samples 416, 417, 418, 419, 420)
  3. Record “Tube Tare (g)” weight (without cap) of labeled 15-mL centrifuge tube.
  4. Add 0.5±0.01-g soil to each tube. Weigh and record “Soils (g)”
  5. Add 12-mL of appropriate groundwater to each tube as defined in Table 1. Note there are 4 different types of groundwater. Put on shaker for overnight. Let sit for 1 hr. Decant liquid. If solids do not separate from the liquid, centrifuge at 15 min 6000 rpm. Then decant liquid. Throw away liquid. Error on the side of leaving liquid in tube rather than accidentally losing clays down the drain. Add 10 mL of groundwater to each tube. Also add 10-mL groundwater to the “No-soil Controls. Record weight of each tube in “Tube + soils + Equil + GW (g)”
  6. Add 74- $\mu$ L 4-M NaOH to each tube. This is to compensate for the acidity in the radionuclide spike solution.<sup>2</sup>

<sup>2</sup> Initially, 64  $\mu$ L of 4M NaOH was added to each tube. pH measured for tube #138 (sand) was ~3; pH for tube #130 (clay) was ~2-3. For tube #110 (clay), another 50  $\mu$ L of 4M NaOH was added; pH was then ~12-13. For tubes #130 (clay) and #138 (sand), another 10  $\mu$ L of 4M NaOH was added; pH was ~5 for tube #130 and ~6 for tube #138. 100  $\mu$ L of 1M HCl was added to tube #110 (clay) in an effort to bring down the pH; after the addition, pH was ~10. Based on these pH tests, it was decided to add 10  $\mu$ L of 4M NaOH to the other tubes to make a total of 74  $\mu$ L of 4M NaOH added to each tube (excluding tube #110).

7. Measure pH of 402-A, 405-A, 416-A, and 417-A.
8. Move rack of tubes to hood. Add 80- $\mu$ L of radionuclide spike solution to each tube. Also add this to the No-soil Controls, Tube #421-A, -B, &-C. Gently shake or swirl tube sediment and solution in each tube.
9. Using litmus paper, test the pH of a few samples (e.g., 402-A, 405-A, 416-A, and 417-A).
10. Leave samples in rad hood for minimum of 2 days.
11. Collect liquids by drawing liquids into a syringe and then passing liquid through a 0.1- $\mu$ m filter.
12. Submit to Analytical Development Section for gamma spec analysis, rush order. Note in your order that the only important rads are  $^{60}\text{Co}$ ,  $^{90}\text{Sr}$ , and  $^{137}\text{Cs}$ .

**Hazards Assessment Package:**

SRNL-EST-2006-00-93: for everything except for breaking up rocks.  
SRNL-ADD-2006-00111: for breaking up rocks (step 1 in procedure).

**Hazards:**

Radionuclides, ~3 mL of strong acids, ~3 mL of strong base

**Hazards Mitigation:**

Radionuclides: Follow training of Rad Worker II,  
Strong Acid: wear appropriate gloves and as always protective eye ware  
Strong Base: wear appropriate gloves and as always protective eye ware,

Table 14. Sediment descriptions and weights during Kd measurements.

Tube #	Boring	Part. Size (µm)	Rep	Ground-water	Tube Tare (g)	Soil (g)	Tube + Soil + Equil + GW (g)
Method Step				(5)	(3)	(4)	(6)
401-A	B-205, 98-100 bgs	10	1	205a			
401-B			2				
402-A		10,000	1	205a			
402-B			2				
403-A	B-212/212a (86-96 bgs)	10	1	205a			
403-B			2				
404-A		10,000	1	205a			
404-B			2				
405-A		10	1	205a			
405-B			2				
406-A		100	1	205a			
406-B			2				
407-A		1000	1	205a			
407-B			2				
408-A		10,000	1	205a			
408-B			2				
409-A	B-305 (64-64.5 bgs)	10	1	305a			
409-B			2				
410-A		10,000	1	305a			
410-B			2				
411-A	B-305 (59-60 bgs)	10	1	327			
411-B			2				



Tube #	Boring	Part. Size (µm)	Rep	Ground-water	Tube Tare (g)	Soil (g)	Tube + Soil + Equil + GW (g)
Method Step					(3)	(4)	(6)
412-A	B-327 (56-59 bgs)	10	1	327			
412-B			2				
413-A	B-333 (63-65 bgs)	10	1	327			
413-B			2				
414-A	B-627 (66.5 -76.5 bgs)	10	1	305a			
414-B			2				
415-A		10,000	1	305a			
415-B			2				
416-A	B-212/212a (#14 (48.5-50 bgs)	Soil	1	212			
416-B			2				
417-A	B-212/212a (#18 (48.5-50 bgs)	Soil	1	212			
417-B			2				
418-A	B-620 #12 (38.5-40 bgs)	Soil	1	212			
418-B			2				
419-A	B-627 #9 (23.5-25 bgs)	Soil	1	327			
419-B			2				
420-A	B-627 #14 (47-48.5bgs); #15 (53.5-55 bgs)		1	327			
420-B			2				
421-A	No-soil control		1	205a			
421-B			2				
421-C			3				

**APPENDIX B. Raw Kd Data and Statistics**

**Table 15. Raw Data Used to Calculate  $K_d$  values.**

Tube #	Boring	Part. Size (microns)	Rep	Groundwater	Soil (g)	Aqueous Vol (mL)	Co (dpm/mL)	Sr (dpm/mL)	Cs (dpm/mL)	Co Kd (mL/g)	Sr Kd (mL/g)	Cs Kd (mL/g)
401-A	B-205, 98-100 bgs	10	1	205a	0.4983	10.0458	1.02E+02	1.61E+04	3.52E+03	282.6	12.9	125.3
401-B			2		0.4994	10.0232	4.24E+01	1.52E+04	4.74E+03	705.1	14.8	87.5
402-A		10,000	1	205a	1.8253	10.0126	1.22E+02	2.29E+04	1.75E+04	63.4	0.8	2.5
402-B			2		1.794	9.9656	9.42E+01	1.72E+04	1.55E+04	84.8	3.0	3.5
403-A	B-212 /212a (86-96 bgs)	10	1	205a	0.5002	10.1285	4.84E+01	1.08E+04	4.57E+03	620.7	29.2	92.3
403-B			2		0.4935	10.1001	3.60E+01	9.01E+03	3.50E+03	850.5	39.5	128.1
404-A		10,000	1	205a	1.9086	10.0281	2.96E+02	2.22E+04	1.83E+04	21.9	1.0	2.0
404-B			2		1.287	10.0589	3.41E+02	2.37E+04	1.98E+04	27.3	0.9	2.2
405-A		10	1	205a	0.5082	10.0837	3.53E+02	2.10E+04	1.77E+04	66.3*	5.1*	8.6
405-B			2		0.4997	10.0557	9.62E+01	1.87E+04	1.80E+04	300.3	8.3	8.3
406-A		100	1	205a	0.5029	10.0184	8.78E+01	1.95E+04	1.63E+04	327.7	7.0	11.1
406-B			2		0.4948	9.9967	7.52E+01	1.88E+04	1.63E+04	391.4	8.2	11.3
407-A		1000	1	205a	0.5043	10.0076	8.65E+01	2.30E+04	2.20E+04	331.6	2.9	3.1
407-B			2		0.5067	9.9719	1.56E+02	2.43E+04	2.22E+04	173.6	1.7	2.8
408-A		10,000	1	205a	1.3694	9.9514	3.19E+02	2.32E+04	2.19E+04	27.6	1.0	1.2
408-B			2		1.4298	9.9106	3.38E+02	2.48E+04	2.29E+04	24.5	0.4	0.8
409-A	B-305 (64-64.5 bgs)	10	1	305a	0.492	10.3344	6.04E+01	1.66E+04	1.22E+04	511.8	12.4	22.7
409-B			2		0.4939	10.0486	5.13E+01	1.48E+04	1.10E+04	587.2	15.9	26.6

Tube #	Boring	Part. Size (microns)	Rep	Groundwater	Soil (g)	Aqueous Vol (mL)	Co (dpm/mL)	Sr (dpm/mL)	Cs (dpm/mL)	Co Kd (mL/g)	Sr Kd (mL/g)	Cs Kd (mL/g)
410-A		10,000	1	305a	0.9814	9.9052	2.32E+03	1.14E+04	1.51E+04	-3.4*	13.3	6.9
410-B			2		1.228	9.8521	4.52E+02	1.90E+04	1.45E+04	19.2	3.1	6.0
411-A	B-305 (59-60 bgs)	10	1	327	0.494	9.9629	5.87E+01	6.18E+03	5.37E+03	506.2	66.0	75.2
411-B			2		0.5025	10.0217	2.80E+01	4.35E+03	2.25E+03	1071.3	101.1	205.2
412-A	B-327 (56-59 bgs)	10	1	327	0.4968	10.0296	3.41E+01	3.70E+03	2.80E+03	886.8	123.9	162.9
412-B			2		0.4996	9.7496	6.66E+01	7.43E+03	8.85E+03	429.4	49.8	36.5
413-A	B-333 (63-65 bgs)	10	1	327	0.4993	10.2923	3.25E+01	3.06E+03	7.03E+02	951.1	157.2	724.2
413-B			2		0.5015	10.3322	< 1.63E+01	4.09E+03	6.91E+02	> 1915.8	112.4	736.7
414-A	B-627 (66.5 -76.5 bgs)	10	1	305a	0.502	10.1386	2.24E+02	9.07E+03	6.49E+02	117.9	38.6	770.2
414-B			2		0.503	10.1697	4.57E+01	1.08E+04	3.89E+02	657.6	29.2	1299.9
415-A		10,000	1	305a	1.6772	9.9074	7.46E+02	9.23E+03	1.99E+03	6.2	11.0	69.5
415-B			2		1.1186	9.8384	1.19E+03	9.43E+03	1.76E+03	2.5	15.8	118.1
416-A	B-212/212a (#14 (48.5-50 bgs)	Soil	1	212	0.5082	10.4313	4.24E+01	6.55E+03	4.74E+03	721.1	62.2	89.5
416-B			2		0.4987	10.0924	4.76E+01	6.51E+03	4.70E+03	631.1	61.8	89.1
417-B			2		0.5033	10.1283	2.07E+02	1.08E+04	5.12E+03	652.8	29.1	79.7
418-A	B-620 #12 (38.5-40 bgs)	Soil	1	212	0.5402	10.0511	5.02E+01	8.34E+03	5.60E+03	128.8	40.3	65.8
418-B			2		0.4979	9.9322	1.01E+02	9.29E+03	5.27E+03	549.2	36.7	76.2
419-A	B-627 #9 (23.5-25 bgs)	Soil	1	327	0.5026	10.4105	2.93E+01	7.76E+03	1.58E+03	282.6	49.8	312.3

Tube #	Boring	Part. Size (microns)	Rep	Groundwater	Soil (g)	Aqueous Vol (mL)	Co (dpm/mL)	Sr (dpm/mL)	Cs (dpm/mL)	Co Kd (mL/g)	Sr Kd (mL/g)	Cs Kd (mL/g)
419-B			2		0.5076	10.6337	1.52E+01	5.17E+03	7.25E+02	2090.5	86.0	713.0
420-A	B-627 #14 (47- 48.5bgs); #15 (53.5- 55 bgs)		1	327	0.505	10.4612	6.40E+01	6.46E+03	3.30E+02	475.2	63.9	1573.7
420-B			2		0.4985	10.4005	1.82E+01	5.37E+02	1.81E+02	1735.3	1004.8*	2907.0
421-A	No-soil control		1	205a	0	10.1762	1.71E+02	3.03E+04	2.90E+04			
421-B			2		0	9.8936	4.23E+03	1.79E+04	1.69E+04			
421-C			3		0	9.8578	1.95E+02	3.10E+04	3.03E+04			

\* Suspect analytical results, this replicate not included in  $K_d$  calculations.

Table 16. Regression Analysis of Particle Size vs. Co, Sr, and Cs K<sub>d</sub> Values

**Co K<sub>d</sub> (mL/g) vs. Particle Size (Table 7; 100nm to 10,000 nm)**

<i>Regression Statistics</i>	
Multiple R	0.916345
R Square	0.839689
Adjusted R Square	0.799611
Standard Error	72.31891
Observations	6

ANOVA					<i>Significance F</i>
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>F</i>
Regression	1	109576.7	109576.7	20.95148	0.010204
Residual	4	20920.1	5230.024		
Total	5	130496.8			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	324.5962	38.32658	8.46922	0.001065	218.1844	431.0081
<b>Co K<sub>d</sub> (mL/g)</b>	-0.03023	0.006605	-4.57728	0.010204	-0.04857	-0.01189

**Cs K<sub>d</sub> (mL/g) vs. Particle Size (Table 7; 100nm to 10,000 nm)**

<i>Regression Statistics</i>	
Multiple R	0.710299
R Square	0.504525
Adjusted R Square	0.380656
Standard Error	3853.491
Observations	6

ANOVA					<i>Significance F</i>
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>F</i>
Regression	1	60482428	60482428	4.073057	0.113733
Residual	4	59397572	14849393		
Total	5	1.2E+08			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	7305.441	2380.422	3.068969	0.03733	696.3161	13914.57
<b>Cs K<sub>d</sub> (mL/g)</b>	-715.775	354.6632	-2.01818	0.113733	-1700.48	268.9302

**Sr-Kd (mL/g) vs. Particle Size (Table 7; 100nm to 10,000 nm)**

<i>Regression Statistics</i>	
Multiple R	0.726353
R Square	0.527588
Adjusted R Square	0.409486
Standard Error	3762.735
Observations	6

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	63247300	63247300	4.467193	0.102078
Residual	4	56632700	14158175		
Total	5	1.2E+08			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	7560.588	2386.64	3.16788	0.033927	934.1994	14186.98
<b>Sr Kd (mL/g)</b>	-1087.48	514.5202	-2.11357	0.102078	-2516.02	341.0638

**Log(Sr Kd) vs. Particle Size (Table 7; 100nm to 10,000 nm)**

<i>Regression Statistics</i>	
Multiple R	0.983973
R Square	0.968203
Adjusted R Square	0.960254
Standard Error	976.1975
Observations	6

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	1.16E+08	1.16E+08	121.7973	0.000383
Residual	4	3811846	952961.6		
Total	5	1.2E+08			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	21788.96	1686.815	12.91722	0.000207	17105.6	26472.32
<b>logSr Kd</b>	-8626.23	781.6319	-11.0362	0.000383	-10796.4	-6456.07

**Log(Cs Kd) vs. Particle Size (Table 7; 100nm to 10,000 nm)**

<i>Regression Statistics</i>	
Multiple R	0.877924
R Square	0.77075
Adjusted R Square	0.713438
Standard Error	2621.187
Observations	6

<i>ANOVA</i>					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	92397512	92397512	13.4482	0.021444
Residual	4	27482488	6870622		
Total	5	1.2E+08			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>
Intercept	8096.537	1606.996	5.038306	0.007291	3634.791	12558.28
<b>logCs Kd</b>	-8846.51	2412.347	-3.66718	0.021444	-15544.3	-2148.75



**Table 17.** XRF raw data (data used to create Table 12).

XRF VC Summer Samples											
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	LOI
236511a	60.01	15.48	7.23	0.114	2.27	4.44	4.29	2.07	1.21	0.624	2.19
236511b	60.10	15.52	7.23	0.112	2.26	4.44	4.28	2.06	1.22	0.576	2.21
236512a	48.25	17.74	11.53	0.192	8.29	9.98	1.24	0.49	0.88	0.184	1.24
236512b	48.25	17.71	11.54	0.192	8.31	10.00	1.24	0.49	0.88	0.182	1.22
236513a	55.47	15.82	9.13	0.179	4.35	9.46	3.46	0.83	0.68	0.136	0.50
236513b	55.49	15.81	9.15	0.178	4.36	9.46	3.45	0.82	0.68	0.108	0.49
236514a	63.66	16.27	5.12	0.119	1.28	2.98	3.50	4.54	0.90	0.295	1.35
236514b	63.71	16.30	5.12	0.118	1.28	2.97	3.50	4.52	0.90	0.240	1.34
236515a	70.75	14.98	3.01	0.087	0.95	2.41	3.03	1.95	0.53	0.049	2.25
236515b	71.00	14.92	2.96	0.087	0.91	2.39	3.01	1.92	0.52	0.091	2.21
236516a	67.05	15.21	5.69	0.16	0.96	0.45	0.71	4.38	0.91	0.093	4.40
236516b	66.94	15.28	5.71	0.16	0.96	0.45	0.72	4.39	0.92	0.162	4.33
236517a	60.72	16.24	7.52	0.086	1.85	2.03	3.73	3.24	1.26	0.395	2.93
236517b	60.63	16.25	7.55	0.087	1.86	2.06	3.75	3.27	1.27	0.357	2.90

**Table 18.** Raw BET data (used to make Table 13).

---

David Missimer  
3/20/2007

Subject: BET results

Dan

Your samples were all degassed at 200 C for 3 hrs. The standard was degassed at 220C for 4hrs.  
The surface area for the standard is 2.85 +/- 0.09 m<sup>2</sup>/g.  
We ran a standard before and after your standards.

We are the results:

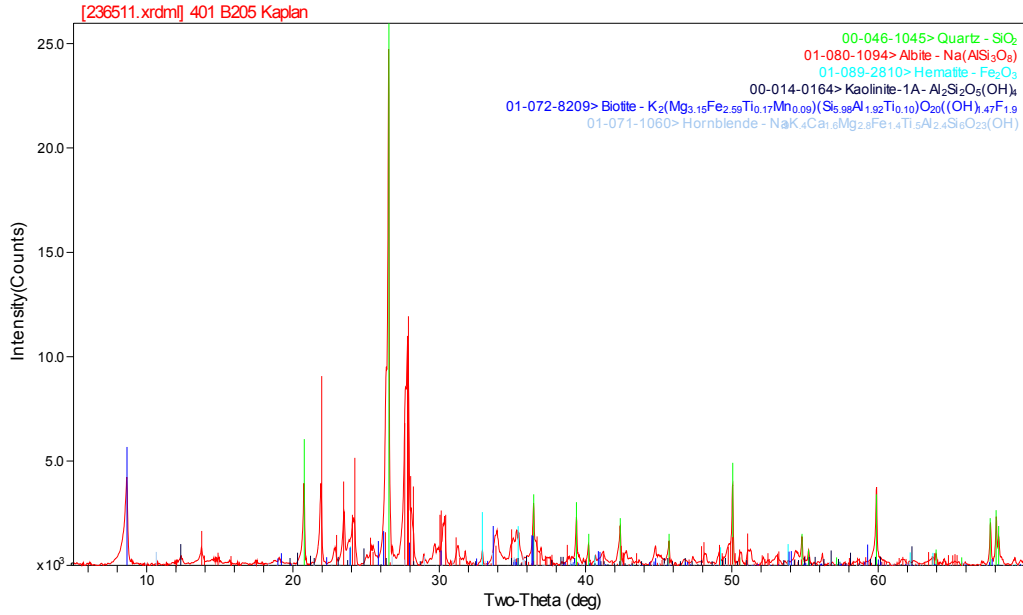
		Sample #
Standard	2.84 m <sup>2</sup> /g	
236511	7.4 m <sup>2</sup> /g	401
236512	6.2 m <sup>2</sup> /g	403
236513	2.2 m <sup>2</sup> /g	409
236514	6.0 m <sup>2</sup> /g	411
236515	3.2 m <sup>2</sup> /g	412
236516	11.3 m <sup>2</sup> /g	413
236517	8.8 m <sup>2</sup> /g	414
Standard	2.84 m <sup>2</sup> /g	

---

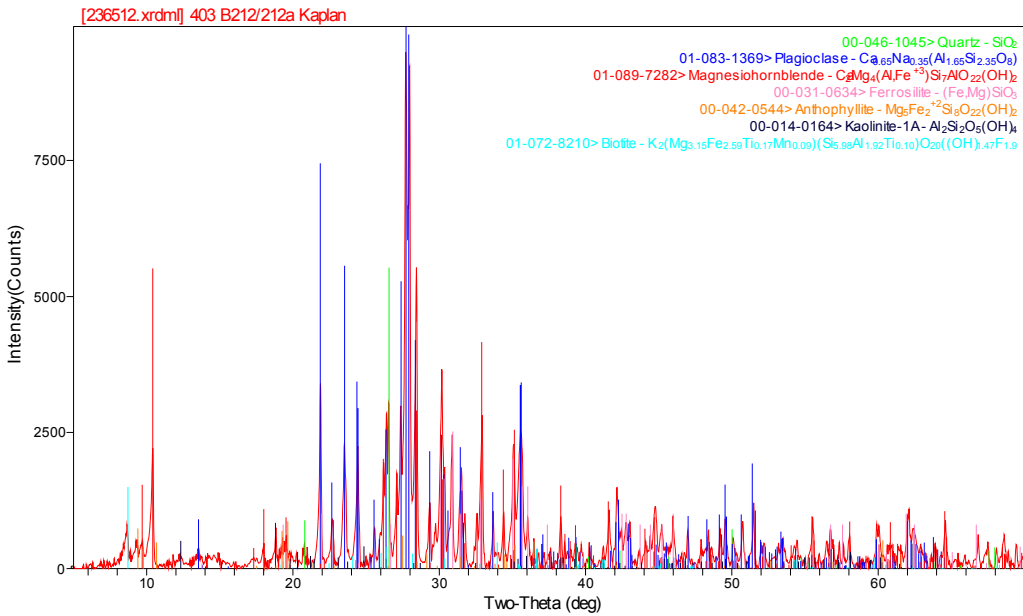
## **APPENDIX C. XRD Diffractograms**

Figure 1. XRD diffractograms.

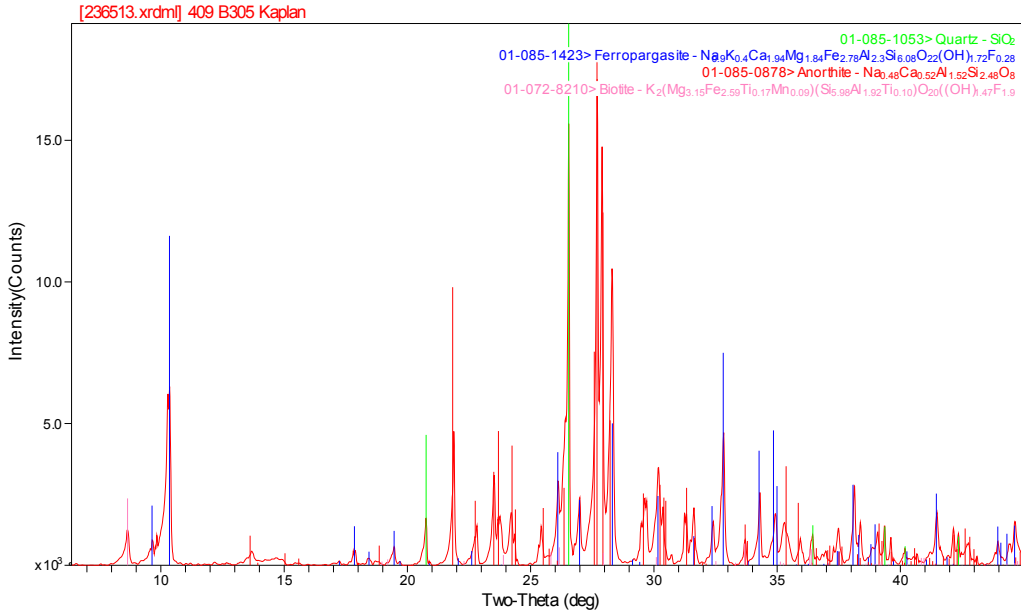
B-205, 98-100 bgs



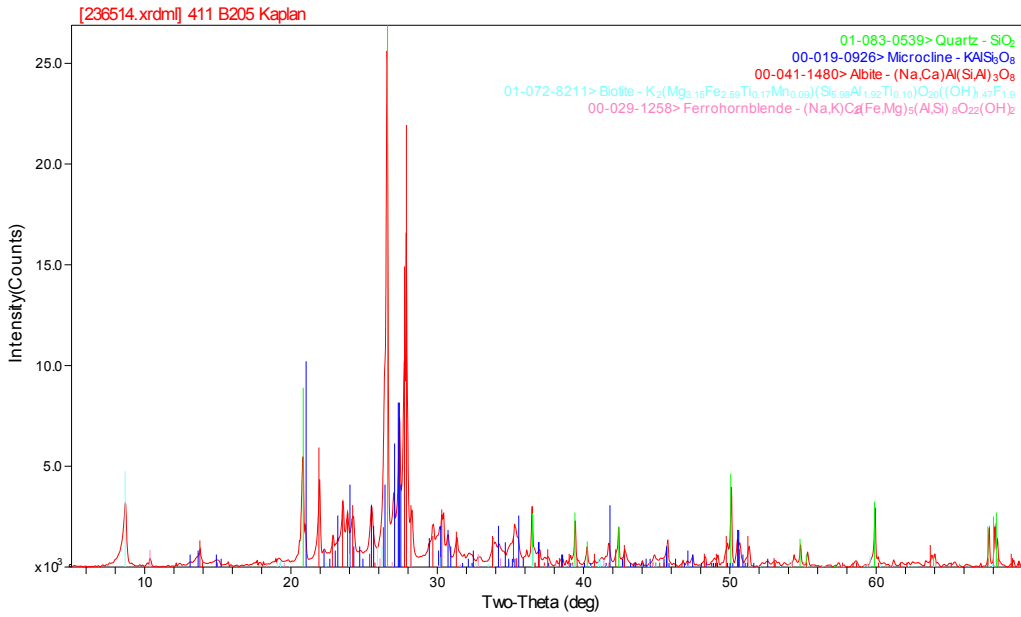
B-212/212a (86-96 bgs)



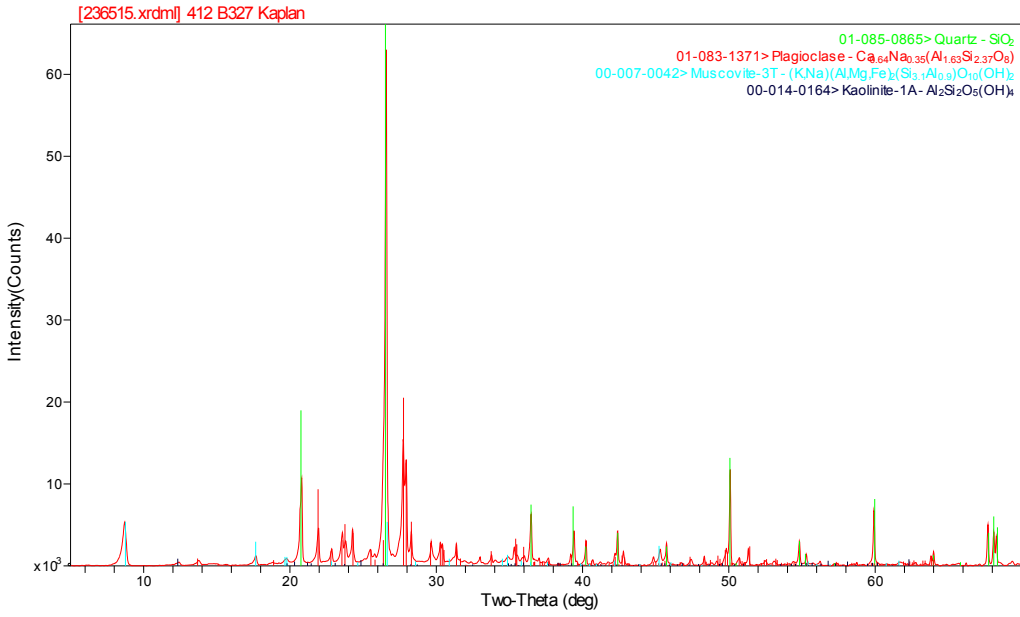
**B-305 (64-64.5 bgs)**



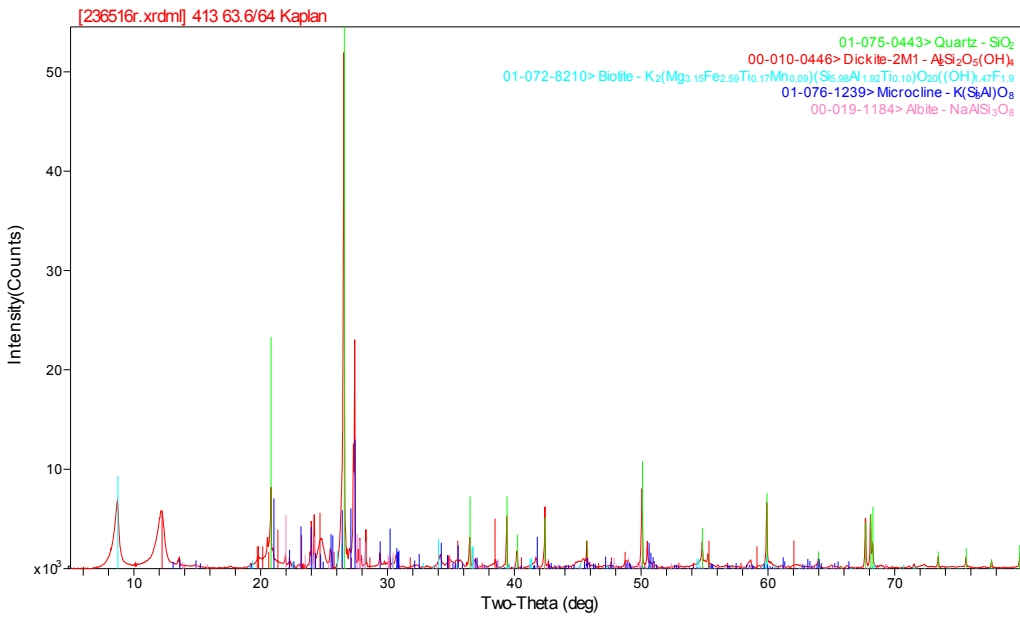
**B-305 (59-60 bgs)**



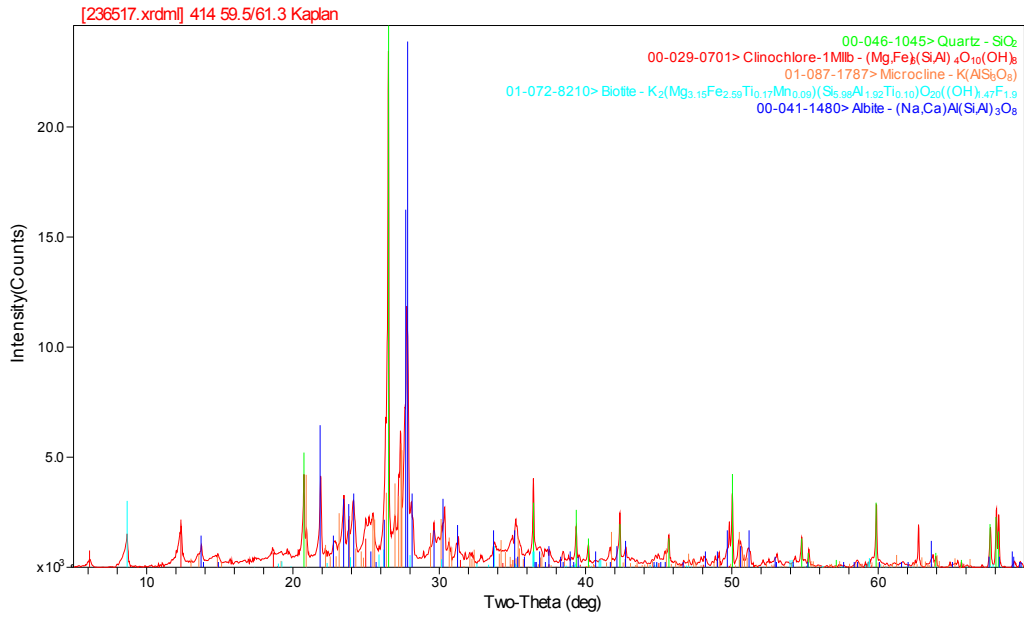
**B-327 (56-59 bgs)**



**B-333 (63-65 bgs)**



B-627 (66.5 -76.5 bgs)



**APPENDIX D. Photographs of selected samples after crushing**



**Table 19.** Photographs of selected samples after grinding. Clockwise starting from top left, using SRNL's labeling system: 408 (10,000 $\mu$ m), 402 (10,000 $\mu$ m), and 404 (10,000 $\mu$ m) and 403 (10 $\mu$ m).

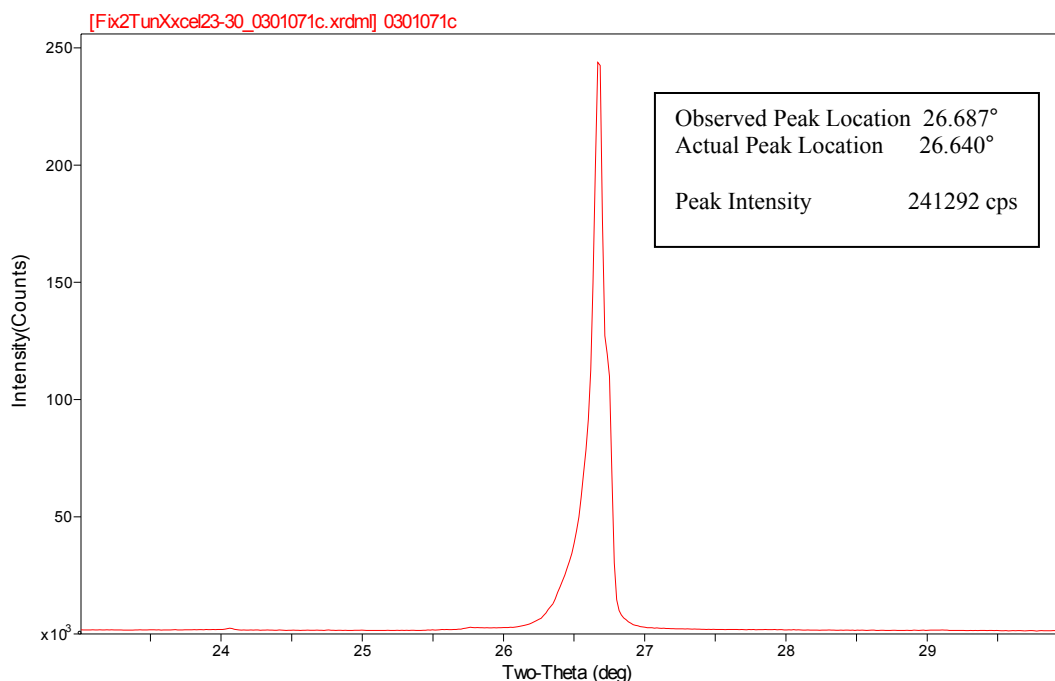


**APPENDIX E. QA Results for Gamma Analyses, XRD, and XRF**

## XRD Analyses QA

One standard was run at the start of the XRD analyses, which were completed all in one day. The standard was provided by the manufacturer of the XRD instrument, PANalytical, Lelyweg 1, 7602 EA ALMELO, The Netherlands; <http://www.panalytical.com/>. To check that the instrument is operating correctly, two parameters are checked: the peak location ( $26.640^\circ$ ) and the peak intensity. Peak intensity does not have a critical value; instead this value is compared to other values previously measured from this sample. When it is  $\sim 20\%$  of the original value (when the radiation source used in the XRD was new) than it indicates that the radiation source needs to be replaced. In this case the peak intensity denoted in Figure 2 indicated  $<5\%$  reduction, indicating that the gamma source did not need replacing.

**Figure 2.** XRD diffractogram of microcrystalline quartz standard (novaculite).



### Gamma Analyses QA

Internal (SRNL) QA standards are measured each morning prior to analyzing any samples. All analyses for this project were conducted on four days, March 26, 27, 28, and 29, 2007. Following are the QA print outs for each morning's QA run. As noted on the bottom of each page, a mark in the "Deviation/Flags" column of "In" means the measured value needs to be "investigated", but the instrument is within specifications of the standard. A "Deviation/Flags" comment of "Ac" means the measured value is outside the acceptable range and "action" is required. During the four days, there were a total of nine "investigate" flags and zero "action" flags. This indicates that all measurement QA standards were within specification. There are 2 pages of reports per day.

**Figure 3.** Gamma analyses QA reports for March 26, 27, 28, and 29, 2007.

Last Measurement Q.A. Report 3/23/07 7:15:13 AM Page 1

\*\*\*\*\*  
 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/23/07 7:15:13 AM

*MM  
 3-23-07*

QA File: C:\LOWLEV\LLDQA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/23/07 6:59:36 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.2 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags			
		< LU	: SD	: UD	: BS >
Am-241 Peak energy [0.00+/-0.00]	5.9562E+001		0.00		
Cs-137 Peak energy [0.00+/-0.00]	6.6189E+002	<	:	:	>
Co-60 Peak energy [0.00+/-0.00]	1.3328E+003	<	:	:	>
Am-241 Peak FWHM [ 7.8680E-001+/- 0.039]	7.4079E-001	<	:	:	>
Cs 137 Peak FWHM [ 1.4132E+000+/- 0.052]	1.4455E+000	<	:	:	>
Co-60 Peak FWHM [ 2.0209E+000+/- 0.094]	1.9896E+000	<	:	:	>
Am-241 activity [0.00+/-0.00]	1.8677E-002		0.00		
Co-60 activity [0.00+/-0.00]	7.6834E-003	<	:	:	>
Cs-137 activity [0.00+/-0.00]	1.4755E-002	<	:	:	>
		<	:	:	>

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 3/23/07 7:15:12 AM Page 1

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 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/23/07 7:15:12 AM

*MM*  
*3-23-07*

QA File: C:\SPC2\SPC2QA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/23/07 6:59:58 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.8 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags			
		< LU	: SD	: UD	: BS >
Am-241 Peak energy [0.00+/-0.00]	5.9523E+001	<	:	:	>
Cs-137 Peak energy [0.00+/-0.00]	6.6148E+002	<	:	:	>
Co-60 Peak energy [0.00+/-0.00]	1.3323E+003	<	:	:	>
Am-241 Peak FWHM [0.00+/-0.00]	7.4621E-001	<	:	:	>
Cs-137 Peak FWHM [0.00+/-0.00]	1.3232E+000	<	:	:	>
Co-60 Peak FWHM [0.00+/-0.00]	1.7211E+000	<	:	:	>
Am-241 activity [0.00+/-0.00]	1.8075E-002	<	:	:	>
Co-60 activity [0.00+/-0.00]	1.1213E-002	<	:	:	>
Cs-137 activity [0.00+/-0.00]	1.5771E-002	<	:	:	>

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 3/26/07 8:06:29 AM Page 1

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 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/26/07 8:06:29 AM

*MH*  
*3-26-07*

QA File: C:\LOWLEV\LLDQA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/26/07 7:51:20 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.2 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
Am-241 Peak energy [0.00+/-0.00]	5.9541E+001	0.00 < : : : >
Cs-137 Peak energy [0.00+/-0.00]	6.6191E+002	0.00 < : : : >
Co-60 Peak energy [0.00+/-0.00]	1.3329E+003	0.00 < : : : >
Am-241 Peak FWHM [ 7.8666E-001+/- 0.039]	7.7686E-001	-2.5013E-001 < : : : >
Cs-137 Peak FWHM [ 1.4133E+000+/- 0.052]	1.4630E+000	9.5310E-001 < : : : >
Co-60 Peak FWHM [ 2.0208E+000+/- 0.094]	2.0185E+000	-2.4748E-002 < : : : >
Am-241 activity [0.00+/-0.00]	1.7718E-002	0.00 < : : : >
Co-60 activity [0.00+/-0.00]	7.7060E-003	0.00 < : : : >
Cs-137 activity [0.00+/-0.00]	1.4545E-002	0.00 < : : : >

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report      3/26/07 8:06:06 AM      Page 1

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 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/26/07 8:06:06 AM

*MV*  
 3-26-07

QA File:                    C:\SPC2\SPC2QA00.QAF  
 Sample ID:                QA  
 Sample Quantity:        1.0000E+000  
 Measurement Date:      3/26/07 7:50:55 AM  
 Elapsed Live Time:     900.0 seconds  
 Elapsed Real Time:     902.7 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
Am-241 Peak energy [0.00+/-0.00]	5.9527E+001	< : : >
Cs-137 Peak energy [0.00+/-0.00]	6.6146E+002	< : : >
Co-60 Peak energy [0.00+/-0.00]	1.3322E+003	< : : >
Am-241 Peak FWHM [0.00+/-0.00]	7.1975E-001	< : : >
Cs-137 Peak FWHM [0.00+/-0.00]	1.2805E+000	< : : >
Co-60 Peak FWHM [0.00+/-0.00]	1.6491E+000	< : : >
Am-241 activity [0.00+/-0.00]	1.6708E-002	< : :In : >
Co-60 activity [0.00+/-0.00]	1.0791E-002	< : : : >
Cs-137 activity [0.00+/-0.00]	1.5487E-002	< : :In : >

Flags Key:    LU = Lower/Upper Bounds Test    (Ab = Above, Be = Below)  
               SD = Sample Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               UD = User Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               BS = Measurement Bias Test        (In = Investigate, Ac = Action)



Last Measurement Q.A. Report                      3/27/07 7:00:30 AM                      Page 1

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 \*\*\*\*\* G E N I E Q U A L I T Y A S S U R A N C E \*\*\*\*\*  
 \*\*\*\*\*

Last Results Report  
 3/27/07 7:00:30 AM

MM  
 3-27-07

QA File:    C:\LOWLEV\LLDQA00.QAF  
 Sample ID:    QA  
 Sample Quantity:                                   1.0000E+000  
 Measurement Date:                                 3/27/07 6:45:19 AM  
 Elapsed Live Time:                                900.0 seconds  
 Elapsed Real Time:                                902.2 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
Am-241 Peak energy [0.00+/-0.00]	5.9591E+001	0.00 < : : >
Cs-137 Peak energy [0.00+/-0.00]	6.6190E+002	0.00 < : : >
Co-60 Peak energy [0.00+/-0.00]	1.3328E+003	0.00 < : : >
Am-241 Peak FWHM [ 7.8664E-001+/- 0.039]	7.8952E-001	7.3377E-002 < : : >
Cs-137 Peak FWHM [ 1.4134E+000+/- 0.052]	1.4789E+000	1.2572E+000 < : : >
Co-60 Peak FWHM [ 2.0208E+000+/- 0.094]	1.8772E+000	-1.5333E+000 < : : >
Am-241 activity [0.00+/-0.00]	1.7601E-002	0.00 < : : >
Co-60 activity [0.00+/-0.00]	7.5799E-003	0.00 < : : >
Cs-137 activity [0.00+/-0.00]	1.4397E-002	0.00 < : : >

Flags Key:    LU = Lower/Upper Bounds Test                      (Ab = Above, Be = Below)  
                   SD = Sample Driven N-Sigma Test                (In = Investigate, Ac = Action)  
                   UD = User Driven N-Sigma Test                    (In = Investigate, Ac = Action)  
                   BS = Measurement Bias Test                        (In = Investigate, Ac = Action)

Last Measurement Q.A. Report      3/27/07 7:00:51 AM      Page 1

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 \*\*\*\*\* G E N I E   Q U A L I T Y   A S S U R A N C E \*\*\*\*\*  
 \*\*\*\*\*

Last Results Report  
 3/27/07 7:00:51 AM

*11*  
*3-27-07*

QA File:                    C:\SPC2\SPC2QA00.QAF  
 Sample ID:                QA  
 Sample Quantity:        1.0000E+000  
 Measurement Date:      3/27/07 6:45:42 AM  
 Elapsed Live Time:     900.0 seconds  
 Elapsed Real Time:     902.8 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags			
		< LU	: SD	: UD	: BS >
Am-241 Peak energy [0.00+/-0.00]	5.9550E+001	<	:	:	>
Cs-137 Peak energy [0.00+/-0.00]	6.6158E+002	<	:	:	>
Co-60 Peak energy [0.00+/-0.00]	1.3324E+003	<	:	:	>
Am-241 Peak FWHM [0.00+/-0.00]	7.9467E-001	<	:	:	>
Cs-137 Peak FWHM [0.00+/-0.00]	1.2833E+000	<	:	:	>
Co-60 Peak FWHM [0.00+/-0.00]	1.7274E+000	<	:	:	>
Am-241 activity [0.00+/-0.00]	1.8215E-002	<	:	:	>
Co-60 activity [0.00+/-0.00]	1.1085E-002	<	:	:	>
Cs-137 activity [0.00+/-0.00]	1.5708E-002	<	:	:	>

Flags Key:    LU = Lower/Upper Bounds Test    (Ab = Above, Be = Below)  
               SD = Sample Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               UD = User Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               BS = Measurement Bias Test        (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 3/28/07 9:06:08 AM Page 1

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 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/28/07 9:06:08 AM

*MH*  
*3-28-07*

QA File: C:\SPC2\SPC2QA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/28/07 8:50:57 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.8 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
Am-241 Peak energy [0.00+/-0.00]	5.9584E+001	< : 0.00 : >
Cs-137 Peak energy [0.00+/-0.00]	6.6159E+002	< : 0.00 : >
Co-60 Peak energy [0.00+/-0.00]	1.3325E+003	< : 0.00 : >
Am-241 Peak FWHM [0.00+/-0.00]	8.0683E-001	< : 0.00 : >
Cs-137 Peak FWHM [0.00+/-0.00]	1.2949E+000	< : 0.00 : >
Co-60 Peak FWHM [0.00+/-0.00]	1.6395E+000	< : 0.00 : >
Am-241 activity [0.00+/-0.00]	1.6944E-002	< : 0.00 : In >
Co-60 activity [0.00+/-0.00]	1.1095E-002	< : 0.00 : >
Cs-137 activity [0.00+/-0.00]	1.5714E-002	< : 0.00 : >

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 3/28/07 7:12:59 AM Page 1

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 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/28/07 7:12:59 AM

3-28-07

QA File: C:\LOWLEV\LLDQA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/28/07 6:57:49 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.2 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
Am-241 Peak energy [0.00+/-0.00]	5.9563E+001	0.00 < : : : >
Cs-137 Peak energy [0.00+/-0.00]	6.6191E+002	0.00 < : : : >
Co-60 Peak energy [0.00+/-0.00]	1.3328E+003	0.00 < : : : >
Am-241 Peak FWHM [ 7.8665E-001+/- 0.039]	7.8250E-001	-1.0600E-001 < : : : >
Cs-137 Peak FWHM [ 1.4135E+000+/- 0.052]	1.5060E+000	1.7753E+000 < : : : >
Co-60 Peak FWHM [ 2.0206E+000+/- 0.094]	1.9667E+000	-5.7570E-001 < : : : >
Am-241 activity [0.00+/-0.00]	1.8009E-002	0.00 < : : : >
Co-60 activity [0.00+/-0.00]	7.4268E-003	0.00 < : : In >
Cs-137 activity [0.00+/-0.00]	1.4687E-002	0.00 < : : : >

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report      3/29/07 7:25:01 AM      Page 1

\*\*\*\*\*  
 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/29/07 7:25:01 AM

*AM*  
*3-29-07*

QA File:                    C:\LOWLEV\LLDQA00.QAF  
 Sample ID:                QA  
 Sample Quantity:        1.0000E+000  
 Measurement Date:      3/29/07 7:09:31 AM  
 Elapsed Live Time:     900.0 seconds  
 Elapsed Real Time:     902.2 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags			
		< LU	: SD	: UD	: BS >
Am-241 Peak energy [0.00+/-0.00]	5.9568E+001	<	:	:	>
Cs-137 Peak energy [0.00+/-0.00]	6.6192E+002	<	:	:	>
Co-60 Peak energy [0.00+/-0.00]	1.3329E+003	<	:	:	>
Am-241 Peak FWHM [ 7.8664E-001+/- 0.039]	7.6044E-001	<	:	:	>
Cs-137 Peak FWHM [ 1.4136E+000+/- 0.052]	1.4693E+000	<	:	:	>
Co-60 Peak FWHM [ 2.0205E+000+/- 0.094]	2.0619E+000	<	:	:	>
Am-241 activity [0.00+/-0.00]	1.8516E-002	<	:	:	>
Co-60 activity [0.00+/-0.00]	7.4487E-003	<	:	:In	>
Cs-137 activity [0.00+/-0.00]	1.4762E-002	<	:	:	>

Flags Key:    LU = Lower/Upper Bounds Test    (Ab = Above, Be = Below)  
               SD = Sample Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               UD = User Driven N-Sigma Test    (In = Investigate, Ac = Action)  
               BS = Measurement Bias Test        (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 3/29/07 7:23:36 AM Page 1

\*\*\*\*\*  
 \*\*\*\*\* GENIE QUALITY ASSURANCE \*\*\*\*\*  
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Last Results Report  
 3/29/07 7:23:36 AM

MH  
 3-29-07

QA File: C:\SPC2\SPC2QA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 3/29/07 7:08:25 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 902.8 seconds

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags			
		< LU	: SD	: UD	: BS >
Am-241 Peak energy [0.00+/-0.00]	5.9530E+001	<	:	:	>
Cs-137 Peak energy [0.00+/-0.00]	6.6161E+002	<	:	:	>
Co-60 Peak energy [0.00+/-0.00]	1.3325E+003	<	:	:	>
Am-241 Peak FWHM [0.00+/-0.00]	7.9813E-001	<	:	:	>
Cs-137 Peak FWHM [0.00+/-0.00]	1.2400E+000	<	:	:	>
Co-60 Peak FWHM [0.00+/-0.00]	1.6737E+000	<	:	:In	>
Am-241 activity [0.00+/-0.00]	1.7156E-002	<	:	:	>
Co-60 activity [0.00+/-0.00]	1.0824E-002	<	:	:In	>
Cs-137 activity [0.00+/-0.00]	1.6188E-002	<	:	:In	>

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

Last Measurement Q.A. Report 5/10/07 7:32:33 AM Page 1

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 \*\*\* GENIE QUALITY ASSURANCE \*\*\*  
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Last Results Report  
 5/10/07 7:32:33 AM

*111  
5-10-07*

QA File: C:\SPC2\SPC2QA00.QAF  
 Sample ID: QA  
 Sample Quantity: 1.0000E+000  
 Measurement Date: 5/10/07 7:17:22 AM  
 Elapsed Live Time: 900.0 seconds  
 Elapsed Real Time: 901.8 seconds

*S+*

Parameter Description [Mean +/- Std. Dev.]	Value	Deviation/Flags < LU : SD : UD : BS >
m-241 Peak energy [0.00+/-0.00]	5.9575E+001	0.00 < : : >
s-137 Peak energy [0.00+/-0.00]	6.6163E+002	0.00 < : : >
Co-60 Peak energy [0.00+/-0.00]	1.3326E+003	0.00 < : : >
m-241 Peak FWHM [ 7.6346E-001+/- 0.048]	7.5657E-001	-1.4304E-001 < : : >
s-137 Peak FWHM [ 1.2735E+000+/- 0.055]	1.2608E+000	-2.2882E-001 < : : >
Co-60 Peak FWHM [ 1.6911E+000+/- 0.089]	1.5721E+000	-1.3380E+000 < : : >
m-241 activity [0.00+/-0.00]	1.8563E-002	0.00 < : : >
Co-60 activity [0.00+/-0.00]	1.0678E-002	0.00 < : ;In : >
s-137 activity [0.00+/-0.00]	1.5960E-002	0.00 < : : : >

Flags Key: LU = Lower/Upper Bounds Test (Ab = Above, Be = Below)  
 SD = Sample Driven N-Sigma Test (In = Investigate, Ac = Action)  
 UD = User Driven N-Sigma Test (In = Investigate, Ac = Action)  
 BS = Measurement Bias Test (In = Investigate, Ac = Action)

**Table 20.** XRF analysis of QA standards W1 and STM-1 (USGS Standards).

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>
Actual W1	52.51	15.01	11.12	0.17	6.62	10.99	2.16	0.64	1.07
Measured W1	52.79	14.94	11.08	0.17	6.13	10.77	2.09	0.64	1.08
Actual STM-1	59.64	18.39	5.22	0.22	0.101	1.09	8.94	4.28	0.135
Measured STM-1	60.05	18.52	5.21	0.22	0.076	1.12	8.61	4.200	0.134

The “measured” values are those measured by SRNL and the “actual” values are those assigned to the standard. An acceptable value is ±10%.



**APPENDIX F. Gamma Spectroscopy Data**

ADP FILE/NOTEBOOK

REF WSRC-NB-2003-00207 pg 85

Sample ID 300237126  
 Comment tube 401\_a  
 Count Date 3/27/2007  
 Count Time 7:50  
 Count Duration (sec) 7200

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	1.02E+02	3.89%
Sr-85	1.61E+04	1.77%
Cs-134	3.52E+03	0.86%

Sample ID 300237127  
 Comment tube 401\_b  
 Count Date 3/28/2007  
 Count Time 13:29  
 Count Duration (sec) 3600

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	4.24E+01	12.7%
Sr-85	1.52E+04	1.80%
Cs-134	4.74E+03	0.95%

Sample ID 300237128  
 Comment tube 402\_a  
 Count Date 3/28/2007  
 Count Time 15:13  
 Count Duration (sec) 7200

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	1.22E+02	4.36%
Sr-85	2.29E+04	1.77%
Cs-134	1.75E+04	0.68%

Sample ID 300237129  
 Comment tube 402\_b  
 Count Date 3/28/2007  
 Count Time 17:18  
 Count Duration (sec) 7200

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	9.42E+01	4.90%
Sr-85	1.72E+04	1.78%
Cs-134	1.55E+04	0.70%

Sample ID 300237130  
 Comment tube 403\_a  
 Count Date 3/28/2007

Count Time 19:22  
 Count Duration (sec) 7200  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 4.84E+01 5.98%  
 Sr-85 1.08E+04 1.79%  
 Cs-134 4.57E+03 0.84%

Sample ID 300237131  
 Comment tube 403\_b  
 Count Date 3/28/2007  
 Count Time 21:26  
 Count Duration (sec) 7200  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 3.60E+01 7.35%  
 Sr-85 9.01E+03 1.79%  
 Cs-134 3.50E+03 0.88%

Sample ID 300237132  
 Comment tube 404\_a  
 Count Date 3/28/2007  
 Count Time 23:30  
 Count Duration (sec) 7200  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 2.96E+02 2.42%  
 Sr-85 2.22E+04 1.77%  
 Cs-134 1.83E+04 0.68%

ADP FILE/NOTEBOOK REF WSRC-NB-2003-00207 pg 86

Sample ID 300237127  
 Comment tube 401\_a  
 Count Date 3/28/2007  
 Count Time 8:50  
 Count Duration (sec) 7201  
 Radioisotope Activity (dpm/mL) 2 Sigma %Unc  
 Co-61 1.00E+04 -0.86%  
 Sr-86 1.17E+04 -2.37%  
 Cs-135 1.34E+04 -3.89%

Sample ID 300237128  
 Comment tube 401\_b  
 Count Date 3/29/2007  
 Count Time 14:29  
 Count Duration (sec) 3601  
 Radioisotope Activity (dpm/mL) 2 Sigma %Unc  
 Co-61 1.14E+04 -6.6%  
 Sr-86 1.37E+04 -12.46%  
 Cs-135 1.61E+04 -18.33%

Sample ID 300237129  
 Comment tube 402\_a  
 Count Date 3/29/2007  
 Count Time 16:13  
 Count Duration (sec) 7201

Radioisotope	Activity (dpm/mL)	2 Sigma %Unc
Co-61	3.08E+04	-1.41%
Sr-86	3.95E+04	-3.25%
Cs-135	4.82E+04	-5.09%

Sample ID 300237130  
 Comment tube 402\_b  
 Count Date 3/29/2007  
 Count Time 18:18  
 Count Duration (sec) 7201

Radioisotope	Activity (dpm/mL)	2 Sigma %Unc
Co-61	2.63E+04	-1.74%
Sr-86	3.39E+04	-3.84%
Cs-135	4.16E+04	-5.94%

Sample ID 300237131  
 Comment tube 403\_a  
 Count Date 3/29/2007  
 Count Time 20:22  
 Count Duration (sec) 7201

Radioisotope	Activity (dpm/mL)	2 Sigma %Unc
Co-61	9.66E+03	-2.27%
Sr-86	1.19E+04	-4.84%
Cs-135	1.42E+04	-7.41%

Sample ID 300237132  
 Comment tube 403\_b  
 Count Date 3/29/2007  
 Count Time 22:26  
 Count Duration (sec) 7201

Radioisotope	Activity (dpm/mL)	2 Sigma %Unc
Co-61	7.65E+03	-3.13%
Sr-86	9.39E+03	-6.37%
Cs-135	1.11E+04	-9.60%

Sample ID 300237133  
 Comment tube 404\_a  
 Count Date 3/29/2007  
 Count Time 0:30

Count Duration (sec)	7201		
	Radioisotope	Activity (dpm/mL)	2 Sigma %Unc
	Co-61	3.16E+04	-0.12%
	Sr-86	4.06E+04	-0.99%
	Cs-135	4.96E+04	-1.86%

ADP FILE/NOTEBOOK  
REF

	WSRC-NB-2003-00207 pg 85		
Sample ID	300237140		
Comment	tube 408_a		
Count Date	3/27/2007		
Count Time	7:50		
Count Duration (sec)	7200		
	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
	Co-60	3.19E+02	2.39%
	Sr-85	2.32E+04	1.77%
	Cs-134	2.19E+04	0.67%

Sample ID	300237141		
Comment	tube 408_b		
Count Date	3/27/2007		
Count Time	15:57		
Count Duration (sec)	7200		
	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
	Co-60	3.38E+02	2.33%
	Sr-85	2.46E+04	1.77%
	Cs-134	2.29E+04	0.68%

Sample ID	300237142		
Comment	tube 409_a		
Count Date	3/27/2007		
Count Time	18:02		
Count Duration (sec)	7200		
	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
	Co-60	6.04E+01	6.20%
	Sr-85	1.66E+04	1.78%
	Cs-134	1.22E+04	0.71%

Sample ID	300237143		
Comment	tube 409_b		
Count Date	3/27/2007		
Count Time	20:06		
Count Duration (sec)	7200		
	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
	Co-60	5.13E+01	6.65%

Sr-85	1.48E+04	1.78%
Cs-134	1.10E+04	0.74%

Sample ID	300237144		
Comment	tube 410_a		
Count Date	3/27/2007		
Count Time	22:10		
Count Duration (sec)	7200		
Radioisotope	Activity (dpm/mL)	1 Sigma	%Unc
Co-60	2.32E+03		1.03%
Sr-85	1.14E+04		1.79%
Cs-134	1.51E+04		0.72%

Sample ID	300237145		
Comment	tube 410_b		
Count Date	3/28/2007		
Count Time	0:14		
Count Duration (sec)	7200		
Radioisotope	Activity (dpm/mL)	1 Sigma	%Unc
Co-60	4.52E+02		1.93%
Sr-85	1.90E+04		1.77%
Cs-134	1.45E+04		0.72%

Sample ID	300237146		
Comment	tube 411_a		
Count Date	3/28/2007		
Count Time	2:19		
Count Duration (sec)	7200		
Radioisotope	Activity (dpm/mL)	1 Sigma	%Unc
Co-60	5.87E+01		5.65%
Sr-85	6.18E+03		1.82%
Cs-134	5.37E+03		0.82%

Sample ID	300237147		
Comment	tube 411_b		
Count Date	3/28/2007		
Count Time	4:23		
Count Duration (sec)	7200		
Radioisotope	Activity (dpm/mL)	1 Sigma	%Unc
Co-60	2.80E+01		7.97%
Sr-85	4.35E+03		1.83%
Cs-134	2.25E+03		0.97%

Sample ID	300237148		
Comment	tube 412_a		

Count Date 3/28/2007  
 Count Time 6:27  
 Count Duration (sec) 7200  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 3.41E+01 7.58%  
 Sr-85 3.70E+03 1.84%  
 Cs-134 2.80E+03 0.92%

Sample ID 300237149  
 Comment tube 412\_b  
 Count Date 3/28/2007  
 Count Time 8:23  
 Count Duration (sec) 3600  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 6.66E+01 7.49%  
 Sr-85 7.43E+03 1.84%  
 Cs-134 8.85E+03 0.84%

Sample ID 300237150  
 Comment tube 413\_a  
 Count Date 3/28/2007  
 Count Time 9:14  
 Count Duration (sec) 3600  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 3.25E+01 11.1%  
 Sr-85 3.06E+03 1.96%  
 Cs-134 7.03E+02 1.71%

Sample ID 300237151  
 Comment tube 413\_b  
 Count Date 3/28/2007  
 Count Time 10:18  
 Count Duration (sec) 3600  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 <1.63E+01 MDA  
 Sr-85 4.09E+03 1.91%  
 Cs-134 6.91E+02 1.71%

Sample ID 300237152  
 Comment tube 414\_a  
 Count Date 3/28/2007  
 Count Time 11:22  
 Count Duration (sec) 3600  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 2.24E+02 12.4%  
 Sr-85 9.07E+03 1.83%

Cs-134 6.49E+02 1.74%

Sample ID  
 Comment  
 Count Date  
 Count Time  
 Count Duration (sec)

300237153  
 tube 414\_b  
 3/28/2007  
 12:26  
 3600  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 4.57E+01 7.95%  
 Sr-85 1.08E+04 1.81%  
 Cs-134 3.89E+02 2.17%

ADP FILE/NOTEBOOK  
 REF

WSRC-NB-2003-0027 pg

Sample ID  
 Comment  
 Count Date  
 Count Time  
 Count Duration (sec)

300237154  
 tube 415\_a  
 3/23/2007  
 11:20  
 14400  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 7.46E+02 1.17%  
 Sr-85 9.23E+03 1.77%  
 Cs-134 1.99E+03 0.88%

Sample ID  
 Comment  
 Count Date  
 Count Time  
 Count Duration (sec)

300237155  
 tube 415\_b  
 3/23/2007  
 15:24  
 14400  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 1.19E+03 1.02%  
 Sr-85 9.43E+03 1.77%  
 Cs-134 1.76E+03 0.91%

Sample ID  
 Comment  
 Count Date  
 Count Time  
 Count Duration (sec)

300237156  
 tube 416\_a  
 3/23/2007  
 19:28  
 14400  
 Radioisotope Activity (dpm/mL) 1 Sigma %Unc  
 Co-60 4.24E+01 4.82%  
 Sr-85 6.55E+03 1.79%  
 Cs-134 4.74E+03 0.75%



Sample ID 300237157  
 Comment tube 416\_b  
 Count Date 3/23/2007  
 Count Time 23:32  
 Count Duration (sec) 14400

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	4.76E+01	4.39%
Sr-85	6.51E+03	1.78%
Cs-134	4.70E+03	0.76%

Sample ID 300237158  
 Comment tube 417\_a  
 Count Date 3/24/2007  
 Count Time 3:36  
 Count Duration (sec) 14400

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	4.62E+01	4.35%
Sr-85	6.59E+03	1.78%
Cs-134	4.98E+03	0.74%

Sample ID 300237159  
 Comment tube 417\_b  
 Count Date 3/24/2007  
 Count Time 7:40  
 Count Duration (sec) 14400

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	2.07E+02	1.98%
Sr-85	1.08E+04	1.77%
Cs-134	5.12E+03	0.75%

Sample ID 300237160  
 Comment tube 418\_a  
 Count Date 3/24/2007  
 Count Time 11:44  
 Count Duration (sec) 14400

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	5.02E+01	4.77%
Sr-85	8.34E+03	1.78%
Cs-134	5.60E+03	0.74%

Sample ID 300237161  
 Comment tube 418\_b

Count Date	3/24/2007		
Count Time	15:48		
Count Duration (sec)	14400		
		Activity	
	Radioisotope	(dpm/mL)	1 Sigma %Unc
	Co-60	1.01E+02	3.01%
	Sr-85	9.29E+03	1.78%
	Cs-134	5.27E+03	0.74%

Sample ID	300237162		
Comment	tube 419_a		
Count Date	3/23/2007		
Count Time	11:18		
Count Duration (sec)	14400		
		Activity	
	Radioisotope	(dpm/mL)	1 Sigma %Unc
	Co-60	2.93E+01	5.52%
	Sr-85	7.76E+03	1.77%
	Cs-134	1.58E+03	0.89%

Sample ID	300237163		
Comment	tube 419_b		
Count Date	3/26/2007		
Count Time	8:21		
Count Duration (sec)	14400		
		Activity	
	Radioisotope	(dpm/mL)	1 Sigma %Unc
	Co-60	1.52E+01	8.02%
	Sr-85	5.17E+03	1.79%
	Cs-134	7.25E+02	1.07%

Sample ID	300237164		
Comment	tube 420_a		
Count Date	3/26/2007		
Count Time	12:25		
Count Duration (sec)	14400		
		Activity	
	Radioisotope	(dpm/mL)	1 Sigma %Unc
	Co-60	6.40E+01	3.33%
	Sr-85	6.46E+03	1.78%
	Cs-134	3.30E+02	1.39%

Sample ID	300237165		
Comment	tube 420_b		
Count Date	3/26/2007		
Count Time	10:40		
Count Duration (sec)	14400		

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	1.82E+01	6.49%
Sr-85	5.37E+03	1.78%
Cs-134	1.81E+02	1.69%

Sample ID 300237166  
 Comment tube 421\_a  
 Count Date 3/26/2007  
 Count Time 14:45  
 Count Duration (sec) 6032.7

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	1.71E+02	4.23%
Sr-85	3.03E+04	1.77%
Cs-134	2.90E+04	0.65%

Sample ID 300237167  
 Comment tube 421\_b  
 Count Date 3/27/2007  
 Count Time 15:49  
 Count Duration (sec) 7200

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	4.23E+03	0.89%
Sr-85	1.79E+04	1.78%
Cs-134	1.69E+04	0.69%

Sample ID 300237168  
 Comment tube 421\_c  
 Count Date 3/28/2007  
 Count Time 7:15  
 Count Duration (sec) 3600

Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	1.95E+02	5.16%
Sr-85	3.10E+04	1.78%
Cs-134	3.03E+04	0.70%

To confirm  
 Duplicates did not match

		<b>Recount</b>
Sample ID	300237166	300237166
Comment	tube 421_a	tube 421_a
Count Date	3/26/2007	3/28/2007
Count Time	14:45	11:05
Count Duration (sec)	6032.7	3600

Radioisotope	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	Co-60	1.53E+02	5.68%
Sr-85	Sr-85	3.00E+04	1.78%
Cs-134	Cs-134	2.93E+04	0.68%

		<b>Recount</b>
Sample ID	300237167	300237167
Comment	tube 421_b	tube 421_b
Count Date	3/27/2007	3/29/2007
Count Time	15:49	13:32
Count Duration (sec)	7200	3600

Radioisotope	Radioisotope	Activity (dpm/mL)	1 Sigma %Unc
Co-60	Co-60	4.30E+03	1.04%
Sr-85	Sr-85	1.75E+04	1.79%
Cs-134	Cs-134	1.71E+04	0.76%

**Gamma Blanks for VC Summer**

Sample ID	300238881		
Comment	OW-205A		
Count Date	5/10/2007		
Count Time	9:40		
Count Duration (sec)	3600		
		Activity	1 Sigma
	Radioisotope	(dpm/mL)	%Unc
	Co-60	<9.87E-01	MDA
	Sr-85	<1.39E+00	MDA
	Cs-134	<1.06E+00	MDA

Sample ID	300238882		
Comment	OW-205A		
Count Date	5/10/2007		
Count Time	10:44		
Count Duration (sec)	3600		
		Activity	1 Sigma
	Radioisotope	(dpm/mL)	%Unc
	Co-60	<1.18E+00	MDA
	Sr-85	<1.31E+00	MDA
	Cs-134	<1.04E+00	MDA

Sample ID	300238883		
Comment	OW-212		
Count Date	5/10/2007		
Count Time	11:48		
Count Duration (sec)	3600		
		Activity	1 Sigma
	Radioisotope	(dpm/mL)	%Unc
	Co-60	<1.14E+00	MDA
	Sr-85	<1.22E+00	MDA
	Cs-134	<1.04E+00	MDA

Sample ID	300238884		
Comment	OW-212		
Count Date	5/10/2007		
Count Time	12:51		
Count Duration (sec)	3600		
		Activity	1 Sigma
	Radioisotope	(dpm/mL)	%Unc
	Co-60	<1.10E+00	MDA

Sr-85	<1.34E+00	MDA
Cs-134	<9.90E-01	MDA

Sample ID	300238885		
Comment	OW-305A		
Count Date	5/10/2007		
Count Time	13:55		
Count Duration (sec)	3600		
	Activity		1 Sigma
Radioisotope	(dpm/mL)		%Unc
Co-60	<1.21E+00	MDA	
Sr-85	<1.39E+00	MDA	
Cs-134	<1.11E+00	MDA	

Sample ID	300238886		
Comment	OW-305A		
Count Date	5/10/2007		
Count Time	14:59		
Count Duration (sec)	3600		
	Activity		1 Sigma
Radioisotope	(dpm/mL)		%Unc
Co-60	<1.09E+00	MDA	
Sr-85	<1.30E+00	MDA	
Cs-134	<9.76E-01	MDA	

Sample ID	300238887		
Comment	OW-327		
Count Date	5/10/2007		
Count Time	16:03		
Count Duration (sec)	3600		
	Activity		1 Sigma
Radioisotope	(dpm/mL)		%Unc
Co-60	<1.07E+00	MDA	
Sr-85	<1.20E+00	MDA	
Cs-134	<9.34E-01	MDA	

Sample ID	300238888		
Comment	OW-327		
Count Date	5/11/2007		
Count Time	9:09		
Count Duration (sec)	3600		
	Activity		1 Sigma
Radioisotope	(dpm/mL)		%Unc
Co-60	<1.17E+00	MDA	

Sr-85	<1.47E+00	MDA
Cs-134	<1.11E+00	MDA

**APPENDIX G. ASTM 4646-03: 24 h Batch  $K_d$  Test**





Designation: D 4646 – 03

## Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments<sup>1</sup>

This standard is issued under the fixed designation D 4646; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method describes a procedure for determining the sorption affinity of waste solutes by unconsolidated geologic material in aqueous suspension. The waste solute may be derived from a variety of sources such as wells, underdrain systems, or laboratory solutions such as those produced by waste extraction tests like the Test Method D 3987 shake extraction method.

1.2 This test method is applicable in screening and providing relative rankings of a large number of geomeedia samples for their sorption affinity in aqueous leachate/geomeedia suspensions. This test method may not exactly simulate sorption characteristics that would occur in unperturbed geologic settings.

1.3 While this procedure may be applicable to both organic and inorganic constituents, care must be taken with respect to the stability of the particular constituents and their possible losses from solution by such processes as degradation by microbes, light, or hydrolysis. This test method should not be used for volatile chemical constituents (see 6.1).

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards.<sup>2</sup>

- D 1129 Terminology Relating to Water
- D 1193 Specification for Reagent Water
- D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

Current edition approved Nov. 1, 2003. Published January 2004. Originally published as E8 10 – 85. Last previous edition D 4646 – 87.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

D 3987 Test Method for Shake Extraction of Solid Waste with Water

D 4319 Test Method for Distribution Ratios by the Short-Term Batch Method

### 3. Terminology

3.1 *Definitions*—For definition of terms used in this test method refer to Terminology D 1129.

3.1.1 *solute*—chemical species (for example, ion, molecule, etc.) in solution.

3.1.2 *sorbate*—chemical species sorbed by a sorbent.

3.1.3 *sorbent*—a substance that sorbs the solute from solution (for example, soil, sediment, till, etc.).

3.1.4 *sorption*—depletion of an amount of solute initially present in solution by a sorbent.

3.1.5 *sorption affinity*—the relative degree of sorption that occurs by a geomeedia.

3.1.6 *unconsolidated geologic material (geomeedia)*—a loosely aggregated solid natural material of geologic origin (for example, soil, sediment, till, etc.).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *distribution coefficient,  $K_d$* —the ratio of the concentration of solute sorbed on the soil or other geomeedia divided by its concentration in solution. A 24-h  $K_d$  is the analogous ratio evaluated after 24 h of contact of the solute with the geomeedia.

3.2.1.1 *Discussion*—The dimensions of  $K_d$  reduce to units of volume per mass. It is convenient to express  $K_d$  in units of millilitres (or cubic centimetres) of solution per gram of geomeedia. Dissimilar  $K_d$  values may be obtained if different initial solute concentrations are used, depending on the sorption behavior of the solute and the properties of the geomeedia (that is, nonlinear sorption curve). This concentration dependency may be absent where the solute concentrations are sufficiently low or the characteristics of the particular solute-sorbent combination yield  $K_d$  values that are independent of the concentration of solute (that is, linear sorption curve).

### 4. Summary of Test Method

4.1 Distilled water, natural water, waste leachate, or other aqueous solution containing a known concentration of a solute is mixed with a known amount of unconsolidated geologic

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**9. Procedure**

9.1 Geomedia samples are spread out on a flat surface, no more than 2 to 3 cm deep, and allowed to air dry for 7 days or until constant weight (a change that is less than 5%/24-h period) is achieved (do *not* oven dry).

9.2 After the sample has air dried, it is passed through a 2-mm screen sieve. Large aggregates are to be crushed, without grinding, using a clean mortar and a rubber-tipped pestle.

9.3 Mix the sieved material until the sample is homogeneous. Use a riffle splitter, or other unbiased splitting procedure, to obtain subsamples of appropriate size.

9.4 Remove subsamples and determine the moisture content of the air-dried sample (refer to Test Method D 2216).

9.5 Determine the mass of geomedia sample, corrected for moisture content:

Determination of air-dried soil mass equivalent to the desired mass of oven-dried soil:

$$A = M_s[1 + (M/100)] \tag{1}$$

where:

- A = air dry soil mass
- M<sub>s</sub> = mass of oven-dried soil desired, and
- M = moisture, %.

9.6 Place between 5 and 70 g (oven-dried basis) of the weighed air-dried sample into the appropriate container. The samples should be weighed to a minimum of three significant figures.

9.7 Add to the container an amount of solute solution necessary to yield a 1:20 soil-to-solution ratio. This is determined on the oven-dried basis:

Determination of solution volume needed per sample for a soil-to-solution ratio of 1:20:

$$V = (M_s \times 20)/\rho \tag{2}$$

where:

- ρ = density of solution, g/cm<sup>3</sup>,
- V = volume of solution per sample, cm<sup>3</sup>, and
- M<sub>s</sub> = mass of soil to be used, g, (oven-dried basis).

9.8 Retain a separate, appropriately preserved aliquot of the initial solute solution for analysis.

9.9 Close the container and place it on the rotary extractor (Test Method D 3987).

9.10 Agitate continuously for 24 ± 0.5 h at 29 ± 2 r/min at room temperature (22 ± 5°C).

9.11 Open the container. Note the temperature of the solution and any changes in the sample or solution (that is, color, odor, etc.).

9.12 Separate the solution phase from the majority of the solid phase by decantation.

9.13 Filter the solution phase through a 0.45-μm pore size membrane filter (see 7.2), or centrifuge a subsample at the predetermined rate of rotation and time for the centrifugation equipment employed at constant temperature (the temperature recorded after 24 h):

$$t = 9/2 \left( \frac{\eta}{\omega^2 r_p^2 (\rho_p - \rho)} \right) \ln (R_b/R_t) \tag{3}$$

where:

$$\omega^2 = \frac{4\pi^2 (r/min)^2}{60}$$

- = angular velocity,
- r<sub>p</sub> = particle radius, cm,
- η = viscosity of water, 8.95 × 10<sup>-3</sup> g/s-cm at 25°C,
- ρ<sub>p</sub> = particle density,
- ρ = density of solution,
- r/min = revolutions per minute,
- R<sub>t</sub> = distance from center of centrifuge rotor to top of solution in centrifuge tube, cm,
- R<sub>b</sub> = distance from center of centrifuge rotor to bottom of centrifuge tube, cm, and
- t = time, min.

To remove particles >0.1-μm radius and 2.65-g/cm<sup>3</sup> density from solution:

$$t = \left( \frac{3.71 \times 10^8}{(r/min)^2} \right) \ln (R_b/R_t) \tag{4}$$

Note that if filtration is used, the affinity of the filtration membrane for the solute must be evaluated. Failure to do so may lead to erroneous results.

9.14 After a clear solution has been obtained, place an aliquot in an appropriate container (see 7.3), and analyze or store in a refrigerator at 4 ± 2°C until analyzed.

9.15 Each geomedia sample is to be subjected to the procedure in three or more replicates. The number of blanks (that is, solute solution without geomedia) carried through all steps of the procedure should be a minimum of 5% of the total number of geomedia samples, but not less than three.

**10. Calculation**

10.1 Calculate the distribution as follows:

$$K_d = \frac{(A - B)V}{(M_s)B} \tag{5}$$

where:

- A = initial concentration of the solute defined as the mean concentration of the blanks, μg/mL,
- B = final concentration of the solute after 24 h in contact with the geomedia, μg/mL,
- V = volume of solution used, mL,
- M<sub>s</sub> = mass of soil expressed on an oven-dried basis, g, and
- K<sub>d</sub> = distribution coefficient, mL/g.

**11. Report**

11.1 The K<sub>d</sub> value must be clearly marked as nonequilibrium 24-h distribution coefficient.

11.2 Both the initial solute concentration (A in 10.1) and the final solute concentration (B in 10.1) must be reported.

11.3 The initial and final solute concentration for each blank (solution without geomedia) must be reported.

11.4 The mass of the sorbent (M<sub>s</sub> in 10.1), volume of solution (V in 10.1), and the room temperature at which the rotary extractor was operated must be reported.

11.5 Report the temperature of the solution and any changes noted in 9.11.

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material (geomedia) for 24 h. After 24 h, equilibrium between the solid and solution phase is presumed to occur. The concentration of solute remaining in solution is measured and the amount of solute adsorbed is calculated. Given that the mass of solid phase is known, the distribution coefficient for the specified experimental conditions can then be calculated.

### 5. Significance and Use

5.1 This test method is meant to allow for a rapid (24 h) index of a geomedia's sorption affinity for given chemicals or leachate constituents. A large number of samples may be run using this test method to determine a comparative ranking of those samples, based upon the amount of solute sorbed by the geomedia, or by various geomedia or leachate constituents. The 24-h time is used to make the test convenient and also to minimize microbial degradation which may be a problem in longer-timed procedures. While  $K_d$  values are directly applicable for screening and comparative ranking purposes, their use in predictive field applications generally requires the assumption that  $K_d$  be a fixed value.

5.2 While this test method may be useful in determining 24-h  $K_d$  values for nonvolatile organic constituents, interlaboratory testing has been carried out only for the nonvolatile inorganic species, arsenic and cadmium. However, the procedure has been tested for single laboratory precision with polychlorinated biphenyls (PCBs) and is believed to be useful for all stable and nonvolatile inorganic, and organic constituents. This test method is not considered appropriate for volatile constituents.

5.3 The 24-h time limit may be sufficient to reach a steady-state  $K_d$ . However, to report this determination as a steady-state  $K_d$ , this test method should be conducted for intermediate times (for example, 12, 18 and 22 h) to ensure that the soluble concentrations in the solution have reached a steady state by 24 h. Refer to Test Method D 4319 for an alternate procedure of longer duration.

### 6. Interferences

6.1 When dealing with solutes of unknown stability either in contact with the geomedia or when used as blanks, care must be taken to determine if volatilization, hydrolysis, photodegradation, microbial degradation, oxidation-reduction (for example,  $\text{Cr}^{3+}$  to  $\text{Cr}^{6+}$ ) or other physicochemical processes are operating at a significant rate within the time frame of the procedure. The stability and hence loss from solution may affect the outcome of this procedure if the aforementioned reactions are significant. The compatibility of the method and the solute of interest may be assessed by determining the differences between the initial solute concentration (see 9.8) and the final blank concentration of the solute (see 9.15). If this difference is greater than the expected precision of the method (10%), then the  $K_d$  value generated may be unreliable and must be carefully evaluated.

### 7. Apparatus

7.1 *Agitation Equipment*—The agitation equipment to be used is the rotary solid waste extractor<sup>3</sup> specified in Test Method D 3987.

7.2 *Phase Separation Equipment*—A filtration apparatus made of materials compatible with the solutions being filtered and equipped with a 0.45- $\mu\text{m}$  pore size membrane filter, or a constant temperature centrifuge capable of separating particles with diameters greater than 0.1  $\mu\text{m}$  (see Section 9). If organic compounds are being measured, the filtration apparatus, centrifuge tubes etc., should be compatible with the compounds being measured (that is, glass or stainless steel). Sorption of solute onto the filtration membrane may be significant for some solutes, and must be evaluated by the use of blanks through all steps of the procedure.

7.3 *Containers*—Round, wide-mouth bottles compatible with the rotary extractor (Test Method D 3987) and of composition suitable to the nature of the solute(s) under investigation and the analysis to be performed will be used. For nonvolatile inorganic constituents, high-density, linear polyethylene bottles should be used with the size of the bottle dictated by sample size, and the need for the solution to occupy 70 to 80% of the container volume (that is, 125 mL, 250 mL, or 2-L bottles for sample sizes of 5, 10, or 70 g respectively). For nonvolatile organic constituents, TFE-fluorocarbon, glass bottles, or stainless steel containers with water-tight closures made of chemically inert materials should be used with size requirements being the same as for nonvolatile inorganics. Containers should be cleaned in a manner consistent with the analyses to be performed. Samples of the solutions to be analyzed should be stored in similar chemically compatible bottles.

7.4 *Balance*, having a minimum capacity of 70 g and a sensitivity of  $\pm 0.005$  g shall be used.

### 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the American Chemical Society, where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean Type IV reagent water of Specification D 1193.

<sup>3</sup> Diamondstone, B. T., Burke, R. W., and Garner, E. L., "Improved Leach Measurements on Solid Wastes," *ASTM Standardization News*, June 1982, pp. 28-33.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analyst Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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11.6 Note and report negative  $K_d$  values when and if they occur. Negative  $K_d$  values may occur if the geomeedia contains the test solute prior to the application of the method.

11.7 It is suggested that the pH of the sorbent-solute mixture be determined prior to separating the sorbent from the liquid and reported where feasible.

**12. Precision and Bias**

12.1 An interlaboratory round-robin test was conducted at a soil-to-solution ratio of 1:20 using 70.0 g of soil. Intralaboratory testing using a 1:20 soil-to-solution ratio was carried out with no significant loss of precision for soil masses of 5.00, 10.0, and 70.0 g. Therefore, it is specified that the soil-to-solution ratio be 1:20 with the working mass of soil (on an oven-dried basis) between 5 and 70 g.

**12.2 Precision:**

12.2.1 The precision of this test method is limited by the ability to obtain a homogeneous sample of geomeedia, and the precision of the various methods used to carry out the procedure (that is, mass determinations, initial and 24-h concentration of constituents).

12.2.2 A comprehensive overall precision statement, covering all species, is not feasible. Interlaboratory testing of this procedure, using cadmium and arsenic as sorbates, with five independent laboratories, and single laboratory testing with PCBs indicated that a coefficient of variation of less than 10% is obtainable.

12.2.3 For a summary of interlaboratory testing for the 24-h batch-type  $K_d$  determination see Table 1.

**TABLE 1 Summary of Interlaboratory Testing for the 24-h Batch-Type  $K_d$  Determination**

Initial Concentration, $\mu\text{g/mL}$	Soil 1	Soil 1	Soil 2	Soil 3	Soil 4
	10	200	100	100	100
<b>Cadmium Carbonate (as <math>\text{CdCO}_3</math>):</b>					
$\bar{X} K_d$ , mL/g	1508	96.3	69.50	69.74	28.94
Standard deviation, mL/g	$\pm 156$	$\pm 6.32$	$\pm 7.73$	$\pm 7.87$	$\pm 2.64$
Coefficient of variation, %	9.95	6.56	11.13	11.29	9.14
Number of replicates	12	12	10	12	9
<b>Arsenic (as <math>\text{KH}_2\text{AsO}_4</math>):</b>					
$\bar{X} K_d$ , mL/g	15.42	2.75	4.95	3.25	2.99
Standard deviation, mL/g	$\pm 0.92$	$\pm 0.85$	$\pm 0.27$	$\pm 0.26$	$\pm 0.20$
Coefficient of variation, %	5.99	30.9	5.53	7.88	6.52
Number of replicates	12	12	12	12	12

12.2.4 For a summary of single laboratory testing for an organic solute see Table 2.

12.3 *Bias*—A determination of the bias for this procedure is not possible since no standard soil or alternate technique exists.

**TABLE 2 Summary of Single Laboratory for an Organic Solute**

Initial Concentration, $\mu\text{g/mL}$	Soil 1 <sup>A</sup>	Soil 1 <sup>B</sup>	Soil 2 <sup>C</sup>	Soil 2 <sup>D</sup>
	0.218 (0% Acetone)	0.187 (20% Acetone)	0.216 (0% Acetone)	0.187 (20% Acetone)
<b>PCBs (as Aroclor 1242):</b>				
$\bar{X} K_d$ , mL/g	708	78.62	23.83	2.28
Standard deviation, mL/g	$\pm 18.41$	$\pm 3.45$	$\pm 1.48$	$\pm 0.11$
Coefficient of variation, %	2.31	4.39	6.21	4.82
Number of replicates	4	4	4	4

<sup>A</sup> Soil 1—Callin silt loam.  
<sup>B</sup> Soil 2—Sangamon paleosol.  
<sup>C</sup> Soil 3—Kaolinic clay.  
<sup>D</sup> Soil 4—Vandalla III, unaltered phase.

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**APPENDIX H. XRD, XRF, BET, and Gamma Sample Preparation and  
Analysis Procedures**

The following procedures were sent electronically in a folder entitled "Appendix H\_ XRD, XRF, BET, & Gamma Procedures." Within this folder are the following files:

- |                        |   |
|------------------------|---|
| 1. ADS-1120 XRD.pdf :  | XRD procedure                                       |
| 2. ADS-2420 gamma.pdf: | Gamma sample preparation and analysis procedure     |
| 3. ADS-1115 XRF.pdf    | Rigaky ZSX 101E Wavelength dispersion XRD procedure |
| 4. ADS-1114 BET.pdf    | Operating the Micromeritics ASAP 2010 BET procedure |

**APPENDIX I. M&TE Approval of Balance Used During Study**

# Certificate of Calibration

*copy*

Issued by  
**Savannah River Standards Laboratory**  
Westinghouse SRC, Building 736-A  
Aiken, SC 29801

<b>M &amp; TE #:</b> IW-0105	<b>Cal Procedure:</b>
<b>Description:</b> BALANCE	BAL-CAL/11.4 (08/16/05)
<b>Manufacturer:</b> SARTORIUS	
<b>Model#:</b> R-200D	
<b>Serial#:</b> 30200365	<b>Cal Calculation:</b>
<b>Calibrated:</b> 02/05/2007	N/A
<b>Frequency:</b> 12 Months	
<b>Expires:</b> 02/05/2008	

**NIST Traceable Standard(s) used, (Expiration Date):**  
SL-5KGSET (04/8/07)

**Print Specification:** N/A  
**The estimated uncertainty (k=2) of the standard(s) and measurement process at SRSL is:**  
SEE ATTACHED SHEET

The measurements made on IW-0105 were obtained at 23 +/- 2.0 °c and 10% RH

OM/RUSH **CALIBRATION DATA FOR IW-0105**

SEE ATTACHED FOR BALANCE CALIBRATION DATA.

THIS INSTRUMENT PASSED CALIBRATION. AS FOUND = AS LEFT.

The assigned uncertainty of the UUT as determined by tolerance test is:  
SEE ATTACHED

**Metrologist:** D. R. SMITH *[Signature]* **Signature** *[Initials]* **PR Init.** *[Signature]* **Engr. Init.**

**Note 1:** This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.  
**Note 2:** The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.





cal11v4 8/16/05

Modified by JP Clark 8-16-05  
 Approved by WA Henderson 8-16-05

**SRS Method**

Analyst Information	
Calibrator Initials:	DRS
Calibrator ID #	W0894
Date:	2/5/2007
Balance Information	
Location	773-A/B122
Unique ID Number	IW-0105
Manufacturer	Sartorius
MODEL#	R200D
Confidence Level	3-Stdev
QA Limit (%)	0.0100
Sen. Limit (g)	0.0006
1 Bal. Maint. SD	0.0002
Wt. Set Selection	SL-5KGSET

Function Tests	
Power	Pass
Load	Pass
Taring	Pass
P/NDisplay	Pass

Visual Inspection Parameters	
Damaged	Pass
Level	Pass
Clean	Pass
Complete	Pass

Cornerloading(g)	
Position	Reading
Top	99.9993
Right	99.9993
Bottom	99.9994
Left	99.9994

Environmental Conditions			
	Before	After	Average
Temp (C)	23	23	23
Humidity (%)	10	11	10

MEASUREMENTS (in grams)				
Stacking Information	High Wt(s)	Mid Wt(s)	Low Wt.	Sensitivity Wt.
High Weights(g)	200	100	50	1
200	199.9994	99.9994	49.9998	1.0000
No stacking	199.9994	99.9994	49.9998	1.0000
Mid-Weights(g)	100	50	1	
100	99.9994	49.9998	1.0000	
No stacking	99.9994	49.9998	1.0000	

page 2 of 4

QA BALANCE CERTIFICATION  
Next Certification due date:

5-Feb-08

111v4 8-16-05

Modified by JPClark 8-16-05  
Approved by WA Henderson 8-16-05

Analyst Information	
Initials:	BRS
Calibrator ID #	W0894
Date:	2/5/2007

Balance Information	
Manufacturer	Sartorius
MODEL#	R200D
ID Number	1W-0105
Lab/Room#	773-A/B122

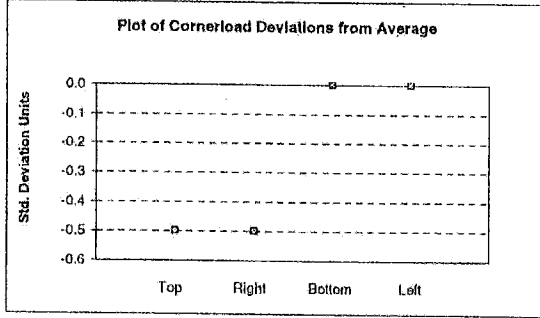
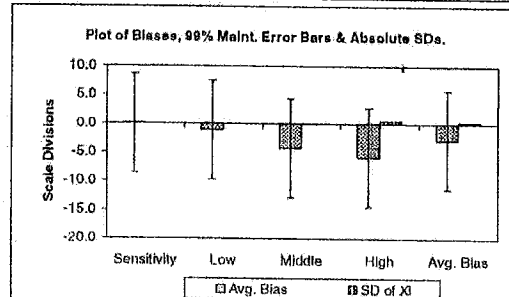
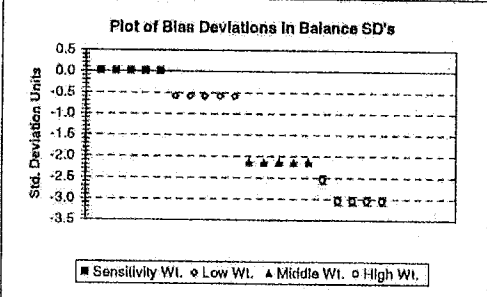
Weight Set Information	
Expiration	03/04/07
Manufacturer	DOT
Wt. Set ID	SL-5KGSET

Functionality		Statistics				Bal/Wt Uncertainty Ratio		
Power	Pass	AVERAGE	High Wt(e)	Mid Wt(e)	Low Wt.	Minimal Wt.	HighWt(e)	21.05283
Load	Pass	Bias (abs)	-0.000582	-0.000430	-0.000117	0.000002	MidWt(e)	19.61229
Taring	Pass	% Bias	-0.000295	-0.000430	-0.000234	0.000190	LowWt.	38.09524
P/NDisplay	Pass	% RSD	0.000022	0.000000	0.000000	0.000000	Minimal Wt.	666.66667
Visual Inspection		SD(abs)	0.000045	0.000000	0.000000	0.000000	Cornerloading	
Max SD	0.000201	0.000201	0.000200	0.000200	0.000200	0.000200	Position	t-Statistic
Calculated t	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant	Top	0.50
Level	Pass	Pooled Abs. Std. Dev., 16 df =	0.000022	Tab. Chi Sq =	32.0	Right	0.50	
Clean	Pass	Tabled t, 16 df (99% Conf.) =	2.921	Cal. Chi Sq =	0.2	Bottom	0.00	
Complete	Pass	Tabled F, 2, 16 df (99% Conf.) =	6.23	F Statistic =	Not Significant	Left	0.00	

Total Uncertainty 95% CL, (K=2)		
	Grams	Percent
High Wt.	0.00072	0.00036%
Middle Wt.	0.00059	0.00059%
Low Wt.	0.00042	0.00083%
Minimal Wt.	0.00040	0.00000%

Control Limits				
	Mean Wt.	3 DEV'S	Low Limit	High Limit
High Wt.	199.99942	0.000802701	199.9986173	200.000227
Middle Wt.	99.9994	0.000803144	99.99859686	100.0000031
Low Wt.	49.9998	0.000800826	49.999001174	50.000000003
Minimal Wt.	1	0.000800003	0.999199997	1.000800003

ACCEPTANCE CRITERIA	
Summary:	Balance passes QA Limits
Linearity(Bias)	Balance passes
Precision (SD)	Balance passes
Corner Loading	Balance passes
Disposition	AS LEFT = AS FOUND
3.0 grams minimum quantity that can be weighed to maintain QA Test Limits with 99.7% confidence	
0.3 grams minimum quantity that can be weighed to maintain 3 Standard Deviation Limits @ 0.1% Tolerance per USP	





Johnp.Clark/SRNL/Srs  
02/05/2007 10:25 AM

To Edward Polz/SRNL/Srs@Srs  
cc Frederick.Leach@srs.gov, Raymond Gay/SRNL/Srs@Srs,  
Ricky Smith/SRNL/Srs@Srs  
bcc  
Subject Re: Priority Balances

Ed,

Ricky is checking on the location of these balances and will make them his number 1 priority for today.

John P. Clark  
Phone: 803-725-3654 pager 10178  
SRS 738-A  
Aiken, SC 29808  
Edward Polz/SRNL/Srs



Edward Polz/SRNL/Srs  
02/05/2007 09:26 AM

To Johnp.Clark/SRNL/Srs@Srs, Ricky Smith/SRNL/Srs@Srs,  
Raymond Gay/SRNL/Srs@Srs  
cc Frederick.Leach@srs.gov@Srs  
Subject Priority Balances

Need IW-0101 and IW-0105 DONE ASAP. Please do today if possible. Need completion by COB tomorrow in preparation for nuclear compliance audit.

Ed Polz  
Metrology Manager  
Savannah River National Laboratory  
803-725-0955

"The most terrifying words in the English language are: I'm from the government and I'm here to help."  
- Ronald Reagan

*Pg. 4064*

*copy*

### Certificate of Calibration

Issued by  
**Savannah River Standards Laboratory**  
Westinghouse SRC, Building 736-A  
Aiken, SC 29801

M & TE #: WP-550 Cal. Procedure: L10.1-301, REV. 2  
Description: WEIGHT SET, 1MG-100G  
Manufacturer: TROEMNER  
Model#: N/A  
Serial#: 53514  
Calibrated: 08/08/2005  
Frequency: 24 Months  
Expires: 08/08/2007

NIST Traceable Standard(s) used, (Expiration Date):  
SL-1005 (CBU), SL-749A (CBU), SL-20KGWTSET (04/21/08)

Print Specification: n/a  
The estimated uncertainty (k=2) of the standard(s) and measurement process at SRSL is:  
SEE ATTACHED DATA SHEET

The measurements made on WP-550 were obtained at 23 +/- 2.0 °c and 33% RH



SEE ATTACHED INSTRUMENT TEST REPORT FOR MEASUREMENT DATA

NOTE: CUSTODIAN REQUESTED TOLERANCE: NONE  
NOTE: THE WEIGHT SET HAS MET CLASS 2 TOLERANCES PREVIOUSLY. THE WEIGHT SET MEETS CLASS 2 "EXCEPT" THE 20MG WEIGHT. IT MEETS CLASS 3.

THIS INSTRUMENT PASSED CALIBRATION. AS FOUND = AS LEFT

The assigned uncertainty of the UUT as determined by tolerance test is:  
SEE ATTACHED SHEET

Metrologist: D. R. SMITH

*D.R. Smith*  
Signature

*[Signature]*  
PR Init.

*[Signature]* 8/19/05  
Engr. Init.

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.  
Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.



### Mass Calibration Data Summary


Issued By  
**Savannah River Standards Laboratory**  
 Westinghouse SRC, Building 736-A  
 Aiken, SC 29801

Calibration Serial No.	M&TE No.	Metrologist	Date			
WP-550-2005001	WP-550	D Smith	08/08/2005			
Tolerance Class						
ASTM 3						
Comments:						
CUSTODIAN TOLERANCE REQUEST: NONE. THE WEIGHT SET HAS MET CLASS 2 TOLERANCES PREVIOUSLY. THE WEIGHT SET MEETS CLASS 2 "EXCEPT" THE 20MG WEIGHT. IT MEETS CLASS 3.						
Uncertainties are calculated using the GUM method of RSS. They are reported at K=2, 95% Confidence. Standards used are 1 piece ASTM Class 0 Weights Calibrated at NIST 5/15/01.						
Nom Wt	Wt # or Marking	True Mass	App. Mass at 8g/cc	Uncertainty (mg)	Density (g/cc)	Method
1	mg	1.011583330 mg	1.011285430 mg	0.0020	2.70	Comparison
2	mg	2.008920000 mg	2.008328400 mg	0.0020	2.70	Comparison
2	mg *	2.010420000 mg	2.009827950 mg	0.0020	2.70	Comparison
5	mg	4.990903330 mg	4.989433570 mg	0.0021	2.70	Comparison
10	mg	9.990190000 mg	9.987248000 mg	0.0020	2.70	Comparison
20	mg	19.99070667 mg	19.98481963 mg	0.0020	2.70	Comparison
20	mg *	19.99627333 mg	19.99038466 mg	0.0020	2.70	Comparison
50	mg	50.00707667 mg	50.01096334 mg	0.0020	16.60	Comparison
100	mg	100.0143592 mg	100.0221325 mg	0.0020	16.60	Double Substitution
200	mg	200.0054846 mg	200.0210295 mg	0.0020	16.60	Double Substitution
200	mg *	200.0037489 mg	200.0192937 mg	0.0020	16.60	Double Substitution
500	mg	499.9703844 mg	500.0092433 mg	0.0020	16.60	Double Substitution
1	g	1.000033040 g	1.000033040 g	0.0026	8.00	Double Substitution
2	g	2.000031400 g	2.000031400 g	0.0026	8.00	Double Substitution
2	g *	2.000019300 g	2.000019300 g	0.0026	8.00	Double Substitution
5	g	5.000008760 g	5.000008760 g	0.0031	8.00	Double Substitution
10	g	9.9999887 g	9.9999887 g	0.012	8.00	Double Substitution
20	g	19.999987 g	19.999987 g	0.013	8.00	Double Substitution
20	g *	19.999987 g	19.999987 g	0.013	8.00	Double Substitution
50	g	49.999907 g	49.999907 g	0.016	8.00	Double Substitution
100	g	100.00002 g	100.00002 g	0.023	8.00	Double Substitution

**APPENDIX J. List of Equipment Used in Study**

**List of Equipment Used for MACTEC's VC Summer Projects**

<b>Instrument</b>	<b>Room</b>	<b>Building</b>
Balance	B122	773A
Balance	C142	773A
Balance	B046	773A
BET Analyzer	B046	773A
XRF Analyzer	C142	773A
XRD Analyzer	C130	773A
Gamma Analyzer	B022	773A



2/9/2007

**APPENDIX K. Personnel Qualification**



**Approval of Qualification of Personnel Working on VC Summer MACTEC Project.**

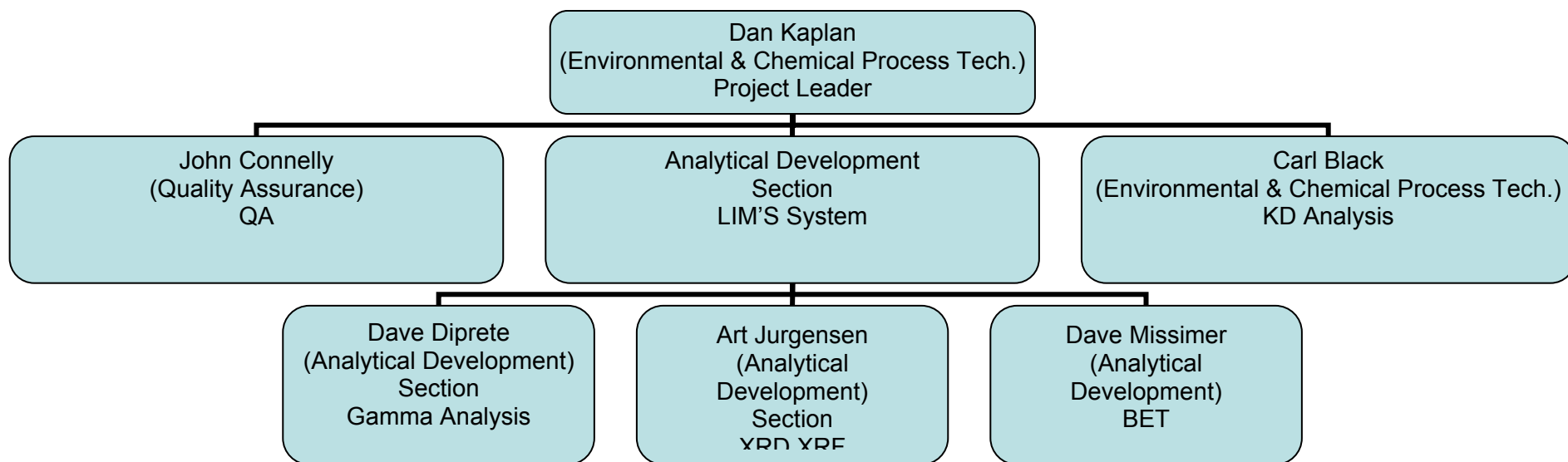
<b>Individuals</b>	<b>Task</b>
Daniel Kaplan	Project Leader, Scientist
Carl Black	Kd Measurement, geochemistry measurements
Dave Diprete	gamma radiation measurements
Art Jurgensen	XRD, XRF
Dave Missimer	BET
John Connelly	QA

I approve that the above personnel are qualified to complete their respective tasks:



2/9/2007

Figure 4. Organizational chart of the personnel working on project.



Personnel qualifications are presented in a separate folder sent together with this document entitled "Appendix K\_Personnel Qualifications" Within that folder are the following five files:

1. Daniel Kaplan.pdf: resume of Dan Kaplan
2. Connelly.pdf: resume of John Connelly
3. Black.pdf: SRNL training record for Carl Black
4. Jugensen.pdf: SRNL training record for Art Jurgensen
5. Missimer.pdf: SRNL training record for David Missimer
6. DiPrete.pdf: resume of David DiPrete

**APPENDIX L. Chain of Custody of Rock/Soil and Groundwater Samples**

Chain of Custody Form



Project Name: SCE&G COL PROJECT  
 Project No. 0234-06-3534  
 Shipped by: Matthew F. Cooke  
 Shipped to: Savannah River National Laboratories

Date: 10/4/2006

Attention of: Don Kaplan

Hazardous materials suspected? yes/no NO

Sample/Test File No	Depth	Material	Field ID No.	Date Sampled	Sample Type (quantity)	Sampled By	Comments
B-212	48.5-50.0	Soil	14	05/08/05	Jar (1)	B. Sharp	
B-212	58.5-60.0	Soil	18	05/08/05	Jar (1)	B. Sharp	
B-620	38.5-40.0	Soil	12	04/07/05	Jar (1)	C. Gaskins	
B-627	23.5-25.0	Soil	9	04/12/05	Jar (1)	C. Gaskins	
B-627	47-48.5, 53.5-55.0	Soil	14/15	4/12/2005 & 4/17/06	Jar (1)	C. Gaskins	
B-205	98.6-99.8	Rock	Run 10	05/09/05	Rock Core	J. Harmon	
B-212A	95.8-97.7	Rock	Run 7	05/15/05	Rock Core	B. Sharp	
B-305	63.3-65.85	Rock	Run 2	05/01/05	Rock Core	J. Liles	
B-305	59.0-59.9	Rock	Run 1	05/01/05	Rock Core	J. Liles	
B-327	59.3-59.1	Rock	Run 2	04/19/05	Rock Core	B. Sharp	
B-335	63.2-64.4	Rock	Run 7	04/19/05	Rock Core	M. Harvey	
B-335	63.2-64.4	Rock	Run 2	04/19/05	Rock Core	C. Gaskins	
B-627	56.8-61.3	Rock	Run 2	04/19/05	Rock Core	C. Gaskins	
OW-265A	NA	Groundwater	NA	09/01/05	1 Liter Bottle (4)	D. Snyder	
OW-212	NA	Groundwater	NA	08/28/05	1 Liter Bottle (4)	D. Snyder	
OW-305A	NA	Groundwater	NA	09/01/05	1 Liter Bottle (4)	D. Snyder	
OW-327	NA	Groundwater	NA	08/28/05	1 Liter Bottle (4)	D. Snyder	
Note:	B-627 Soil Samples 14 and 15 Combined by MFC at site						

Relinquished By (Site Coordinator):

Matt Cooke *[Signature]*  
 Print/Sign

Date/Time:

10/4/06 1230

Received By (Transporter):

Matt Cooke *[Signature]*  
 Print/Sign

Date/Time:

10/4/06 1230

Relinquished By (Transporter):

Matt Cooke *[Signature]*  
 Print/Sign

Date/Time:

10/5/06 1450

Received By (Laboratory):

Daniel Kaplan *[Signature]*  
 Print/Sign

Date/Time:

10/5/06 1450

DCN SC 381

**DISTRIBUTION**

Clay Sams	MACTEC Engineering and Consulting, Inc., 2801 Yorkmont Road, Suite 100, Charlotte, NC 28208 (5 copies)
Dan Kaplan	Savannah River National Laboratory, 773-43A, Rm. 215, Aiken, SC 29808
WPT File	Savannah River National Laboratory, 773-43A, Rm. 213, Aiken, SC (2 copies)

Appendix H

SAVANNAH RIVER NATIONAL LABORATORY  
ANALYTICAL DEVELOPMENT SECTION  
ADS ANALYTICAL OPERATING PROCEDURES

Manual: L16.1  
Procedure: ADS-1114  
Revision: 2  
Page: 1 of 19  
Effective Date: 7/15/05

TECHNICAL REFERENCE

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**Procedure for Operating the Micromeritics ASAP 2010  
Surface Area and Pore Volume Analyzer (U)**

---

Minor Revision

Author/Task Supervisor:

SIGNED COPY ON FILE  
D. M. Missimer

Peer Review:

(Not required for minor revision)  
A. R. Jurgensen

Spectroscopic Research &  
Instrumental Analysis Manager

SIGNED COPY ON FILE  
F. M. Pennebaker

ADS Procedures Coordinator:

SIGNED COPY ON FILE  
L. Hillary

## I. PURPOSE

This procedure provides information and instructions required for routine operation of the Micromeritics Corporation Accelerated Surface Area and Pore Analyzer (ASAP) Model 2010 by technical analysts.

## II. SCOPE AND APPLICATION

This procedure describes the requirements for the use of an automated gas adsorption apparatus to measure adsorption isotherms using nitrogen or krypton at liquid nitrogen temperatures. Included are sample handling and preparation procedures, instructions for sample data acquisition and reduction using Micromeritics Windows 95-based software, required operating conditions, and quality assurance procedures. This procedure applies to the determination of BET surface areas by nitrogen and krypton adsorption, the determination of Langmuir surface areas by nitrogen adsorption, and the determination of pore size distributions by nitrogen adsorption. Other operations, including argon adsorption experiments, micropore adsorption measurements, and density functional theory (DFT) analysis of micropore adsorption data must be performed under direct supervision of the Task Supervisor. **This procedure is suitable for research and development samples only.**

## III. RESPONSIBILITIES

**The Task Supervisor** has overall responsibility for safe, efficient and effective operation of the Micromeritics ASAP 2010. These responsibilities include laboratory safety; analyst training, methods development, and sample scheduling.

**The Technical Analyst** is responsible for all aspects of routine operation such as routine sample analysis, routine maintenance, scheduling service calls, ordering replacement parts, gases, and liquid nitrogen, and keeping notebooks current. The analyst shall notify the Task Supervisor immediately of safety issues related to operations, scheduling problems, or any other issue affecting routine operation of the equipment.



#### IV. PROCEDURE

##### A. Manufacturer

Accelerated Surface Area and Pore Analyzer

Model Number: ASAP 2010

Serial Number: 845

ID Number: 00381999

Micromeritics Instrument Corporation

One Micromeritics Drive

Norcross, Georgia 30093

(770) 662-3666

##### B. DESCRIPTION

###### 1. Principles of Operation

When atoms or molecules of a gas or vapor are subjected to the forces of attraction which exist in the vicinity of the surface of a solid, there is known to be an increase in the concentration of the gas near the interface relative to the bulk gas phase. The accumulation of gas molecules at a surface is known as adsorption. Physical adsorption, or physisorption, occurs as the result of van der Waals forces which act between the adsorbate (gas) molecule and the adsorbent (solid) surface to form the surface excess of gas (adsorbed) molecules. The physical adsorption process may proceed from very low coverage to saturation, where the solid surface is covered with several molecular layers of adsorbed molecules and pores and voids within the solid are filled with gas molecules in a liquid-like state. Note that the opposite of adsorption - desorption - involves the removal of gas from the surface. The state of an adsorbed phase is dependent upon many physicochemical variables, including temperature, pressure, and the nature of both the gas and solid. Surface properties of solid materials, including their state of subdivision, surface area, and porosity, have enormous impact on innumerable natural processes, and knowledge of these properties is fundamental to much of science and technology. Amongst the various means of measuring such properties, methods employing physical adsorption are broadly applied in scientific investigations, product development, and process and quality control. Adsorption data are measured at constant temperature and presented as isotherms with the quantity adsorbed on the y-axis and the equilibrium adsorption pressure on the x-axis.

## 2. Instrument Description

The Micromeritics ASAP 2010 is a state-of-art automated apparatus for measuring adsorption isotherms and calculating surface properties of solid materials from the data

The ASAP 2010 system consists of the analyzer, an interface controller, a computer (referred to as the control module), and printer. The instrument is configured to measure adsorption and desorption isotherms with nitrogen, argon, or krypton at liquid nitrogen temperatures.

The analyzer contains two high vacuum systems, pressure measurement transducers, thermocouples and thermistors for measuring temperature, ports for mounting sample tubes, and a variety of electronic and electrical controls.

The interface controller, the link between the computer and the analyzer, controls the experimental process, manages power failure and power recovery processes, and can operate the analyzer independently in the event of a computer failure.

The Hewlett Packard Vectra 100 computer contains the software for conducting experiments and calculating results. The operator cannot access the interface controller directly and must enter all commands through the computer.

## C. SAFETY PRECAUTIONS

1. All SRS, SRTC, and ADS safety rules apply.
2. For Liquid Nitrogen safety precaution, refer to Procedure ADS-1401.
3. The operator shall be familiar with procedures for using compressed gas cylinders as outlined in the SRS Safety manuals. Argon, helium, krypton, and nitrogen are all non-toxic. A pressure relief valve set at 20 psig is attached to the regulator for each gas cylinder.

D. TRAINING

An Analyst must successfully complete the applicable training package to be qualified. If an analyst is not qualified, data is reviewed by the Task Supervisor.

E. QUALITY ASSURANCE/QUALITY CONTROL1. Registered Notebooks

At the direction of the Task Supervisor, all quality control data, instrument status data, records of preventive maintenance and instrument repairs, and analyst training will be recorded in bound notebooks registered with SRTC Records.

2. Instrument Calibration and QC Standards

There are no certified standards for surface area and pore structure characterization measurements. A number of working reference materials for both surface area measurements and pore characterization are available from Micromeritics Instrument Corporation. The National Institute of Standards and Technology (NIST) has several standard reference materials for BET surface area determination by nitrogen adsorption only. For routine QA applications, an appropriate NIST, Micromeritics, or other reference material will be used as indicated below. For special applications and customer-assisted QA, applicable reference materials will be chosen on an individual basis. Results will be documented in a notebook.

3. Calibration Frequency

The frequency of calibration of the ASAP 2010 is based in part on the one-station configuration of the apparatus and elapsed times requirement for a run. A calibration run for a nitrogen five-point BET analysis requires approximately six hours. At best, only two surface area runs can be made in one normal workday, so it is impractical to bracket each sample with calibration runs. As indicated above, customer assisted QA requirements will be developed specific to each case. For ADS Routine QA, the apparatus shall be calibrated with a BET Surface Area reference material of surface of appropriate surface area range as follows:

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- a. at the beginning of the normal workweek;
- b. whenever analysis gas is changed -e.g. from nitrogen to krypton;
- c. following maintenance on the analysis vacuum system;
- d. following calibration of the calibrated manifold;
- e. when changing sample surface area ranges - e.g. from low surface area to high surface area.

Standard reference material surface area determinations shall fall within the uncertainty stated on the certificate before customer analyses are run. All analyses run within an interval preceding a failed calibration run shall be rerun.

## F. OPERATING PROCEDURES

### 1. Preliminary Information

#### a. Turning the ASAP 2010 System On

Place the power switches for the computer and printer in the ON position.

Place the power switch for the Interface controller in the ON position

Place the analyzer main power switch in the ON position, and verify that the red power supply indicators on the back of the analyzer are illuminated.

#### b. Turning the ASAP 2010 System Off

Always exit from the ASAP 2010 program before turning the system off.

Turn system components OFF in the same order as they were turned ON (see above).

#### c. Vacuum Systems

The ASAP 2010 contains two separate vacuum systems. The degas vacuum system is capable of attaining vacuum of about 0.0005 Torr and is used to remove water and other adsorbed contaminants from samples before they are analyzed. The degas system is connected to two sample degas ports on the front of the instrument. The analysis

vacuum system is capable of reaching 0.000005 Torr and is connected to the single analysis sample port. Periodic maintenance requirements, routine maintenance procedures, and leak test procedures for the vacuum systems are given in Chapter 9 of the ASAP 2010 Operator's Manual. **System maintenance shall be performed under the direction of the Task Supervisor or a Senior Analyst and are not within the scope of this procedure.**

d. Filling the Cold Trap Dewar

Both vacuum systems are serviced by cold traps cooled by liquid nitrogen dewar within the COLD TRAP dewar. The COLD TRAP dewar must be filled about every three days.

To fill the COLD TRAP dewar, proceed as follows:

remove the blue insulator/stopper from the dewar

lift the dewar up slightly above the mounting hook and lower until clear of the two glass cold trap tubes

fill the dewar with liquid nitrogen to about 5 cm from the top;

hang the dewar on the mounting hook support and place the blue insulator/stopper over the opening.

**Note** About once each week, allow the cold trap tubes to warm to room temperature before replacing the dewar. This will allow condensed vapors within the traps to be pumped off and assure the maintenance of good vacuua. Dry the exterior of the cold traps with paper towels before reinstalling the dewar.

e. Filling the Analysis Dewar

The sample tube is immersed in liquid nitrogen within the ANALYSIS dewar during analysis. The ANALYSIS dewar must be filled and installed on the dewar elevator just prior to beginning a run as follows:

Fill the analysis dewar to the correct level with liquid nitrogen. The correct level is indicated when liquid just covers the hole in the analysis dewar dipstick.

Place the analysis dewar on the dewar elevator.

The analysis dewar contains enough cryogen for about forty-eight hours of operation.

**NOTE: The analysis dewar should never be allowed to become depleted of liquid nitrogen during an analysis. To do so could result in high gas pressures within the vacuum system and possible damage to the apparatus.**

## 2. PREPARING FOR AN ANALYSIS

### a. Creating a Sample Information File

The ASAP 2010 runs sample analyses and processes adsorption data on the basis of sample information files. Each sample and associated files are identified by five labels as defined below:

**SEQUENCE NUMBER** a number of the form XXX-XXX beginning with 000-000 and incremented by 1 automatically whenever a new sample information file is created.

e.g. **000.001**

**SAMPLE FILENAME** The sample filename is simply the sequence number "dot" smp

e.g. **000-000.smp**

**SAMPLE ID NUMBER** a maximum of 40 characters sample identifier of the form [Sample Number - ADS Sample Number - Description]

e.g. **000-100.smp-300066666-soil from tank farm**

**OPERATOR ID** any entry of up to 40 characters,

e.g. **Ronny and Donnie**

**SUBMITTER ID** any entry of up to 40 characters,

e.g. **Frank and Jessie James-DWPF**

A sample information file is divided into the following five sections:

**SAMPLE INFORMATION  
ANALYSIS CONDITIONS**

**ADSORPTIVE PROPERTIES  
REPORT OPTIONS  
COLLECTED DATA**

Each section and applicable information and parameters will be discussed below.

**SAMPLE INFORMATION**

To create a new sample file in preparation for a sample run, click on the FILE header, select OPEN, and then select SAMPLE INFORMATION to the right and the open sample information dialogue window will appear. Type the desired sample filename into the filename box and click OK. The prompt "This file does not exist would you like to create it" will appear. Click OK, and the Sample Information window will appear. Enter the SAMPLE ID, OPERATOR ID, and SUBMITTER ID in the appropriate fields. Under TYPE OF DATA click the circle labeled AUTOMATICALLY RECORDED. Enter the Sample Weight, then click the SAVE button at the lower left of the window.

**ANALYSIS CONDITIONS**

Click on the top folder tab labeled analysis conditions and the analysis conditions window will appear. In the Sample Preparation box, turn the Fast Evacuation switch off - no X in the box. In the Unrestricted Evacuation box enter 5.0, and 0.5 in the Evacuation Time box. In the Analysis Data box enter:

Equilibration Time	10.0 sec;
Click on the Maximum Volume Increment box and enter	25.00 cc/gm @ STP;
Relative Target Tolerance	5.0 %;
Absolute Target Tolerance	5.000 mm Hg;
Minimum Equilibration Delay	600 sec.

Next, click on the FREESPACE button at the upper right of the window. Enter the following information:

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Freespace	click Measure;
Post Freespace Evacuation	click Lower Dewar;
Evacuation time	0.5 hours;
Leak Test Duration	180 sec.

Click OK and the display will return to the Analysis Conditions window. Next, click on the Pressure button. The pressure table box will appear. Pressure data points are entered into the table as **relative pressure** values. Attachment B gives a relative pressure table for nitrogen and krypton BET analysis. Attachment C gives a relative pressure table for complete nitrogen adsorption and desorption isotherms for BET analysis and BJH pore analysis. Enter values from the appropriate table in the attachments or other pressure values as directed by the Task Supervisor. When completed, click OK and return to Analysis Conditions.

Next, click the Low Pressure Option button and assure that both the Fixed Dose Mode and the Incremental Dose Mode are off e.g. no X in either box. Click OK and return to Analysis Conditions.

Next Click the P0 and T button and enter the following information:

P0	click on measure;
Analysis Bath Temperature	Click on Calculate from measured Psat;
Measurement Interval	120 minutes.

Click on OK, return to Analysis Conditions and click on the Backfill button and enter the following information:

Backfill Sample at Start of Analysis	click On;
Backfill Sample a End of Analysis	click On;
Backfill Gas	if the analysis gas is krypton, click Psat gas;



if the analysis gas is nitrogen,  
click Analysis Gas.

Click OK and return to the Analysis Conditions window.

## ADSORPTIVE PROPERTIES

Click on the Adsorptive Properties tab at the top of the window. If the analysis gas is nitrogen, click on the Replace button and the Adsorptive file window will appear. Scroll through the files and find Nitrogen @ 77.35 Degrees Kelvin. Click on the file and then click OK. The Adsorptive Properties window will return and the appropriate values for nitrogen adsorption will be in the tables. If the analysis gas is krypton, repeat the above process using Krypton @ 77.35 Degrees Kelvin. Finally, under the Dosing Method, click on Normal, then on Save at the bottom of the window.

## REPORT OPTIONS

Next, click on the Report Options button and enter the options appropriate to the analysis to be conducted as follows:

<b>Krypton BET or Nitrogen BET</b>	Isotherm BET Surface Area Options
<b>Nitrogen Adsorption and Desorption Pore Analysis</b>	Isotherm BET Surface Area Langmuir Surface Area t - Plot Surface Area BJH Adsorption BJH Desorption Options

Click on Save and Close. The completed sample information file may now be called up for analysis or for additional editing.

### b. Preparing the Sample

Samples of porous materials submitted for analysis may vary in form from extremely fine powders and soft agglomerates to hard chunks or combinations of physical forms. If necessary, very large chunks may be broken into smaller pieces so that they may be introduced into the sample cell. Do not alter the state of subdivision of the sample material more than is

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necessary to load the sample unless instructed to do so by the customer or Task Supervisor.

All samples and reference materials to be analyzed on the ASAP 2010 contain adsorbed water and other vapor or gaseous contaminants that must be removed before analysis by degassing the sample. To prepare and degas a sample, proceed as follows:

1. Weighing the Sample

Obtain a clean and dry sample, tube seal frit, and filler rod (if necessary). As a general rule, use a filler rod if the total surface area in the tube is (or is suspected of being) less than about 20 square meters/gram.

Weigh the tube and seal frit and record the empty weight on an ASAP 2010 Sample Data Worksheet (Attachment A).

Add an appropriate amount of sample or reference material to the tube. Reference materials data certificates will give recommended sample weights for the reference material. Appropriate sample weights for unknowns must sometimes be determined by trial and error. The Task Supervisor or a Senior Analyst will recommend an appropriate weight for a sample of unknown surface area.

Weigh the tube, seal frit, and sample and record the BEFORE DEGAS weight on the Sample Data Worksheet.

2. Degassing the Sample

Remove the degas port plug by holding the plug with one hand and turning the knurled connector nut counterclockwise.

Place the connector nut, steel ferrule, and O-ring onto the sample tube. Firmly insert the seal frit into the sample port. Be sure to push the sample tube into the port to a full stop. Tighten the connector nut clockwise until the tube is tightly secured.

Place a heating mantle on the sample tube and secure the mantle in place with a mantle clip. Insert the heating mantle power plug into the appropriate connector (left or right). Insert the mantle thermocouple plug into the appropriate connector.

All degas operations are managed from the control panel on the lower front of the Analyzer. Temperature controls for each degas station are clearly labeled. The current temperature of the heating mantle will be displayed in the Degas Temperature indicator. Set the desired degassing temperature for the degas port in degrees Celsius (ambient to 350 degrees C) at the Heating Indicator set-switches. Use the - switches to reduce a digit and the + switches to increase a digit.

Set the Degas Vac Set switches to 500 micro torr unless instructed by the Task Supervisor or Senior Analyst to use another setting.

Place the AUTOMANUAL switch in the manual position. Press LOAD, then the desired degas port switch, LEFT or RIGHT, and then BEGIN. Next press the heater ENABLE switch for the degas port being used. The analyzer will begin the automatic degas cycle. The analyst must manually time the degas cycle from the time the ready light on the degas panel is lit until the appropriate time has elapsed.

When the degas cycle is completed press UNLOAD, press the degas port switch, LEFT or RIGHT, and than press BEGIN. Press the ENABLE switch for the heater controller to turn the heating mantle off. The degas system will automatically back fill the sample tube with nitrogen to a pressure of one atmosphere and evacuate the degas vacuum system. Remove the heating mantle and allow the tube to cool to room temperature before removing it from the degas port.

### 3. Transferring the Sample to the Analysis Port

Remove the sample from the degas port, weigh the degassed tube, sample, and seal frit and record the degassed weight on the Sample Data Worksheet. Place a filler rod in the sample tube if necessary.

Place an Isothermal Jacket on the sample tube such that the bottom of the jacket rests on the sample bulb.

Remove the plug from the analysis port. Place the connector nut, ferrule, and O-ring on the tube and insert the tube firmly into the analysis port. Tighten the connector nut **firmly** finger tight.

#### 4. Preparing for an Analysis Run

Check that the cold Trap dewar was filled within the previous twenty-four hours.

Fill the analysis dewar with liquid nitrogen and place it on the elevator. Visually inspect the dewar and sample tube to make certain they are properly aligned.

Check analysis gasses. All appropriate gas cylinder valves and shutoff Nupro valves, and the correct analysis gas Nupro shutoff valve on the back of the analyzer must be open. Set regulated pressure to 6 to 10 psig. NOTE - If the analysis gas is krypton, open the krypton cylinder valve briefly and then close it with the regulated pressure at 6 to 10 psig. This will provide more than enough krypton gas to run an analysis.

### 3. CONDUCTING AN ANALYSIS

To begin an analysis, select Analyze on the Main Menu to display the Start Analysis dialogue box. Under Analysis Settings, select no report after analysis by clicking the box until there is no "x" in the box. Select the filename for the sample to be analyzed by clicking on it in the file box. This will cause the correct filename to be displayed in the filename box at the top of the window. When ready to begin, click OK at the bottom of the window and the analysis will begin.

During the automatic analysis, the ASAP 2010 software displays ongoing pressure measurements, the current analysis step, the latest data point taken, and the elapsed time. Additionally, a report may be presented based on the data taken up to that time. Simply click on the report button at the lower left of the ASAP 2010 display. Route the report to the screen when the dialogue box appears, and click OK. The report will appear on the screen and the analyst may scroll through the various pages using the Nxt>> and <<Prev buttons. To exit the report and return to the 2010 display, simply click close.

Three software buttons labeled SKIP, SUSPEND, and CANCEL are displayed on the left side of the window. The analyst shall not use the SKIP or SUSPEND button options unless instructed to do so by the Task Supervisor or a Senior Analyst. The CANCEL button will cause the apparatus to abort the current run and safely return to the idle condition. The analyst may use the CANCEL button option to terminate a run for

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reasons relating to safety or operational necessity without consulting the Task Supervisor.

The ASAP 2010 is fully automated and will complete the adsorption analysis before returning to the ready condition. When an analysis is completed, the sample dewar will be in the down position and the Display will show the instrument status as Idle.

#### 4. Printing Reports

To print an analysis report, first check to be sure the printer power switch is ON. Select Start Report from the Reports menu to display the Start Report dialogue box. Select and highlight the file from which the report is to be generated from the filename box. Select Printer under Destination, and click OK.

### V. REFERENCE

Micromeritics ASAP 2010 Operator's Manual  
Manual L16.1, Procedure ADS-1401, "Liquid Nitrogen Dispensing (U)." |

### VI. ATTACHMENTS

- Attachment A: Micromeritics ASAP 2010 Sample Data Worksheet
- Attachment B: Relative Pressure Table for Five-Point BET Nitrogen or Krypton Surface Area analysis
- Attachment C: Relative Pressure Table for Full Nitrogen Adsorption/Desorption Surface and Pore Analysis

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ATTACHMENT A  
Micromeritics ASAP 2010 Sample Data Worksheet

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ATTACHMENT B  
Relative Pressure Table for Five-Point BET  
Nitrogen or Krypton Surface Area analysis

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Relative Pressure	BT	LA	TP	BA	BD	TP	HK	DU	MP
0.01000	X								
0.03000	X								
0.05000	X								
0.08000	X								
0.10000	X								
0.15000	X								
0.20000	X								
0.25000	X								

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ATTACHMENT C  
 Relative Pressure Table for Full Nitrogen  
 Adsorption/Desorption Surface and Pore Analysis

Relative Pressure	BT	LA	TP	BA	BD	TP	HK	DU	MP
0.01000			X						
0.03000			X						
0.06000	X	X	X						
0.10000	X	X	X						
0.15000	X	X	X	X					
0.20000	X	X	X	X					
0.25000	X	X	X	X					
0.30000		X	X	X					
0.35000		X	X	X					
0.40000		X	X	X					
0.45000		X	X	X					
0.50000		X	X	X					
0.55000		X	X	X					
0.60000		X	X	X					
0.65000			X	X					
0.70000				X					
0.75000				X					
0.80000				X					
0.85000				X					
0.90000				X					
0.92500				X					
0.95000				X					
0.97500				X					
0.98000				X					
0.99500				X					
0.98000				X					
0.97500				X					
0.95000				X					
0.94000				X					



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ATTACHMENT C (Continued)  
 Relative Pressure Table for Full Nitrogen  
 Adsorption/Desorption Surface and Pore Analysis

Relative Pressure	BT	LA	TP	BA	BD	TP	HK	DU	MP
0.93000					X				
0.90000					X				
0.85000					X				
0.80000					X				
0.75000					X				
0.70000					X				
0.65000					X				
0.60000					X				
0.55000					X				
0.50000					X				
0.45000					X				
0.40000					X				
0.35000					X				
0.30000					X				
0.25000					X				

RIGAKU ZSX 101E


Wavelength Dispersive X-Ray Fluorescence System (U)

Signature

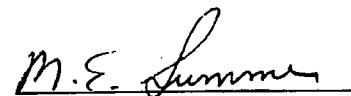
Author:

  
D. M. Missimer

Analytical Task Supervisor:

  
A. R. Jorgensen

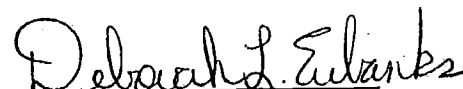
Technical Reviewer:

  
M. E. Summer

AD Group Manager:

  
F. Pennebaker

AD Procedure Coordinator:

  
D. Eubanks

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## 1.0 PURPOSE

This procedure provides requirements for the wavelength dispersive x-ray fluorescence analysis of non-radioactive macroscopic samples by the Analytical Development (AD).

## 2.0 SCOPE

This procedure applies to all wavelength dispersive x-ray fluorescence analyses of non-radioactive macroscopic materials performed by AD. This includes qualitative, semi-quantitative, and quantitative determinations.

### 2.1 Definitions

- A. X-Rays - X-rays may be defined as electromagnetic radiation of wavelength  $\sim 10^{-5}$  to  $100 \text{ \AA}$  produced by the deceleration of high-energy electrons and/or electron transitions in the inner orbits of atoms.
- B. X-Ray Fluorescence - X-ray fluorescence is simply the characteristic secondary spectral line x-ray emission produced by an element. The mechanism of production of these characteristic x-ray lines involves the removal of one of the inner shell electrons to create a vacancy in the electronic structure of the atom. This vacancy is filled by one of the electrons in a higher energy shell. To maintain conservation of energy an x-ray photon, whose energy is the difference between the two levels, is produced.

### 2.2 Responsibilities

A. Technical Analyst is responsible for:

- Preparing samples for x-ray fluorescence. This includes grinding the sample to the proper particle size range, fusing sample with appropriate flux, pressing pellets with binder, polishing samples, fabricating custom mounts if necessary, and positioning the sample properly in the mount and spectrometer.
- Obtaining x-ray fluorescence patterns. This involves operating the x-ray generator, autosampler, filters, detectors, collimators,

mask size, safety interlocks, Rigaku software, and computer peripherals.

- Performing qualitative elemental analysis of sample data.
- Making simple electrical and mechanical repairs to the instrument and printer.
- Maintaining safety inspections records, the XRF sample logbook, XRF Calibration notebook, XRF maintenance notebook, and all other general documentation required for laboratory operation.

**B. Task Supervisor is responsible for:**

- Developing specimen preparation methods for proper presentation of the sample to the instrument.
- Performing data reduction, analysis, and presentation of results using Rigaku software programs or other software packages, and associated computer equipment. This includes elemental identification, semi-quantitative, and quantitative analysis.
- Keeping abreast of equipment and technology developments to maintain a current complement of equipment, procedures, and software.
- Communicating with customers to make sure that they are obtaining the correct and most effective analysis.

**C. Level 4 Manager (or designee) is responsible for reviewing and approving the operating procedure.**

### **3.0 PRECAUTIONS/LIMITATIONS**

#### **3.1 Safety**

##### **A. Radiation Control**

- Access to the laboratory is limited by combination locks on the doors per DOE G 441.1-5, Radiation Generating Devices Guide,

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for use with Title 10, Code of Federal Regulations, Part 835, Occupational Radiation Protection. Only authorized equipment operators and Building Services personnel are permitted in the laboratory.

- Personnel using this facility are subject to requirements as specified in the 5Q and L1 Manuals, Radiation Worker II Training, Radiation Generating Device Training, and the XRD Operating Procedure.

### B. X-ray Exposure

- The main hazard associated with the use of x-ray equipment is overexposure to x-rays. The spectrometer, as currently configured, is an open beam instrument. However, because the equipment enclosure provides a radiation shield, there should be no personnel exposure unless it is bypassed or fails. This procedure complies with WSRC 5Q1.1-524, Ionizing Radiation Generating Device Control Program and WSRC 5Q1.1-525 Inspection and Survey of RGD's.
- X-ray equipment may only be used following posted operating procedures, or with a written job plan approved by the Task Supervisor.
- RCO will conduct a semi-annual radiological survey to check for any radiation leakage and to test equipment safety interlocks as required by WSRC 6Q1.1-525, Inspection and Survey of RGD's..
- A series of interlocks on the equipment doors is provided to enable repairs and alignment with x-rays on. The Task Supervisor must approve the use of the interlocks. Any repairs or alignment must be done at as low x-ray power as possible.

### C. High Voltage

The other main danger associated with operating x-ray equipment is from high voltage. The generator can produce up to 60 kV at 100 mA, but the total power is limited to 4 kilowatts. The unit should be shut down immediately if any electrical discharge is heard or seen. If any insulation is worn on any wiring, the unit should not

be used. Entry into the generator cabinet requires vendor-trained personnel with either x-ray personnel trained on 18 Q procedure 2, completed copy of an OSR 20-202, and additional SRNL Requirements (SRQEW000), or a Work Clearance Permit with E & I present.

#### D. Modification Requirement

Modification to and/or substitution of critical components in the control circuitry of existing x-ray apparatus must be approved by RCPO and be in compliance with the intent of ANSI N43.2 and ANSI N543. Proper documentation of the modification and/or substitution must be provided for custodial and RCO files. Appropriate revisions of schematics and prints are required.

#### 4.0 PREREQUISITE ACTIONS

**NOTE:** Task Supervisor must review work done by the Analyst before being submitted to customer.

#### 5.0 PERFORMANCE

##### 5.1 General Information

###### 5.1.1 Description of Method

The Rigaku ZSX 101E system is a computer-controlled x-ray fluorescence spectrometer used for non-destructive elemental analyses of elements from B (Z=4) to Pu (Z=94). Basically, a sample is irradiated with x-rays, which cause the loss of inner shell electrons from the constituent atoms. These inner orbital vacancies can be filled by electrons in a higher energy shell. To maintain conservation of energy an x-ray photon, whose energy is the difference between the two levels, is produced. The energy of each emitted x-ray photon is well-defined and characteristic of the element from which it originated. The rate at which x-ray photons are emitted by a particular element is related to the concentration of the element.

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The ZSX system features two detectors: a scintillation detector and a gas flow proportional counter (detector). It also has nine analyzing crystals, three collimators, a programmable mask, and a twelve-position auto-sampler. Analysis can either be done in air, vacuum, or helium.

### 5.1.2 Instrumentation

#### 5.1.2.1 MS&E

Model #	ZSX101E
Serial #	ZR03005
Capital Equipment #	00397418

#### 5.1.2.2 M&TE

The M&TE Program Controls the four place balance.

### 5.1.3 Manufacturer

Rigaku/MSC, Inc.  
9009 New Trails Drive  
The Woodlands, TX 77381  
Telephone: (281) 363-3628

## 5.2 Calibration and Measurement Control Program

If the results for any of the tests are out of specifications, see the Task supervisor. It may be necessary to generate a **Non-Conformance Report (NCR)** for any data generated since the last acceptable calibration check.

Calibration curves are generated from standards, which bracket the composition of the unknown sample, per section 5.4.4.1 section F and G.

These tests can be performed easily and quickly and shall be repeated at least once a month by the Analyst to ensure optimum system performance. If the results are out of specifications, see the Task supervisor.

### 5.2.1 Pulse Height Analyzer Adjustment Check

This check shall be performed monthly by the analyst to monitor the detectors performance.

- 5.2.1.1 Open valve for the primary water.
- 5.2.1.2 Turn the **Rigaku** and the **Haskris** chiller on.
- 5.2.1.3 Set the rate of flow at **P-10** gas to **50 ml/min**.
- 5.2.1.4 Turn on the main power switch to the instrument, the printer, and the computer.
- 5.2.1.5 After the computer has booted up and the desktop appears, type in "**RIGAKU**" in for the password.
- 5.2.1.6 When the conductivity is **below 1 $\mu$ S/cm** on the chiller, click the **ZSX** button on the desktop. Wait for the system to initialize. The **Spectrometer Status** menu will appear.
- 5.2.1.7 Click on the **Startup/Shutdown** box on the main tool bar. A drop down menu will appear.
- 5.2.1.8 Select the **Startup** option.
- 5.2.1.9 Click on the **Turn on X-ray** and the **Age X-ray tube** boxes.

**NOTE: Only use the Age X-ray Tube options if the system was shutdown over the weekend, power outage, or changing out the P-10 gas.**

- 5.2.1.10 Click the **Start** button.



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- 5.2.1.11 After the X-ray tube and the Aging process is complete, Click on the "X" in the upper right hand corner to close the window.
- 5.2.1.12 Click the **Startup/Shutdown** option on the main toolbar. A drop down menu will appear with several options.
- 5.2.1.13 Select the **Tube/Atmosphere** option on the drop down menu.
- 5.2.1.14 Click on the **Voltage and Current** Radial box and use the pull down scroll bars to increase the voltage and current gradually to 50, 50. If the Aging process was done, the voltage and current will be at 50, 50.

**NOTE: When increasing the voltage and current use small increments over a 15 minute time frame. (E.g. Increase the voltage to 25 and the current to 10 and wait a couple of minutes.)**

- 5.2.1.15 Click the **Start** button.
- 5.2.1.16 The process is complete when the radial box does not contain a black dot.
- 5.2.1.17 Repeat steps 5.2.1.14 through 5.2.1.16 until the voltage and current is 50, 50.
- 5.2.1.18 After setting the voltage and current, click on the **Switch Atmosphere** radial box and **Select Vacuum** with the scroll bar.
- 5.2.1.19 The process is complete when the radial box does not contain a black dot.
- 5.2.1.20 Click the "X" on the upper right hand side of the menu to close the **Tube/Atmosphere Change** menu.

- 5.2.1.21 Place the **PHA** sample in a sample cup with a 30mm mask. The **PHA** sample is stored in the desiccator on the counter top.
- 5.2.1.22 Place the Sample cup in **Position 1** on the sample wheel.
- 5.2.1.23 Click on the **Startup/Shutdown** option on the main toolbar.
- 5.2.1.24 Click on the **Startup** option from the drop down menu.
- 5.2.1.25 Select the **Adjust PHA** radial box and Wait radial box.
- 5.2.1.26 Select a **Time** for the **Wait** option.
- 5.2.1.27 Click **Position 1** with the pull down scroll bar.
- 5.2.1.28 Click the boxes next to **PC** and **SC**.
- 5.2.1.29 Click the **Start** button.
- 5.2.1.30 Limits for both detectors are as follows:
- |                       | <u>ER</u>  | <u>%</u> |
|-----------------------|------------|----------|
| Scintillation counter | around 200 | <40      |
| Proportional counter  | around 200 | <35      |
- 5.2.1.31 If there is a problem, contact the Task Supervisor.
- 5.2.1.32 After the measurement is completed, click on the "X" on the upper right hand corner of the Startup menu.
- 5.2.1.33 Input the information into the **Rigaku Calibration Notebook**.

## 5.2.2 Intensity Measurement Check

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This check shall be done monthly by the analyst to monitor the x-ray tube performance.

- 5.2.2.1 Open valve for the primary water.
- 5.2.2.2 Turn the **Rigaku** and the **Haskris** chillers on.
- 5.2.2.3 Set the rate of flow at **P-10** gas to **50 ml/min**.
- 5.2.2.4 Turn on the main power switch to the instrument, the printer, and the computer.
- 5.2.2.5 After the computer has booted up and the desktop appears, type in "**RIGAKU**" in for the password.
- 5.2.2.6 When the conductivity is below  $1\mu\text{S/cm}$  on the Rigaku chiller, double click the **ZSX** button on the desktop. Wait for the system to initialize. The **Spectrometer Status** menu will appear.
- 5.2.2.7 Click on the **Startup/Shutdown** option on the main tool bar. A drop down menu will appear.
- 5.2.2.8 Select the **Startup** option.
- 5.2.2.9 Click the **Turn on X-ray** and the **Age X-ray Tube** boxes.

**NOTE: Only use the Age X-ray Tube option if the system was shutdown over the weekend, power outage, or after changing out the P-10**

- 5.2.2.10 Click the **Start** button.
- 5.2.2.11 After the X-ray tube and the Aging process is complete Click the "**X**" in the upper right hand corner to close the window.
- 5.2.2.12 Click the **Startup/Shutdown** option in the main tool bar.

- 5.2.2.13 Select the **Tube/Atmosphere** option from the drop down menu.
- 5.2.2.14 Click on the **Voltage and Current** Radial box and use the pull down scroll bars to increase the voltage and current gradually to 50, 50. If the Aging process was done, the voltage and current will be at 50, 50.

**NOTE: When increasing the voltage and current use small increments over a 15minute time frame. (E.g. Increase the voltage to 25 and the current to 10 and wait a couple of minutes)**

- 5.2.2.15 Click the **Start** button.
- 5.2.2.16 The process is complete when the radial box does not contain a black dot.
- 5.2.2.17 Repeat steps 5.2.2.14 through 5.2.2.16 until the voltage and current is 50, 50.
- 5.2.2.18 After setting the voltage and current, click on the **Switch Atmosphere** radial box and **Select Vacuum** with the pull down scroll bar.
- 5.2.2.19 The process is complete when the radial box does not contain a black dot.
- 5.2.2.20 Click the **"X"** on the upper right hand side to close the **Tube/Atmosphere Change** menu.
- 5.2.2.21 Wait a  $\frac{1}{2}$  hour after turning up the generator before starting the measurement to let the instrument warm up.
- 5.2.2.22 Place the **WCP Blend Glass** standard in a sample cup with a 30mm mask.
- 5.2.2.23 Place the sample cup into **Position 1** on the sample wheel.

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- 5.2.2.24 Click on the **Analysis** button on the main tool bar.
- 5.2.2.25 Double Click on the first **BLANK** line. A **Sample ID** setting menu will appear.
- 5.2.2.26 Click on the **Quant** button.
- 5.2.2.27 Select **Position "1"** with the scroll bar.
- 5.2.2.28 Input **WCP Blend Glass** for the **Sample Name**.
- 5.2.2.29 Select the **Intensity Meas.** radial box.
- 5.2.2.30 Select **WCP Blend Glass** from the **Sample Type** in the Analytical Condition.
- 5.2.2.31 To remove the Result Output Information, click on the box next to Trans Format. This is used for sending information to Uniquant.
- 5.2.2.32 Click **OK**.
- 5.2.2.33 Click on the **Analyze** button.
- 5.2.2.34 Input the information into the **Rigaku Calibration Notebook**. As the x-ray tube ages the intensity will steadily decrease, an abrupt lowering of the intensities indicates that one of the crystals is loose or the x-ray tube needs to be replaced.

**NOTE: The program BLEN is already set up. Changes can be made to BLEN by clicking the Quant button on the main toll bar. Click on modify and select glass.**

### 5.3 Standards

The following is a series standards and manufacturers: Metal Alloys, Glasses, and Geological standards from NIST, BCS, ARM, Brammer,

and USGS. The Certificate of Analyses for the standards is kept in the lab. None of these standards have an expiration date.

## 5.4 Operating Procedure

### 5.4.1 Emergency Shutdown

- Have Building Operations shut off Disconnect Switch C on wall inside of C-142/146.
- Call Task Supervisor.

Emergency shutdown is required in the event of general fire, and flooding. This procedure is intended primarily for Building Operations or Fire Department personnel when problems develop off-shift.

### 5.4.2 Reagents and Chemicals

Small amounts (<1 g) of spectroblend, paraffin wax, copolywax, or cellulose are used to prepare pressed powdered samples. Lithium metaborate, lithium tetraborate, and lithium fluoride are used to make fused sample disks. None of the reagents and chemicals used to prepare the samples has an expiration date.

### 5.4.3 Normal Operating Procedure for Rigaku X-ray Spectrometer

#### 5.4.3.1 Specimen Preparation

##### 1. Bulk Solids

When bulk solid specimens are analyzed directly, a major cause of error arises from improper surface preparation. This effect is more severe when longer wavelength (low energy) x-rays are being measured. The penetration depth of the x-rays into a solid surface is very shallow. Minor surface irregularities act as heterogeneities in the path of the primary exciting x-rays and have even a greater effect on the

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secondary fluorescent x-rays. The amount of allowable surface roughness is dependent upon the mass absorption coefficient of the whole sample and the x-ray energy of the measured analyte photon. For quantitative determinations most samples will have to be polished using a series of graded abrasives (silicon carbide paper and diamond pastes) to as low as a 0.1  $\mu\text{m}$  finish. See Task supervisor for more details.

### 2. Powders

Powdered samples can be analyzed qualitatively in loose form. For quantitative determinations it is necessary to eliminate or reduce the wide density and compositional variations and particle size effects that occur when a powder is poured into a specimen holder. These variations can be reduced by pressing the <400 mesh powder in a die at pressures ranging from 20,000 to 100,000 psi. Often binders (copolywax, spectroblend, or paraffin wax) are added (~10 wt%) prior to pressing in order to form a durable pellet.

Another method to reduce or eliminate interelement and particle size effects is fusing the sample into a borate glass matrix. The method consists of weighing 1 g of the sample, 5 g of flux (lithium metaborate and/or lithium tetraborate), and 2 drops of a 33 wt% lithium bromide solution. The glass is cast to form a disk having a smooth surface. See Task Supervisor for more details and Attachment 4.

### 3. Liquids

Liquids represent a nearly ideal specimen type. The sample is homogeneous and obviously no particle size effects are present. A thin film sample support is positioned over a disposable polyethylene cell, and a snap-on ring is placed on it and pushed downward to complete assembly. The cell is inverted and a

sample is introduced through the top opening and presented for analysis. Several thin film sample supports are available including: Prolene (best transmission for low energy x-rays), Kapton (highest purity), polycarbonate, Mylar, and polypropylene. See Task Supervisor for more details.

**NOTE:** When using liquids, make sure the vacuum is **OFF**.

#### 5.4.3.2 Turning the Instrument On

- 5.4.3.2.1 Open the valve for the primary water and let the primary water flow into the heat exchanger. The valve is located on the inside wall of C142/146.
- 5.4.3.2.2 Turn on the **Rigaku** and the **Haskris** chillers. The switch is located on the right-hand side of the Rigaku chiller. The switch for the Haskris chiller is on the front panel. (The conductivity must be below 1  $\mu\text{S}/\text{cm}$  before turning the x-ray generator on.)
- 5.4.3.2.3 Adjust the rate of flow of **P-10** gas from **5ml/min to 50ml/min**. A flow gauge is located behind a hinged door on the front panel of the instrument.
- 5.4.3.2.4 Turn on the main power switch to the instrument, the printer, and the computer.
- 5.4.3.2.5 After the computer has booted up and the desktop appears, type in "**RIGAKU**" in for the password.
- 5.4.3.2.6 When the conductivity is below 1  $\mu\text{S}/\text{cm}$  on the Rigaku chiller, double click the **ZSX** button on the desktop. Wait for the system



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to initialize. The **Spectrometer Status Menu** will appear.

- 5.4.3.2.7 Click on the **Startup/Shutdown** button on the main tool bar. A drop down menu will appear.
- 5.4.3.2.8 Select the **Startup** option.
- 5.4.3.2.9 Click on the **Turn on X-ray** and the **Age X-ray tube** boxes.

**NOTE: Only use the Age X-ray Tube option if the system was shutdown over the weekend, power outage, or changing out the P-10 gas.**

- 5.4.3.2.10 Click the **Start** button.
- 5.4.3.2.11 After the X-ray tube is turned on and the Aging process is complete, click on the "X" in the upper right hand corner to close the window.
- 5.4.3.2.12 Click on the **Startup/Shutdown** button on the main tool bar.
- 5.4.3.2.13 Select the **Tube/ Atmosphere** option on the drop down menu.
- 5.4.3.2.14 Click on the **Voltage and Current Radial** box and use the pull down scroll bars to increase the voltage and current gradually to 50, 50. If the Aging process was done, the voltage and current will be at 50, 50.

**NOTE: When increasing the voltage and current use small increments over a 15minute time frame. (E.g. Increase the voltage to 25 and the current to 10 and wait a couple of minutes)**

- 5.4.3.2.15 Click the **Start** button.
- 5.4.3.2.16 The process is complete when the radial box does not contain a black dot.
- 5.4.3.2.17 Repeat steps 5.4.3.2.14 through 5.4.3.2.16 until the voltage and current is 50, 50.
- 5.4.3.2.18 After setting the voltage and current click on the **Switch Atmosphere** radial box and **Select Vacuum** with the scroll bar.
- 5.4.3.2.19 The process is complete when the radial box does not contain a black dot.
- 5.4.3.2.20 Click on the "X" on the upper right hand side to close the Tube/Atmosphere Change menu.
- 5.4.3.2.21 Wait a **1/2 hour** after turning up the generator before starting any measurement to let the instrument warm up.

#### 5.4.4. Procedure for General Quantitative and Qualitative Analysis

##### 5.4.4.1 Quantitative Analysis

###### A Creating an Application File

1. Click on the **Quant Application** button on the main tool bar.
2. Click on the **Application File** box. The **Application File** box is located on the left side of the screen and is highlighted in yellow.
3. An **Application File** window will appear on the screen with the following options: create a new application, modify an existing application,

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rename and save the application, delete an application, and print application information.

4. Click on the radial box associated with **Create a New Application**.
5. Click on the **Next** button.
6. A **Create a New Application** window will appear. The window will have 8 tabs. The tabs are labeled as follows: **Polymer, Filter, Film & Coating, Liquid, Metal & Alloy, Powder, Fusion Bead, and Ceramics & Glass**.

**NOTE:** The following directions are for the **Glass Fusions**.

7. Select the **Fusion Bead** tab.
8. The **Fusion Bead** tab has the following four folders: **Mineral, Cement, Refractory, and Ceramics**.
9. Click on the **Mineral Folder**.
10. Click on the **Next** button.
11. A **Select an Application Template** window will appear. The window has the following two options: **Use an Application Template and Create your own Application**.
12. Click on the radial box next to create your own application.
13. Click the **Next** button.
14. An **Application Name Input** window will appear. Type in the **Name** for the application, select the radial box next to **Fundamental**

**Parameters**, and input a description. The description is optional.

15. Click on the **Finish** button.

**B** Setting of Application Information

1. After clicking on the **Finish** button under the **Creating an Application File** section, a **Component Selection** window will appear. The window has three parts: **Type of components to add, periodic table, and a list of components selected.**
2. Click on the radial box next to **Oxide** under the **Type of Component** to add.
3. Select the components you want to analyze for by clicking on the **Elements** in the periodic table.
4. The select elements from the periodic table will be inputted as oxides in the list of components selected.
5. In the list of components the **Unit** should be **mass %** and the **Analysis** should be **Measure (FP)**. If **mass %** is not the desired unit, click on the analysis box associated with the element in question. A scroll bar will appear in the lower right corner of the box. Click on the **Scroll Bar** and select the appropriate unit.
6. Click on the radial box next to **Special Compound**.
7. Select **Li** in the periodic table.
8. A **Special Compound** window will appear.
9. Click the radial box next to **All Compounds**.

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10. Select "IG" from the list. **IG** is a special compound for **Loss on Ignition**.
11. Click on the **OK** button on the bottom of the Special compound window.
12. Check to make sure that **IG** was added to the list of components.
13. Double click on the **Analysis** box associated with **IG**.
14. Change the **Analysis** from **Fixed** to **Input** by using the scroll bar.
15. Click on the **OK** button on the bottom of the **Compound Selection** window.
16. A **Sample Preparation Method for Fusion Bead** window will appear.
17. One can select what kind of pulverizing container, additive agent, sample size, fusing condition, fusion time, sampling condition, and a memorandum. The only information that needs to be inputted is the sampling condition.
18. Use the scroll bar associated with the flux box to select **LITLIF1**.
19. Enter 7.5000g for the **Flux weight** and 1.5000g for the **Sample weight**.
20. Click on the **OK** button on the bottom of this window.

#### C Setting of Standard Samples

1. After clicking the **OK** button on the above step, a **Standard Samples** window will appear.

2. Double click on the **Name** cell and enter a standard sample name.
3. Use the **Tab** button on the keyboard to move to the first Oxide mass% column. The mass% values are the LOI correct mass%.
4. Input the mass% for this oxide.
5. **Tab** to the next Oxide and enter the next mass%.
6. Repeat this process until the entire mass% for this standard is inputted.
7. Input the sample to flux ratio for the standard into the column labeled **LITLIF1**. (e.g. (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> + LiF)/LOI sample weight)
8. Leave the column marked **IG** blank.
9. The mass% for the row should equal **99.00 through 101 mass%**.
10. Repeat steps 2 – 9 for the rest of the standards.
11. Click on the **Next** button at the bottom of this window. A **Standard Samples** dialog will appear and Query "Save Modified Data?" Click on the **Yes** button.
12. A **Composition Information** window will appear.
13. This window will display the average composition based on the standards inputted for each oxide and **LITLIF1**.
14. Click on the **OK** button on the bottom of the window.

D Setting of Analysis Information

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1. After clicking on the **OK** button, a **Parameters window** will appear. This window has three tabs: **Analysis Information**, **Measuring Condition**, and **Calculation Parameter**.
2. Make sure that the **LITLIF1** and the **IG Type** column state **INPUTTED**. If they don't click on either the **LITLIF1** or the **IG** lines and make the changes.
3. Look at the information on the **Analysis Information** tab. Make sure the **TYPE** is **FP** and the **Element Line** is **Correct**.
4. If the **Element Line** is not correct, click on the **Line** in question and select the **Property** button. A **Property Sample** window will appear. The window will have the following information: **element line, component type, component value, etc.** Select the **Correct Element Line** and click the **OK** button when finished. Repeat this process if there are any other mistakes.
5. Click on the **Advance** button. An **Advance** window will appear.
6. Select **"1"** for the **STD sample prep. Time**, either add a description of the application in the appropriate box or leave this box **BLANK**. Select the following **Print Options: Peak/BG Intensity, Internal std/overlap correction, Uncorrected/Unnormalized result, and Application result**.
7. Click on the **OK** button when finished.
8. Click on the **Evaluation** button. An **Evaluation** window will appear. This window

will let the user know if there are any problems with the setup.

9. Click on the **OK** button when finished.
10. Double Click on the **Measuring Condition** tab. The information in the spreadsheet will change. Make sure that the **voltage** and **current** are **50, 50** and the crystal identified are correct.
11. Click on one of the **Elements** in the spreadsheet. A **Measuring Condition** window will appear.
12. Change the Tube Voltage and the Tube Current to **50, 50** using the scroll bar associated with each box.
13. Look at the **Selected Crystal**. If the select crystal is wrong, select the correct one using the **Scroll Bar** associated with the crystal box. **Also, one can change the background positions. Click on the box labeled BG1 and input a value 1.5 -2° to the left of the peak value. Do the same for BG2 but add 1.5 - 2° to the peak.**

**NOTE: Make sure the crystals for the light elements are correct. The computer selects the TAP crystal for Na and Mg, and the PET for the Si. We use the RX35 crystal for Na and Mg, and the RX4 crystal for Si.**

14. Make sure the **detector, slit, attenuator, and filter** are correct.
15. Make sure the **Private** is selected for the **Type** box. This will make the measuring condition only available for this application.
16. Click on the **OK** button.



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17. Repeat steps 10 – 16 for each measuring condition.
18. Click on the **Advanced** button on the **Parameters** window. An **Advanced Setting Measuring Condition** window will appear.
19. Select the following **Sample Spin, Optimize Measuring Order, and APC for the Vacuum setting**. The **Measuring Multiplier** should be 1, and the **Evacuation Speed** should be set to **Slow**.
20. The **Parameter** tab will not be used. The parameter tab is used to change  $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$ .
21. Click on the **Next** button on the bottom of the **Parameters** window.
22. A **Parameters** dialog box will appear and Query "Save the Modified Data?". Click the **Yes** button.
23. A **Special Standards** window will appear. This is used when the composition of an unknown sample is different from that of the standard samples. The setting procedure is the same as that of an ordinary standard sample.
24. Click on the **Next** button on the bottom of the **Special Standards** window.

#### E. Measuring Condition Setup (Optimize MC's)

1. After clicking on the **Next** button on the **Special Sample** window, the **Add Condition – Optimize MC's** window will appear.
2. Select the **Optimize MC** radial box.
3. Click on the **Add** button.

4. Click on the boxes associated with **2-theta** and **PHA** scan if they are not pre-selected.
5. Click on the **Select All** button.
6. Type in the standard or the unknown sample name for the sample name.
7. Select the position on the window as **Position 1**.
8. Click on the **Sample Spin** box.
9. Put the standard or unknown sample in a sample cup with a 30mm mask.
10. Place the sample cup in **Position 1** on the sample wheel.
11. Click on the **OK** button on the bottom of the window.
12. Click on the **Start** button in the middle right hand side of the **Optimize MC's** window.

**NOTE: One can measure a standard with high concentrations and one with low concentrations.**

13. When the measurement is complete, the **Start** button will reappear.
14. Repeat this process if two standards are being used to optimize the measuring conditions.
15. Select a **Component**. Each component will have a 2-theta and PHA scan associated with it.
16. Select the first component line. The 2-theta scan will appear for this component on the bottom half of the window. If two standards were used,

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the other standard can be brought up by selecting **View** and then **Select Reference Data**. Select the **same Component** and then click on the **OK** button. Both 2-theta scans will appear on the bottom half of the window.

17. To update the **Peak angle**, **Select 2-theta scan-Peak Angle Setting-Use Peak search angle**. Click **OK**. To change the **Background points**, **Time for the peak or Background**, or the **PHA**, select **File** then **Save Measuring Condition**.
18. A **Save Measuring Condition** window will appear.
19. The **Peak Angle** should update automatically.
20. Check the time period for the **Peak angle**. Make sure the time select will give a **1,000,000cts**.
21. To change the **Time** for the peak angle, click on the **Time Line** under **Peak 2-theta**.
22. A window will appear. Click in the box associated with the time and type in the **Time** you want.
23. Click the **OK** button.
24. Check the background position. If the background positions are not in the right location or the time is too long or too shorter. Click on the line associated with the **Background Position 1**. A window will appear. Click in the associated box and change the background position and the time if needed. Repeat for this operation for the other background position.

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25. Click on the **OK** button on the bottom of the **Save Measuring Condition** window.
26. Select the **PHA** scan associated with the above 2-theta scan. The **PHA** scan will appear on the bottom half of the window. If two standards were used, the other **PHA** scan can be brought up by selecting **View** and the **select Reference Data**. Select the same component **PHA** scan and then click on the **OK** button. Both **PHA** scans will appear on the bottom half of the window.
27. To change the **PHA lower and upper limit**, Select **File** then **Save Measuring Condition**.
28. A **Save Measuring Condition** window will appear.
29. Click on the **PHA line**. A **PHA** window will appear. Click the box associated with the lower limit and type in the change for the lower limit. Repeat this process for the upper limit.
30. Click on the **OK** button on the bottom of the **Save Measuring Condition**.
31. If there are any **overlapping elements** (e.g.  $\text{CrK}\beta_1$  and  $\text{MnK}\alpha$ ), a **Peak Deconvolution** must be carried out.
32. Click on the tool bar button that has an arrowhead at either end.
33. Designate a range by dragging the mouse from left to the right of an object peak.
34. Select **2-theta Scan/Peak Deconvolution/Function**.
35. Select the **Type of Background Calculation**.
36. Click on the **OK** button.

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37. Check the **net intensities etc.**
38. Click on the **OK** button.
39. Select **2-theta Scan/Peak Deconvolution/ Calculate BG Coefficient.**
40. Select a **Correcting Line.**
41. Click on the **OK** button.
42. Check the **coefficients etc.**
43. Click on the **OK** button.
44. Click on the **Next** button on the bottom of the **Optimize MC's** window.

**NOTE: The highest intensity that does not cause loss is about 1000 kcps for the SC detector and about 2000 kcps for the PC detector. If the X-ray intensities approach these intensity rates, take some precautions to determine any counting loss effects.**

### F. Measuring the Standards

1. After clicking the **Next** button on the **Optimize MC's** window, the **Run Standards** window will appear.
2. Select the **ALL** button.
3. Enter the Position of the first sample.
4. Enter the **Sample Name.**
5. Click on the **OK** button.

6. A **Run Standards** window will appear. Double click on the second line and change the sample name to the Application.
7. Repeat this process for all of the standards.
8. Put each standard sample cup with a 30mm mask.
9. Place the sample cups on the sample wheel starting with **Position 1**.
10. Click the **Analyze** button.
11. After each standard has been analyzed, the **Drift Correction Samples** window will appear. The drift correction is performed to update prepared calibration curves for a long period. This correction is typically not performed. For information about how to perform this correction refer to the **Instruction Manual vol1 starting with page 3-77**.
12. Click the **Next** button at the bottom of the **Drift Correction Samples** window.

#### G Regression Calculation (Calibration)

1. After clicking on the **Next** button at the bottom of the **Drift Correction Samples** window, the **Calibration** window will appear.
2. Select the first component. Make sure the Type is **Fundamental Parameters**, and the **Formula is Linear** if there are two or more data points for the first component. If there is only **One Data Point** the **Formula** box must be changed to **Ratio**.
3. If there is an overlap between two components, make sure the overlapping component is part of

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the standard table. If this is not true one cannot perform an overlap correction.

4. Click on the **Correction** button. The **Overlap Correction** window will appear for the selected component.
5. Choose between **Measured** or **Theoretical Intensity** for the Method. Use the pull down bar next to the Method box.
6. Click on the **List Possible Overlap** button. A list of possible overlaps will be displayed.
7. Click on the **OK** button on the **Possible Overlap Lines** window.
8. Select a **Correcting Component** in the **Correcting Line** box.
9. Select **Calculate** in the **Coefficient Frame**.
10. Click on the **OK** button.
11. Click on the **OK** button.
12. Click on the **Calibrate** button. The **FP** calibration graph for the selected component will appear.
13. Look at the curve. If there is a standard out of place, click on the appropriate data point. Click on **Recalibrate**.
14. Click on the **Save** button.
15. Click the **"X"** on the upper right hand corner of the **FP calibration graph**. One might have to close out more than one FP calibration Graph for the same component depending on how many times the curve was recalibrated.

16. Repeat this process for each component.
17. Click on the **Next** button on the bottom of the **Calibration** window.
18. The **Constants Input** window will appear. This option will not be used. For more information about this option refer to the Instruction Manual vol. 1 starting with page 3-122.
19. Click on the **Next** button.

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This option will not be used refer to the Instruction Manual vol. 1. Starting with page 3-127, click on the **Exist** button on the left-hand flow chart.

#### I Analyzing Unknown Samples

1. Place the unknown sample(s) into sample cup(s) with 30mm mask.
2. Place the first unknown sample into **Position 1** on the sample wheel. Place the next unknown sample in **Position 2** and so on.
3. Click on the **Analysis** button on the main tool bar.
4. Double click on the first BLANK line. A **Sample ID Setting** window will appear.
5. Click the **QUANT** button.
6. Select "1" for the position of the first unknown sample.
7. Input the **Sample Name**.



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8. Select the name you gave the model from the list of Analytical Condition.
9. Click on the **Manual Input** box.
10. A **Manual Input** window will appear. This window will contain two lines. One for **IG** and the other for **LITLIF1**.
11. Double click in the box associated with **IG**. Input the **LOI** value for the first unknown sample.
12. Double click in the box associated with **LITLIF1**. Input the **Flux to Sample Ratio** value.
13. Click on the **OK** button.
14. Click on the **OK** button.
15. Repeat this process if there are more than one unknown sample.
16. After input all the information for all the unknown samples, Click on the **Analyze** button.

### J Printing the Application Information

1. Click on the **Quant** button on the main tool bar.
2. Click on the **Application File** box on the flow bar to the left.
3. An **Application File** window will appear.
4. Select the radial box associated with the **Print Application Information**.
5. Click on the **Next** button.
6. An **Application to Print** window will appear.

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7. Select a **Folder**.
8. Double click the folder of an object type on the tree display.
9. Click on the **Next** button.
10. A **Print Quant Application** window will appear.
11. Select the necessary items on the Print Quant Application window.
12. If necessary, click the Select buttons and make selections for detailed items.
13. Click on the **Finish** button.
14. Click the **Exist** button on the Flow chart on the left-hand side of the screen.

**K** Printing the Results

1. Click on the **Data Processing** button on the main tool bar.
2. Click on **Result Display** option from the drop down menu. A **Result Display** window will appear.
3. Using the **Scroll Bar** look for the **Sample Name** you want to Print.
4. Click on the line for the **Sample Name**.
5. Right click on the line, and select **Print**. A **Print** window will appear.
6. Select the **Options** you want from the **Print** window.

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7. Click on the **OK** button.
8. Click the "**X**" on the **Result Display** window to close the window.

### L. Turning the Instrument Off.

1. Click on the **Startup/Shutdown** button on the main tool bar.
2. A drop down menu will appear. Select **Tube/Atmosphere** option on the drop down menu.
3. Click on the **Switch Atmosphere** radial box and select **AIR** with the pull down scroll bar.
4. Click on the **Start** button.
5. The process is complete when the radial box is white.
6. On the same window, click on the **Voltage and Current** radial boxes. Using the pull down scroll, decrease the Voltage and Current slowly. Start by decrease the Voltage and Current to 40, 40. Click the **Start** button. Repeat the process until the Voltage and Current is 20, 2.

**NOTE: 20kV and 2mA is the setting the instrument is placed in when not in**

7. Click on the "**X**" on the upper right hand corner to close the **Tube/Atmosphere** window.
8. Click on the **Startup/Shutdown** button on the main tool bar.

9. Click on the **Spectrometer Shutdown** option. A **Spectrometer Shutdown** window will appear.
10. Click the radial box associated with **Manual Shutdown**.
11. Select the **Turn Off X-ray** option.
12. Click on the **Start** button.
13. When the X-ray Tube is turned off, click the "X" on the upper right hand side of the window.
14. Close the **ZSX** software.
15. Shutdown the computer
16. Turn off the spectrometer main unit.
17. Turn off both the Haskris and the Rigaku chillers.
18. Close the valve for the primary water. The valve is located on the inside wall of C142/146

#### 5.4.4.2 Qualitative Analysis

##### A Creating an Application File

1. Click on the **Qual Application** button on the main tool bar.
2. An **Application File** window will appear on the screen with the following options: **Create a New Application, Modify an Existing Application, Rename and Save the Application, Delete an Application, and Print Application Information.**

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3. Click on the radial box associated with **Create a New Application**.
4. Click on the **Next** button.
5. A Select an **Application Template** window will appear. The window will have 8 tabs. The tabs are labeled as follows: **Polymer, Filter, Film & Coating, Liquid, Metal & Alloy, Oxide Powder, Fusion Bead, and Qualitative**.

**NOTE:** The following directions are for the **Qualitative**.

6. Select the **Qualitative Tab**.
7. Click on radial box next to **Create your own Application**.
8. Click the **Next** button.
9. An **Application Name Input** window will appear. Type in the **Name** for the application, and input a description. The description is optional.
10. Click on the **Finish** button.

### **B** Selecting Element Range

1. After clicking on the **Finish** button under the **Creating an Application File** section, a **Select Element Range** window will appear.
2. Select the **Components** you want to analyze for by clicking on the elements in the periodic table or one can press the **B-U, F-U, or the Ti-U** buttons.

3. The select elements from the periodic table will be inputted into the **Qualitative** box on the left-hand side of the screen.

**NOTE:** When there is a component of special importance, measure it by adding a fixed angle analysis. The fixed angle measurement cannot be set for a component for which the qualitative analysis is not made. Setting cannot be made for Ti-U unless the full range qualitative analysis is carried out

4. Click on the **OK** button. A **Parameters** window will appear.

**C. Parameters**

1. Select the **Measuring Diameter**.
2. Select **YES** for **Sample Spin**.
3. Select **Vacuum** for **Atmosphere**.
4. Check to make sure the following radial boxes are check: **Metal, Balance calculation NO, Matching Library NO, and Flux (dilution/additive) No.**
5. Click on the **OK** button. A **Check Measuring Condition** window will appear.

**D. Check Measuring Conditions**

1. Check the following information on the **Check Measuring** window: the **Voltage and Current, Detector, Slit, Attenuator, Filter and Crystal** are set correctly.

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2. To make changes, click on the First line then click on the **Property** button.
3. A **Scan Condition** window will appear. Use the scroll bar next to the **Voltage and Current** boxes to change the **Voltage and Current** to **50, 50**. While this window is opened, check on the following items, the crystal, filter, slit attenuator, the peak angle, the start angle, end angle, the step and the speed are what you want.

**NOTE: Make sure the Crystal for the light elements are the one you want to use. The computer selects the TAP crystal for NA and MG. We use the RX35 crystal for these elements.**

4. Click the **OK** button.
5. Repeat for each line in the table.
6. Click the **OK** button on the Property window when finished.
7. Click on the **OK** button on the bottom of the **Check Measuring Condition** window. An **Output Information** window will appear.

**NOTE: The Qualitative tab is the only tab where one can change the voltage, current, filter attenuator, slit, crystal, and the detector. The other tabs one can only change the following information: current and the attenuator. All the other information is pre-selected for you.**

### E Output Information

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1. Select the items you want to **Print** from the **Output Information** window.
2. Click on the box associated with smoothing, and select **11point** using the scroll bar.
3. Leave the **Peak Detection Level** at 1.0.
4. Click the **OK** button.
5. Click on the **Exist** button on the left-hand side flow chart.

F Analyzing Unknown Samples

1. Place the unknown sample(s) into sample cup(s) with 30mm mask.
2. Place the first unknown sample into **Position 1** on the sample wheel. Place the next unknown sample in **Position 2** and so on.
3. Click on the **Analysis** button on the main tool bar.
4. Double click on the first **BLANK** line. A **Sample ID Setting** window will appear.
5. Click the **QUAL** button.
6. Input "1" for the position.
7. Input the **Sample Identification** for the sample name.
8. Input your name for the operator.
9. Select the name you gave the **Model** from the list of **Analytical Condition**.
10. Input the **Result Output Filename**.



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11. Click on the **OK** button.
12. Repeat this process if there are more than one unknown sample.
13. After input all the information for all the unknown samples, click on the **Analyze** button.

### G Printing the Application Information

1. Click on the **Qual** button on the main tool bar.
2. Click on the **Application File** box on the flow bar to the left.
3. An **Application File** window will appear.
4. Select the radial box associated with the **Print Application Information**.
5. Click on the **Next** button.
6. An **Application to Print** window will appear.
7. Select a **Folder**.
8. Double click the folder of an object type on the tree display.
9. Click on the **Next** button.
10. A **Print Qual Application** window will appear.
11. Select the necessary items on the **Print Qual Application** window.
12. Click on the **Finish** button.
13. Click the **Exist** button on the Flow chart on the left-hand side of the screen.

## H Printing the Results

1. Click on the **Data Processing** button on the main tool bar.
2. Click on **Qual Results** then **Qual Data Handling** option from the drop down menu. A **Qual Data Handling: File** window will appear.
3. Click the **Browse** Button. A **File Load** window will appear.
4. Click on the line for the **Filename** of interest.
5. Click on the **OK** button.
6. Click on the **ALL** button
7. Click on the **OK** button.
8. To print the **Peak List**, click on **File** then **Print Peak List**.
9. Select the **ALL** button.
10. Click on the **OK** button.
11. To Print the **Spectrum**, click on **File** then **Print Active Chart**.
11. Click on the **OK** button on the **Active Chart** window.
12. To print a chart with a couple of spectra overlays, click on the **Data Processing** button on the main tool bar.
13. Click on **Qual Results** then **Qual Data Handling** option from the drop down menu. A **Qual Data Handling: File** window will appear.

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14. Click on the **Browse** button associated with the **Target File**.
15. Select a **Filename**.
16. Click on the **OK** button.
17. Click on the **Reference Files Tab**.
18. Click on the **Browse** button associated with the **Condition Reference File**.
19. Select a **Filename**.
20. Click on the **OK** button.
21. To add more spectra repeat steps 18 through 21.
22. Click on **File** then **Print Active Chart**.
23. Click on the **OK** button on the Active Chart window.
24. To close the **Qual Data Handling** window, select **File** and **Close All Qual** windows.

### I Data Processing

1. Click on the **Data Processing** button on the main tool bar.
2. Click on the **Qual Results** then **Qual Data Handling** option from the drop down menu. A **Qual Data Handling: File** window will appear.
3. Click on the **Browse** button. A **File Load** window will appear.
4. Click on the line for the **Filename**.
5. Click on the **OK** button.

6. Click on the **ALL** button.
7. Click on the **OK** button.
8. Using the shortcut keys or the main heading option, one can do any Data Processing. (E.g. Background, Peak Deconvolution.)
9. To Close, Select **File** and then **Close All windows**.

**J Turning the Instrument Off.**

1. Click on the **Startup/Shutdown** button on the main tool bar.
2. A drop down menu will appear. Select **Tube/Atmosphere** option on the drop down menu.
3. Click on the **Switch Atmosphere** radial box and select **AIR** with the pull down scroll bar.
4. Click on the **Start** button.
5. The process is complete when the radial box is white.
6. On the same window, click on the **Voltage and Current** radial boxes. Using the pull down scroll decrease the **Voltage and Current** slowly. Start by decrease the Voltage and Current to 40, 40. Click the Start button. Repeat the process until the **Voltage and Current** is 20, 2.

**NOTE: 20kV and 2mA is the setting the instrument is placed in when not in**

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7. Click on the "X" on the upper right hand corner to close the **Tube/Atmosphere** window.
8. Click on the **Startup/Shutdown** button on the main tool bar.
9. Click on the **Spectrometer Shutdown** option. A **Spectrometer Shutdown** window will appear.
10. Click the radial box associated with **Manual Shutdown**.
11. Select the **Turn Off X-ray** option.
12. Click on the **Start** button.
13. When the **X-ray Tube** is turned **Off**, click the "X" on the upper right hand side of the window.
14. Close the **ZSX** software.
15. Shutdown the computer
16. Turn off the spectrometer main unit.
17. Turn off both the **Haskris** and the **Rigaku** chillers.
18. Close the valve for the primary water. The valve is located on the inside wall of C142/146

### 5.4.5. Preventive Maintenance

1. When not in use, idle the system with the x-ray tube at **20 kV and 2mA**.
2. Change the vacuum pump oil yearly.
3. Change the P-10 gas as needed.
4. Do a **DSAVE** at the beginning of every month.

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## 6.0 RECORDS

- 6.1 The X-Ray Fluorescence Logbook shall be handled in accordance with the Records Management procedures.
- 6.2 A copy of all the x-ray fluorescence raw data shall be stored for one year on the computer's hard drive.
- 6.3 A copy of the Maintenance Reports shall be entered into the Rigaku Maintenance Notebook.
- 6.4 The Calibration results shall be entered into the Rigaku Calibration Notebook.
- 6.5 Results for all routine analytical samples will be entered into the AD Laboratory Information System and the XRF Logbook using AD LIMS numbers.

## 7.0 REFERENCES

### A. Rigaku ZSE Manuals and Prints

Rigaku ZSX101e X-ray Fluorescence Spectrometer Instruction Manual  
Volumes 1-3.

Rigaku ZSX101e X-ray Fluorescence Spectrometer Maintenance Manual.

Rigaku ZSX101e Circuit Diagram of ZSX101e.

### B. Haskris

Model RW175F1 Refrigerated Closed Circuit Water Chiller Instruction  
Manual.

### C. Claisse

Claisse M4 Fluxer Instruction Manual

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8.0 ATTACHMENTS

- 8.1 Attachment 1: DSAVE/DLOAD
- 8.2 Attachment 2: Changing the P-10 cylinder
- 8.3 Attachment 3: Overlap Correction
- 8.4 Attachment 4: Making Glass Fused Pellets using the Claisse M4 Fluxer in Automatic or Manual Modes

## ATTACHMENT 1

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### DSAVE/DLOAD

#### DSAVE

1. Open the ADS Cluster on the File server.
2. Open the **Rigaku** folder
3. Create a new folder using **DSAVE** and the date. (E.g.DSAVE41103)
4. Close the **Rigaku** folder.
5. Click on the **Utility** button on the main tool bar.
6. Click on the **Data Save/Load** option on the drop down menu.
7. Select the **Data Save** radial box.
8. Click on the **Next** button.
9. Click on the radial box, which is associated with the **Specify a Folder** option.
10. Click on the **Browse** button.
11. Click on the **My Computer** Icon.
12. Click on the **ADS Cluster**.
13. Click on the **Rigaku** folder.
14. Click on the **DSAVE** folder that was created above.
15. Click on the **OK** button.
16. Click on the **Next** button.
17. Select the **User Setting Data: Qual Condition, Quant Condition, Qual Data, and Quant Data**.
18. Click on the **Next** button.
19. Click on the **OK** button.
20. Click on the **OK** button.



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#### DSAVE/DLOAD

#### DLOAD

1. Click on the **Utility** button on the main tool bar.
2. Click on the **Data Save/Load** option on the drop down menu.
3. Select the **Data Load** radial box.
4. Click on the **Next** button.
5. Click on the radial box, which is associated with the **Specify a Folder** option.
6. Click on the **Browse** button.
7. Click on the **My Computer** Icon.
8. Click on the **ADS Cluster**.
9. Click on the **Rigaku** folder.
9. Click on the latest **DSAVE** folder.
10. Click on the **OK** button.
11. Click on the **Next** button.
12. Select the following items to load: **Qual Condition, Quant Condition, Qual Data, and Quant Data.**
13. Click on the **Next** button.
14. Click on the **OK** button.
15. Click on the **OK** button.

## ATTACHMENT 2

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### CHANGING THE P-10 GAS CYLINDER

1. Click on the **Startup/Shutdown** button on the main tool bar.
2. A drop down menu will appear. Select **Tube/Atmosphere** option on the drop down menu.
3. Click on the **Switch Atmosphere** radial box and select **AIR** with the pull down scroll bar.
4. Click on the **Start** button.
5. The process is complete when the radial box is white.
6. On the same window, click on the **Voltage and Current** radial boxes. Using the pull down scroll decrease the Voltage and Current slowly. Start by decrease the **Voltage and Current to 40, 40**. Click the **Start** button. Repeat the process until the **Voltage and Current is 20, 2**.

**NOTE: 20kV and 2mA is the setting the instrument is placed in when not in use.**

7. Click on the "X" on the upper right hand corner to close the **Tube/Atmosphere** window.
8. Click on the **Startup/Shutdown** button on the main tool bar.
9. Click on the **Spectrometer Shutdown** option. A **Spectrometer Shutdown** window will appear.
10. Click the radial box associated with **Manual Shutdown**.
11. Select the **Turn Off X-ray** option.
12. Click on the **Start** button.

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13. When the **X-ray Tube** is turned off, click the "X" on the upper right hand side of the window.
14. Close the **ZSX** software.
15. Shutdown the computer
16. Turn off the spectrometer main unit.
17. Close the valve on the cylinder.
18. Remove the regulator. (NOTE: The regulator has reverse threads.)
19. Replace the empty cylinder with a full cylinder.
20. Wait 15 minutes before turning the instrument on.
21. To turn instrument on, go to Step 16 and follow the directions down to Step 1.

ATTACHMENT 3

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OVERLAP CORRECTION

1. Examples of Overlaps: Mo L $\alpha$  with S K $\alpha$ , Cr K $\alpha$  with Fe K $\beta$ , and Pb L $\alpha$  with As K $\alpha$ . There are other overlaps. See Task Supervisor for more information.
2. Use the same kV-mA and measuring diameter as the analysis.
3. Measure the interfering element at the analyte's wavelength and at its own wavelength.
4. Calculate the Overlap Coefficient as follows:

Overlap Coefficient =  $\frac{\text{interfering element at the analyte's wavelength}}{\text{interfering element at its own wavelength}}$

$$\text{(O.C. = } \frac{\text{Mo @ S K}\alpha}{\text{Mo @ Mo L}\alpha}$$

5. The measured analyte intensity is:  
= the actual analyte intensity + the interference.
6. The corrected analyte intensity is:  
= the measured analyte intensity - overlap coefficient \* the interfering element at its own wavelength.

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### ATTACHMENT 4

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#### MAKING GLASS FUSED PELLETS USING THE CLAISSE M4 FLUXER IN AUTOMATIC OR MANUAL MODES

##### 1 Automatic Operations

The procedure for making glass disks is described in the following paragraphs. The **automatic** operation means that once the program is launched, every function is executed in numerical sequence.

###### 1.1. Prepare the instrument:

- a) Raise the arm as far as it can go; it should stop at about 20° backwards from the vertical. The crucible clips automatically rotate until they reach a position easy for loading.
- b) Put 1, 2 or 3 crucibles along with the corresponding molds into the clips; make sure that they will not slip out of the latter during rotation.
- c) Plug in the fan unit in front of the burners.

###### 1.2. Load the crucibles:

- a) Have the mixture to fuse ready. The usual quantities vary between 6 and 10 grams for molds with a diameter of 30 to 40 mm.
- b) Transfer the mixture to fuse into the crucibles.
- c) If required, add a solution containing a non-wetting agent.
- d) If the arm is tilted backwards, pull the arm towards you; it should stop at about 20° forward.

###### 1.3. Open the propane cylinder and adjust the in-line propane regulator to 8-10 psi.

###### 1.4. Select the burners to be supplied with gas by turning on the appropriate toggle switches located at the front panel of the instrument.

###### 1.5. Turn the instrument on if required.

###### 1.6. Close the security cabinet.

###### 1.7. If required, transfer the program from the locked bank to one of the programs 0 to 9.

- a) Enter the "transfer mode" by typing the code **K 8 3 7 0**.
- b) Type **P**, followed by the number of the program you wish to overwrite (0 to 9), followed by the number of the locked program you want to transfer

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**MAKING GLASS FUSED PELLETS USING THE CLAISSE M4 FLUXER IN  
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(100 to 999). For example, if one wishes to transfer the locked program 127 into program 4, one must type **P 4 1 2 7**.

c) Press **STOP**, and then **RESET** to exit the "transfer mode".

- 1.8. Choose the program. If the desired program is not the one shown on the display, type **P** and the number of the desired program, for example **P 4**.
- 1.9. Press **START** and let the program take place. A countdown of the remaining time until the end of the ongoing program is displayed. If you press **7** a countdown of the remaining time until the end of the ongoing function is displayed. The process ends when the program countdown reaches 00.00.

**NOTE:** If you must stop a fusion in progress, press **STOP** on the remote control. After this procedure, you must either press **RESET** or turn the power off, then on again to start another fusion.

- 1.10. Pick up the disks using the suction cup provided.
- 1.11. Turn the power off to the instrument.
- 1.12. Turn of the select the burners by turning off the appropriate toggle switches located at the front panel of the instrument
- 1.13. Close the in-line regulator and close the cylinder valve.

**Note:** Make sure the propane gas is turned off at the cylinder at the end of the day when the instrument is in use.

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#### MAKING GLASS FUSED PELLETS USING THE CLAISSE M4 FLUXER IN AUTOMATIC OR MANUAL MODES

##### 2 Manual Operations

In manual mode, one can decide how long each function will last during the fusion process. This is useful when trying to define function parameters for fusing a new kind of material.

The process is the same as above, except that instead of pressing **START** to initiate the process, one presses **F 0**. This starts function 0 of the selected program, but there is no time limit. When desired, one may press **F 1** to continue to function 1, or even **F 2** (or any valid function number) to jump directly to that function. Note that going backwards is impossible.

Also, if you are in automatic mode, you can switch to manual mode by pressing **LOAD 6 2 6 (M A N)**. In this mode, there is no time definition so the function will go on until you manually change a parameter.

When a program contains more than 9 functions, pressing **F 1** will not jump immediately to function 1, but only after a short delay (2-3 seconds). This delay is provided in the event the user wishes to jump to a two-digit function number, function F12 for example. In that case, the user has 2-3 seconds to type the second digit before the program jumps to function 1.

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PANALYTICAL X'PERT PRO 3040  
X-RAY DIFFRACTION SYSTEM (U)

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Signature

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## 1.0 PURPOSE

This procedure provides requirements for the x-ray diffraction analysis of non-radioactive samples by the Analytical Development Section using a PANalytical X'Pert Pro 3040 X-Ray Diffractometer.

## 2.0 SCOPE

This procedure applies to most of the x-ray diffraction analyses of non-radioactive materials performed by ADS. This includes, but is not limited to, qualitative, quantitative, crystallite size, lattice parameter, and microstress determinations.

### 2.1 Definitions

- A. X-Rays - X-rays may be defined as electromagnetic radiation of wavelength  $\sim 10^{-5}$  to 100 Å produced by the deceleration of high-energy electrons and/or electron transitions in the inner orbits of atoms.
- B. X-Ray Diffraction - The phenomenon of diffraction involves the scattering of x-rays by atoms of a crystal and the reinforcement of scattered rays in definite directions away from the crystal. Reinforcement of the scattered rays is quantitatively related to the distance of atomic planes as defined by Bragg's law.

$$n\lambda = 2d \sin \Theta$$

When a collimated beam of monochromatic x-rays of wavelength  $\lambda$  strikes a crystal, the rays penetrate and are partially scattered from many successive planes within the crystal. For a given interplanar spacing,  $d$ , there is a critical angle,  $\Theta$ , at which rays scattered from successive planes will be in phase along a front as they leave the crystal. The angle between the normal to the emerging wave front and the atomic planes will equal the angle between the normal to the primary wave front and the atomic planes. Diffraction from a succession of equally spaced lattice planes results in a diffraction maximum, which has sufficient intensity to be recorded.

Since vast majority of compounds have different interatomic distances in three dimensions, the angles at which diffraction occurs will be distinct for a particular compound. The unique array of diffraction maxima serves to identify that compound. The

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intensity of these maxima can also be used to quantify the amount of this compound in a mixture.

- C. Goniometer - A goniometer is an instrument for measuring crystal angles. It consists of a sample holder, source & detector slits, collimators, detector, and drive mechanisms for rotating the sample and detector.
- D. Proportional Detector - This detector consists of a cylindrical chamber filled with a xenon/methane gas mixture. The window of the detector is made out of beryllium and measures 20mm by 24mm. An anode wire is positioned in the axial center of the cylinder. The detector process can be described as follows: When an X-ray photon enters the detector and collides with an inert gas atom, it may ionize the atom by removing an electron from the outer shell of the atom. A primary electron ion-pair is formed. The total number of primary electron ion-pairs produced by an X-ray photon is defined by the ratio of the energy of the X-ray photon and the ionization energy of the inert gas atom. The electrons from the electron ion-pair are then accelerated towards the anode wire by its high potential. On its way to the anode wire the accelerating electron can produce another electron ion-pair. Each primary electron may give rise to many secondary electrons which in turn are accelerated towards the anode wire. The electrons (primary and secondary) create an electrical current in the wire, this electrical current is then converted into an electrical pulse by the pulse forming circuit. The height of the pulse is proportional to the energy of the detected X-ray photon. Conversely, the positively charged ions are attracted towards the ground casing. The inert gas is mixed with a quenching gas to prevent ions from creating an avalanche. This quenching gas neutralizes the positive ions without any other reactions.
- E. X'celerator Detector – This detector is an ultra fast X-ray detector based on Real Time Multiple Strip. The detector operates as if there were an array of over a hundred detectors working simultaneously. The Real Time Multiple Strip technology offers direct detection of diffracted X-rays, and the ability to efficiently process high count rates without any compromise on resolution.

## 2.2 Responsibilities

**A. Technical Analyst is responsible for:**

- Preparing samples for x-ray diffraction analysis. This includes grinding the sample to the proper particle size range, fixing with collodion or Vaseline, back-loading and side-loading techniques, fabricating custom mounts if necessary, and positioning the sample properly in the mount and diffractometer.
- Obtaining x-ray diffraction patterns.
- Troubleshooting the x-ray generator, goniometer, detector, water recirculator, computer and printer.
- Maintaining safety inspection records, the XRD sample logbook, and all other general documentation required for laboratory operation.

**B. Task Supervisor or designee is responsible for:**

- Performing data reduction, analysis, and presentation of results using several XRD software programs and associated computer equipment. This includes phase identification, cell refinement, indexing, crystallite size determinations, preferred orientation assessment, pattern simulation, and quantitative analysis.
- Mounting special attachments or different setups on instruments, e.g., the high temperature attachment, capillary stage.
- Reviewing the data generated by the Analyst.
- Entering results in the ADS Laboratory Information System.
- Ensuring safety controls have been implemented.

**C. Level 4 Manager (or designee) is responsible for reviewing and approving the operating procedure.****3.0 PRECAUTIONS AND LIMITATIONS****3.1 Safety****3.1.1 Radiation Control**

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- Access to the laboratory is limited by combination locks on the doors per DOE G 441.1-5, Radiation Generating Devices Guide, for use with Title 10, Code of Federal Regulations, Part 835, Occupational Radiation Protection. Only authorized instrumentation operators and Building Services personnel are permitted in the laboratory.
- Personnel using this facility are subject to requirements as specified in the 5Q and L1 Manuals, Radiation Worker II Training, Radiation Generating Device Training, and the XRD Operating Procedure.

## 3.1.2 X-Ray Exposure

The main hazard associated with use of x-ray equipment is overexposure to x-rays. The goniometer, as currently configured, is an open beam instrument. However, because of the equipment enclosure, there should be no personnel exposure unless it is bypassed or fails. This procedure complies with WSRC 5Q1.1-524, Ionizing Radiation Generating Device Control Program and WSRC 5Q1.1-525 Inspection and Survey of RGD's.

- X-ray equipment may only be used following operating procedures or with a written job plan approved by the Task Supervisor.
- The x-ray diffraction goniometer must be at the rear of the equipment enclosure to ensure adequate distance from the front of the enclosure and proper orientation of the x-ray beam.
- SHO will conduct a semi-annual audit to check for radiation leaks and to test equipment interlocks.
- A compilation of all interlocks and protection devices are listed in Attachments 1 and 2. Do not disable the interlocks or protection devices.

## 3.1.3 High Voltage

The other main danger associated with operating x-ray equipment is from high voltage. The generator can produce up to 55 kV at 60 mA. The unit should be shut down immediately if any electrical discharge is heard or seen. The detector is

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supplied with a potential of up to 1,000 volts. If any insulation is worn on any wiring, the unit should not be used. Entry into the generator cabinet requires vendor-trained personnel with either x-ray personnel trained on Safe Practices on or Near Electrical Conductors, Electrical Systems Safety, and First Aid Blood Borne Pathogens or a Work Clearance Permit with an E & I technician present.

## 3.1.4 Modification Requirement

Modification to and/or substitution of critical components in the control circuitry of existing x-ray apparatus must be approved by SHO and be in compliance with the intent of ANSI N43.2 and ANSI N543. Proper documentation of the modification and/or substitution must be provided for custodial and SHO files. Appropriate revisions of schematics and prints are required.

## 4.0 PREREQUISITE ACTIONS

Radiation Generating Device Operator training is required for instrument operation.

**NOTE:** Task Supervisor must review the analytical results generated by the Analyst before they are transmitted to customer.

## 5.0 PERFORMANCE

## 5.1 General Information

## 5.1.1 Description of Method

Collimated x-rays are directed at the sample by a set of slits. The x-ray tube and detector are then rotated (the detector about the sample and at twice the rate of the x-ray tube), and the x-ray intensity recorded as a function of angle. If the sample is a single crystalline phase, the pattern recorded will be characteristic of that phase. For multiple phases, characteristic patterns are additive. Since the intensity of the pattern is proportional to the amount of a phase present, quantitative techniques are available. Various crystallographic and solid solution information can also be obtained.

## 5.1.2 Quality Control (Calibration frequency, QC information)

Calibration and Measurement Control Program

Constant calibration checking is inherent within the search/match software used during the identification of phases from the ICDD (International Centre for Diffracted Data) database of standard materials. If the results for any of the tests are out of specification, see the Task Supervisor. It may be necessary to generate a **Non-Conformance Report (NCR)** for any data generated since the last acceptable calibration check.

## 5.1.3 Manufacturer

PANalytical, INC.  
12 Michigan Drive  
Natick, MA 01760  
Telephone: 800-234-9729

## 5.1.4 Instrumentation

MS&EXRD

Model #	X'pert Pro
Serial #	DY1743
Capital Equipment #	00397460

M&TE

The M&TE Program controls the four-place balance.

## 5.1.5 Reagents and Chemicals

Small amounts (<0.1 cc) of collodion or Vaseline are used to mount powdered samples. Standards, Si (NIST SRM640c), alumina (NIST SRM 676), LaB<sub>6</sub> (NIST SRMs 660 and 660a), and fluorophlogopite (NIST SRM 675), are used for quantitative and high precision determinations. None of these standards or reagents used to mount the samples has an expiration date.

## 5.2 Monthly Calibration Check

This test can be performed easily and quickly and should be repeated at least once a month by the Analyst to ensure optimum system performance. This test scans two quartz regions to examine several peaks for intensity, resolution, and placement. If the results are out of specifications, see the Task Supervisor. It may be necessary to perform the extensive calibration check, which may help identify the problem. If the extensive calibration check is passed, but the intensities are still low, the x-ray tube or the detector may be defective. Record the test results in the XRD Calibration Notebook.

**NOTE:** The quartz intensities will decrease progressively as the x-ray tube ages.

**NOTE:** There are several different configurations for this system. The following is a list of the different configuration: Mirror-Mirror with the Programmable Receiving Slit and Prop detector, Incident Mirror with the Programmable Receiving Slit and Prop detector, Incident Mirror with the two beam tunnels and the X'celerator detector, Fixed Divergent slit with the Programmable Receiving slit and Prop detector, Fixed Divergent slit with the Parallel Plate Collimator, Prop detector, Incident Mirror with the Parallel Plate Collimator, and Fixed Divergent slit with the two beam tunnels and the X'celerator detector. Initially, the quartz standard will be run for each of the above configurations. The quartz standard shall be run for each the different configurations yearly to ensure that the prefix for each of the different components has not been bumped. Monthly, the quartz standard shall be run on the configuration, which is being used.

**NOTE:** All configurations should meet the specifications in step 5.3

### 5.2.1 The Mirror – Mirror (Programmable Receiving slit) with the Prop Detector Configuration

#### 5.2.1.1 Insert the following:

	<u>Instrument</u>
Divergence Mirror Slit	1/2°
Mirror Mask	Remove
Mirror Soller Slit	0.04 Rad
Beam Knife Edge	Installed
SC (Flat Sample Stage)	Novaculite
Mirror Soller Slit	Remove
Mirror Mask	Remove
Mirror Slit	1/2°
Programmable Receiving Slit	0.2mm

**NOTE:** The mirrors must be run at 45kV and 40 mA.

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

- 5.2.1.2 Double click on the **X'Pert Data Collector** Icon on the desktop.
- 5.2.1.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).
- 5.2.1.4 After inputting the User ID and Password, click the OK button.
- 5.2.1.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.1.6 Click on the Mirror – Mirror configuration.
- 5.2.1.7 Click the OK button.
- 5.2.1.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.1.9 Click the OK button.



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- 5.2.1.10 The Mirror – Mirror configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.1.11 Click on the Instrument Setting tab.
- 5.2.1.12 Double click the Generator: PW 3040 (mpps) line.
- 5.2.1.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.1.14 Click the X-ray tab.
- 5.2.1.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.1.16 Type in 45.
- 5.2.1.17 Repeat the process for the Current (mA).
- 5.2.1.18 Type in 40.
- 5.2.1.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.1.20 Click Measure/Program on the main toolbar.
- 5.2.1.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.
- 5.2.1.22 Click on the CalMMquartz name on the list.
- 5.2.1.23 Click the OK button.
- 5.2.1.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.
- 5.2.1.25 Make sure the Filename starts off with F:\XRD-Current Year.
- 5.2.1.26 Click OK. This will start the scan.

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5.2.1.27 The scan is complete when you hear the shutter closing on the x-ray tube.

5.2.1.28 The following are steps to locate the data for Data Evaluation:

- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

5.2.2 The Incident Mirror – Programmable Slit with the Prop Detector Configuration

5.2.2.1 Insert the following:

	<u>Instrument</u>
Divergence Mirror Slit	$1/2^{\circ}$
Mirror Mask	Remove
Mirror Soller Slit	0.04 Rad
Beam Knife Edge	Installed
SC (Flat Sample Stage)	Novaculite
Programmable Receiving Slit	0.2mm

**NOTE:** The mirrors must be run at 45kV and 40 mA.

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

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- 5.2.2.2 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).
- 5.2.2.3 After inputting the User ID and Password, click the OK button.
- 5.2.2.4 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.2.5 Click on the Flat Sample Stage configuration.
- 5.2.2.6 Click the OK button.
- 5.2.2.7 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.2.8 Click the OK button.
- 5.2.2.9 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.2.10 Click on the Instrument Setting tab.
- 5.2.2.11 Double click the Generator: PW 3040 (mppc) line.
- 5.2.2.12 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.2.13 Click the X-ray tab.
- 5.2.2.14 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.2.15 Type in 45.
- 5.2.2.16 Repeat the process for the Current (mA).
- 5.2.2.17 Type in 40.
- 5.2.2.18 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes,

but the window will stay open. The close button will have to be depressed for the window to close.

- 5.2.2.19 Click Measure/Program on the main toolbar.
- 5.2.2.20 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.
- 5.2.2.21 Click on the CalMPRSquartz name on the list.
- 5.2.2.22 Click the OK button.
- 5.2.2.23 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.
- 5.2.2.24 Make sure the Filename starts off with F:\Xrd-Current Year.
- 5.2.2.25 Click OK. This will start the scan.
- 5.2.2.26 The scan is complete when you hear the shutter closing on the x-ray tube.
- 5.2.2.27 The following are steps to locate the data for Data Evaluation:
- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
  - Click on **File**, and then **Patterns**.
  - Select the **ADSCLUSTER** Drive.
  - Double click on the **XRD-Current Year**.
  - Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
  - Double click on the correct filename for range 1.
  - Click on File, and then Patterns.
  - Select the correct filename for range 2 and add this to range 1.
  - Click Edit/Merge Segments.

5.2.3. The Incident Mirror with the Two beam Tunnels and X'celerator Detector Configuration

- 5.2.3.1 Insert the following:

	<u>Instrument</u>
Divergence Mirror Slit	1/2°
Mirror Mask	Remove
Mirror Soller Slit	0.04 Rad
Beam Knife Edge	Installed
SC (Flat Sample Stage)	Novaculite
Short Beam Tunnel	3.4
Long Beam Tunnel	5.5
Attenuator	Ni
Programmable Receiving Slit	0.2mm

**NOTE:** The mirrors must be run at 45kV and 40 mA.

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

- 5.2.3.2 Double click on the **X'Pert Data Collector** Icon on the desktop.
- 5.2.3.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).
- 5.2.3.4 After inputting the User ID and Password, click the OK button.
- 5.2.3.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.3.6 Click on the Flat Sample Stage) configuration.
- 5.2.3.7 Click the OK button.
- 5.2.3.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.3.9 Click the OK button.

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- 5.2.3.10 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.3.11 Click on the Instrument Setting tab.
- 5.2.3.12 Double click the Generator: PW 3040 (mppc) line.
- 5.2.3.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.3.14 Click the X-ray tab.
- 5.2.3.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.3.16 Type in 45.
- 5.2.3.17 Repeat the process for the Current (mA).
- 5.2.3.18 Type in 40.
- 5.2.3.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.3.20 Click Measure/Program on the main toolbar.
- 5.2.3.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.
- 5.2.3.22 Click on the CalMXquartz name on the list.
- 5.2.3.23 Click the OK button.
- 5.2.3.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.
- 5.2.3.25 Make sure the Filename starts off with F:\Xrd-Current Year.
- 5.2.3.26 Click OK. This will start the scan.

## TECHNICAL REFERENCE

5.2.3.27 The scan is complete when you hear the shutter closing on the x-ray tube.

5.2.3.28 The following are steps to locate the data for Data Evaluation:

- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

5.2.4 The Fixed Divergent slit with the Programmable Receiving slit and Prop Detector Configuration.

5.2.4.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	$1/2^\circ$
Beam Mask	10mm
Divergent Antiscatter Slit	$1^\circ$
SC (Flat Sample Stage)	Novaculite
Programmable Receiving Slit	0.2mm

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

5.2.4.2 Double click on the **X'Pert Data Collector** Icon on the desktop.

5.2.4.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).

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- 5.2.4.4 After inputting the User Id and Password, click the OK button.
- 5.2.4.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.4.6 Click on the Flat Sample Stage configuration.
- 5.2.4.7 Click the OK button.
- 5.2.4.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.4.9 Click the OK button.
- 5.2.4.10 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.4.11 Click on the Instrument Setting tab.
- 5.2.4.12 Double click the Generator: PW 3040 (mpps) line.
- 5.2.4.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.4.14 Click the X-ray tab.
- 5.2.4.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.4.16 Type in 45.
- 5.2.4.17 Repeat the process for the Current (mA).
- 5.2.4.18 Type in 40.
- 5.2.4.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.4.20 Click Measure/Program on the main toolbar.



5.2.4.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.

5.2.4.22 Click on the CalFPRSquartz name on the list.

5.2.4.23 Click the OK button.

5.2.4.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.

5.2.4.25 Make sure the Filename starts off with F:\XRD-Current Year.

5.2.4.26 Click OK. This will start the scan.

5.2.4.27 The scan is complete when you hear the shutter closing on the x-ray tube.

5.2.4.28 The following are steps to locate the data for Data Evaluation:

- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

5.2.5 The Fixed Divergent slit with the 0.18 Parallel Plate Collimator and Prop Detector Configuration

5.2.5.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	$1/2^\circ$

## TECHNICAL REFERENCE

Beam Mask	10mm
Divergent Antiscatter Slit	1°
SC (Flat Sample Stage)	Novaculite
Parallel Plate Collimator Slit	0.18
Parallel Plate Collimator	
Soller Slit	0.04 Rad

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

- 5.2.5.2 Double click on the **X'Pert Data Collector** Icon on the desktop.
- 5.2.5.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).
- 5.2.5.4 After inputting the User ID and Password, click the OK button.
- 5.2.5.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.5.6 Click on the Flat Sample Stage configuration.
- 5.2.5.7 Click the OK button.
- 5.2.5.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.5.9 Click the OK button.
- 5.2.5.10 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.5.11 Click on the Instrument Setting tab.
- 5.2.5.12 Double click the Generator: PW 3040 (mppc) line.

## Appendix H

SAVANNAH RIVER NATIONAL LABORATORY  
ANALYTICAL DEVELOPMENT SECTION  
ADS ANALYTICAL OPERATING PROCEDURES

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- 5.2.5.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.5.14 Click the X-ray tab.
- 5.2.5.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.5.16 Type in 45.
- 5.2.5.17 Repeat the process for the Current (mA).
- 5.2.5.18 Type in 40.
- 5.2.5.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.5.20 Click Measure/Program on the main toolbar.
- 5.2.5.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.
- 5.2.5.22 Click on the CalFPPquartz name on the list.
- 5.2.5.23 Click the OK button.
- 5.2.5.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.
- 5.2.5.25 Make sure the Filename starts off with F:\XRD-Current Year.
- 5.2.5.26 Click OK. This will start the scan.
- 5.2.5.27 The scan is complete when you hear the shutter closing on the x-ray tube.
- 5.2.5.28 The following are steps to locate the data for Data Evaluation:

## TECHNICAL REFERENCE

- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

## 5.2.6 The Incident Mirror with the 0.18 Parallel Plate Collimator and Prop Detector Configuration.

## 5.2.6.1 Insert the following:

	<u>Instrument</u>
Divergence Mirror Slit	$1/2^{\circ}$
Mirror Mask	Remove
Mirror Soller Slit	0.04 Rad
Beam Knife Edge	Installed
SC (Flat Sample Stage)	Novaculite
Parallel Plate Collimator Slit	0.18
Parallel Plate Collimator	
Soller Slit	0.04 Rad

**NOTE:** The mirrors must be run at 45kV and 40 mA.

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

5.2.6.2 Double click on the **X'Pert Data Collector** Icon on the desktop.

5.2.6.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).

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- 5.2.6.4 After inputting the User ID and Password, click the OK button.
- 5.2.6.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.6.6 Click on the Flat Sample Stage configuration.
- 5.2.6.7 Click the OK button.
- 5.2.6.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.6.9 Click the OK button.
- 5.2.6.10 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.6.11 Click on the Instrument Setting tab.
- 5.2.6.12 Double click the Generator: PW 3040 (mpps) line.
- 5.2.6.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.6.14 Click the X-ray tab.
- 5.2.6.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.6.16 Type in 45.
- 5.2.6.17 Repeat the process for the Current (mA).
- 5.2.6.18 Type in 40.
- 5.2.6.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.6.20 Click Measure/Program on the main toolbar.

## TECHNICAL REFERENCE

5.2.6.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.

5.2.6.22 Click on the CalMPPquartz name on the list.

5.2.6.23 Click the OK button. This will start the scan.

5.2.6.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.

5.2.6.25 Make sure the Filename starts off with F:\XRD-Current Year.

5.2.6.26 Click OK

5.2.6.27 The scan is complete when you hear the shutter closing on the x-ray tube.

5.2.6.28 The following are steps to locate the data for Data Evaluation:

- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

5.2.7 The Fixed Divergent Slit with the Two beam Tunnels and X'celerator Detector Configuration.

5.2.7.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	$1/2^\circ$

## TECHNICAL REFERENCE

Beam Mask	10mm
Divergent Antiscatter Slit	1°
SC (Flat Sample Stage)	Novaculite
Short Beam Tunnel	3.4
Long Beam Tunnel	5.5
Attenuator	Ni

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

- 5.2.7.2 Double click on the **X'Pert Data Collector** Icon on the desktop.
- 5.2.7.3 The software will ask for a User ID and a Password. The User ID is User-1 and the Password is galaxy (case sensitive).
- 5.2.7.4 After inputting the User ID and Password, click the OK button.
- 5.2.7.5 Click on Instrument/Connect on the main toolbar. A Connect dialog window will appear.
- 5.2.7.6 Click on the Flat Sample Stage configuration.
- 5.2.7.7 Click the OK button.
- 5.2.7.8 An X'Pert Data Collector dialog box will appear with yellow triangles with exclamation points in them. This window will tell you what things must be changed.
- 5.2.7.9 Click the OK button.
- 5.2.7.10 The Flat Sample Stage configuration window will appear. The window has three tabs: Instrument Settings, Incident Beam Optics, and Diffracted Beam Optics.
- 5.2.7.11 Click on the Instrument Setting tab.
- 5.2.7.12 Double click the Generator: PW 3040 (mppc) line.

## Appendix H

SAVANNAH RIVER NATIONAL LABORATORY  
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- 5.2.7.13 The Instrument Settings window will appear. This window has three tabs: Position, Sample Stage, and X-ray.
- 5.2.7.14 Click the X-ray tab.
- 5.2.7.15 Click in the Tension (kV) box with the left mouse button and highlight the value.
- 5.2.7.16 Type in 45.
- 5.2.7.17 Repeat the process for the Current (mA).
- 5.2.7.18 Type in 40.
- 5.2.7.19 Click either the OK or the Apply button. The OK button will make the changes and automatically close the window. The Apply button will make the changes, but the window will stay open. The close button will have to be depressed for the window to close.
- 5.2.7.20 Click Measure/Program on the main toolbar.
- 5.2.7.21 The Open Program window will appear. Use the scroll bar adjacent to the List Programs of Type dialog box, and select General Batch.
- 5.2.7.22 Click on the CalFXquartz name on the list.
- 5.2.7.23 Click the OK button. This will start the scan.
- 5.2.7.24 A Start window will appear. On the bottom of the window there are two tabs: Filename and Filename settings.
- 5.2.7.25 Make sure the Filename starts off with F:\XRD-Current Year.
- 5.2.7.26 Click OK.
- 5.2.7.27 The scan is complete when you hear the shutter closing on the x-ray tube.
- 5.2.7.28 The following are steps to locate the data for Data Evaluation:



- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
- Click on **File**, and then **Patterns**.
- Select the **ADSCLUSTER** Drive.
- Double click on the **XRD-Current Year**.
- Change the file extension to Philips PC-XML Ascii Pattern Files (\*.xrd).
- Double click on the correct filename for range 1.
- Click on File, and then Patterns.
- Select the correct filename for range 2 and add this to range 1.
- Click Edit/Merge Segments.

### 5.3 The following specifications should be met for satisfactory instrument performance:

For the quartz (101)  $K\alpha_1/K\alpha_2$  peak:

#### Scintillation

Peak Intensity >20,000 cts/s

Peak  $2\theta$  value  $26.65^\circ \pm 0.03^\circ$

For the quartz (302)  $K\alpha_1$  peak:

#### Scintillation

Peak Intensity > 1,500 cts/s

Peak  $2\theta$  value  $68.32^\circ \pm 0.03^\circ$

Resolution (H1/H2) 2

where H1 is the peak intensity of the  $K\alpha_1(212)$  peak

H2 is the average of the three troughs between the line pairs.

$K\alpha_1(212)$ :  $K\alpha_2(212)$ ;  $K\alpha_2(212)$ :  $K\alpha_1(203)$

$K\alpha_1(301)$ :  $K\alpha_2(301)$

All intensities are background subtracted.

5.3.1 Three additional specifications need to be run to determine if the extensive calibration is needed. The tests are run using the Si powder standard, which came with the system, in standard Bragg - Brentano configuration. These tests, Linearity,

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Modulation Depth, and Reproducibility, are run with the use of the X'Pert Data Collector software. All the tests are done with 45 kV and 40 mA.

## 5.3.2 Linearity Test

## 5.3.2.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	1°
Beam Mask	10mm
Divergent Antiscatter Slit	2°
SC (Flat Sample Stage)	Si Powder Sample
Long Beam Tunnel	8.7
Attenuator	Ni
Detector	X'Celerator

## 5.3.2.2 The following 2theta angles are measured (#steps, step size, and time are included below):

Angle (°2 $\Theta$ )	Steps	Step size (°2 $\Theta$ )	Time (s)
28.443	31	0.005	1.00
47.303	21	0.010	1.25
56.123	21	0.010	2.00
69.131	21	0.010	5.00
76.377	21	0.010	5.00
88.032	21	0.010	5.00
94.954	21	0.015	5.00
106.711	21	0.015	7.50
114.094	21	0.015	15.00
127.547	21	0.015	15.00
136.897	21	0.020	20.00

## 5.3.2.3 Record the following information:

The intensity of the 28.443° reflection (at measured maximum position) and the measured peak positions (profile fit the peaks).

- 5.3.2.4 Calculate the difference between the Actual peak position and the observed peak positions.
- 5.3.2.5 Calculate the Bandwidth. The bandwidth is the most positive peak position difference minus the most negative peak position difference. The bandwidth should be  $<0.030^\circ$  2theta.
- 5.3.2.6 If the bandwidth is greater than  $0.030^\circ$  2theta check the following:
- Check the sample height
  - Check if the sample is correctly inserted (not tilted)
  - Check the tube focus alignment (tube height)

### 5.3.3 Modulation Depth Test

- 5.3.3.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	$1^\circ$
Beam Mask	10mm
Divergent Antiscatter Slit	$2^\circ$
SC (Flat Sample Stage)	Si Powder Sample
Long Beam Tunnel	8.7
Attenuator	Ni
Detector	X'Cellerator

- 5.3.3.2 Measure the 56.123° with 21 steps, a step size of 0.010, and a dwell time of 2.00 seconds.
- 5.3.3.3 Determine the following: Resolution 2-theta FWHM, Modulation Depth, and the  $K\alpha_1/K\alpha_2$  Intensity ratio. The modulation depth is the ratio between the valley (of  $K\alpha_1$  and  $K\alpha_2$ ) and the top of the  $K\alpha_2$ .

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5.3.3.4 The following are the specifications of the three determinations:

- Resolution 2-theta FWHM < 0.12°
- Modulation Depth < 75%
- $K\alpha_1/K\alpha_2$  Intensity ratio ~50%

5.3.3.5 If the above specifications are not met, check the following:

- Check the position of the receiving slit (radius)
- Check the opening of the receiving slit
- Check the calibration of the receiving slit
- Check the Theta alignment and/or calibration.

#### 5.3.4 Reproducibility Test

5.3.4.1 Insert the following:

	<u>Instrument</u>
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	1°
Beam Mask	10mm
Divergent Antiscatter Slit	2°
SC (Flat Sample Stage)	Si Powder Sample
Long Beam Tunnel	8.7
Attenuator	Ni
Detector	X'CeLerator

5.3.4.2 Measure the 28.443° peak of the Si powder sample ten times in a general batch program with a step size of 0.01° and a dwell time of 1 second.

5.3.4.3 Determine the reproducibility of the measurement. The required reproducibility is < 0.00200° 2 theta.

5.3.4.4 If the reproducibility of the measurement is  $> 0.002^\circ$  2 theta check the following:

- Check the movement of 2 theta
- Check the 2 theta alignment and/or calibration.

## 5.4 Standards

Qualitative – A vendor supplied novaculite (quartz) plate is used for calibration.

Quantitative – The following is a list of NIST standards, which are used: Si (NIST SRM640c), alumina (NIST SRM 676),  $\text{LaB}_6$  (NIST SRMs 660 and 660a), and fluorophlogopite (NIST SRM 675). None of these standards has as an expiration date.

## 5.5 Operating Procedure

### 5.5.1 Emergency Shutdown

**Note:** Emergency shutdown is required in the event of general fire, flooding, or massive contamination. This procedure is intended primarily for Building Services or Fire Department personnel when problems develop off-shift.

5.5.1.1 Close PCW valve in cabinet in personnel corridor outside C-123.

5.5.1.2 Call Task Supervisor.

### 5.5.2 Normal Operating Procedures for Bruker D8 Advance X-Ray Diffractometer

#### 5.5.2.1 Specimen Preparation

The specimen usually will have to be ground to a particle size range of 0.2 to 10  $\mu\text{m}$  to achieve maximum and reproducible intensities. Metal plates and other types of monoliths do not have to be ground if they are sufficiently fine grained and do not have crystallographic texture. If there are texture

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phenomena, the intensities will have to be corrected for quantitative determinations.

For qualitative analysis, the powdered specimen must be applied to an amorphous support, which has a low x-ray diffraction background, such as quartz (cut along the [100] plane), plate glass (most common), or lexan. The most common qualitative preparation methods are as follows:

- 5.5.2.1.1 Coat a 2" diameter glass plate with a thin film of Vaseline. Sprinkle approximately 100 mg of finely ground sample onto the plate and using a microscope slide smear the material to as thin a layer as possible (<0.1 mm). The sample need only cover the center portion (1" dia) of the disk.
- 5.5.2.1.2 Place approximately 100 mg of finely ground sample in a mound in the center of the 2" diameter glass plate. Add a couple of drops of 10:1 mixture of amyl acetate/collodion to the material and using a paper clip spread the sample to as thin a layer as possible (<0.1 mm). The sample need only cover the center portion (1" dia) of the disk.
- 5.5.2.1.3 The first two methods should be appropriate for most materials. However, for samples that show preferred orientation, such as clays and other micaceous minerals, see the Task Supervisor for instructions on preparing back-loaded or dusted mounts.

For quantitative analysis, a relatively thick sample must be prepared for good reproducibility, to ensure the sample is infinitely thick, and to reduce texture effects on intensity measurements.

- 5.5.2.1.4 Pour finely ground sample into a 1" dia. by 1/8" deep depression in a 2" dia. Lexan disk. Compact sample evenly with a spatula; scrape off any excess quantity. Repeat this process

several times with a thin layer of powder until the cup is filled to its edge as evenly as possible. The top of the sample must be flush with the top of the Lexan disk.

5.5.2.1.5 This method should be appropriate for most materials. However, for samples that show preferred orientation, such as clays and other micaceous minerals, see the Task Supervisor for instructions on preparing back-loaded or side-loaded mounts.

5.5.2.1.6 Load sample in goniometer. Make sure that the sample is seated properly in the sample holder.

#### 5.5.2.2 X'Pert Pro

For normal operation insert or set the following:

<u>Instrument</u>	
Incident Soller Slit	0.04 Rad
Fixed Divergent Slit	1/2°
Beam Mask	10mm
Divergent Antiscatter Slit	1°
SC	Novaculite
Short Beam Tunnel	3.4
Long Beam Tunnel	5.5
Attenuator	Ni

**NOTE:** The fixed slits are placed in the component with the v notch at the top. There is only one way and one opening that the slits will go into the component.

5.5.2.3 Recirculating Chiller - Haskris R100 Serial number H-A13853

5.5.2.3.1 Turn **On-Off Switch** to "ON".

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- 5.5.2.3.2 Tank Level Indicator light to the left of the switch will illuminate for the Haskris chiller. If off, refill the tank with deionized water.
- 5.5.2.3.3 Set temperature at 72 °F on the Haskris. Set GPM water gauge at 1.4 GPM.

#### 5.5.2.4 Electronics/Generator High Voltage Supply

- 5.5.2.4.1 Turn the **HT Key Switch** on the control and display panel clockwise.
- 5.5.2.4.2 Make sure the front sliding and rear doors are closed.
- 5.5.2.4.3 Press the **Power On** button on the control and display panel. The “**Standby**” indicator lamp will go out and the “**Power On**” will be lit.
- 5.5.2.4.4 Wait until the LEDs on the control and display panel are lit. When no dots are shown on the “Shutter Open” LED the system starts its initialization procedure. The HT generator will switch on to a generator setting of 15 kV, 5mA. The X-rays On Lamp on top of the enclosure will light.

**NOTE:** If three moving dots are shown on the “Shutter Open” LED it indicates that the enclosure doors and/or the rear panels are not correctly closed or that the HT key is not turned on. In this condition, the system cannot be initialized; either close the doors or turn the HT key switch clockwise, in order to let the system continue its initialization procedure.

**NOTE:** If the system has been shut down completely, the x-ray tube must be Breed. The breeding process is found in the X'Pert Data Collector software. Click on the Instrument Setting tab and double click on the Generator PW3040 header. An Instrument Settings window will appear. Click on the Breed button located in the middle of this window.



## 5.5.2.5 Computer - On the Desktop:

## 5.5.2.5.1 If an attachment has been replaced:

- Select the **X'Pert Data Collector** Icon on the Desktop
- Enter **User-1** in the User box, and **galaxy** for the Password on the Login window.
- If the sample stage is different then the one that is attached to the instrument, select Tools/Exchange Sample Stage from the main toolbar. A Change Sample Stage window will appear. Select from one of the 4 sample stages (PW3071/xx Bracket, Capillary Spinner, Anton Paar HTK 1200 Oven, and the Anton Paar XRK-900). There is one other sample stage in the list, which is the PW 3072/xx bracket. This is a spinner stage, which we do not have.
- After selecting the desired sample stage, click the Apply button.
- The X'Pert Wizard will appear. The Wizard will go through the steps of how to change to a different sample stage. After each step is complete, click the Next button. Click the Finish button when complete.
- Select Instrument/Connect from the main toolbar.
- Select appropriate configuration based on the above stage selection, and click OK. There are several configurations. They are as follows: Anton Paar HTK 1200, Anton Paar XRK 900, Capillary Spinner, Flat sample stage, and Mirror-mirror. A X'Pert Data Collector window will appear with upside down yield signs. Select the OK button.

- Based on your configuration selection, a window will appear with the name of the configuration.
- Select the Instrument Settings tab.
- Double click on the Generator header. An Instrument setting window will appear. Type in 45kV and 40mA for the voltage and current in the appropriate boxes.
- Click the OK button. This will adjust the voltage and current, and the window will close when the generator reaches the inputted values.

5.5.2.5.2 Select Measure/Program from the main toolbar. An Open Program window will appear.

5.5.2.5.3 Select the program by using the scroll bar adjacent to the list programs of type dialog box. The type of program depends on the sample stage selected and the type of measurement to be performed. The following is a list of the types of programs: Relative scan, Absolute scan, 2-Axes measurement, Stationary measurement, General batch, and non-ambient program. After making a selection, all the programs for that type will appear.

5.5.2.5.4 Select the appropriate Name (model) by moving the scroll bar and clicking on that line.

**NOTE:** There are several models available for slower or longer scans, and different configurations. Consult the Task Supervisor. See Attachment 5 on how to create a **Program/Model**.

5.5.2.5.5 Click the OK button. A Start window will appear.

5.5.2.5.6 In the Name box, highlight everything up to the .xrdml and delete it. Type in the ADS number for the name.

- 5.5.2.5.7 Click on the Folder Icon to the right.
- 5.5.2.5.8 Click on the arrow next to the Save in box, and select the Adscluster.
- 5.5.2.5.9 The Adscluster will appear. Select the current year XRD folder. (XRD-20xx)
- 5.5.2.5.10 Click the save button.
- 5.5.2.5.11 Type the Customer's identification into the Sample ID box.
- 5.5.2.5.12 Click the OK button. The instrument will start the measurement.
- 5.5.2.5.13 The scan is complete when the numbers for Omega and 2Theta, located on the left corner of the X'Pert Data Collector window, stopped increasing.
- 5.5.2.5.14 The following are steps to locate the data for Data Evaluation:
- Double click on the **JADE** Icon on the desktop of the data evaluation computer.
  - Click on **File**, then **Patterns**.
  - Select the **ADSCLUSTER** drive.
  - Select the **XRD- Current Year**.
  - Select the file extension for Philips PC-XML Ascii Pattern Files (\*.xrd).
  - Double click on the correct filename.
- 5.5.2.5.15 Use various options in the MDI Jade+ software to obtain diffraction results after the scan is completed. See appropriate manuals for more information.
- 5.5.2.5.16 Enter results in the XRD Sample Logbook and into the ADS LIMS.

## 5.6 Normal Shutdown

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- 5.6.1 Double click on the Generator PW3040 header on the Instrument Setting tab for the particular configuration that was used.
- 5.6.2 Type in 20 and 10 for the kV and mA. Click on the OK button.
- 5.6.3 Switch the HT generator off by turning the HT key switch on the control and display panel counter-clockwise. The “X-rays On” lamp on top of the enclosure will go out.
- 5.6.4 Press the “Standby” button on the control and display panel. The “Power On” indicator lamp will go out and the “Standby” indicator lamp will light.
- 5.6.5 Turn the **ON/OFF** switch on the Haskris recirculating chiller to the “OFF” position.

**6.0 RECORDS**

- 6.1 The X-Ray Diffraction Logbook shall be handled in accordance with Records Management procedures.
- 6.2 A copy of all the x-ray diffraction raw data shall be stored for five years on the ADS file server in a location separate from the XRD facility.
- 6.3 A copy of the Maintenance Reports shall be entered into the Maintenance Notebook.
- 6.4 The calibration results shall be entered into the Calibration Notebook.
- 6.5 Results for all routine analytical samples will be entered into the ADS Laboratory Information System and the XRD Logbook using the ADS LIMs numbers.

**7.0 REFERENCES**

- 7.1 PANalytical X’Pert Pro Manuals and Prints
  - X’Pert Pro User’s Guide
  - X’Pert Pro Service Manual
- 7.2 Anton Paar
  - Anton Paar HTK 1200 Manual
  - Anton Paar XRK 900 Manual

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- Anton Paar TCU 1000 Manual
- Anaton Paar TCU 750 Manual
- LeyBold TurboVac Manual
- LeyBold TriiVacB Rotary Vane Vacuum Pump Manual

7.3 Haskris

Model R100C Refrigerated Closed Circuit Water Chiller Instruction Manual.

7.4 Materials Data Inc.

Jade for Windows Manual

7.5 WSRC 8Q, Employee Safety Manual 12, General Site Safety Requirements.

**8.0 ATTACHMENTS**

8.1 Attachment 1: Interlocks for the PANalytical X'Pert Pro

8.2 Attachment 2: Creating a New Program File

8.3 Attachment 3: Creating a New Non-ambient Program File

8.4 Attachment 4: Scans: Quartz (Main Reflection and the Five Finger Region)

## ATTACHMENT 1

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**INTERLOCKS for the PANalytical X'Pert Pro**

This table lists the interlocks for which routine testing is not necessary. This list and the PANalytical schematics are required to troubleshoot the systems when x-rays cannot be produced or tube shutters will not open. A number of the interlocks are synergistic. See reference section for a list of the PANalytical schematics documenting interlocks logic.

<u>Purpose</u>	<u>Fault</u>	<u>Device(s) Controlled</u>	<u>Interlocks</u>
Radiation Safety	No X-ray On light on top of equipment enclosure.	X-ray power	1 lamp
Radiation Safety	Burned out yellow Shutter open light	1 shutter on x-ray tube	1 LED
Radiation Safety	Burned out X-ray On Lamp on the control Panel	X-ray power	1 light
Radiation Safety	X-ray tube out of position	X-ray power	"Rip Cord" pigtail connector at bottom of tube housing unplugged
Radiation Safety	Back panel not properly positioned	X-ray power	2 microswitches
Radiation Safety	Front doors not properly closed	X-ray power	4 microswitches
Radiation Safety	Shutter does not open	X-ray power	1 microswitch
Protect Generator and Tube	X-ray generator and tube switched off	X-ray power	The cooling water fell below 3.5 l/min.
Protect generator	X-ray tube or generator will not turn on	X-ray power	1. The tube indicator plug is not correctly fitted into the sockets

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Purpose	Fault	Device(s) Controlled	Interlocks
			in the tube shield and the X-ray tube. 2. The cylinder through which the HT cable passes from the HT tank to the X-ray enclosure is not correctly fitted.
Protect Water Recirculator Compressor	PCW water flow	Compressor	Thermostat
Protect Water Recirculator	Excess input current	Power from Main	1. 15A breaker 2. Fuse
Protect Water Recirculator	Water temperature	Compressor	Thermostat
Protect Water Recirculator	Water Flow	Pump	Flowmeter

**ATTACHMENT 2**

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**CREATING A NEW PROGRAM FILE**

**The Program file specifies all of the instrument operating conditions.**

1. Double click on the Data Collector Icon on the desktop.
2. The Login window will appear.
3. Log on with the following information:  
User: User-1 (case sensitive)  
Password: galaxy
4. Press the OK button.
5. Select File/New Program from the main toolbar. The New Program window will appear.
6. Use the scroll bar adjacent to the Program type box to select the type of Program. There are 6 different program types to select from which include the following: Relative scan, Absolute scan, 2-Axes measurement, Stationary measurement, General batch, and Non-ambient program.

Note: The direction from here forward will be for an absolute scan. Direction for a General batch and a Non-ambient program can be found in the next attachment. For all other programs see the Task supervisor.

7. Press the OK button. The Prepare Absolute scan window will appear.
8. Select the sample stage by using the scroll bar right underneath the Configuration header. The flat sample stage will be used in this example.

Note: See the Task Supervisor for more information on the other sample stages.

9. Select Diffracted beam path 1 by clicking the radial box.
10. Select the Gonio for the Scan Axis by using the scroll bar underneath the Scan Axis header.

Note: There are several different scan axis. The following is a list of the different scan axis: Gonio, Omega, 2Theta, Omega-2Theat, and 2Theta-Omega. See the Task Supervisor for more information.



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11. Select Continuous for the Scan mode.
12. Input the following information: Start angle, End angle, Step size, Time per step, Scan speed.

**Note:** If the X'celerator detector is selected for the program the step size, time per step, and the scan speed will need to be changed. See the Task supervisor for more details.

13. Click on the Settings button. The Program1:Settings window will appear.
14. Expand the Incident beam path by clicking on the "+" in front of the Incident beam path Icon.
15. Click on the PreFix Module line. Using the scroll bar on the bottom half of the window, select the Incident PreFix module that will be used. Select either Fixed Div. Slit module or the X-ray Mirror Cu module (MPD). For this program the Fixed Incident Div. Slit module will be used. See the Task Supervisor for information regarding the X-ray Mirror Cu module.
16. Click on the Incident Filter line.
17. Select None by using the scroll bar on the bottom half of the window.
18. Click on the Soller slit line.
19. Select Soller 0.4 rad. by using the scroll bar on the bottom half of the window.
20. Click on the Beam Attenuator line.
21. Select None by using the scroll bar on the bottom half of the window.
22. Click on the Mask line.
23. Select the Inc. Mask Fixed 10mm (MPD/MRD) by using the scroll bar on the bottom half of the window.
24. Click on the Divergence slit line.
25. Select the Slit Fixed  $1/2^\circ$  by using the scroll bar on the bottom half of the window.
26. Click on the Anti-scatter line.
27. Select the Slit Fixed  $1^\circ$  by using the scroll bar on the bottom half of the window.

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28. Click on the Beam knife line.
29. Select None by using the scroll bar on the bottom of the window.
30. Click on the Mirror line.
31. Select None by using the scroll bar on the bottom of the window.
32. Expand the Diffracted beam path by clicking on the "+" in front of the Diffracted beam path Icon.
33. Click on the PreFix Module line. Using the scroll bar on the bottom half of the window select the Diffracted PreFix module that will be used. Select from the following: Parallel Plate Collimator 0.18°, X'celerator Module, or the X'celerator Monochromator for Cu. For this program the X'celerator Module will be used. See the Task Supervisor for information regarding the other Diffracted beam path PreFix Modules.
34. Click on the Filter line.
35. Select Actual by using the scroll bar on the bottom of the window.
36. Click on the Soller slit line.
37. Select None by using the scroll bar on the bottom of the window.
38. Click the Detector line.
39. Select the X'celerator by using the scroll bar on the bottom of the window.
40. After selecting the X'celerator two boxes will appear. They are Scan Mode and Active length (mm).
41. Select Scanning mode by using the scroll bar.
42. Type in 2.10 for the Active length.
43. Click on the scroll bar but do not change the Scan mode. The Active length will change to 2.12.
44. Click on the Anti-scatter line.
45. Select None by using the scroll bar on the bottom of the window.
46. Click on the Collimator line.
47. Select None by using the scroll bar on the bottom of the window.
48. Click on the Mask line.
49. Select None by using the scroll bar on the bottom of the window.
50. Click on the Monochromator line.

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51. Select None by using the scroll bar on the bottom of the window.
52. Click on the Receiving slit line.
53. Select None by using the scroll bar on the bottom of the window.
54. After everything is inputted into this file, click the OK button.
55. Make sure the Step size, Time per step, and Scan speed is correct.
56. Click on the "X" in upper right hand corner. A X'Pert Data Collector window will appear and the following question will be contained in the window: "Do you want to save the changes?"
57. Click the Yes button. A Save Program As window will appear.
58. Type a Name into the Name box.
59. Click on the box adjacent to Available to all users.
60. Click the OK button.

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**ATTACHMENT 3**

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**CREATING A NON AMBIENT PROGRAM**

**The following directions are for either the XRK900 or the HTK1200 high temperature stages.**

1. Double click on the Data Collector Icon on the desktop.
2. The Login window will appear.
3. Log on with the following information:  
User ID: User-1 (case sensitive)  
Password: galaxy
4. Press the OK button.
5. Select File/New Program from the main toolbar. The New Program window will appear.
6. Use the scroll bar adjacent to the Program type box to select the Absolute scan option.
7. Press the OK button. The Prepare Absolute scan window will appear.
8. Select the sample stage, either the Anton Paar XRK-900 or the Anton Paar HTK-1200, by using the scroll bar right underneath the Configuration header.
9. Select Diffracted beam path 1 by clicking the radial box.
10. Select the Gonio for the Scan Axis by using the scroll bar underneath the Scan Axis header.
11. Select Continuous for the Scan mode.
12. Input the following information: Start angle, End angle, Step size, Time per step, Scan speed.

Note: If the X'celerator detector is selected for the program, the step size, time per step, and the scan speed will need to be changed. See the Task supervisor for more details.

13. Click on the Settings button. The Program1:Settings window will appear.
14. Expand the Incident beam path by clicking on the "+" in front of the Incident beam path Icon.

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15. Click on the PreFix Module line. Using the scroll bar on the bottom half of the window select the Incident PreFix module that will be used. Select either Fixed Div. Slit module or the X-ray Mirror Cu module (MPD). For this program, the Fixed Incident Div. Slit module will be used. See the Task Supervisor for information regarding the X-ray Mirror Cu module.
16. Click on the Incident Filter line.
17. Select None by using the scroll bar on the bottom half of the window.
18. Click on the Soller slit line.
19. Select Soller 0.4 rad. by using the scroll bar on the bottom half of the window.
20. Click on the Beam Attenuator line.
21. Select None by using the scroll bar on the bottom half of the window.
22. Click on the Mask line.
23. Select the Inc. Mask Fixed 10mm (MPD/MRD) by using the scroll bar on the bottom half of the window.
24. Click on the Divergence slit line.
25. Select the Slit Fixed  $1/2^\circ$  by using the scroll bar on the bottom half of the window.
26. Click on the Anti-scatter line.
27. Select the Slit Fixed  $1^\circ$  by using the scroll bar on the bottom half of the window.
28. Click on the Beam knife line.
29. Select None by using the scroll bar on the bottom of the window.
30. Click on the Mirror line.
31. Select None by using the scroll bar on the bottom of the window.
32. Expand the Diffracted beam path by clicking on the "+" in front of the Diffracted beam path Icon.
33. Click on the PreFix Module line. Using the scroll bar on the bottom half of the window, select the Diffracted PreFix module that will be used. Select from the following: Parallel Plate Collimator  $0.18^\circ$ , X'celerator Module, or the X'celerator Monochromator for Cu. For this program the X'celerator Module

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34. will be used. See the Task Supervisor for information regarding the other Diffracted beam path PreFix Modules.
35. Click on the Filter line.
36. Select Actual by using the scroll bar on the bottom of the window.
37. Click on the Soller slit line.
38. Select None by using the scroll bar on the bottom of the window.
39. Click the Detector line.
40. Select the X'celerator by using the scroll bar on the bottom of the window.
41. After selecting the X'celerator two boxes will appear. They are Scan Mode and Active length (mm).
42. Select Scanning mode by using the scroll bar.
43. Type in 2.10 for the Active length.
44. Click on the scroll bar but do not change the Scan mode. The Active length will change to 2.12.
45. Click on the Anti-scatter line.
46. Select None by using the scroll bar on the bottom of the window.
47. Click on the Collimator line.
48. Select None by using the scroll bar on the bottom of the window.
49. Click on the Mask line.
50. Select None by using the scroll bar on the bottom of the window.
51. Click on the Monochromator line.
52. Select None by using the scroll bar on the bottom of the window.
53. Click on the Receiving slit line.
54. Select None by using the scroll bar on the bottom of the window.
55. After everything is inputted into this file, click the OK button.
56. Make sure the Step size, Time per step, and Scan speed is correct.
57. Click on the "X" in upper right hand corner. An X'Pert Data Collector window will appear and the following question will be contained in the window: "Do you want to save the changes?"
58. Click the Yes button. A Save Program As window will appear.
59. Type a Name into the Name box.

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**ATTACHMENT 3**

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60. Click on the box adjacent to Available to all users.
61. Click the OK button.
62. Click on File/New Program from the main toolbar. A New Program window will appear.
63. Select either the Anton Paar XRK-900 or the Anton Paar HTK-1200 configuration by using the scroll bar adjacent to the configuration box.
64. Click on the OK button. The Prepare Non-ambient Program window will appear.
65. The program will contain three lines, Start, reset, and finish.
66. There will be a black arrow to the right of the start line.
67. Click on the gray box in front of the Reset box. This will move the black arrow down and the Insert Program will not be grayed out.
68. Click on the Insert to the left of the Measurement button. A Non-ambient Program step window will appear.
69. Click on the Browse button. A Open Program window will appear.
70. Select the Absolute scan option by using the scroll bar adjacent to the List Programs of type box. All the available scans will appear.
71. Select the absolute scan program which was made at the beginning of this attachment.
72. Click the OK button on the Open Program window.
73. Click the OK button on the Non-ambient Program Step window.
74. Click on the Insert button to the left of Non-ambient setting button. A Non-ambient Setting window will appear.
75. Change the temperature and the slope by highlighting the information in the boxes and typing in the new temperature and slope values. See the Task Supervisor for more information.
76. Click the OK button.
77. Click on the Insert button to the left of the Timer setting. A Timer setting window will appear.
78. Type in a hold time. See the Task Supervisor for more details.
79. Click the OK button.

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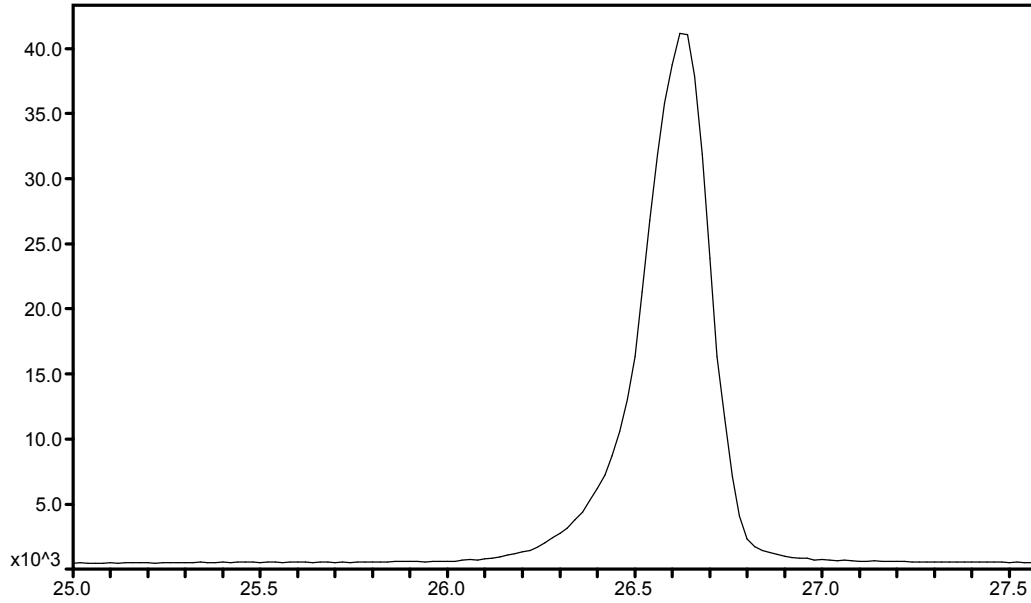
80. Repeat steps 67 through 78 to add more lines to the program. See Task Supervisor for more information.
81. Click on the Profile button at the bottom of the window. This will show a plot of the program.
82. Click on the "X" in the upper right hand corner to close this plot.
83. Click on the Check button at the bottom of the window. This will let you know if anything needs to be replaced.
84. Click the OK button.
85. Click on the "X" in upper right hand corner. A X'Pert Data Collector window will appear and the following question will be contained in the window: "Do you want to save the changes?"
86. Click the Yes button. A Save Program As window will appear.
87. Type a Name into the Name box.
88. Click on the box adjacent to Available to all users.
89. Click the OK button.



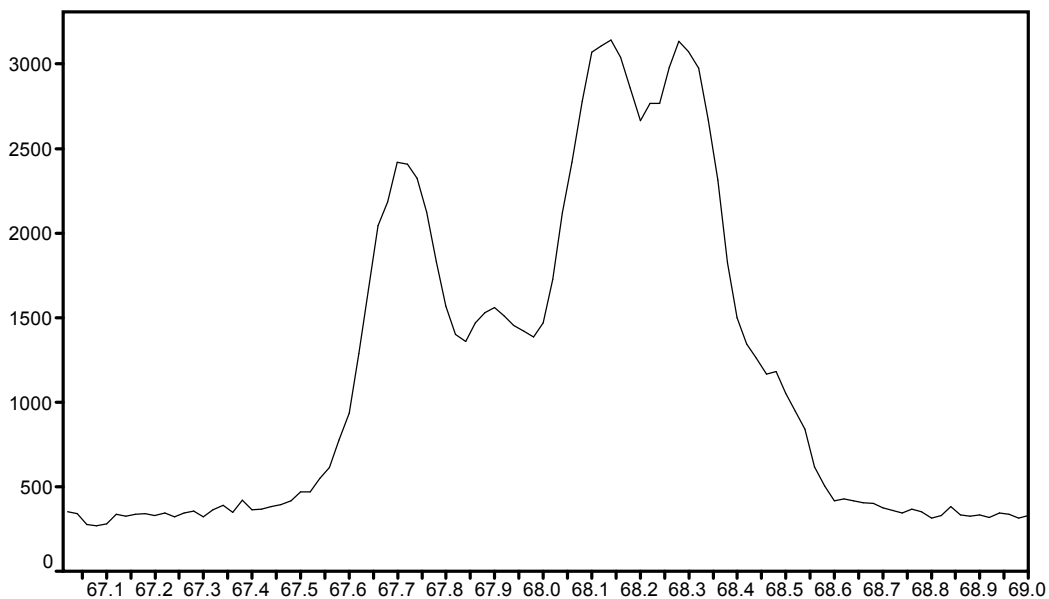
ATTACHMENT 4

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SCANS: QUARTZ (Main Reflection and the Five Fingers Region)



ATTACHMENT 4  
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**Gamma Sample Preparation and Analysis ( $\gamma$ -PHA) (U)**

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Signature

Originator:

SIGNED COPY ON FILE  
J. D. Leyba

Author:

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C. C. DiPrete

Analytical Task Supervisor:

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D. P. DiPrete

Reviewer:

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R. A. Sigg

ADS Procedure Coordinator:

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ADS Radioanalytical Services Group Manager:

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## 1.0 Purpose

This procedure covers the application of high-purity, high resolution germanium detectors to the detection, identification and quantification of gamma rays from a variety of low to moderate density sample matrices.

## 2.0 Scope

This procedure applies to gamma Pulse Height Analysis of a variety of sample types.

Although gamma Pulse Height Analysis (PHA) is relatively simple, specific and rapid, there are limitations that must be taken into account. Detectors must be carefully energy calibrated with known standards. Detection efficiencies for gamma-rays vary with energy and must be obtained during the calibration process. The counting geometry (position of counting package relative to the detector) is established during calibration and must be carefully duplicated for accurate work. The relative amount of activity in the counting vial must be within an optimum range for accurate work. The Multichannel Analyzer (MCA) displays a "dead time" percent on the screen as the sample counts. During this "dead time" the MCA does not accept additional events. If the "dead time" is >10%, the sample should be diluted for accurate counting. Also, the relative abundance of the various gamma nuclides in the samples has an effect on the quality of the data. Higher energy gammas can interfere with, or even mask low energy gammas. Non-calibrated geometries can be analyzed, but only if a standard is used and only with the approval of the Analytical Task Supervisor.

## 3.0 Precautions/Limitations

FOLLOW THE SRTC RADIOLOGICAL WORK PRACTICES  
REQUIREMENTS

FOLLOW THE ADS RADIATION SAFETY GUIDELINES

FOLLOW THE ADS LABORATORY SAFETY GUIDELINES

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Remember:

1. Use a fume hood when necessary.
2. Add acid to water.
3. Wear plastic gloves when handling chemicals.
4. Avoid contact with skin and breathing of vapors.
5. Voice contact or supervision present for off shift work
6. Supervision present for high activity work.
7. Do not exceed:
  - hood and radiobench activity limits
  - self-monitoring limits
  - unsupervised off-shift limits.

#### 4.0 Prerequisite Actions

Laboratory personnel performing this analysis must have:

- Completed on-the-job training in this method under the supervision of the Analytical Task Supervisor.
- Be qualified in this method by the Analytical Task Supervisor or the group supervisor, after processing samples supplied by the Analytical Task Supervisor.

#### 5.0 Performance

##### 5.1 Description

Liquid and low-density solid samples in a variety of container types can be analyzed by gamma-PHA. The primary limitations are that a suitable efficiency standard be available for each sample type and that the efficiency standard and the sample be counted in the same geometry.

##### 5.2 Equipment

High Purity germanium detectors, and associated gamma spectroscopy electronics

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Canberra Genie 2K gamma spectroscopy software  
 Changer Labs sample changer  
 Calibrated pipets

5.3 Calibrated analytical balances  
 Reagents

Purity of reagents - Unless otherwise stated, ACS reagent grade chemicals shall be used.

Purity of water - Unless otherwise indicated, reference to water shall be understood to mean the distilled water, which is provided in the building (773-A) distilled water system.

5.4 Calibration Frequency

Energy calibrations are made as specified by the Analytical Task Supervisor, but are evaluated daily by the daily check standard. Efficiency calibrations are made as needed, but are at least evaluated annually. Other standards may be run, as called for in specific procedures or at the direction of the Analytical Task Supervisor, for special samples or geometries. Such standards measurements will cross-reference the sample for which they are run.

A three peak daily check source is counted each day that samples are run. Gamma energies for the check standard should be within  $\pm 0.5$  keV and are required to be within  $\pm 1.0$  keV of the true peak energy. The radionuclide activities are required to be within  $2\sigma$  of the true value. Recalibration for energy or efficiency is required if either the energy calibration or the efficiency calibration is outside of these acceptance criteria.

5.5 Calibration and Standardization

This section defines the minimum calibration and service checks for all germanium detectors used for gamma PHA service analyses in ADS.

5.5.1 Energy Calibration:

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Energy calibration is performed by counting known standards containing several gamma ray peaks with well established energies. In general, or as required by the customer, the calibration peaks will span the entire energy range to be reported. Because of the presence of several common high-energy background gamma rays, however, this may be optional for some samples, as determined by the customer and the Analytical Task Supervisor.

In some cases (as determined by the Analytical Task Supervisor), it may be desirable to determine the energy calibration from the sample itself. This is often possible in environmental samples that exhibit a few well-known natural background peaks at high energies (e.g. 2614.4 keV of Tl-208). In all cases where this option is done, it shall be recorded in the detector logbook, along with the sample number, date, time and analyst.

The energy calibration tolerance is 1.0 keV; that is, the assigned peak energy in the spectrum must be 1.0 keV or less from the actual peak energy. No corrections for counting geometry are required.

#### 5.5.2 Efficiency Calibration:

Known standards of various gamma-emitting radioisotopes are run through the data acquisition procedure to determine detection efficiencies over the energy range of interest. No corrections for counting geometry are required as long as the same geometry is used for both standards and unknown samples. Deadtime corrections are made from the MCA; summing corrections (only needed at very high count rates or for radionuclides with cascade gamma rays) are done at the direction of the Analytical Task Supervisor.

A full efficiency calibration, using NIST-traceable standards, shall be performed as determined by the Analytical Task Supervisor. Radionuclide activities determined during the

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efficiency calibration must be within  $\pm 2\sigma$  of the expected radionuclide activities for the standard.

### 5.5.3 Daily Check Standard and Control Charts

A (minimum) 3-peak check standard shall be counted on each detector on each day that the detector is to be used to count samples. The measured peak energies in the check standard should be within  $\pm 0.5$  keV and must be within  $\pm 1.0$  keV. The measured radionuclide activities shall be within  $2\sigma$  of the expected values for the sample. If either requirement is not met, the detector shall be calibrated as indicated by the check standard results.

The full-width at half maximum (FWHM) width of the largest peak in the check standard shall be monitored at each check standard court. The FWHM shall be within 0.3 keV of the average value for that peak.

The following items shall be reviewed daily, and the QC reports containing this data shall be filed for future reference, if necessary. Items a) and b) shall be control charted. The control charts are reviewed monthly.

- a). the measured peak energies of three peaks in the check standard.
- b). the measured activities of three radionuclides in the check standard.
- c). the FWHM of the largest peak.

### 5.5.4. Background Check

A background spectrum will be collected for each detector as determined by the Analytical Task Supervisor, but at least monthly.

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The spectrum should be accumulated over a period of time sufficient to properly determine a true background spectrum for the energy region normally used for sample analyses. Preferably the spectrum will be acquired for 200,000 seconds or at a minimum of 50,000 seconds, unless otherwise directed by the Analytical Task Supervisor.

## 5.6 Analysis

### 5.6.1. Liquid Samples

- a. Prepare sample in geometry and dilution as directed by Analytical Task Supervisor.
- b. Record prep information.
- c. Have samples cleared to be transported to final counting facility.
- d. Count sample on detector as directed by Analytical Task Supervisor. Ensure detector has had QA successfully completed prior to counting the sample.
- e. Record sample ID, date, time, detector, geometry, and initials in log book.
- f. After data has been reviewed by Analytical Task Supervisor, remove results from LIMS as directed by Analytical Task Supervisor.
- g. After analysis, check data and enter into the ADS Laboratory Information Management System as directed by the Analytical Task Supervisor.

### 5.6.2. Solid Samples

- a. Prepare sample in geometry as directed by Analytical Task Supervisor.
-

## Appendix H

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- b. Record prep information.
- c. Have samples cleared to be transported to final counting facility.
- d. Count sample on detector as directed by Analytical Task Supervisor. Ensure detector has had QA successfully completed prior to counting the sample.
- e. Record sample ID, date, time, detector, geometry, and initials in log book.
- g. After data has been reviewed by Analytical Task Supervisor, remove results from LIMS, as directed by Analytical Task Supervisor.

### 6.0 Records

All lab notebooks, raw data, and electronic data will be kept indefinitely.

### 7.0 REFERENCES

1. Nuclear and Radiochemistry, G. Friedlander, J.W. Kennedy, E.S. Macias, J.M. Miller, 3rd Ed., pp. 243-286.
  2. ANSI N42.14-1991. American National Standard Calibration and Use of Germanium Spectrometers for Measurement of Gamma-Ray Emission Rates of Radionuclides.
  3. Genie-PC Basic Operation, Canberra Industries, Inc. 1994.
  4. Genie-PC Advanced Topics, Canberra Industries, Inc. 1994.
  5. Changer Labs Operations and Set-up manual, Westinghouse Savannah River Technical Center, Changer Labs 1996.
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## 8.0 Attachments

None

**APPENDIX M**

**SOIL SAMPLE TESTS BY MACTEC AND STL**

## **APPENDIX M LABORATORY TESTING - GEOTECHNICAL**

Laboratory testing was performed on disturbed soil samples obtained during the subsurface investigation. All testing was performed in accordance with ASTM standards or other standards where applicable. Selection of the samples to be tested and the tests to be performed on the samples were done by Bechtel. Bechtel provided Kd Analysis Laboratory Test Assignment Sheet.

Testing of soil samples, except for chemical tests, was done in MACTEC's laboratory in Charlotte, North Carolina.

Chemical testing for Cation Exchange Capacity (CEC) of selected soil samples as assigned by Bechtel was done by Severn Trent Laboratories, Inc. (STL), a subcontractor to MACTEC.

The distribution coefficient, Kd, was determined by the Savannah River National Laboratory, located at the Savannah River Site of the DOE in Aiken, South Carolina and operated by the Washington Savannah River Company. The Washington Savannah River Company LLC, under a Work for Others Agreement with MACTEC, performed the Kd tests. The Kd tests are presented within this Attachment H.

### **1.1 IDENTIFICATION TESTS**

- Sieve and hydrometer analysis, ASTM D 422-63 (2002)
- Chemical analysis, (pH) ASTM D 4972-01

### **1.2 REPORTING**

Except for the Kd tests, the laboratory test reports on the soil samples, consisting of individual test data and results sheets as required by the testing standard, are contained in this Appendix M of Attachment H. A summary of the test results on soil samples in Appendix M is found in Table M-1 of this Appendix.

### **1.3 QUALITY ASSURANCE**

Quality related activities performed by MACTEC and its subcontractors organizations during the work herein presented were in accordance with the MACTEC Quality Assurance Manual and the MACTEC Quality Assurance Project Document. The MACTEC QA program complies with NQA-1 Subpart 2.2 and to the requirements of 10CFR50 Appendix B.

### **1.4 DESCRIPTIONS**

A brief description of the tests performed and reported in this Attachment H Appendix M is as follows:

### Particle Size Analysis ASTM D 422-63 (2002)

Sieve Analysis – The dried soil sample is separated into a series of fractions using a standard set of nested sieves. The sieving operation is conducted by means of a lateral and vertical motion of the nest of sieves, accompanied by jarring action to keep the sample moving continuously over the surface of the sieves. The weights retained on each of the set of nested sieves are used to calculate the percent of the sample passing each sieve size.

Hydrometer Analysis – The portion of the soil sample passing the No. 10 (2.0 mm) sieve is soaked in water and dispersed using a dispersing agent. The solution is placed in a cylinder and stirred, and the density of the solution is monitored over time with a hydrometer to observe the settling out of suspended soil particles. Diameters corresponding to the readings of the hydrometer are then calculated using Stoke's law.

Section 5.1.1 of ASTM D 422-63 (2002) gives minimum sample mass requirements (the minimum depends on the maximum particle size present) for each test. In cases where there was not enough sample to meet the appropriate recommended mass, the test was completed using the available sample and it was noted in the Remarks section of the Particle Size Distribution Report.

### Chemical Analysis (pH) ASTM D 4972-01

Chemical analyses consisted of soil pH testing. Soil pH is measured using a pH meter with results reported to 0.1 pH units. The pH test is performed in a solution of calcium chloride and also in a solution of distilled water.

### Chemical Analysis (CEC) SW846 9081

Chemical Analysis for Cation Exchange Capacity (CEC) was performed by Severn Trent Laboratories, Inc. (STL) in Earth City, Missouri. Results of the CEC testing are included in the Analytical Report (Lot # F6L220224) by STL included in this Appendix and summarized in Table M-1.

## **1.5 ATTACHMENTS**

Table M-1 Summary of Test Results – 1 page  
Particle Size Distribution Reports – 15 pages  
Analytical Report (Lot # F6L220224) by STL – 14 pages

**Table M-1**  
**Summary of Test Results**  
**SCE&G COL Project**  
**MACTEC Engineering and Consulting, Inc.**  
**Project No. 6234-06-3534**

Source of Sample	Sample No.	Depth (ft)	Sample Type	Gravel <sup>(1)</sup> (%)	Sand <sup>(1)</sup> (%)	Fines <sup>(1)</sup> (%)	Silt <sup>(1)</sup> (%)	0.005 mm Clay <sup>(1)</sup> (%)	USCS <sup>(2)</sup>	pH in Water <sup>(3)</sup>	pH in Calcium Chloride <sup>(3)</sup>	CEC (meq/100g)	Percent Moisture (%)
B-212 / B-212A	14 (13)	48.5	SPT	10	40	49	40	9	SM	6.0	6.0	22.3	28.8
B-212 / B-212A	18	58.5	SPT	69	15	16	12	4	GM	6.4	6.3	22.3	28.3
B-620	12 (11)	38.5	SPT	0	57	43	34	9	SM	6.2	6.2	27.2	16.4
B-627	9 (10)	23.5	SPT	0	53	47	38	9	SM	5.9	5.8	40.2	22.3
B-627	14 (15)	47.0	SPT	3	67	30	24	6	SM	6.9	6.3	29.4	13.4

(1) Due to computer roundoff, particle size fractions may total 100 ± 1. Fines include silt plus clay.

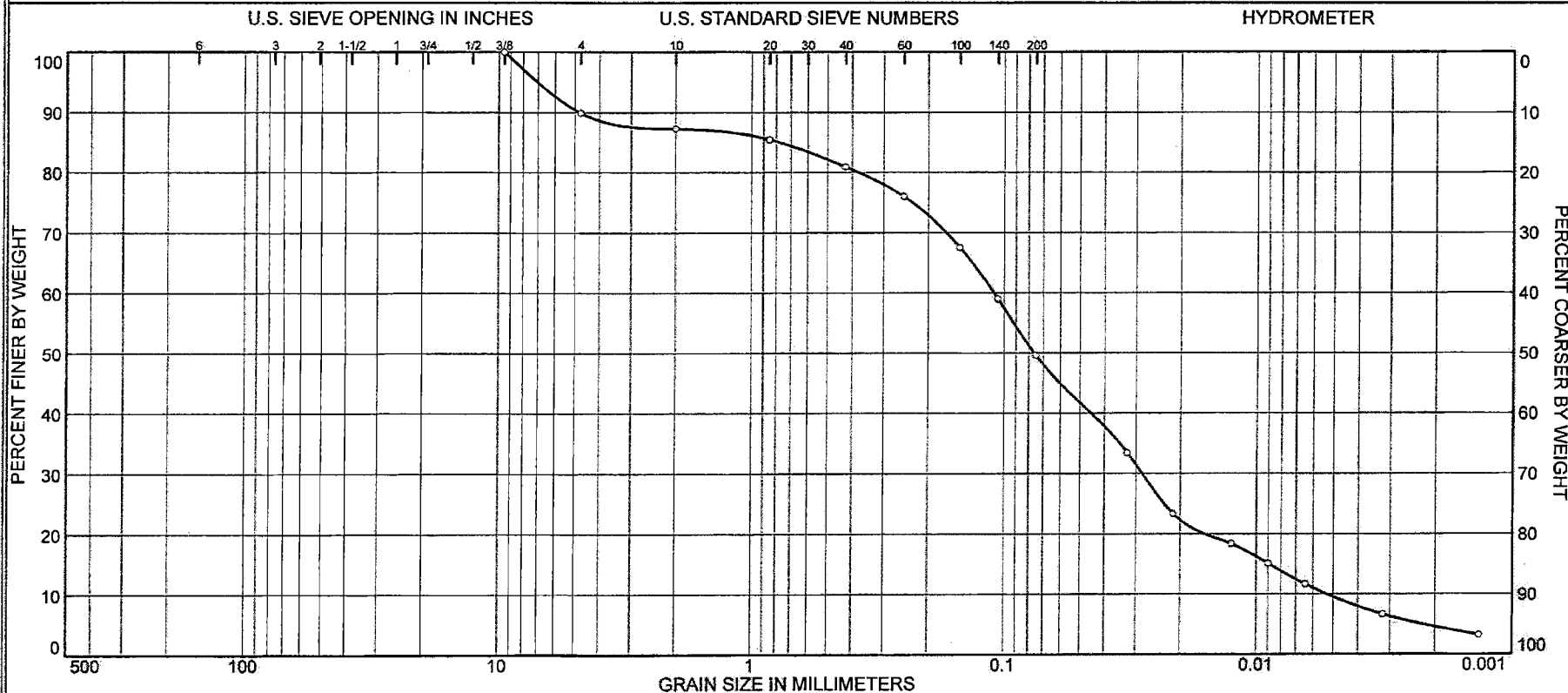
(2) USCS Symbol is based on visual-manual method.

(3) pH reported based on ASTM D 4972-01 Method A.

Prep By / Date: BAR 4/24/2007

Check By / Date: JRS. 4/24/2007

# Particle Size Distribution Report



% COBBLES	% GRAVEL		% SAND			% FINES	
	COARSE	FINE	COARSE	MEDIUM	FINE	SILT	CLAY
0	0	10	3	6	31	40	9

SOURCE	SAMPLE #	DEPTH/ELEV.	DATE SAMPLED	USCS	MATERIAL DESCRIPTION	NM %	LL	PL
B-212/B-212A	14 (13)	48.5'	12-21-06					

Client Bechtel Power Corporation Project SCE&G COL	<b>MACTEC, INC.</b>  <b>Charlotte, North Carolina</b>	Tested by: JA Reviewed by: MH <i>MAA 4-24-07</i> ASTM D422-63 (2002)
Project No. 6234-06-3534	Figure	



---

**GRAIN SIZE DISTRIBUTION TEST DATA**

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**Client:** Bechtel Power Corporation  
**Project:** SCE&G COL  
**Project Number:** 6234-06-3534

---

**Sample Data**

---

**Source:** B-212/B-212A  
**Sample No.:** 14 (13)  
**Elev. or Depth:** 48.5' **Sample Length(in./cm.):**  
**Location:** B-212/B-212A  
**Description:**  
**Date:** 12-21-06 **Natural Moisture:**  
**Liquid Limit:** **Plastic Limit:** **USCS Class.:**  
**Testing Remarks:** Tested by: JA Reviewed by: MH  
ASTM D422-63 (2002)

---

**Mechanical Analysis Data**

---

**Initial**

**Dry sample and tare=** 150.68  
**Tare =** 0.00  
**Dry sample weight =** 150.68  
**Sample split on number 10 sieve**  
**Split sample data:**  
Sample and tare = 52.05 Tare = .00 Sample weight = 52.05  
Cumulative weight retained tare= .00  
Tare for cumulative weight retained= .00

Sieve	Cumul. Wt. retained	Percent finer
.375 inch	0.00	100
# 4	15.15	90
# 10	19.08	87
# 20	1.08	86
# 40	3.82	81
# 60	6.71	76
# 100	11.82	68
# 140	16.85	59
# 200	22.50	50

---

**Hydrometer Analysis Data**

---

**Separation sieve is #10**  
**Percent -#10 based upon complete sample= 87**  
**Weight of hydrometer sample: 53.36**  
**Hygroscopic moisture correction:**  
Moist weight & tare = 51.01  
Dry weight & tare = 50.04  
Tare = 11.50  
Hygroscopic moisture= 3 %  
**Calculated biased weight= 59.62**  
**Table of composite correction values:**  
Temp, deg C: 14.0 24.0 40.2  
Comp. corr: -5.0 -3.0 0.0

**Meniscus correction only= 1**  
**Specific gravity of solids= 2.65**

---

Specific gravity correction factor= 1.000

Hydrometer type: 152H

Effective depth  $L = 16.294964 - 0.164 \times R_m$

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	Rm	Eff. depth	Diameter mm	Percent finer
2.00	23.6	23.0	19.9	0.0131	24.0	12.4	0.0325	33
5.00	23.6	17.0	13.9	0.0131	18.0	13.3	0.0213	23
15.00	23.6	14.0	10.9	0.0131	15.0	13.8	0.0125	18
30.00	23.6	12.0	8.9	0.0131	13.0	14.2	0.0090	15
60.00	23.6	10.0	6.9	0.0131	11.0	14.5	0.0064	12
250.00	23.6	7.0	3.9	0.0131	8.0	15.0	0.0032	6.6
1440.00	23.6	5.0	1.9	0.0131	6.0	15.3	0.0013	3.2

---

Fractional Components

---

Gravel/Sand based on #4

Sand/Fines based on #200

% COBBLES =                    % GRAVEL = 10    (% coarse =                    % fine = 10)

% SAND = 40    (% coarse = 3                    % medium = 6                    % fine = 31)

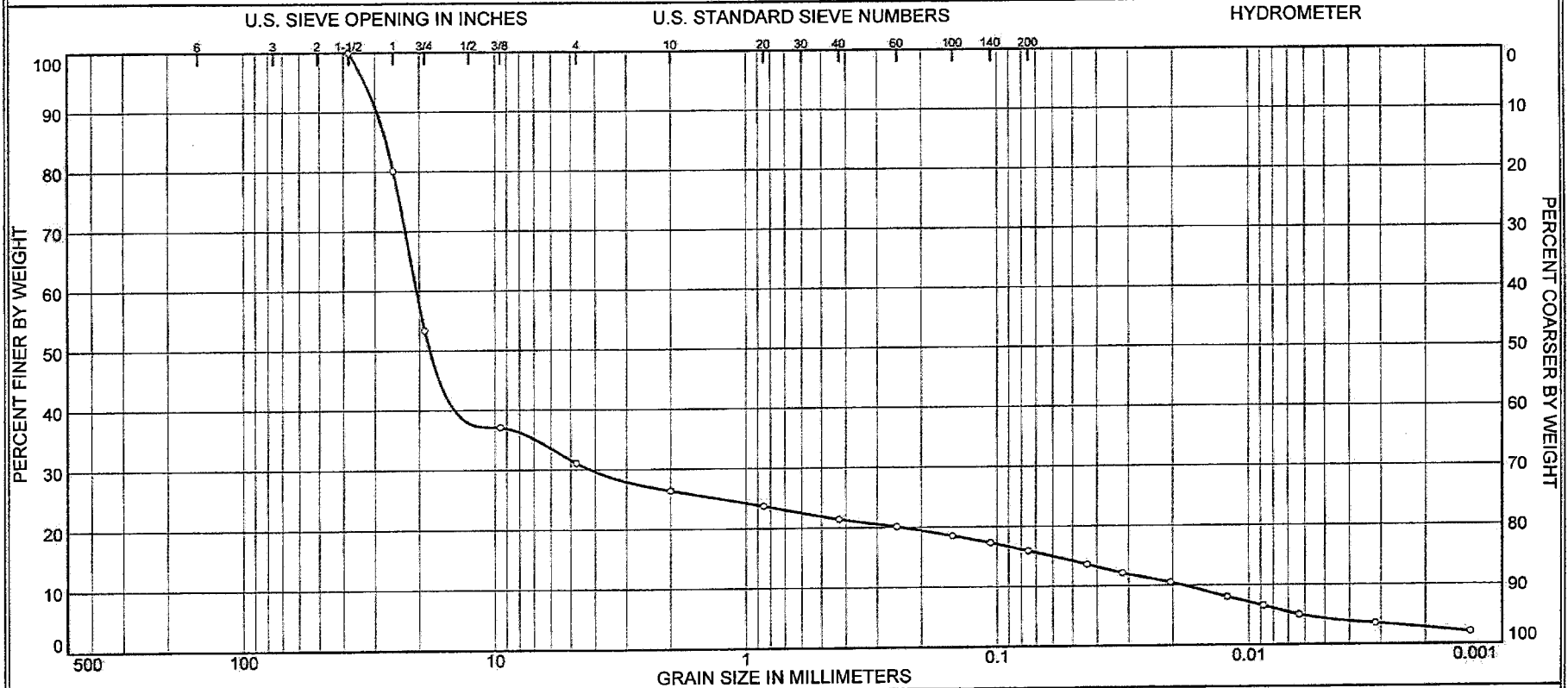
% SILT = 40                    % CLAY = 9

D<sub>85</sub>= 0.77    D<sub>60</sub>= 0.11    D<sub>50</sub>= 0.08

D<sub>30</sub>= 0.03    D<sub>15</sub>= 0.01    D<sub>10</sub>= 0.01

C<sub>c</sub>= 1.3704    C<sub>u</sub>= 20.6061

# Particle Size Distribution Report



% COBBLES	% GRAVEL		% SAND			% FINES	
	COARSE	FINE	COARSE	MEDIUM	FINE	SILT	CLAY
0	47	22	5	5	5	12	4

SOURCE	SAMPLE #	DEPTH/ELEV.	DATE SAMPLED	USCS	MATERIAL DESCRIPTION	NM %	LL	PL
B-212/B-212A	18	58.5'	1-12-07					

Client <b>Bechtel Power Corporation</b>	<b>MACTEC, INC.</b>	◯ Tested by: JA Reviewed by: MH <i>MA 4-21-07</i> ASTM D422-63 (2002) Sample mass was less than minimum required by ASTM.
Project <b>SCE&amp;G COL</b>		
Project No. <b>6234-06-3534</b>		
Figure	<b>Charlotte, North Carolina</b>	



Comp. corr: -5.0 -3.0 0.0

Meniscus correction only= 1

Specific gravity of solids= 2.65

Specific gravity correction factor= 1.000

Hydrometer type: 152H

Effective depth L= 16.294964 - 0.164 x Rm

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	Rm	Eff. depth	Diameter mm	Percent finer
1.00	23.5	30.0	26.9	0.0131	31.0	11.2	0.0438	14
2.00	23.5	27.0	23.9	0.0131	28.0	11.7	0.0316	12
5.00	23.5	24.0	20.9	0.0131	25.0	12.2	0.0204	11
15.00	23.5	19.0	15.9	0.0131	20.0	13.0	0.0122	8.0
30.00	23.5	16.0	12.9	0.0131	17.0	13.5	0.0088	6.5
60.00	23.5	13.0	9.9	0.0131	14.0	14.0	0.0063	5.0
250.00	23.5	10.0	6.9	0.0131	11.0	14.5	0.0031	3.5
1440.00	23.5	7.0	3.9	0.0131	8.0	15.0	0.0013	2.0

---

Fractional Components

---

Gravel/Sand based on #4

Sand/Fines based on #200

% COBBLES =           % GRAVEL = 69 (% coarse = 47 % fine = 22)

% SAND = 15 (% coarse = 5 % medium = 5 % fine = 6)

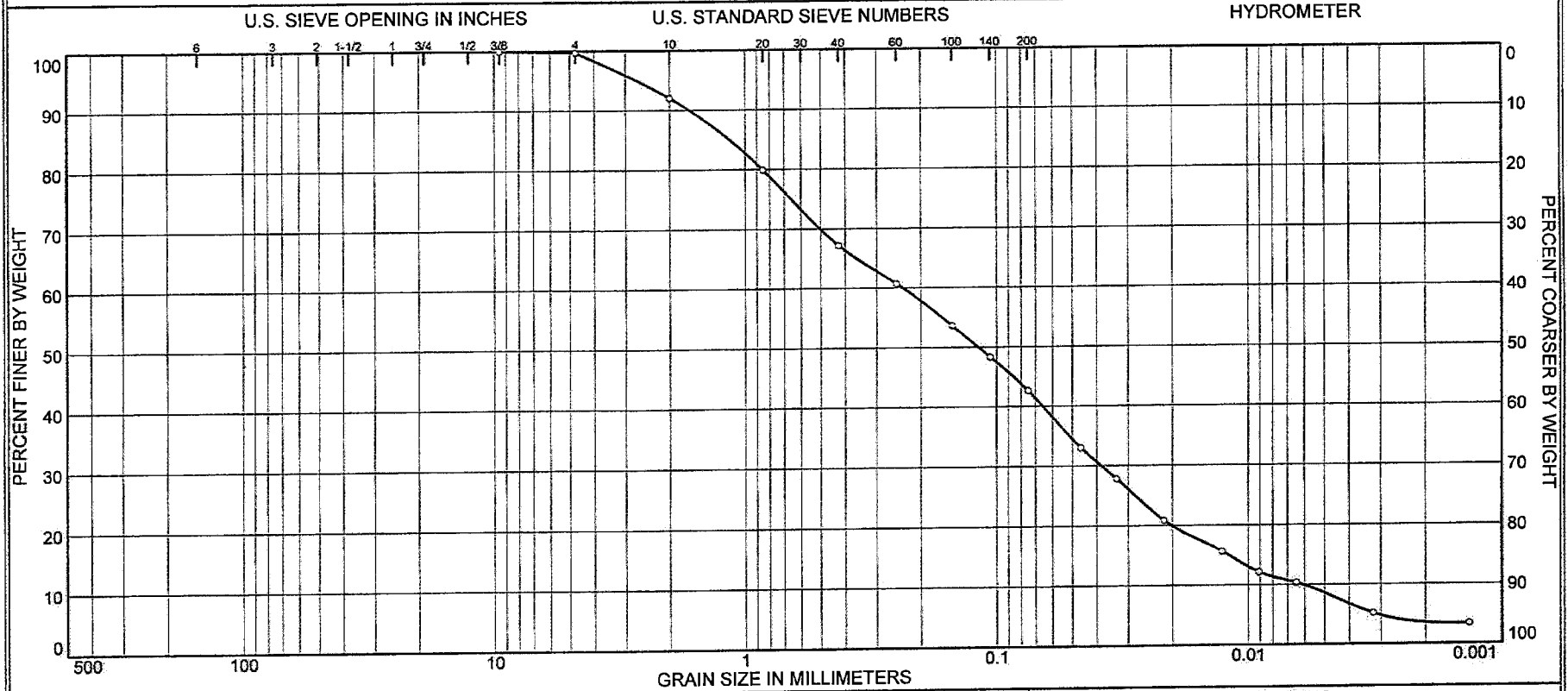
% SILT = 12 % CLAY = 4

D<sub>85</sub>= 27.13 D<sub>60</sub>= 20.54 D<sub>50</sub>= 18.19

D<sub>30</sub>= 4.19 D<sub>15</sub>= 0.06 D<sub>10</sub>= 0.02

C<sub>c</sub>= 46.8703 C<sub>u</sub>= 1127.148

# Particle Size Distribution Report



% COBBLES	% GRAVEL		% SAND			% FINES	
	COARSE	FINE	COARSE	MEDIUM	FINE	SILT	CLAY
0	0	0	8	25	24	34	9

SOURCE	SAMPLE #	DEPTH/ELEV.	DATE SAMPLED	USCS	MATERIAL DESCRIPTION	NM %	LL	PL
B-620	12 (11)	38.5'	12-21-06					

Client Bechtel Power Corporation	<b>MACTEC, INC.</b>  <b>Charlotte, North Carolina</b>	◯ Tested by: JA Reviewed by: MH <i>MMA</i> 4-24-07 ASTM D422-63 (2002)
Project SCE&G COL		
Project No. 6234-06-3534		

---

**GRAIN SIZE DISTRIBUTION TEST DATA**

---

**Client:** Bechtel Power Corporation  
**Project:** SCE&G COL  
**Project Number:** 6234-06-3534

---

**Sample Data**

---

**Source:** B-620  
**Sample No.:** 12 (11)  
**Elev. or Depth:** 38.5' **Sample Length(in./cm.):**  
**Location:** B-620  
**Description:**  
**Date:** 12-21-06 **Natural Moisture:**  
**Liquid Limit:** **Plastic Limit:** **USCS Class.:**  
**Testing Remarks:** Tested by: JA Reviewed by: MH  
ASTM D422-63 (2002)

---

**Mechanical Analysis Data**

---

**Initial**

**Dry sample and tare=** 138.05  
**Tare =** 0.00  
**Dry sample weight =** 138.05  
**Sample split on number** 10 sieve  
**Split sample data:**  
**Sample and tare =** 52.74 **Tare =** .00 **Sample weight =** 52.74  
**Cumulative weight retained tare=** .00  
**Tare for cumulative weight retained=** .00

Sieve	Cumul. Wt. retained	Percent finer
.375 inch	0.00	100
# 4	0.37	100
# 10	11.09	92
# 20	6.93	80
# 40	14.19	67
# 60	17.95	61
# 100	22.00	54
# 140	25.08	48
# 200	28.31	43

---

**Hydrometer Analysis Data**

---

**Separation sieve is** #10  
**Percent -#10 based upon complete sample=** 92  
**Weight of hydrometer sample:** 54.48  
**Hygroscopic moisture correction:**  
**Moist weight & tare =** 44.75  
**Dry weight & tare =** 43.67  
**Tare =** 10.89  
**Hygroscopic moisture=** 3 %  
**Calculated biased weight=** 57.33  
**Table of composite correction values:**  
**Temp, deg C:** 14.0 24.0 40.2  
**Comp. corr:** -5.0 -3.0 0.0

**Meniscus correction only=** 1  
**Specific gravity of solids=** 2.65

---

Specific gravity correction factor= 1.000

Hydrometer type: 152H

Effective depth L= 16.294964 - 0.164 x Rm

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	Rm	Eff. depth	Diameter mm	Percent finer
1.00	23.6	22.0	18.9	0.0131	23.0	12.5	0.0462	33
2.00	23.6	19.0	15.9	0.0131	20.0	13.0	0.0333	28
5.00	23.6	15.0	11.9	0.0131	16.0	13.7	0.0216	21
15.00	23.6	12.0	8.9	0.0131	13.0	14.2	0.0127	16
30.00	23.6	10.0	6.9	0.0131	11.0	14.5	0.0091	12
60.00	23.6	9.0	5.9	0.0131	10.0	14.7	0.0065	10
250.00	23.6	6.0	2.9	0.0131	7.0	15.1	0.0032	5.1
1440.00	23.6	5.0	1.9	0.0131	6.0	15.3	0.0013	3.3

---

Fractional Components

---

Gravel/Sand based on #4

Sand/Fines based on #200

% COBBLES =            % GRAVEL = 0    (% coarse =            % fine = 0)

% SAND = 57    (% coarse = 8    % medium = 25    % fine = 25)

% SILT = 34    % CLAY = 9

D<sub>85</sub>= 1.16    D<sub>60</sub>= 0.24    D<sub>50</sub>= 0.12

D<sub>30</sub>= 0.04    D<sub>15</sub>= 0.01    D<sub>10</sub>= 0.01

C<sub>c</sub>= 1.0179    C<sub>u</sub>= 38.5723





---

**GRAIN SIZE DISTRIBUTION TEST DATA**

---

**Client:** Bechtel Power Corporation  
**Project:** SCE&G COL  
**Project Number:** 6234-06-3534

---

**Sample Data**

---

**Source:** B-627  
**Sample No.:** 9 (10)  
**Elev. or Depth:** 23.5' **Sample Length(in./cm.):**  
**Location:** B-627  
**Description:**  
**Date:** 12-21-06 **Natural Moisture:**  
**Liquid Limit:** **Plastic Limit:** **USCS Class.:**  
**Testing Remarks:** Tested by: JA Reviewed by: MH  
ASTM D422-63 (2002)

---

**Mechanical Analysis Data**

---

**Initial**  
**Dry sample and tare=** 119.26  
**Tare =** 0.00  
**Dry sample weight =** 119.26  
**Sample split on number 10 sieve**  
**Split sample data:**  
**Sample and tare = 50.71 Tare = .00 Sample weight = 50.71**  
**Cumulative weight retained tare= .00**  
**Tare for cumulative weight retained= .00**

<b>Sieve</b>	<b>Cumul. Wt. retained</b>	<b>Percent finer</b>
# 4	0.00	100
# 10	1.37	99
# 20	1.67	96
# 40	6.43	86
# 60	12.35	75
# 100	18.69	62
# 140	22.59	55
# 200	26.38	48

---

**Hydrometer Analysis Data**

---

**Separation sieve is #10**  
**Percent -#10 based upon complete sample= 99**  
**Weight of hydrometer sample: 52.74**  
**Hygroscopic moisture correction:**  
**Moist weight & tare = 41.25**  
**Dry weight & tare = 40.07**  
**Tare = 10.65**  
**Hygroscopic moisture= 4 %**  
**Calculated biased weight= 51.27**  
**Table of composite correction values:**  
**Temp, deg C: 14.0 24.0 40.2**  
**Comp. corr: -5.0 -3.0 0.0**

**Meniscus correction only= 1**  
**Specific gravity of solids= 2.65**  
**Specific gravity correction factor= 1.000**

---

Hydrometer type: 152H

Effective depth  $L = 16.294964 - 0.164 \times R_m$

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	R <sub>m</sub>	Eff. depth	Diameter mm	Percent finer
0.50	23.6	26.0	22.9	0.0131	27.0	11.9	0.0636	45
1.00	23.6	23.0	19.9	0.0131	24.0	12.4	0.0459	39
2.00	23.6	19.0	15.9	0.0131	20.0	13.0	0.0333	31
5.00	23.6	17.0	13.9	0.0131	18.0	13.3	0.0213	27
15.00	23.6	13.0	9.9	0.0131	14.0	14.0	0.0126	19
30.00	23.6	11.5	8.4	0.0131	12.5	14.2	0.0090	16
60.00	23.6	9.0	5.9	0.0131	10.0	14.7	0.0065	12
250.00	23.6	7.0	3.9	0.0131	8.0	15.0	0.0032	7.6
1440.00	23.6	5.0	1.9	0.0131	6.0	15.3	0.0013	3.7

---

Fractional Components

---

Gravel/Sand based on #4

Sand/Fines based on #200

% COBBLES =

% GRAVEL =

% SAND = 53 (% coarse = 1 % medium = 13 % fine = 39)

% SILT = 38 % CLAY = 9

D<sub>85</sub> = 0.40 D<sub>60</sub> = 0.13 D<sub>50</sub> = 0.09

D<sub>30</sub> = 0.03 D<sub>15</sub> = 0.01 D<sub>10</sub> = 0.01

C<sub>c</sub> = 1.2606 C<sub>u</sub> = 23.8945



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**GRAIN SIZE DISTRIBUTION TEST DATA**

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**Client:** Bechtel Power Corporation  
**Project:** SCE&G COL  
**Project Number:** 6234-06-3534

---

**Sample Data**

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**Source:** B-627  
**Sample No.:** 14 (15)  
**Elev. or Depth:** 47' **Sample Length(in./cm.):**  
**Location:** B-627  
**Description:**  
**Date:** 12-26-06 **Natural Moisture:**  
**Liquid Limit:** **Plastic Limit:** **USCS Class.:**  
**Testing Remarks:** Tested by: JA Reviewed by: MH  
ASTM D422-63 (2002)

---

**Mechanical Analysis Data**

---

**Initial**

Dry sample and tare= 130.05  
Tare = 0.00  
Dry sample weight = 130.05  
Sample split on number 10 sieve  
Split sample data:  
Sample and tare = 55.04 Tare = .00 Sample weight = 55.04  
Cumulative weight retained tare= .00  
Tare for cumulative weight retained= .00

Sieve	Cumul. Wt. retained	Percent finer
.375 inch	0.00	100
# 4	3.61	97
# 10	25.43	80
# 20	6.17	71
# 40	11.90	63
# 60	17.90	54
# 100	25.35	43
# 140	30.04	37
# 200	34.44	30

---

**Hydrometer Analysis Data**

---

Separation sieve is #10  
Percent -#10 based upon complete sample= 80  
Weight of hydrometer sample: 56.47  
Hygroscopic moisture correction:  
Moist weight & tare = 61.70  
Dry weight & tare = 60.41  
Tare = 10.74  
Hygroscopic moisture= 3 %  
Calculated biased weight= 68.46  
Table of composite correction values:  
Temp, deg C: 14.0 24.0 40.2  
Comp. corr: -5.0 -3.0 0.0

Meniscus correction only= 1  
Specific gravity of solids= 2.65

---

Specific gravity correction factor= 1.000

Hydrometer type: 152H

Effective depth  $L = 16.294964 - 0.164 \times R_m$

Elapsed time, min	Temp, deg C	Actual reading	Corrected reading	K	R <sub>m</sub>	Eff. depth	Diameter mm	Percent finer
0.50	23.6	21.0	17.9	0.0131	22.0	12.7	0.0658	26
1.00	23.6	17.5	14.4	0.0131	18.5	13.3	0.0476	21
2.00	23.6	14.0	10.9	0.0131	15.0	13.8	0.0343	16
5.00	23.6	11.0	7.9	0.0131	12.0	14.3	0.0221	12
15.00	23.6	10.0	6.9	0.0131	11.0	14.5	0.0128	10
30.00	23.6	8.0	4.9	0.0131	9.0	14.8	0.0092	7.2
60.00	23.6	8.0	4.9	0.0131	9.0	14.8	0.0065	7.2
250.00	23.6	6.0	2.9	0.0131	7.0	15.1	0.0032	4.3
1440.00	23.6	5.0	1.9	0.0131	6.0	15.3	0.0013	2.8

---

Fractional Components

---

Gravel/Sand based on #4

Sand/Fines based on #200

% COBBLES =            % GRAVEL = 3    (% coarse =            % fine = 3)

% SAND = 67    (% coarse = 17    % medium = 17    % fine = 33)

% SILT = 24            % CLAY = 6

D<sub>85</sub>= 2.64    D<sub>60</sub>= 0.35    D<sub>50</sub>= 0.20

D<sub>30</sub>= 0.07    D<sub>15</sub>= 0.03    D<sub>10</sub>= 0.01

C<sub>c</sub>= 1.2695    C<sub>u</sub>= 27.4203



STL St. Louis  
13715 Rider Trail North  
Earth City, MO 63045

Tel: 314 298 8566 Fax: 314 298 8757  
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## ANALYTICAL REPORT

PROJECT NO. SCE&G COL PROJECT

VC Summer

Lot #: F6L220224

Brian Reinicker

MACTEC Engineering & Consultin  
2801 Yorkmont Rd  
Suite 100  
Charlotte, NC 28208

SEVERN TRENT LABORATORIES, INC.

  
Terry Romanko  
Project Manager

January 8, 2007

**Case Narrative**  
LOT NUMBER: F6L220224

This report contains the analytical results for the five samples received under chain of custody by STL St. Louis on December 22, 2006. These samples are associated with your VC Summer project.

The analytical results included in this report meet all applicable quality control procedure requirements.

The test results in this report meet all NELAP requirements for parameters in which accreditations are held by STL St. Louis. Any exceptions to NELAP requirements are noted in the case narrative. The case narrative is an integral part of this report.

All chemical analysis results are based upon sample as received, wet weight, unless noted otherwise. All radiochemistry results are based upon sample as dried and ground with the exception of tritium, unless requested wet weight by the client.

Observations/Nonconformances

Reference the chain of custody and condition upon receipt report for any variations on receipt conditions and temperature of samples on receipt.

There were no nonconformances or observations noted with any analysis on this lot.



**METHODS SUMMARY**

F6L220224

<u>PARAMETER</u>	<u>ANALYTICAL METHOD</u>	<u>PREPARATION METHOD</u>
Cation-Exchange Capacity	SW846 9081	SW846 9081
Percent Moisture	MCAWW 160.3 MOD	MCAWW 160.3 MOD

**References:**

- MCAWW "Methods for Chemical Analysis of Water and Wastes",  
EPA-600/4-79-020, March 1983 and subsequent revisions.
- SW846 "Test Methods for Evaluating Solid Waste, Physical/Chemical  
Methods", Third Edition, November 1986 and its updates.

**SAMPLE SUMMARY**

F6L220224

<u>WO #</u>	<u>SAMPLE#</u>	<u>CLIENT SAMPLE ID</u>	<u>SAMPLED DATE</u>	<u>SAMP TIME</u>
JL201	001	B-212/B/212A, SAMPLE 14 (13)	05/08/06	
JL21C	002	B-212/B/212A, SAMPLE 18	05/08/06	
JL21E	003	B-620, SAMPLE 12 (11)	04/07/06	
JL21J	004	B-627, SAMPLE 9 (10)	04/12/06	
JL21K	005	B-627, SAMPLE 14 (15)	04/17/06	

**NOTE(S):**

- The analytical results of the samples listed above are presented on the following pages.
- All calculations are performed before rounding to avoid round-off errors in calculated results.
- Results noted as "ND" were not detected at or above the stated limit.
- This report must not be reproduced, except in full, without the written approval of the laboratory.
- Results for the following parameters are never reported on a dry weight basis: color, corrosivity, density, flashpoint, ignitability, layers, odor, paint filter test, pH, porosity pressure, reactivity, redox potential, specific gravity, spot tests, solids, solubility, temperature, viscosity, and weight.

MACTEC Engineering and Consulting Inc

Client Sample ID: B-212/B/212A, SAMPLE 14 (13)

General Chemistry

Lot-Sample #...: F6L220224-001    Work Order #...: JL201    Matrix.....: SOLID  
Date Sampled...: 05/08/06    Date Received...: 12/22/06  
% Moisture.....: 29

PARAMETER	RESULT	RL	UNITS	METHOD	PREPARATION- ANALYSIS DATE	PREP BATCH #
Cation Exchange Capacity	22.3	5.0	meq/100g	SW846 9081	12/28-01/03/07	6362222
			Dilution Factor: 10	Analysis Time...: 00:00		
Percent Moisture	28.8	0.10	%	MCAWW 160.3 MOD	12/27-12/28/06	6361070
			Dilution Factor: 1	Analysis Time...: 00:00		

STL ST. LOUIS

MACTEC Engineering and Consulting Inc

Client Sample ID: B-212/B/212A, SAMPLE 18

General Chemistry

Lot-Sample #...: F6L220224-002    Work Order #...: JL21C    Matrix.....: SOLID  
Date Sampled...: 05/08/06    Date Received...: 12/22/06  
% Moisture.....: 28

<u>PARAMETER</u>	<u>RESULT</u>	<u>RL</u>	<u>UNITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>PREP BATCH #</u>
Cation Exchange Capacity	22.3	5.0	meq/100g	SW846 9081	12/28-01/03/07	6362222
			Dilution Factor: 10	Analysis Time...: 00:00		
Percent Moisture	28.3	0.10	%	MCAHW 160.3 MOD	12/27-12/28/06	6361070
			Dilution Factor: 1	Analysis Time...: 00:00		

STL ST. LOUIS

MACTEC Engineering and Consulting Inc

Client Sample ID: B-620, SAMPLE 12(11)

General Chemistry

Lot-Sample #....: F6L220224-003

Work Order #....: JL21E

Matrix.....: SOLID

Date Sampled....: 04/07/06

Date Received...: 12/22/06

% Moisture.....: 16

<u>PARAMETER</u>	<u>RESULT</u>	<u>RL</u>	<u>UNITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>PREP BATCH #</u>
Cation Exchange Capacity	27.2	5.0	meq/100g	SW846 9081	12/28-01/03/07	6362222
			Dilution Factor: 10	Analysis Time...: 00:00		
Percent Moisture	16.4	0.10	%	MCAWW 160.3 MOD	12/27-12/28/06	6361070
			Dilution Factor: 1	Analysis Time...: 00:00		

MACTEC Engineering and Consulting Inc

Client Sample ID: B-627,SAMPLE 9(10)

General Chemistry

Lot-Sample #...: F6L220224-004    Work Order #...: JL21J    Matrix.....: SOLID  
Date Sampled...: 04/12/06    Date Received...: 12/22/06  
% Moisture.....: 22

<u>PARAMETER</u>	<u>RESULT</u>	<u>RL</u>	<u>UNITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>PREP BATCH #</u>
Cation Exchange Capacity	40.2	5.0	meq/100g	SW846 9081	12/28-01/03/07	6362222
			Dilution Factor: 10	Analysis Time...: 00:00		
Percent Moisture	22.3	0.10	%	MCAWW 160.3 MOD	12/27-12/28/06	6361070
			Dilution Factor: 1	Analysis Time...: 00:00		

MACTEC Engineering and Consulting Inc

Client Sample ID: B-627,SAMPLE 14(15)

General Chemistry

Lot-Sample #...: F6L220224-005    Work Order #...: JL21K    Matrix.....: SOLID  
Date Sampled...: 04/17/06    Date Received...: 12/22/06  
% Moisture.....: 13

<u>PARAMETER</u>	<u>RESULT</u>	<u>RL</u>	<u>UNITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>PREP BATCH #</u>
Cation Exchange Capacity	29.4	5.0	meq/100g	SW846 9081	12/28-01/03/07	6362222
			Dilution Factor: 10	Analysis Time...: 00:00		
Percent Moisture	13.4	0.10	%	MCANW 160.3 MOD	12/27-12/28/06	6361070
			Dilution Factor: 1	Analysis Time...: 00:00		

STL ST. LOUIS

METHOD BLANK REPORT

General Chemistry

Client Lot #...: F6L220224

Matrix.....: SOLID

<u>PARAMETER</u>	<u>RESULT</u>	<u>REPORTING LIMIT</u>	<u>UNITS</u>	<u>METHOD</u>	<u>PREPARATION- ANALYSIS DATE</u>	<u>PREP BATCH #</u>
Cation Exchange Capacity	ND	0.50	meq/100g	SW846 9081	12/28-01/03/07	6362222

Work Order #: JL7XM1AA MB Lot-Sample #: F6L280000-222  
Dilution Factor: 1  
Analysis Time...: 00:00

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.









STL St. Louis

Lot #(s): F6L220224

- 5579 -

Client: MacTech COC/RFA No: N/A Date: 12-22-06  
Quote No: 71996 Initiated By: [Signature] Time: 1315

Condition Upon Receipt Form

Shipper Name: Fed Ex  
Shipping # (s):\* 1. 7982 7132 1740  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
4. \_\_\_\_\_  
5. \_\_\_\_\_

Shipping Information

Multiple Packages Y (N) N/A  
Sample Temperature (s):\*\*  
1. Ambient 6. \_\_\_\_\_  
2. \_\_\_\_\_ 7. \_\_\_\_\_  
3. \_\_\_\_\_ 8. \_\_\_\_\_  
4. \_\_\_\_\_ 9. \_\_\_\_\_  
5. \_\_\_\_\_ 10. \_\_\_\_\_

\*Numbered shipping lines correspond to Numbered Sample Temp lines

\*\*Sample must be received at 4°C ± 2°C- If not, note contents below. Temperature variance does NOT affect the following: Metals-Liquid or Rad tests- Liquid or Solids

Condition (circle "Y" for yes, "N" for no and "N/A" for not applicable):

1.	Y (N)	Was sample received broken?	8.	Y (N)	Sample received with Chain of Custody?
2.	Y N (N/A)	Was sample received with proper pH? (If not, make note below) If N/A-Was pH taken by original STL Lab?	9.	Y (N)	Chain of Custody matches sample ID's on container(s)?
3.	Y N	Sample received in proper containers?	10.	Y (N)	Are there custody seals present on cooler?
4.	Y (N)	Sample volume sufficient for analysis?	11.	Y N (N/A)	Do custody seals on cooler appear to be tampered with?
5.	Y (N)	Headspace in VOA or TOX liquid samples? (If Yes, note sample ID's below)	12.	Y (N)	Are there custody seals present on bottles?
6.	Y N (N/A)	Were contents of the cooler frisked after opening?	13.	Y N (N/A)	Do custody seals on bottles appear to be tampered with?
7.	Y (N)		14.	Y N	Was Internal COC/Workshare received?

<sup>1</sup> For DOE-AL (Pantex, LANL, Sandia) sites, pH of ALL containers received must be verified, EXCEPT VOA, TOX and soils.

Notes: All jars are 20% full

Corrective Action:

- Client Contact Name: \_\_\_\_\_
- Sample(s) processed "as is"
- Sample(s) on hold until: \_\_\_\_\_

Informed by: \_\_\_\_\_

If released, notify: \_\_\_\_\_ Date: 12/22/06

Project Management Review:

THIS FORM MUST BE COMPLETED AT THE TIME THE ITEMS ARE BEING CHECKED IN. IF ANY ITEM IS COMPLETED BY SOMEONE OTHER THAN THE INITIATOR, THEN THAT PERSON IS REQUIRED TO APPLY THEIR INITIAL AND THE DATE NEXT TO THAT ITEM.

ADMIN-0004, REVISED 03/01/06\slr\01\QA\FORMS\ST-LOUIS\ADMIN\Admin004030106.doc