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Pacific Northwest National Laboratory
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Composition and Technical Basis for K Basin Settler Sludge Simulant for Inspection, Retrieval, and Pump Testing

A. J. Schmidt
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June 2007

Prepared for the U.S. Department of Energy
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This report was originally published in May 2007. In June, 2007, errors were discovered in Table 7, "Recipe for Preparation of ~100 L of as-Settled Simulant." The errors were discovered (and corrected) prior to procurement and make up of the simulant for testing. The values in Table 7 have been corrected in Revision 1. The value for Dry Solids, $Fe(OH)_3$ has been corrected to 2.0 kg (formerly, it was incorrectly given as 6.0 kg). This correction resulted in changes to the mass values given for: As-Added $Fe(OH)_3$, Additional water, Total Dry Solids, and Total As Added. References to a specific sludge transfer pump have also been removed in Rev. 1.

Composition and Technical Basis for K Basin Settler Sludge Simulant for Inspection, Retrieval, and Pump Testing

1.0 Approach and Summary

This report provides the formulation and technical basis for a K Basin Settler Tank sludge simulant that will be used by the K Basin Closure Project (KBC) to test and develop equipment/approaches for Settler Tank sludge level measurement and retrieval in a mock-up test system of the actual Settler Tanks. The sludge simulant may also be used to test pumps for transfer of sludge from the K West (KW) Basin to the Cold Vacuum Drying Facility (CVDF) (~500 ft). As requested by the K Basins Sludge Treatment Project (STP), the simulant is comprised of non-radioactive (and non-uranium) constituents.

The approach used to select and justify the sludge simulant consisted of series of general elements/steps:

- Identify/understand the objectives for the testing to be conducted using the simulant.
- Identify the sludge simulant chemical/physical properties important to meet the test objectives.
- Understand the composition/properties of the actual sludge being modeled (i.e., establish parameter targets for simulants).
- Select appropriate simulant components by matching or bounding the key properties of the sludge being modeled.
- Prepare simulant materials and verify that resulting properties are within the acceptable parameter targets.

Based on the test objectives and the above approach, the simulant (referred to as the "Settler Sludge Simulant") was formulated and is summarized in Table 1. Table 1 also identifies potential suppliers for the simulant components. The simulant was prepared in the laboratory and some key physical properties were measured (Table 2). A photo of a 10 ml sample of the as-settled simulant is provided as Figure 1.

General Caution: Use of this sludge simulant for testing purposes beyond those considered above must be evaluated on a case-by-case basis.

Table 1. Settler Sludge Simulant for Inspection, Retrieval, and Pump Testing

Represents	**U metal**	**Large U oxide**	**Fine U oxide**	**Iron phases**	**Al & blow sand**	**Simulant**
Surrogate Component	W or WC[(a)]	Steel Grit L[(b)]	CeO_2[(c)]	$Fe(OH)_3$[(d)]	Flyash[(e)]	
Wt%, Dry	6	14	68	1.00	11.00	100
Particle density, g/cm^3	14.5 to 18	7.8	7.12	3.02	2.2-2.5	6.0
Size, µm	Particle Size Distribution, Cumulative Percent Less Than					
600	100	100	100	100	100	100
500	85	85	100	100	100	97
250	0	29	100	100	100	84
100	0	0	99.5	100	97.4	79
40	0	0	91.1	100	81	72
20	0	0	70.9	100	65	56
10	0	0	53.8	100	45.6	43
5	0	0	38	100	30	30
1	0	0	17.9	93	7.8	14

(a) Can use tungsten (W) metal alloy SD170 or cobalt-cemented tungsten carbide (WC). Product SD170, ATI Firth Sterling (800-821-4273, www.tungstenprod.com). WC, KENFACE, Reade Advance Materials (Attn. B. Cockren 775-352-100) www.reade.com. [Note: Reade can also supply SD170 and steel grit.]
(b) Virgin Steel Grit Hardness L, Combination of GL-40 and GL-80 to build specified size distribution. Stephenson Pattern Supply (800-873-1912), www.stephensonpattern.com.
(c) Ceric Oxide, 95%+, Item 21, GFS Chemical Inc., (877-534-0795) gfschemicals.com
(d) Iron hydroxide, Code 11177, 99.5%, 13 wt% Slurry, Noah Tech Corp, TX (210-691-2000) www.noahtech.com.
(e) Class F Centralia Flyash. Glacier Northwest, Tacoma (253 896-4650) or Central Pre-Mix, Pasco, WA (509-545-8405).

Table 2. Properties of Settler Sludge Simulant

Property	**Value**	**Unit**
Average Particle Density[(a)]	6.0	g/cm^3
Settled sludge density[(b)]	2.7	g/cm^3
Volume fraction water[(b)]	67	Percent
pH (of supernatant)[(b,c)]	10.4, 11.6	pH units
Shear strength[(b,d)] Mixed, transported, 54 h gel time Mixed, 90 h gel time	 6920 ± 230[d] 3650 ± 700[e]	 Pascal Pascal
Settling Time[(b,f)]	~2	h

(a) Calculated based on vendor provided data.
(b) Based on measurements performed on simulant prepared in laboratory.
(c) pH = 11.6 with 10 cm^3 sludge + 24 mL H_20; pH = 10.4 with 10 cm^3 sludge + 1000 mL H_20.
(d) Performed with Haake – RS600, 1.6 × 1.6 cm shear vane. Sample may have compacted during transportation. Mean ± standard deviation of 3 measurements.
(e) Mean ± standard deviation of 5 measurements.
(f) 10 ml sludge in 1000 ml cylinder (6 cm diameter, 36 cm height). Some cloudiness after 2 h.

Figure 1. Image of As-settled Settler Sludge Simulant (1 hr) in Centrifuge Cone (units in mL).

2.0 Key Uses/Objectives for Settler Sludge Simulant

The simulant will be used in mock-up testing to evaluate the use of ultrasonic level measurement equipment. For this testing, a mock-up KW Basin Settler Tank will be fabricated, partially loaded with simulant, and may be submerged in water. The ultrasonic level measurement equipment will be placed on top of the Settler Tank or mounted on the outside of the mock-up Settler Tank. The equipment will then interrogate the water level (at the sludge/water interface) at a number of locations to estimate the volume of simulant within the tank by subtracting the water level.

Setter tank mock-up testing with simulant will also be conducted to evaluate and refine equipment and approaches for retrieval of sludge from the Settler Tanks.

The simulant may also be used in testing to demonstrate and validate pumps for transferring sludge from the KW Basin to the CVDF (~500 ft via a hose-in-hose system). The pump demonstration testing will likely evaluate pump performance under nominal and bounding sludge conditions. Acceptable performance would be verified by showing:

- The pump check valves will not plug,
- The selected hose and pump internals, are adequately resistant to abrasion,
- Pump recovery can be achieved following an unexpected loss of power under design solids transfer conditions, and
- The sludge transfer velocity is adequate to prevent line/pump plugging.

For these test objectives, a physical (vs. a chemical) simulant had been specified. Use of this simulant for testing purposes beyond those considered above must be evaluated on a case by case basis.

3.0 Key Properties for Simulant

The sludge physical properties most important to the testing objectives are identified below.

Ultrasonic Level Measurement
Volume fraction water, particle size, particle density, and settled sludge density. The ultrasonic level measurement equipment must be able to accurately discern the sludge/water interface.

Retrieval
Settled shear strength, particle size, particle density, volume fraction water, oversized/foreign debris, particle hardness. These parameters will affect particle motivation, particle settling, mixing, bridging/plugging and erosion wear to equipment.

Pump Testing
[As a 2 to 12 volume percent slurry during nominal sludge transfer, and as settled/compacted sludge during off normal events (e.g., loss of power)]: oversized debris/foreign material (note: no oversized debris or foreign material is expected in Setter Tank sludge, as discussed below), particle size, particle hardness, particle density, volume fraction water (settled sludge).

Volume Fraction Water in Settled Sludge and Settled Sludge Density
Volume fraction water in actual K Basin settled sludges is not easily matched with simulants (particle morphology in available simulant material differs greatly from the actual sludge). Typical settled simulants are ~50% by volume water vs. 65% to 75% by volume for actual sludge. In general, the approach to date for K Basin sludge simulant design has been to match other key properties and accept the resulting lower volume fraction water. Because of the importance of this parameter to ultrasonic level measurement, for this Settler Sludge Simulant, the volume fraction water was refined by preparing and evaluating a number of simulant compositions. By matching the volume fraction water and particle density, the settled sludge density will also approximate that of the sludge being modeled.

Particle Size, Density, and Hardness
Several previous K Basin sludge simulants, including the "Simulant for Pump Erosion Testing"[a] were formulated to match particle size, particle density and particle hardness. This previous simulant experience was used to match these properties for the Settler Sludge Simulant.

Shear Strength of Settled Sludge
By matching chemical composition, phases, and particle size distribution, simulants have been prepared that exhibit a shear strength on the same order as actual sludge.[b] However, the KBC/STP project has not attempted to formulate simulants to specifically match settled sludge shear strength. For this Settler Sludge Simulant, this property was evaluated by preparing a number of simulants and qualitatively evaluating the resulting simulant consistency and strength. A 200-ml batch of the final simulant was prepared and shear strength was measured using a shear vane and a Haaka RS-600 measuring head.

(a) Schmidt, AJ. 2004. "Recommended Simulant for Pump Erosion Testing." PNNL Letter Report 46497-RPT04, transmitted by GB Mellinger (PNNL) to WW Rutherford (FH) on November 9, 2004 via Letter 46497-L06, Pacific Northwest National Laboratory, Richland, WA.
(b) BNGA. 2005. "K Basin Containerized Sludge Rheological Simulants." KBC-MO-0041, RCI # A21C-25147-RCI-065, October 31, 2005, BNG America, Richland, WA.

Foreign Materials

In a recent evaluation (see Attachment) it was concluded that oversized debris/foreign material is not expected to be present in Settler Tank sludge. This conclusion was based on the availability and use of engineering controls/features associated with the Settler Tanks, including the 500/600-µm screens upstream of the tanks and the "closed" nature of the Settler Tanks (i.e., operational debris cannot be dropped into the closed Settler Tank sludge system from the grating).

4.0 Composition and Character of the Settler Tanks Sludge

Settler Tank sludge consists of size-segregated material from the spent nuclear fuel, canister, and fuel-scrap cleaning (i.e., KW/KE Internal, Coatings, and Canister sludge). After passing thought the Knockout pots (KOPs) and KOP screens, the undersized material (i.e., less than 500 or 600 µm) from these fuel packaging operations was accumulated in the Settler Tanks. No direct samples of the Settler Tank sludge have been characterized to date. The Settler Tank sludge characteristics are based on data from the characterization of source feed streams.

In the KBC Project Sludge Databook, Rev. 13A (Schmidt 2006) and Plys and Schmidt (2006), the design and safety basis composition of Settler Tank sludge is conservatively defined as 100% size segregated KW Canister sludge, design and safety basis, respectively. The composition of sludge in the Settler Tanks is expected to vary among the 10 settler tubes (or tanks), and vary spatially within each settler tube. That is, the settler tube design and operation results in some sludge stratification/concentration. Consequently, a conservative sludge source, for dose consequence and other safety analyses (higher uranium and radionuclide content) was selected as the representative sludge source for the Settler Tank sludge. However, for formulation of a simulant to evaluate level measurements, retrieval, and pumps, the properties of other sludge source streams to the Setter Tanks must also be considered.

The mass of sludge material accumulated in the Settler Tanks was estimated from a material balance constructed with data collected during spent nuclear fuel (SNF) packaging operations (Moore and Boger 2005). Based on SNF packaging experience, observations, and measurements (weight loss during fuel cleaning), higher quantities of KE Canister/Internal sludge were generated in comparison to original estimates made before completion of fuel processing. Therefore, to construct an updated sludge source-based composition and match the Settler Tank sludge volume estimates made by Moore and Boger (2005), Table 3 was constructed (Plys and Schmidt 2006).

Internal sludge[(a)] from KE (0.92 m^3) and KW fuel (0.31 m^3) is similar in composition to KW Canister sludge (0.96 m^3). Canister Coating sludge[(b)] [0.093 m^3 (from KE) + 0.613 m^3 (from KW)] can be considered to be similar to KE Canister sludge (2.5 m^3). Therefore, it is projected that Settler Tanks contain 2.2 m^3 of KW Canister-type sludge and 3.2 m^3 of KE Canister-type sludge (i.e. ~ 60 vol% KE

(a) Internal sludge is defined as particulate material, already existing and contained within breached and damaged fuel elements. During fuel element washing, internal sludge was liberated through cladding breaches and became part of the IWTS sludge inventory. Internal sludge consists primarily of uranium oxides and uranium metal particulate less than 500 µm.

(b) KE/KW Coatings Sludge: Many KE and KW fuel elements were covered with an adherent layer of material on the cladding surface. For the KE fuel, the coatings consisted predominantly of uranium oxyhydrates; for KW fuel, the coatings are predominately aluminum hydroxides. During fuel element washing, some fraction of the coatings was removed and became part of the IWTS sludge inventory.

Canister type sludge and ~40 vol% KW Canister type sludge). Also, if it is assumed that the Settler Tank sludge composition is approximately proportional to the weight of fuel scrap generation during fuel packaging, then it would be expected that the quantity of sludge derived from KE Fuel washing is approximately 3 times greater than that derived from KW Fuel (Table 4).

Based on these considerations, the target composition for the Settler Sludge Simulant should range from 100% KW Canister sludge to a 25/75 volume percent mixture of KW/KE Canister Sludge.

Table 3. Projected Source Based Compositions of Settler Tank Sludge (from Plys and Schmidt 2006, Appendix F)

Settler Sludge Volume and Source Stream Evaluation	Volume in Settler Tank, m^3	Design Basis Density, g/cm^3 (air)	Design Basis Void Fraction	Average Particle Density, g/cm^3	Apparent Underwater Density, g/cm^3	Total Mass, kg (in air)
KE Canister Sludge	2.5	1.9	0.75	4.6	0.90	4750
KE Coatings	0.093	1.32	0.70	2.07	0.32	123
KE Internal Sludge	0.92	2.97	0.70	7.57	1.97	2730
KW Canister	0.96	3.0	0.65	6.71	2.0	2880
KW Coatings	0.612	1.32	0.70	2.07	0.32	808
KW Internal	0.31	2.97	0.70	7.57	1.97	921
Total	5.40	2.26			1.26	12200
Values for settled density and void fraction (volume fraction water) were taken from the Sludge Databook (SNF-SD-TI-015, Vol 2, Rev 12a) for KE and KW Canister Sludge, and from Pearce (2001) for KE and KW Coatings and Internal Sludge. Total Settler Tank sludge volume, 5.4 m^3, is consistent with estimates developed by Moore and Boger (2005).						

Table 4. KE/KW SNF Scrap Generation During SNF Packaging

Stream	Mass,[(a)] Kg
Scrap from KE Fuel Washing	24430
Scrap from KW Fuel Washing	7330
Total Scrap	31760
	Fraction
Fraction of Total from KE	0.77
Fraction of Total from KW	0.23
(a) Underwater Mass	

5.0 Comparison of Settler Sludge Simulant to Settler Tank Source Streams

Table 5 provides data on settled density, volume percent water, and uranium concentrations (dry solids basis) for design and safety basis KW and KE Canister sludge and design basis mixtures [based on the KBC Sludge Databook (Schmidt 2006)]. These are the dominant source streams to the Settler Tanks, and their compositions provide a reasonable range for the simulant. Table 5 also includes the properties and uranium concentrations (being modeled) for the Settler Sludge Simulant.

Table 5. Comparison of Settler Tank Source Streams and Simulant

Stream	Settled Density, g/cm^3	Volume % Water in Settled Sludge	Average Particle Density, g/cm^3	Wt% Uranium Total in Dry Solids	Wt % Uranium Metal in Dry Solids	Wt% Uranium Oxides in Dry Solids
KW Canister Sludge, Safety Basis	4.00	65	9.6	80	5.97	85
KW Canister Sludge, Design Basis	3.00	65	6.7	80	2.68	94
KE Canister Sludge Safety Basis	2.50	75	7.0	80	7.14	88
KE Canister Sludge Design Basis	1.90	75	4.6	67	3.47	77
50/50 (Vol) KW/KE Can Design Basis	2.45	70	5.8	74	2.94	86
25/75 (Vol) KW/KE Can Design Basis	2.18	73	5.3	70	3.15	81
Settler Sludge Simulant	2.70	67	6.0	74	6.00	82

Screens in the KW Integrated Water Treatment System (IWTS) limit the maximum particle size in the KW Settler to less than 600 µm. Table 6 and Figure 2 depict the size-truncated particle size distributions (PSDs) of various Settler Tank source streams, and compares these distributions to the simulant. The PSD of the Settler Sludge Simulant is a reasonable match to all sludge types except KW Canister sludge. The KW Canister sludge exhibits a significantly finer PSD than that of the simulant (i.e., simulant contains higher concentrations of larger particles). While submicron particles will contribute to shear strength and solution viscosity, it is expected that the larger particle size PSD of the simulant will present a greater challenge to mobilization, transport, pumping, and equipment wear (erosion).

Table 6. Comparison of Particle Size Distributions of Sludge Streams and Settler Simulant

Particle Size, µm	Source Streams			KE/KW Can Mixtures		Settler Simulant
	KE Floor	KE Can	KW Can	50/50 (vol) KE/KW Can	75/25 (vol) KE/KW Can	
	Particle Size Distribution, Cumulative Percent Less Than					
600	100	100	100	100	100	100
500	99	98	99	99	98	97
250	84	90	98	94	92	84
100	81	83	96	89	86	79
40	71	64	91	77	71	72
20	54	51	79	65	58	56
10	35	34	63	48	41	43
5	21	20	44	32	26	30
1	4	4	33	19	11	14

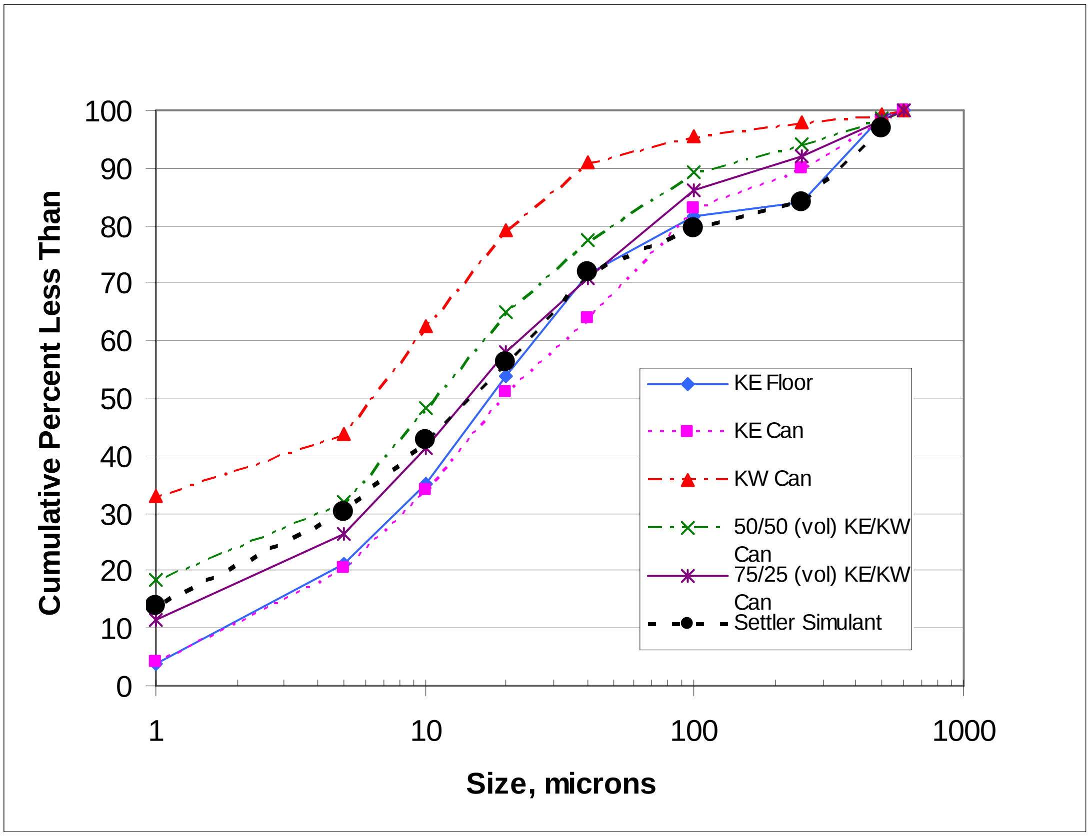

Figure 2. Comparison of Particle Size Distribution of Sludge Streams and the Settler Simulant

The shear strength of the source streams to the Settler Tanks ranges from a low of 30 Pa for KW Canister sludge to a high of 8100 Pa for KE Canister sludge (Plys and Schmidt 2006). The shear strength measurements were performed on the actual sludge samples with a particle size range from < 1 µm to 6350 µm, while Settler Tank sludge only includes particles less than 600 µm (74% of KE Canister and 85% of KW Canister sludge is less than 600 µm).

The Settler Sludge Simulant exhibited a relatively high, but reasonably representative shear strength range: 2600 to 7200 Pa. Two sets of shear strength measurements were performed on the simulant. For the first set, simulant was prepared, mixed, transported by vehicle to the laboratory, and allowed to set up for 54 hours (gel time). The resulting shear strength (average of 3 measurements) was 6920 ± 230 Pa. Vibrations experienced during the transport may have resulted in some compaction of the simulant. For the second set of measurements, the Settler Sludge Simulant was remixed and allowed to settle for 90 hours. The resulting shear strength (average of 5 measurements) was 3650 ± 700 Pa.

6.0 Bases for Simulant Component Selection

In 2004, surveys of the technical literature were performed for the KBC Project to characterize the mechanical properties of K Basin sludge components and, in particular, irradiated uranium metal and uranium dioxide. Measurements of the hardness of typical irradiated uranium metal fuel coupons taken from fuel at the K Basins were also performed. Based on these findings and associated surveys of supplier data, candidate materials were proposed to act as uranium metal and uranium dioxide surrogates. The results of these investigations were provided in Delegard et al. (2004). Based on Delegard et al. (2004) and other considerations, recommendations on surrogate materials for the Settler Sludge Simulant are provided here.

6.1 Surrogate for Uranium Metal

Recommended Material(s)

1) Tungsten (W) metal-based alloy SD170 (90% W, ~7% Fe, 3% Ni), density 17.1 g/cm^3. The Rockwell C hardness for SD170 is 32 vs. 30 ± 8 for irradiated uranium metal. Ultimate tensile strength of SD170 is 120,000 psi compared to 77,000 psi for irradiated uranium metal (Delegard et al. 2004).

2) As an alternative to alloy SD170, Cobalt-cemented tungsten carbide (“WC”), KENFACE (Kennametal, Fallon, NV) (87% W, 6.9% Co, 5.7% C, 0.20% Ti), may be used. WC exhibits a Rockwell C hardness value of 69 to 74, which is much higher than that of the actual irradiated uranium fuel (30 ± 8), and exhibit a lower particle density (14.5 to 15 g/cm^3 vs. 19 g/cm^3 for U metal). However, Cobalt-cemented tungsten carbide is readily available in the particle size ranges needed for the Settler Sludge Simulant.

   With its significantly higher hardness, use of cobalt-cemented tungsten carbide will provide conservative results with respect to system component erosion. As noted later, this material will also resist degradation aspects of the simulant if it is continuously recycled during testing, as has been the case in some past KBC pumping evaluations.

Particle Size Range

250 to 600 µm. Very little uranium metal has been found in size-fractionated K Basin sludge samples that only contain particles below 250 µm. Placing all of the uranium metal surrogate in the upper portion of the Settler Sludge Simulant particle size distribution will increase the challenges associated with mobilization/retrieval and component erosion.

Weight Fraction in Simulant

6 wt% (dry weight basis). As shown in Table 5, 6 wt% uranium metal is at the upper end of the sludge streams considered.

6.2 Surrogates for Uranium Oxide

Recommended Material(s)

Cerium oxide for fine uranium oxides (< 100 µm). Steel grit (L hardness) for uranium oxide agglomerates (100 to 600 µm). The Rockwell C hardness for uranium oxide (UO_2) ranges from 56 to 63 (Delegard et al. 2004). In comparison, the Rockwell C hardness values are 41 to 56 for cerium oxide and 55 to 60 for steel grit (L hardness). The calculated particle density for the representative uranium oxide [Plys and Schmidt (2006), Appendix C] $UO_{2.42}\cdot0.66H_2O$[(a)] is 7.53 g/cm^3. In comparison, the particle densities of cerium oxide and steel grit are 7.13 and 8.2 g/cm^3, respectively.

(a) $UO_{2.42}\cdot0.66H_2O$ (molecular weight = 288.6) is an equal uranium mole fraction mix of U_4O_9 ($UO_{2.25}$), UO_2, and $UO_3\cdot2H_2O$.

Particle Size Range
<1 to 100 μm for fine uranium oxide; 100 to 600 μm for uranium oxide agglomerates.

Weight Fraction in Simulant
Total = 82 wt%, dry basis. As shown in Table 5, 82 wt% falls within the lower range of the streams considered. Selection of a higher uranium oxide content (for the simulant to model) would reduce the quantity of non-uranium solids within the simulant.

To determine the appropriate quantity of uranium oxide to model for the simulant, the uranium metal concentration (6 wt%) was subtracted from the uranium total concentration (74 wt%), and the difference (68 wt%, uranium as uranium oxide) was then converted to an oxide-basis. For the uranium as "oxide" calculation, the masses of oxygen and waters of hydration must be accounted for. Based on analyses in Plys and Schmidt (2006, SNF-7765, Rev. 3, Appendix C) and Schmidt and Delegard (2003), the representative uranium oxide compound in KE Canister and KE Floor sludge is: $UO_{2.42}\cdot 0.66H_2O$. This uranium oxide compound has a molecular weight of 288.6 g/gmol and a particle density of 7.53 g/cm^3. To convert to uranium oxide, the mass of uranium "as oxide" is multiplied by the ratio of molecular weights (i.e., 288.6/238 x 68% = 82 wt%).

KE Canister sludge Samples 96-06 M and 96-06 L are high uranium content sludge samples with detailed information on particle size, and provide insight on the particle size distribution of uranium oxide within the sludge. On a dry weight basis, these samples contain 83 to 84 wt% uranium (Makenas et al. 1997). After sonication, the volume mean particle size of these samples was about 30 to 40 μm. On a number basis, the mean particle diameter for 96-06 M and 96-06 L ranged from 0.36 to about 1.12 μm (Makenas et al. 1997). Hence, the major population of particles high in uranium content is expected to be well below 250 μm.

To determine the fraction of fine uranium oxides vs. uranium oxide agglomerates, sieving and analytical results from KE Canister sludge sample KC-2/3 (a composite prepared with sludge collected from 11 canister barrels in the KE Basin) were extensively evaluated.[a] This sample composite was fractionated (sieved) based on size (at 250 μm). The resulting splits (plus 250-μm and minus 250-μm fractions) were analyzed to determine the total uranium content (Elmore et al. 2000) and uranium metal content (Delegard et al. 2000). Based on a mass balance, approximately 12 wt% of the uranium oxide was greater than 250 μm. The split in uranium oxide at 250 μm was used to determine quantities of cerium oxide (i.e., uranium oxide being less than 100 μm) and steel grit (i.e., uranium oxide agglomerates being greater then 250 μm) for the Settler Sludge Simulant. To fill in the particle size distribution between 100 and 250 μm, additional steel grit was added.

6.3 Surrogates for Non-Uranium Sludge Fraction

Recommended Material(s)
Class F Flyash and ferric hydroxide (slurry).

Particle Size Range
<1 μm to 100 μm.

(a) BNGA. 2007. "Evaluation of Simulant Specified for Design and Fabrication of the Assay Vessel Agitator." PRT-5477-T-0013, Rev. 0, BNG America, Richland WA.

Weight Fraction in Simulant
Non uranium solids = 12 wt%, a determined by difference after accounting for the uranium metal and oxide.

Centralia Class F Flyash, with a particle size distribution of <1 to 100 µm, has been found to act as a good physical simulant for actual sludge, and its successful use in prior sludge simulants is continued here. About 85 to 95 wt% of flyash is composed of aluminosilicate glass. Ferric hydroxide has been identified in KE Canister sludge, and has been added to the simulant in a slurry form at a low concentration to help adjust the water content of the as-settled simulant to be consistent with that observed in the actual sludge.

7.0 Considerations for Simulant Preparation

Based on a settled density of 2.7 g/cm^3, and a volume fraction water of 67%, Table 7 provides the simulant recipe for preparation of ~100 L of as-settled Settler Sludge Simulant. With the exception of the iron hydroxide, the simulant components can be added and mixed as dry materials. The iron hydroxide is available as a 13 wt% slurry, and therefore, must be added as a slurry. For small batches of simulant (e.g., < 10 L), an excess of water should be added, and components can be mixed by hand. For large batches (>10 L), mechanical mixing should be considered (e.g., prepared in ~50 L batches using cement mixer). After mixing and settling (~ several hours) excess supernatant can be withdrawn. Excessive mixing followed by batch settling will result in partial segregation of simulant components. [Note: when adding and mixing dry components, significant dusting may occur. Addition of water and use of appropriate personal protective equipment (PPE) should be considered.]

Table 7. Recipe for Preparation of ~100 L as-Settled Settler Sludge Simulant

Component	Size Range, µm	Wt% (dry basis)	Dry Solids, kg	As Added, kg
SD170[a] or WC[a]	250 to 600	6	12.2	12.2
Steel grit[a], GL-40	250 to 600	10	20.3	20.3
Steel grit[a], GL-80	100 to 250	4	8.1	8.1
Cerium Oxide	<1 to 100	68	138.0	138.0
Flyash Class F	<1 to 100	11.0	22.3	22.3
$Fe(OH)_3$[b]	<1 to 5	1.0	2.0	15.6
Additional water		add a minimum of:		53.4
Total		100	203	270

(a) Component will need to be sieved to obtain target size fraction.
(b) To be added as 13 Wt% Slurry

7.1 Surrogate for Uranium Metal

As noted above, either tungsten alloy SD170 or tungsten carbide (KENFACE) can be used to represent the uranium metal fraction in the Settler Sludge Simulant. The tungsten carbide is likely to be more readily available in the required particle size distribution; however, with respect to particle hardness, the tungsten carbide may be overly conservative. If the simulant is to be extensively recirculated during the testing, and SD170 is used, the simulant should be checked periodically (e.g., every 4 hours) for evidence of particle rounding. If particle rounding is significant, and the test objectives include erosive wear evaluation, then the particles greater than 100 µm may need to be removed and replenished periodically. Tungsten carbide would be significantly less susceptible to rounding.

Potential Suppliers

Can use tungsten (W) metal alloy SD170 or cobalt-cemented tungsten carbide (WC).

- Product SD170, ATI Firth Sterling (800-821-4273, www.tungstenprod.com.
- WC, KENFACE, Reade Advance Materials (Attn. B. Cockren 775-352-100) www.reade.com.

7.2 Surrogates for Uranium Oxide

For procurement of steel grit, it is important to specify that virgin grit is needed. Recycled grit is likely to exhibit significant particle rounding. A vendor supplied sample (Stephenson Pattern Supply) of steel grit GL-80 (hardness L) exhibited a particle size distribution (PSD) nearly identical to that of the target for this component. That is, the as-supplied sample required very little particle size modification to match the target distribution. However, the sample provided to the laboratory may not have been representative. Most likely, quantities of both GL-40 and GL-80 will need to be obtained and size fractionated (sieved) to provide steel grit that meets the target PSD as shown in Table 7.

The cerium oxide tested in the laboratory for the Settler Sludge Simulant was obtained from GSF Chemicals (Item 26). The Erosion Simulant[a] included a cerium oxide manufactured by Molycorp (760-856-6655, Product 5310). Although the particle size distributions of the two cerium oxides are similar, the GSF product contains a higher fraction of submicron material. If cerium oxide from GSF is difficult or costly to obtain, it is possible the Molycorp product could be substituted. However, if a substitution is made, it is recommend that a small batch of simulant is prepared in the laboratory and its properties compared against the simulant made with the GSF cerium oxide.

Potential Suppliers

- Steel Grit: Virgin Steel Grit Hardness L, combination of GL-40 and GL-80 to build specified size distribution. Stephenson Pattern Supply (800-873-1912), www.stephensonpattern.com. Note, Reade Advance Materials www.reade.com, may be able to supply steel grit.
- Ceric Oxide: 95%+, Item 21, GFS Chemical Inc., (877-534-0795) gfschemicals.com

(a) Schmidt, A.J., 2004. "Recommended Simulant for Pump Erosion Testing" PNNL Letter Report 46497-RPT04, Transmitted by GB Mellinger (PNNL) to WW Rutherford (FH) on November 9, 2004 via Letter 46497-L06, Pacific Northwest National Laboratory, Richland WA.

7.3 Surrogates for Non-Uranium Sludge Fraction

Use of Centralia Class F Flyash in previous simulants for testing K Basin sludge sampling equipment was successful (i.e., the Flyash was a good physical simulant for high uranium content sludges). However, in subsequent K Basin sludge simulant testing, with other grades/types of flyash, the testing was not as successful. Therefore, to the extent practicable, Centralia Class F flyash should be used wherever possible.

In addition to the NOAH Technologies $Fe(OH)_3$ iron hydroxide slurry, other iron oxides/hydroxides were evaluated in the laboratory for the Settler Sludge Simulant. The selected $Fe(OH)_3$, added at 1 wt% (dry basis) yields a simulant with the targeted volume fraction water and as-settled density. Therefore, use of alternative iron oxide/hydroxide products is not recommended.

Potential Suppliers

- Iron hydroxide, Code 11177, 99.5%, 13 wt% Slurry, Noah Tech Corp, TX (210-691-2000) www.noahtech.com.
- Class F Centralia Flyash. Glacier Northwest, Tacoma (253 896-4650) or Central Pre-Mix, Pasco, WA (509-545-8405).

8.0 References

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Attachment

Foreign Material in Sludge Simulant

Prepared by: RA Sexton (FH)

Purpose: Define the basis for the type and quantity of foreign material that should be included in simulants used for testing pumps for transfer of sludge from KW to the Cold Vacuum Drying Facility (CVDF).

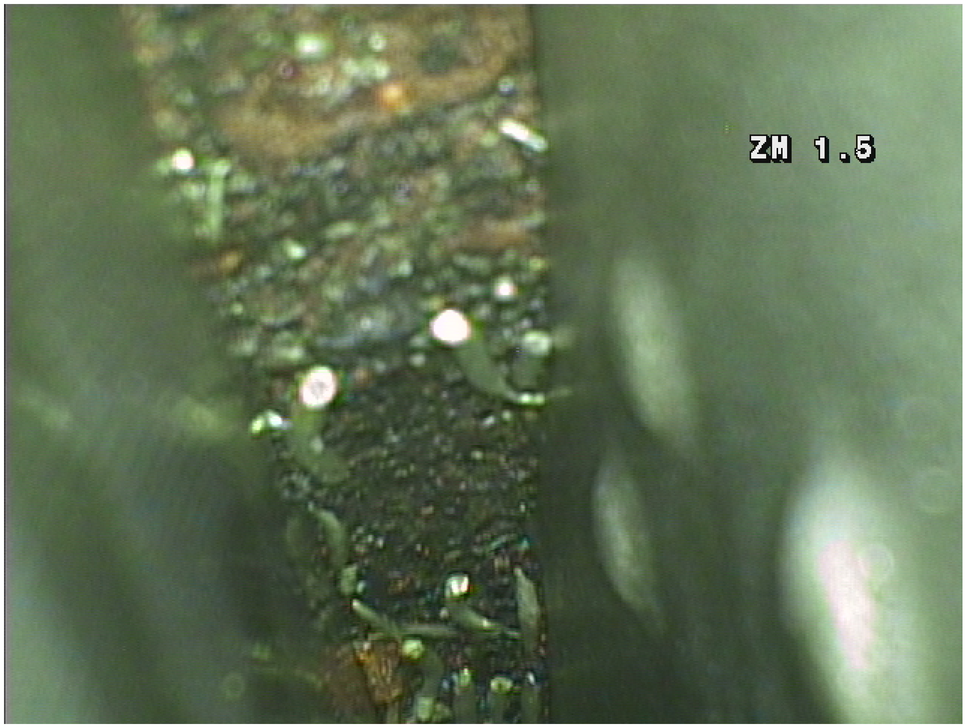
Foreign Material Observed in KE Sludge Containers: A variety of materials have been found in KE sludge containers, or in the Hose-in-Hose Transfer Line (HIHTL) system transferring sludge to KW, including but not limited to (per email from C. R. Miska to E. G. Erpenbeck, dated March 13, 2007):

- Wire ties. Length = various, including wire ties that were previously secured and then cut off.
- 1" conduit nut
- 1" conduit ferrule
- Writing pens in a variety of sizes
- Wads of duct tape
- Maslin survey wipes (they shred)
- Surgeons gloves (an operator reported a plastic bag full of gloves sank in one of the KE sludge containers; individual gloves have also been dropped in the sludge).
- Signal wire (~22 gauge). This was observed in the impellers of the booster station pumps following shutdown due to high vibration. It appeared that a very long length went through the system and was cut to ~ 1" lengths by the up stream pumps.
- Rubber shoe covers (floating on the surface of a KE sludge container)
- Signal wire spool (floating on the surface of a KE sludge container, disintegrating)
- Roll of duct tape (floating on top of tank at one time)
- Poly rope (normally floats, but could be sucked in to a pump)
- Long strands of fibrous material (with the appearance of a disintegrated automotive serpentine belt)

In addition to the observed foreign material, aluminum wires from degraded screened-bottom fuel canisters, are expected to be present in the KE Containerized sludge. According to WHC-SD-SNF-TI-012, 39% of the 3700 fuel storage canisters formerly stored in the KE Basin had screened bottoms. H-1-36935 indicates that this is aluminum wire 0.063" in diameter. Small lengths of this wire have been observed in Primary Cleaning Machine (PCM) cleanout (see photo below)

Foreign Material Transferred to CVDF: Sludge will be transferred to CVDF from three sources, KW sludge containers, knock-out pots, and settlers.

Sludge collected in the KW containers, including sludge transferred by HIHTL and collected by the Fuel and Pit Sludge Retrieval (FPSR) system, has passed through a screen to remove particles larger than ¼". However, items longer than ¼" can pass through strainers if their other two dimensions are under ¼". Small lengths of wire have been observed in transfer pump impellers. The following photo was taken with a boroscope. The impeller clearance is only ¼" and the small wires appear to be shorter than that.



“Buggy springs”, which have been collected as scrap following disassembly of N Reactor fuel assemblies, are too large to pass through the strainers and are not expected in the KW sludge containers. The SWS basket holes maximum size is 0.245" (H-1-86806, sheet 3). The minimum buggy spring width is 0.247 plus it has a minimum thickness of 0.075" (H-3-27584).

Foreign Material in KW Sludge Containers: The larger items noted above in KE sludge containers are believed to have fallen into the containers from above, not transferred via pumping. Containerized Sludge in KW should have a greatly reduced probability of such foreign material, compared to KE containers for the following reasons:

- The KW Containers were video taped before being placed in service to assure that they were free of foreign material.
- The containers were installed with covers in place, with construction cleanliness protocol in effect.
- The facilities have since implemented an administrative control for foreign material exclusion zones.
- KW containers do not have grating directly above them, providing manned access and the resultant dropping of foreign objects. KE containers have grating and manned access directly above them.

Material transferred through the HIHTL system to KW had to pass through a pump with no more than ¼” impeller clearance.

Recommended Composition of Foreign Material for Simulants

Most of the large foreign material observed in KE Containers is expected to be excluded by end effectors or removed by strainers during HIHTL sludge transfer to the KW containers. However, some fraction of wires from degraded screen-bottom fuel storage canisters and other sources (e.g., signal wire) in the KE sludge are expected to be transferred to the KW Containers.

Based on the wire expected from Mark 0 fuel canisters as observed in PCM cleanout and wire observed in the booster pump impeller, it is proposed that foreign material in the containerized sludge be modeled with 1" lengths of wire. Uninsulated 12 gauge wire, (approximately 0.08"), would be considered conservatively large in diameter compared to items that are known to have passed through ¼" strainers. This size is larger than the signal wire found in the KE containerized sludge and larger than the 0.063" wire from screen canister bottoms found during PCM cleanout. Some smaller, uninsulated 22 gauge wire, simulating signal wire should also be used.

The Knock Out Pot (KOP) contains material that passed through a ¼" strainer, but not through a 500 micron strainer. The wire described above can also be used to model foreign material in KOP sludge.

In the KW Basin, fuel was stored in enclosed fuel storage canisters to minimize fuel corrosion. A Grafoil seal was used between the canister lid and body. During KW Canister sludge characterization activities (HNF-1728) and fuel washing, significant quantities of Grafoil pieces and fines were observed. Therefore, Grafoil is anticipated to be present in the KOP and Settler sludge (HNF-SD-SNF-TI-009, Rev. 4), and should be included in the KOP sludge simulant. The Grafoil should be ground or cut into pieces that could pass through a ¼" strainer. This could include pieces approximately 1/8" wide and ½" long. Grafoil particles (less than 600 μm) within the Settler sludge are not expected to present any unique sludge handling issues, and therefore, do not need to be included with a Settler Tank simulant.

Because the settlers only contain material that has passed through a 500 micron filter, foreign material can be neglected in simulating settler sludge.

Quantity of Foreign Material:

Foreign material concentrations are suggested as follows:

Containerized Sludge = Wires, as described above, should be modeled at 0.25 vol% of the simulant settled volume. The containerized sludge is the collection of a large quantity of particles that originally accumulated by settling on the floor. The containerization process screened out larger pieces, except those that found their way through strainers. Some larger items have been dropped into KE containers, but most will have been screened out when moved to KW.

A 3/16" strainer in the transfer line from KE to KW accumulated approximately 1% of the sludge bulk volume transferred, but a significant portion of this would be considered sludge and not foreign material. The percentage of wire-like pieces, therefore, is expected to be a small fraction of the total volume.

KOP Sludge = Wires, as described above, should represent a higher percentage of KOP sludge volume and can be modeled at 2% of the simulant volume, because the lighter components of the sludge will pass through the KOP, tending to concentrate larger pieces.

Some KOP sludge, specifically some of the KOPs that had internal strainers, may have a high volume percentage of Grafoil. Some testing should be done with simulants that contain Grafoil at 20% by volume. This is conservatively higher than the 12% Grafoil concentration listed for nominal KOP sludge in HNF-SD-SNF-TI-009, Rev. 4. Examples of the type of Grafoil pieces found in the KW canister sludge are shown in HNF-1728 figures 1.2 and 1.3.

Settler Sludge = Foreign material is negligible, because the settler sludge has passed through a 500 or 600 micron strainer.

References:

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