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K BASIN SLUDGE/RESIN BEAD SEPARATION TEST REPORT, REV. 0

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Numatec Hanford Corporation, Richland, WA 99352

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Abstract: Experiments with simulants have demonstrated that it is possible to extract a nominal 95% resin bead mass in an elutriation column with a nominal 3.8 cm/s water flow rate. The non-bead sludge component entrained with the separated beads range from 0.15 to 1.4 percent of the total sludge dry mass. The percentage of solids entrained with the beads depends on the type of screen (125 or 300 microns) used before the elutriation column.

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**K BASIN SLUDGE/RESIN BEAD SEPARATION
TEST REPORT**

August 1998

**Don Squier
Numatec Hanford Corporation
Richland, Washington**

**Issued by
NUMATEC HANFORD CORPORATION
for the**

**U.S. DEPARTMENT OF ENERGY
RICHLAND OPERATIONS OFFICE
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K Basin Sludge/Resin Bead Separation Test Report, HNF-3132, Rev. 0

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1.0 INTRODUCTION

The K Basin sludge is an accumulation of fuel element corrosion products, organic and inorganic ion exchange materials, canister gasket materials, iron and aluminum corrosion products, sand, dirt and minor amounts of other organic material. The sludge will be collected and treated for storage and eventual disposal. This process will remove the large solid materials by a 1/4 inch screen. The screened material will be subjected to nitric acid in a chemical treatment process. The organic ion exchange resin beads produce undesirable chemical reactions with the nitric acid. The resin beads must be removed from the bulk material and treated by another process. An effective bead separation method must extract 95% of the resin bead mass without entraining more than 5% of the other sludge component mass.

The test plan HNF-2729, "Organic Ion Exchange Resin Separation Methods Evaluation," proposed the evaluation of air lift, hydro cyclone, agitated slurry and elutriation resin bead separation methods. This follows the testing strategy outlined in section 4.1 of HNF-2574, "Testing Strategy to Support the Development of K Basins Sludge Treatment Process". Engineering study HNF-3128, "Separation of Organic Ion Exchange Resins from Sludge", Rev. 0, focused the evaluation tests on a method that removed the fine sludge particles by a sieve and then extracted the beads by means of a elutriation column.

Ninety-nine percent of the resin beads are larger than 125 microns and 98.5 percent are 300 microns and larger. Particles smaller than 125 microns make up the largest portion of sludge in the K Basins. Eliminating a large part of the sludge's non-bead component will reduce the quantity that is lifted with the resin beads in the elutriation column. Resin bead particle size distribution measurements are given in Appendix A

The Engineering Testing Laboratory conducted measurements of a elutriation column's ability to extract resin beads from a sieved, non-radioactive sludge simulant. A elutriation column uses a constant velocity upward flow stream to segregate materials. In simplistic terms, the dense particles fall to the column's bottom while the flow lifts less dense particles to the column's top. A particle can be streamlined or have a high drag profile; this factor also influences the lift or fall of a particle exposed to the column flow. The sludge components that lift or fall are determined by the fluid velocity.

The column flow velocity needed to lift the bulk of the resin beads will also lift other, non-bead, sludge components. Resin bead treatment and disposal are complicated by large quantities of non-bead material. Tests are necessary to determine a column flow velocity that will collect the bulk of the resin beads and the amount of non-bead sludge components that are also collected. Measurements will compare the effect of elutriation column runs using minimum particle sizes of 125 microns and 300 microns on the mass percent of non-bead material lifted with the beads.

2.0 SIMULANT

2.1 Sludge Simulant

The sludge simulant composition is based on a description provided in Numatec Hanford Corporation internal memo, "Sludge Simulant for Organic Ion Exchange Resin Separation," 8C720-98-JBD:001 (see Appendix B). The simulant components are aluminum oxide, iron oxide, copper (simulates uranium oxide), mixed bed resin beads and blow sand obtained beside the road north of the Hanford site's 300 Area. The resin beads and copper particles were the only simulant components that were used in the supplied particle sizes. The remaining components were sieved into eight particle sizes to emulate the particle size/mass percentages detailed in the memo. These components required ball milling to obtain enough of the fine particles.

2.2 Organic Resin Beads

Purolite, NRW-37, nuclear grade mixed bed resin beads were used in this test series. The resin beads were stored under water to maintain their swollen wet diameters. This condition mimics resin beads in a sludge quantity freshly retrieved from the K Basins. The wet condition poses some problems and special procedures for mass measurements. The bulk of the water is removed by exposure to a vacuum filtration system for 30 seconds. The beads are then placed on a tared weighing pan and subjected to an evacuated bell jar for five minutes. The bead mass drops continuously while resting on a balance; apparently the water readily evaporates from the beads. For this reason, the beads are weighed immediately after removal from the bell jar to maintain a consistent measurement method. This procedure is used for all resin bead mass measurements.

2.3 Additional Simulant Components

The sludge simulant is based on samples that had passed through a 700 micron sieve. The sieve removes 10 to 20% of the original mass in the form of large particles. The sludge retrieval process may collect particles larger than 700 microns. These large particles may be lifted in the column and collect with the resin beads. Large sand particles are added to a simulant batch to test this possibility. Sand is a lighter sludge component and represents a worst case condition.

Zeolite is also present in the K basin sludge, but is not included in the simulant. Zeolon 900 was added to a sludge batch to measure the effects of zeolite.

ZEOLON is a registered trademark of Norton Company

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2.4 Simulant Batches

The simulant batches contained 120 grams or 134 grams of dry components. A unique number is assigned to each simulant batch. The column runs are identified by an S, followed by the simulant batch number used for the run. Appendix C contains additional information on the sludge simulant components and a table listing the particle sizes, masses and components of each batch used in this test series.

3.0 SLUDGE SIEVING

The dry sludge components were mixed with an arbitrary amount of water and then placed onto a 125 or 300 micron sieve. The fine simulant particles were washed through the sieve by a stream of water and collected in a tared beaker. The water was driven from the fines in a 110° C oven so the residue mass could be measured in the beaker. The simulant not passing through the sieve is run through the elutriation column.

The theoretical amount of material passing through the screen can be calculated by summing the component masses with particle sizes smaller than the sieve size. Table 1 compares the dried through sieve material mass with the calculated mass. The calculated and measured masses are within a fractional percent in every batch.

Table 1. Calculated/Sieved Sludge Mass Values							
	Total grams, particles less than 125 microns				Total grams, particles less than 300 microns		
	Sludge Simulant Batch						
	1	2	3	9	4	10	11
Calculated Sludge Mass Passing Through Screen	96.002	95.994	96.009	96.005	99.732	99.732	99.729
Grams of screened dried sludge simulant	96.4	96.78	96.77	95.15	100.18	99.4	100.6
% of Calculated Material Actually Recovered	100.4	100.8	100.8	99.1	100.4	99.7	100.9

4.0 ELUTRIATION COLUMN

4.1 Elutriation Column Description

Figure 1 is an image of the elutriation column test apparatus. The column is constructed of 34.67 mm inside diameter acrylic tubing with a length of 189.9 cm from the inlet at the bottom collection chamber to the column outlet's lowest elevation. The column outlet directs water and sludge particles to the top collection chamber. A standpipe in this chamber permits water to overflow into the reservoir below while collecting the sludge particles lifted by the column's upward flow.

Water is drawn from the reservoir by a variable flow pump. The water is pumped through a rotameter before it is discharged into the bottom collection chamber for recirculation through the column.

Sludge particles not lifted by the flow of the column will fall into the bottom collection chamber. The relatively large diameter of this chamber reduces the velocity of the flowing water so the particles will stay in the chamber rather than rise into the column.

4.2 Elutriation Column Operation

A run begins without water in the column. A hose conducts filtered de-ionized water from the ETL K-Basin mockup pool into the upper end of the column. The water is run until it flows from the outlet, fills the top collection chamber and half of the reservoir. Air is expelled from system by running the pump at an elevated flow rate. The flow is then adjusted to the rate required for the run.

Sludge is introduced into the column through a funnel elevated above the column's outlet. Tubing connects the funnel to an elbow fitting that penetrates the column 82.9 cm above the bottom collection chamber. A quantity of water following the sludge forces the mixture into the flow stream of the column.

The magnetically coupled gear pump must slip under the load for it is necessary to monitor and continuously adjust the flow rate throughout the run. Particles continue to circulate in the column's mid- elevation when no additional particles are observed

Figure 1. Elutriation Column

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passing through the column outlet. The run is ended under these conditions by shutting off the pump. The circulating particles then fall into the bottom collection chamber.

The column is drained by removing the tubing from the rotameter outlet and directing the tubing end to a collection vessel resting on the floor. This drains the water down to the bottom collection chamber's top. The bottom collection chamber may then be removed by loosening the four C-clamps that fasten it to the column flange.

4.3 Flow Rate Calibration

A volume flow rate calibration was performed on the rotameter after completing the elutriation column activities. This approach permitted the calibration data to be focused on the flow indication range used in the test series. Appendix D contains a description of the volume flow rate calibration, the calibration data, and conversion of the volume flow rate into column flow velocity.

4.4 Resin Bead Yield per Column Flow Rate

The elutriation column was fitted with an uncalibrated rotameter (flow rate instrument) that indicated 0 to 100%. The initial column runs were made to determine a flow rate indication that would lift 95 mass percent of the resin beads to the top collection chamber. A column flow rate was established, then a mass of resin beads were introduced into the column to separate into the top or bottom collection chambers. The beads in each collection chamber were dried and weighted so the mass percent lifted could be calculated using the sum of both masses as the 100% value. This process was repeated to obtain flow indications above and below the 95% bead yield value. The data values are contained in Appendix E.

Figure 2 is a plot of the elutriation column bead yield at various flow rates. The equation for the fitted line has a high correlation coefficient with the data points. Lines on the figure show the three flow rates used in the elutriation column sludge simulant tests and expected bead yields. The middle flow rate was selected because it is near the 95% bead yield and corresponds to the rotameter's 50% flow graduation. The maximum and minimum flow rates correspond to the next graduations above and below the 50% graduation.

Figure 2. Elutriation Column Resin Bead Yield

5.0 POST RUN OPERATIONS AND MEASUREMENTS

The beads must be separated from the non-bead material in both the top and the bottom collection chambers so the four quantities of material can be dried and weighted. The material is rinsed from a collection chamber and into a beaker. The reservoir also collects some material lifted by the column so these contents must be added to the contents of the top collection chamber.

Some initial bead separation experiments were made using a 70% by mass sugar solution to float the resin beads. This was an effective resin bead separation method, but it required a lot of time for the beads to rise in the viscous solution. The beads would float in two layers with a space of clear liquid between the layers. Time and resources were not expended on investigating the reason for the two layers. It is speculated that the slight density difference between the anion and cation resin beads would account for the layering.

A sluice box was very effective in removing resin beads from the bulk material separated by the column. The bead's spherical shape rolls off the sluice's lower end while the non-bead material is trapped in the sluice's grooved mat. Zeolon 900 tended to run down the sluice much faster than other sludge components, but just slightly slower than the resin beads.

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The sluice is constructed from a grooved plastic mat. The triangular shaped grooves are 3.175 mm apart and 1.52 mm deep. The grooved mat did not have the ideal profile for separating beads. The groove's closely spaced peaks and valleys tended to trap some of the beads. Rounded groove peaks and valleys with wider spacings would promote a spherical bead's ability to roll out of the grooves. The mat is laid in a length of ten inch channel iron so a 0.9 meter long trough is formed with perpendicular grooves. One end of the channel iron is elevated so the sluice has a 6.5° slope. A bead catch pan is placed at the sluice's lower end.

The sludge mixture is placed on the elevated end of the sluice. A gentle stream of water washes the beads off the sluice and into the catch pan. The washing continues until the non-bead components approach the lower end of the sluice. The beads are then removed from the catch pan by turkey baster suction and transferred into a specimen jar. The material on the upper end of the sluice does not contain beads and is washed into another, non-bead, specimen jar. The material remaining on the mat is consolidated and washed down the sluice again. This process is repeated until no additional beads are separated.

The beads are dried and weighed as previously described. The bulk of the water is removed from the non-bead material by a vacuum filter system operated for thirty seconds. The material is then transferred to a tared weighing pan and exposed to a 110° C oven for ten minutes. The non-bead material is then weighed.

6.0 RESIN BEAD SEPARATION RESULTS

Table 2 summarizes the seven sludge simulant run parameters. Runs with a minimum particle size of 125 microns (S1, S2, S3 and S9) used column velocities of 0.037, 0.038 and 0.039 meters per second that were expected to respectively yield 94%, 96.6% and 97.8% resin beads. Runs with a minimum particle size of 300 microns used sludge composition variations and were only run at a column velocity expected to yield 96.6% of the resin beads.

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Table 2. Run Parameters

Run ID	Simulant Batch Number	Minimum Particle Size - Micron	Column Velocity m/s	Other Parameters
S1	1	125	0.037	
S2	2	125	0.038	
S3	3	125	0.039	
S4	4	300	0.038	
S9	9	125	0.038	
S10	10	300	0.038	Add sand as follows: 7 grams 840 to 590 micron and 7 grams 590 to 420 micron to standard 120 gram stimulant batch
S11	11	300	0.038	Add Zeolon 900 as follows: 7 grams 840 to 297 micron, 7 grams 300 to 420 micron to standard 120 gram stimulant batch

The post-run simulant mass measurement values are presented in Table 3 below.

Table 3. Post Run Mass Measurements

Table 3. Post Run Mass Measurements						
Run	Column Velocity m/s	Notes	Column Collection Position	Beads		Non-bead components grams
				Vacuum filtered dry grams	Vacuum dry grams	
Greater than 125 micron particles						
S1	0.037		Top	12.78	12.016	1.68
			Bottom	0.918	0.774	6.57
S2	0.038		Top	13.955	12.898	1.79
			Bottom	1.436	1.195	6.64
S3	0.039		Top	13.78	12.88	1.77
			Bottom	0.57	0.44	6.65
S9	0.038	Re-Run S2	Top	11.03	10.27	1.878
			Bottom	2.16	1.74	7.388
Greater than 300 micron particles						
S4	0.038		Top	13.61	12.75	0.18
			Bottom	0.74	0.6	5.29
S10	0.038	Add 7 grams 840 to 590 micron sand and 7 grams 590 to 420 micron sand to standard 120 gram stimulant batch	Top	11.215	10.391	0.365
			Bottom	1.77	1.488	18.301
S11	0.038	Add Zeolon 900 as follows: 7 grams 840 to 297 mesh, 7 grams 300 to 420 micron to standard 120 gram stimulant batch	Top	12.147	11.293	9
			Bottom	0.43	0.28	13.572

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Figure 3 is a plot of the bead yield at various column flow velocities. The figure shows a line fitted to the initial runs using only the resin beads in the column. The subsequent sludge simulant runs follow the expected bead yield line in four out of seven runs. Malfunctions with the column flow rate indication were noted for the lowest bead yield runs, S9 and S10.

Figure 3. Resin Bead Yield from Simulant

The variable speed pump requires constant adjustment throughout each run to maintain the desired flow. The runs were plagued by problems with the rotameter flow rate indication. Lint would collect on the instrument's float midway into a run and produce higher than actual flow rate indications. The pump speed would then be reduced to maintain the desired flow rate indication. This effect reduces the resin bead yield. The float was also observed to stick when small particles would wedge between the float and sight glass. This condition produces an unknowable effect on the flow rate and bead yield. These limitations produced bead mass percent data that generally are below the amounts predicted by the beads only runs.

Table 4 summarizes a column's ability to remove resin beads from sludge, the amount of non-bead material entrained with the beads and the effect of sieving to control the minimum particle sizes.

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Table 4. Elutriation Column Effectiveness					
Run	Notes	Column Velocity m/s	% Bead Mass Lifted in Column	Mass % of non-bead components lifted in column	
				100%=mass loaded into column	100%=pre- sieved dry simulant mass
Particles Greater than 125 Microns					
S1		0.037	93.9	12.3	1.4
S2		0.038	91.5	12.2	1.5
S3		0.039	96.7	12.1	1.5
S9	Re-Run S2	0.038	85.5	15.5	1.6
Particles Greater than 300 Microns					
S4		0.038	95.5	1.4	0.15
S10	Add sand as follows: 7 grams 840 to 590 micron and 7 grams 590 to 420 micron to standard ~120 gram stimulant batch	0.038	87.5	3.4	0.3
S11	Add Zeolon 900 as follows: 7 grams 840 to 297 micron, 7 grams 300 to 420 micron to standard 120 gram stimulant batch	0.038	97.6	44.4	6.7

The amount of lifted non-bead material can be expressed as a percentage of the total simulant mass prior to sieving. This makes it possible to make useful comparisons between runs of different minimum particle sizes. The 125 micron minimum particle size column runs lifted consistent percentages of non-bead components in spite of the poor flow rate control. Eliminating the particles sized between 125 and 300 microns reduced this percentage by a nominal factor of ten as shown by the results of run S4 that used the same simulant composition and a mid-range flow rate. Fourteen grams of sand particles larger than 420 microns added to the sludge simulant in run S10 only increased the non-bead components lifted by the column a fractional percent relative to run S4. The 14 grams of Zeolon 900 added to batch 11 resulted in the greatest percentage (6.7%) of non-bead components lifted by the column in this test series.

Post-test operations require the removal of material collected in the top and bottom chambers of the column. The beads are then separated from the bulk material gathered from each chamber. These four material quantities are then subjected to drying and weighing operations. The operations transfer the material from one container to another leaving some material in the previous container. The result is that the measured mass is less than the actual quantity not exposed to the handling operations. Table 5 expresses the post-run recovered mass as a percentage of the initial simulant batch mass prior to sieving.

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Table 5. Total Simulant Mass Balance

Run	Component	Batch Mass Grams	Through Sieve Mass	Mass - Column Top	Mass - Column Bottom	Mass% Recovered
Particles Greater than 125 micron						
S1	Resins	14.4		12.016	0.774	88.8
	Sludge	105.612	96.4	1.68	6.57	99.1
S2	Resins	14.4		12.898	1.195	97.9
	Sludge	105.599	96.78	1.79	6.64	99.6
S3	Resins	14.4		12.88	0.44	92.5
	Sludge	105.614	96.77	1.77	6.65	99.6
S9	Resins	14.4		10.27	1.74	83.4
	Sludge	105.61	95.15	1.878	7.388	98.9
Particles Greater than 300 micron						
S4	Resins	14.402		12.75	0.6	92.7
	Sludge	105.613	100.18	0.18	5.29	100.0
S10	Resins	14.4		10.391	1.488	82.5
	Sludge	105.614	99.4	0.365	18.301	98.7
	Large sand	14				
S11	Resins	14.402		11.293	0.28	80.4
	Sludge	105.61	100.6	9	13.572	103.0
	Zeolon 900	14				

The mass percent of non-bead components recovered is generally higher than the percentage of bead mass recovered. This is attributed to the fact that the bead sluice separation method is not 100% effective and part of the bead mass is contained in the non-bead components. This is especially true of the S11 run, containing Zeolon 900, which did not separate very well from the beads in the column or the sluice.

7.0 CONCLUSIONS/OBSERVATIONS

The basic sludge simulant recipe experiments have demonstrated that it is possible to extract a nominal 95% resin bead mass in a elutriation column with a nominal 0.038 meter per second water flow rate. The non-bead sludge component entrained with the separated beads ranges from 0.15 to 1.4 percent of the total sludge dry mass.

The elutriation column will also lift non-bead sludge components to the top collection chamber. These components are mostly fine particles as evidenced by a factor of ten reduction in the non-bead component when the minimum particle size is increased from 125 microns to 300 microns.

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Sand particles larger than 420 microns only increase the column's lifted non-bead mass by a fractional percent. Large particles denser than sand will not rise and become entrained with the separated beads.

Zeolon 900 added to the sludge simulant produced the greatest mass percentage (6.7%) of non-bead material lifted by the column in the test series. As with the large sand particles, a mass of Zeolon equal to the resin bead mass was added to the sludge simulant. The results of the only test suggest that 63% of the Zeolon mass will be lifted with the beads to the top of the elutriation column.

Appendix A

Resin Bead Particle Size Distributions

NRW-100

wet

Descriptive Statistics

	Mean	Std. Dev.	Std. Error	Count	Minimum	Maximum
Equiv.Diam.(μm)	628.608	200.548	17.390	133	193.710	1192.900

NRW-100

dry

Descriptive Statistics

	Mean	Std. Dev.	Std. Error	Count	Minimum	Maximum
Equiv.Diam.(μm)	661.952	107.980	11.644	86	343.290	996.610

NRW-400

wet

1000 μm

Descriptive Statistics

	Mean	Std. Dev.	Std. Error	Count	Minimum	Maximum
Equiv.Diam.(μm)	788.052	190.875	18.036	112	41.940	1173.320

NRW-400

dry

Descriptive Statistics

	Mean	Std. Dev.	Std. Error	Count	Minimum	Maximum
Equiv.Diam.(μm)	505.559	125.430	8.574	214	238.480	812.890

Appendix B

**Sludge Simulant for Organic Ion Exchange Resin Separation
8C720-98-JBD:001**

K Basin Sludge/Resin Bead Separation Test Report, HNF-3132, Rev. 0

KSUDGE001.JED

TO: Thierry A. Flament
FROM: Jim Duncan
SUBJECT: Sludge Simulant for Organic Ion Exchange Resin Separation
DATE: 23 April 1998
CC: Mike Schliebe

1. Purpose

The purpose of this memorandum is to put forth a selection basis for the K East Basin floor and Weasel Pit simulant for the organic ion exchange resin (OIER) separation unit operation.

2. References:

Analysis of Sludge from Hanford K East Basin Floor and Weasel Pit, WHC-SP-1182, April 1996.
K. Willeke and K.T. Whitby, J. Air Pol Control Assoc. 25:529(1975)
Kirk-Othmer, Encyclopedia of Chemical Technology, Vol. 1 (4th Ed.), 1991
CRC Handbook of Chemistry and Physics, 53rd Ed., 1977-1978
Purolite Technical Data for NRW-37 Nuclear Grade Resin

3. K East Sludge and Weasel Pit

Using Table 4.3 Sludge Characterization Data -- Per Gram As-Settled Sludge (WHC-SP-1182), the following mean values were extracted:

Table 1^A

Composition	
Inorganic Elements	13.4
Water	56.8 ^B
Uranium	2.5 ^C
Acid Insoluble Residue	8.25
Total	80.95
Major Inorganic Elements (> 1%)	Fe 10.8 Al 1.8

^A The values are "as settled sludge", the expected water content of the retrieved sludge is expected to be at a much higher concentration.

^B Averaged values from 222S Lab and PNNL.

^C ICP for total U, 222S Lab

The total analysis does not sum to 100%. This is due to some elements not being analyzed, such as Si. Silicon did appear in compounds detected by XRD analysis. For example, some of the compounds reported

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were: SiO_2 , $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, Si , $\text{CaAl}_2\text{Si}_2\text{O}_8 \cdot 4\text{H}_2\text{O}$. Also, the analysis ignored the mass contribution of bound water (water of hydration).

In Appendix I (WHC-SP-1182), the particle size analysis indicated that the samples were measured "as received", after sonication at 25W (120 sec.), and after sonication at 40W (300 sec.). The data taken into consideration for this report will be the "as received" data for simulant formulation. The use of "as received" data is due to the fact that the sludge retrieval mechanism is not yet known. Appendix A of this memorandum is an analysis of the graphical data presented in Appendix I of WHC-SP-1182, for the "as received" and sonicated samples.

4. Assumptions

1. The separations will be based on physical parameters of the floor and weasel pit sludge.
2. The analyses as reported in WHC-SP-1182 are correct.
3. The fully loaded resin beads have a negligible increase in density (phone conversation with a Purolite Tech Representative, (800) 343-1500).
4. The mean mass balance difference of 19.05% (100% minus 80.95% from Table 1) is due to the mass contribution of compounds that were not considered for analyses (silicon for example), bound water, and ion exchange resin.
5. The resin is Purolite NRW-37 Nuclear Grade, mixed bed. The density of the hydrogen form (cation exchange resin) is 1.24. The density of the Cl form (anion exchange resin) is 1.10. The mesh size is 16 to 40, US standard screen (1.19 to 0.42 mm, respectively).
6. Blow sand in the range of 2.5 to 10 microns (See Appendix B).
7. The only radionuclide of mass consequence is Uranium.
8. The simulant will be derived from the mean values as reported in Table 4.2 (WHC-SP-1182). Adjustments will be made to mimic various conditions.

5. Selection of the Simulant for OIER separation

Table 2 Major Analytes and Simulants

Major Analytes and Simulants			
Uranium (UO_2)	Copper or Chromium	2.5	Cu = 8.9 Cr = 7.2 (10.96)
Iron (Fe_2O_3)	Iron Oxide	10.8	5.24 (5.24)
Aluminum (Al_2O_3)	Aluminum oxide	1.8	3.97 (3.97)
Water	Water	56.8	1.00 (1.00)
Total		71.9	
Silicon (SiO_2)	Blow Sand from K Area, quartz	Sand and the combination of ion exchange resin will make up the balance of 28.1 per cent as delineated in the OIER test plan.	2.64 (2.64)
Organic Ion Exchange Resin	Purolite, NRW-37, mixed bed (Volume Ratio 1.5 anion to 1.0 cation)		Cation Resin = 1.24 Anion Resin = 1.10

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6. Laboratory Protocol for Simulant Make-up

Materials:

- Resin: Resin beads will be obtained from either the vendor or from K Basin operations.
- Aluminum oxide, chromium (or copper) will be obtained from a vendor in varying particle dimensions.
- Iron oxide will be obtained by physically scraping "rusted material" found in storage yards around Bldg 305, 306E, etc.
- Blow sand will be obtained by retrieving the top 2 to 3 mm of sand around the K East basin.
- Water will be de-ionized, conductivity and pH will approximate that of K East basin.

Preparation:

- Grinding:

Individually, the iron oxide, aluminum oxide, and the blow sand will be subjected to a grinding phase such as a ball mill or similar apparatus.

All material will individually be classified using a Rotap, with screen sizes from 25 US Standard mesh (710 μm) to 400 US Standard mesh (37 μm). Material passing the 400 mesh will be collected as 400 minus and subjected to particle analysis.

- Simulant Make-up

Example:

The resin and sand are dependent variables and will vary according to the test protocol. However, the concentrations for iron oxide, aluminum oxide, water, and chromium will be fixed (assumption — normally and independently distributed, (0,1)).

For a total of 1000 g of simulant, and using the particle volume distribution for M13B (Appendix A, Table A-2, as received), particle diameters from 31 μm to 352 μm (mesh size of 400 to 48 respectively). The following table can be derived. The weight of simulant should sum to the gram fraction of 1000 g as indicated. The difference between the independent variables and the total grams required for that particle range will be made from the dependent variables (resin and sand).

Table 3 Example of Simulant Make-up (1000 g)

Mesh Size	24-35	35-42	48 - 60	60 - 80	120 - 80	170 - 120	230 - 170	325 - 230	400 - 325	< 400
Particle Diameter (µm)	704-497	497-352	352-248	248-176	176-125	125-88	88-62	62-44	44-37	< 37
Per Cent Particles in Range	8.94 (89.4g)	5.17 (51.7g)	2.2 (22 g)	2.98 (29.8 g)	2.8 (28g)	3.15 (31.5g)	4.38 (43.8 g)	6.13 (61.3g)	8.23 (82.3g)	56.04 (560 g)
Al ₂ O ₃ @ 1.8 % (g)	1.61	0.93	0.39	0.54	0.50	0.57	0.79	1.1	1.48	10.1
Fe ₂ O ₃ @ 10.8% (g)	9.66	5.58	2.37	3.22	3.02	3.40	4.73	6.62	8.89	60.52
Cr @ 2.5% (g)			0.55	0.83	0.98	1.38	1.9	2.4	2.5	14.6
Water @ 56.8% (g)			7.4	18.7	22.2	31.2	44.9	55.1	57.4	331.1
Total			9.4	23.7	28	39.5	55.3	69.7	72.6	419.2
Difference to be made up with Resin and Blow Sand (g)			3.6	9.3	11	15.5	23.7	27.3	28.4	163.8

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Analyses of Particle Number Distribution and Particle Volume Distribution Graphs
from Appendix I, WHC-SP-1182

Table A-1 Particle Number Distribution (WHC-SP-1182, Appendix I)

	M13T			M13B			T20T			T20B(Duplicate)		
Particle Dia. (µm)	Per Cent in Range			Per Cent in Range			Per Cent in Range			Per Cent in Range		
	40 W	25 W	As Received	40 W	25 W	As Received	40 W	25 W	As Received	40 W	25 W	As Received
0.172 to 0.243	35	--	--	--	--	--	60	64	--	48	37	--
0.243 to 0.344	38	60	--	60	57	--	25	23	--	34	41	33
0.344 to 0.486	13	21	--	23	23	--	8	6	--	11	14	42
0.486 to 0.688	7	9	47	9	10	51	4	2	55	5	6	14
0.688 to 0.972	3	5	27	6	8	25			23			7
0.972 to 1.375	1	3	13	1		12			7			3
1.375 to 1.946			7			5			4			1
1.946 to 2.750			3			3			3			
2.750 to 3.889			2			3			8			
3.889 to 5.500			1			1						
Total	98	98	100	99	98	100	97	95	100	98	98	100

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Particle Volume Distribution (WHC-SP-1182, Appendix I)

	M13T			M13B			T20T			T20B(Duplicate)		
Particle Dia. (µm)	Per Cent in Range			Per Cent in Range			Per Cent in Range			Per Cent in Range		
	40 W	25 W	As Received	40 W	25 W	As Received	40 W	25 W	As Received	40 W	25 W	As Received
0.172 to 0.243	0.75	--	--	--	--	--	1.8	1.7	1.2	0.9	0.8	--
0.243 to 0.344	1.80	0.8	--	1.0	0.4	--	2.1	1.8	1.3	2.0	1.8	1.0
0.344 to 0.486	1.90	1.0	0.3	1.0	1.0	0.8	2.0	1.6	1.2	2.0	1.8	1.0
0.486 to 0.688	1.60	1.1	0.8	1.3	1.1	0.9	2.1	1.5	1.3	1.9	1.8	1.0
0.688 to 0.972	2.6	1.6	1.1	2.0	1.7	1.2	2.7	1.9	1.5	2.7	2.1	1.4
0.972 to 1.375	3.4	2.0	1.6	2.7	2.1	1.6	3.7	2.4	2.1	3.6	2.8	1.9
1.375 to 1.946	4.0	3.2	1.9	3.2	2.5	2.1	3.8	2.8	2.3	3.9	3.2	2.1
1.946 to 2.750	5.0	4.2	2.3	3.8	3.5	2.6	3.9	3.7	2.2	4.2	3.6	2.4
2.750 to 3.889	6.3	5.5	4.1	4.8	4.5	3.8	5.1	4.6	4.0	4.9	4.2	3.2
3.889 to 5.500	7.4	7.7	5.9	6.3	6.0	5.1	6.5	6.1	5.4	5.5	4.7	4.0
5.500 to 7.778	10.8	10.6	8.7	8.1	7.7	6.3	8.1	7.8	6.5	7.4	6.5	5.0
7.778 to 11.000	11.5	11.6	10.3	8.6	8.2	7.1	8.6	8.3	7.5	7.8	6.9	5.2
11.000 to 15.560	11.8	11.8	11.0	8.8	8.7	8.0	9.3	9.4	8.5	7.9	7.4	5.8
15.560 to 22.000	11.9	13.5	14.7	10.1	10.0	9.0	10. 5	11. 0	10.2	8.4	8.2	6.7
22.000 to 31.100	9.3	11.4	14.7	10.9	11.1	10.1	9.6	10. 6	10.4	7.8	8.3	7.5
31.100 to 44.000	5.2	6.9	10.1	10.3	11.4	10.1	7.1	7.1	9.0	7.8	8.3	8.2

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44,000 to 62,230	2.5	3.2	5.6	7.9	9.4	9.7	4.9	5.5	6.7	5.8	6.8	5.9
62,230 to 88,000	1.2	1.8	2.7	4.7	5.9	7.9	2.0	2.7	4.5	4.5	5.4	5.1
88,000 to 124.50	0.6	0.8	0.9	3.2	4.1	5.5	1.6	2.2	3.3	3.4	4.6	4.8
124.50 to 176.00			1.3	1.5	1.9	3.9	1.0	1.8	2.3	2.5	3.5	5.2
176.00 to 248,900				0.7	0.9	3.3	1.2	2.2	2.3	1.1	1.7	4.5
248,900 to 352.00						1.3	1.7	1.8	3.2	1.5	2.0	3.0
352.00 to 497.00									2.2	3.8	4.8	9.6
497.00 to 704.00												10.2
Total	99.6	98.7	98	100.9	102.1	100.3	99.3	98.5	99.1	101.3	101.2	104.7

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Basis for Blow Sand Particle Size

Reference:

Kirk-Othmer, Encyclopedia of Chemical Technology, Vol. 1 (4th Ed.), 1991

K. Willeke and K.T. Whitby, J. Air Pol Control Assoc. 25:529(1975)

Atmospheric aerosols may be classified into three size modes: nuclei, accumulation, and large or coarse-particle modes. The bulk of the aerosol mass usually occurs in the 0.1 to 10 μm size range which encompasses most of the accumulation mode and part of the large-particle mode. From approximately 0.09 μm to approximately 2.5 μm , particles are in the accumulation mode, while particles in the 2.5 μm to 10 μm range tend to have appreciable deposition velocities (Kirk-Othmer).

The characteristics of wind blown dust are (Willeke & Whitby):

Table B-1 Aerosol Characteristics of 2.5 μm to 10 μm Particles

Source	Wind Blown Dust
Fate	Deposit
Atmospheric Lifetime	Hours

Appendix C

Table C-1. Sludge Simulant Components									
Balance: Mettler PM1200			Measurements by: Bob Loper						
Calibration Code 752-06-01-001			Dry Sludge Stimulant Batch						
Calibration Date 10/31/97									
Calibration Expires 10/28/98			1	2	3	4	9	10	11
Component	Micron Size	Target	Actual	Actual	Actual	Actual	Actual	Actual	Actual
Al ₂ O ₃	297 to 420	0.567	0.568	0.567	0.568	0.568	0.567	0.567	0.567
	210 to 297	0.239	0.24	0.24	0.239	0.239	0.24	0.24	0.238
	125 to 210	0.398	0.398	0.398	0.399	0.399	0.398	0.399	0.398
	90 to 125	0.328	0.328	0.328	0.328	0.329	0.329	0.329	0.328
	63 to 90	0.462	0.462	0.462	0.462	0.463	0.463	0.463	0.463
	45 to 63	0.787	0.788	0.786	0.787	0.788	0.788	0.788	0.788
	38 to 45	1.134	1.134	1.135	1.134	1.134	1.134	1.133	1.134
	< 38	11.686	11.685	11.686	11.685	11.68	11.68	11.68	11.687
Fe ₂ O ₃	297 to 420	1.744	1.745	1.745	1.744	1.743	1.744	1.744	1.744
	210 to 297	0.734	0.736	0.734	0.736	0.735	0.734	0.735	0.735
	125 to 210	1.224	1.225	1.226	1.224	1.224	1.225	1.225	1.225
	90 to 125	1.01	1.011	1.009	1.012	1.01	1.009	1.01	1.011
	63 to 90	1.423	1.423	1.421	1.425	1.423	1.424	1.425	1.423
	45 to 63	2.421	2.42	2.421	2.422	2.423	2.422	2.421	2.42
	38 to 45	3.488	3.487	3.487	3.488	3.487	3.489	3.489	3.488
	< 38	35.956	35.957	35.957	35.957	35.95	35.95	35.95	35.956
Copper	> 420	2.566	2.565	2.566	2.566	2.567	2.566	2.567	2.566
	< 10	11.833	11.834	11.834	11.835	11.83	11.83	11.83	11.833
Blow Sand	297 to 420	1.003	1.005	1.003	1.002	1.003	1.003	1.004	1.004
	210 to 297	0.422	0.423	0.422	0.423	0.424	0.423	0.422	0.422
	125 to 210	0.704	0.705	0.704	0.704	0.704	0.705	0.703	0.705
	90 to 125	0.581	0.582	0.581	0.58	0.582	0.58	0.582	0.581
	63 to 90	0.818	0.82	0.816	0.82	0.819	0.818	0.819	0.819
	45 to 63	1.392	1.393	1.392	1.392	1.392	1.392	1.394	1.393
	38 to 45	2.006	2.005	2.004	2.008	2.005	2.007	2.007	2.007
	< 38	20.674	20.673	20.675	20.674	20.67	20.67	20.67	20.675
Resin	As received	14.4	14.4	14.4	14.4	14.4	14.4	14.4	14.402
* Mass measured after vacuum drying hydrated material		Optional materials						Blow Sand Added: 7 g 590 to 840 micron and 7 g 420 to 590 micron	Zeolon 900 Added: 7 g 297 to 840 micron and 7 g 300 to 420 micron

Appendix D

Rotameter Calibration

The rotameter was calibrated by pumping water through the instrument to fill a 2000 ml graduated cylinder and recording the time required at various flow indications. The pump speed is adjusted to provide the desired rotameter reading while the water flow is directed away from the graduated cylinder. Once the desired flow is established, the water is directed into the cylinder while simultaneously starting a stopwatch. As the cylinder approaches full, the stopwatch is stopped while simultaneously the water is directed away from the cylinder. The rotameter reading, time and water volume are recorded. This is repeated at each rotameter increment throughout the range of interest. The volume flow rate is calculated by dividing the water volume by time. The data is entered into a spreadsheet to obtain the equation of a linear regression line.

The elutriation column velocity is calculated by dividing the volume flow rate by the cross sectional area of the column. Column velocities are calculated using volume flow rates predicted by the linear regression line equation:

$$\text{ml/sec} = 0.6687 \times (\text{rotameter reading}) + 2.5118$$

Table D-1. Rotameter Calibration						
Manufacture: Brooks Instrument Div. Model Number 1307-08E1B1A Serial Number 7806H54274 Calibration Date 23-Jun-98 Calibrated By: Don Squier				Column Velocity Column Inside Diameter - Inches 1.365 Column Cross Section - cm ² 9.4411		
Rotameter Reading	Seconds	Volume ml	Flow Rate ml/sec	Regression Line	Column Velocity	Column Velocity
42	65.3	2005	30.70	30.60	3.241	0.032
44	62.5	1990	31.84	31.93	3.383	0.034
46	60	1987	33.12	33.27	3.524	0.035
48	57.5	1992	34.64	34.61	3.666	0.037
50	55	1987	36.13	35.95	3.807	0.038
52	53.4	1990	37.27	37.28	3.949	0.039
54	51.6	1990	38.57	38.62	4.091	0.041

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Rotameter Calibration

Figure D-1. Rotameter Calibration

Appendix E

Table E-1. Resin Bead Yield per Column Flow Rate Data						
Rotameter Indication - % Flow (100% = ~1.09 GPM)	Grams lifted to column top	Grams falling to column bottom	Flow Rate - ml/sec	Velocity in Column - cm/sec	Velocity in Column - m/s	% Resin lifted to column top
40	8.41	3.93	29.260	3.099	0.031	68.2
50	12.58	0.46	35.947	3.807	0.038	96.5
46	14.35	1.61	33.272	3.524	0.035	89.9
52	13.1	0.28	37.284	3.949	0.039	97.9

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