PNNL-15092

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National Laboratory
Operated by Battelle for the
U.S. Department of Energy

Characterization of Compaction and Dryout Properties of KE Basin Sludge During Long-Term Storage

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Summary

The Hanford K Basins, K East (KE) and K West (KW), were built in the early 1950s for underwater storage of spent nuclear fuel (SNF) from the KE and KW Reactors. Since the 1980s, however, their principal use has been to store N Reactor SNF. In 1992, the decision to deactivate the PUREX plant left approximately 2100 metric tons of irradiated fuel in the basins, with no means for near-term disposition. With time, much of this uranium metal SNF degraded due to cladding breaches that occurred during reactor discharge and subsequent corrosion during underwater storage. The fuel corrosion products and corrosion products from structural components of the basins, along with ion exchange media and environmental matter, now make up the approximately 50 m^3 of sludge in the basins. Since the SNF has been removed from the basins and placed into dry storage, the focus of the K Basins Closure Project is on managing the sludge and decontaminating and decommissioning the basins.

As part of studies conducted to support decisions on sludge management, K Basin sludge samples have been collected, characterized, and evaluated since 1995. In the course of these studies, a number of sludge samples, stored in capped jars and vials within shielded hot cell facilities (hot cells), have lost supernatant liquids at unexpectedly high rates. As a result, several samples dried out in as little as 3 to 6 months. Consequently, systematic testing, discussed here, was performed to evaluate long-term sludge sample dryout under controlled conditions. Further, the tests were designed to provide data on long-term sludge compaction, gas bubble formation, and changes in sludge character over time to provide insights into phenomena that could affect the recovery of sludge after long-term storage.

At the time testing was initiated, the plan for sludge management was to collect the sludge in large diameter containers (LDCs) and store the containers at T Plant for a number of years before final processing and offsite disposal. The plan was revised in FY 2004 so that only ~6 m^3 of the less-radioactive sludge from the KE Basin will be transported in LDCs to T Plant. The remaining KE Basin sludge will be collected from the floor and pits into large freestanding containers located in the KE Basin pool. The sludge will settle and consolidate in these containers for a number of months before being transferred to the KW Basin and consolidated into large freestanding containers in the KW pool. The KW floor and pit sludge will also be consolidated into large containers. The sludge will be staged in these containers for up to 2 years before being removed for final treatment. Therefore, the consolidation and physical behavior of sludge during long-term storage in containers continues to be important to the K Basins Closure Project.

This report describes the results of laboratory tests performed to understand and predict sludge drying rates under warm room temperature (hot cell) storage conditions. Tests with six K Basin sludge materials, a control sample of simulated K Basin sludge, and a control sample containing only K Basin supernatant liquid were conducted for a continuous 28-month period from May 14, 2002 to September 22, 2004. All samples were loaded into glass graduated cylinders fitted with threaded plastic caps. Quantitative data were gathered on how the mass and volume of K Basin sludge, and its associated supernatant liquid, change with respect to storage time. The key findings and conclusions are summarized here, along with the results from individual tests.

Key Findings and Conclusions

Overall, the tests showed that K Basin sludge samples stored in screw-cap sealed containers under hot cell conditions can dry out unpredictably, depending on cap seal integrity. However, the integrity of the cap sealing cannot be confirmed when the cap is applied. The ambient hot cell radiation field (~5 Rad/hour) likely contributed to cap seal degradation. Dryout times for a 1-cm cover water depth can range from 5 to 216 months (18 years). Evaporation rates in screw-capped cylinders under hot cell conditions were found to be independent of the type of sample (water *vs*. sludge; radioactive *vs*. non-radioactive sludge). Although water was lost at variable rates from sludge samples during storage in the hot cell (and, presumably, in long-term containerized storage), the sludge itself had no intrinsic propensity to enhance or diminish the rate of water evaporation compared with that exhibited by water stored in the same environment.

Tests of six KE Basin sludges and the simulated sludge showed that most compaction occurred in the first week. Subsequent settling to 28-months time provided little additional compaction for most sludge types, as indicated by settled sludge density. Agitating the settled sludge with a vortex mixer had little to no effect on the density; negligible sludge lofting or compaction was evident within 4 days of the agitation for all sludge types, except one. That sample contained gas bubbles, which were generated from residual reactive uranium metal.

A composite sludge material containing KE floor, KE canister, and residual uranium metal left from prior testing showed gas bubble formation and consequently decreased density after about 78 days settling in the ~34°C hot cell. Visible gas voids subsequently were observed. This induction time behavior shows again that uranium metal-bearing sludge may lie quiescent for long periods, even at comparatively warm temperatures, before initiating gas generation.

When the testing was completed, the sludge samples were removed from the graduated cylinders. Except for a single uranium-rich sample that previously had dried out during storage prior to the present testing, the sludge re-suspended readily in the supernatant liquid at the end of the 28-month settling. Despite being reconstituted for the present testing and maintained in a water-saturated condition during the entire 28-month test interval, the uranium-rich sludge sample self-cemented and could not be re-suspended.

Test Description and Results

Once the graduated cylinders were loaded with sludge samples and capped, they were weighed and allowed to settle. The total volumes, settled sludge volumes, and weights of the cylinders were recorded, with measurements taken more frequently during the initial periods when sludge volume change rates would be greater. Also, the graduated cylinders and their contents were subjected to vibrations using a laboratory vortex mixer at an intermediate point in the sludge compaction tests. The intermediate mixing was performed to understand the potential impacts of transportation on sludge consolidation. Measurements were taken of the settled sludge volume before and after the vibration compaction to obtain quantitative data on the effect of vibration on sludge consolidation.

The table shows the test matrix, sludge compositions, and settled density (including results from separate, earlier studies for comparison) for the sludge samples used in this work. The results of the water loss and sludge compaction tests are summarized below.

Sludge	KE Sludge Origin	U, wt% (dry)	Density, g/ml			% Difference (a)
			Prior Studies	This Study		
				1 week	Final	
KB Sim	Simulated sludge	0.0	2.24	2.39	2.38	+6.2
FE-5	Weasel pit	5.32	1.47	1.57	1.62	+10.2
KC-2/3	Canister sludge composite	59.0	1.30	1.75	1.77	+36.2
KC-4	Floor sludge between canisters	16.6	1.26	1.28	1.28	+1.6
KC-5	Floor sludge from main basin	6.36	1.28	1.29	1.31	+2.3
96-13	Canister sludge that had dried in prior storage	82.0	2.67	2.86	2.95	+10.5
SNF Comp	Floor, canister, and crushed fuel mix	60.2	2.32	2.02	2.20	-5.2
KE NLOP	North Loadout Pit	2.51	1.35	1.33	1.34(b)	-0.7

(a) % Difference in the final settled sludge density in the present tests compared with the density found in prior testing; $\% \text{ Difference} = \frac{\text{Final density (this study)} - \text{Density (prior studies)}}{\text{Density (prior studies)}} \times 100\%$

(b) 2-week test conducted in a glovebox in January 2004.

Drying (Water Loss) Tests:

- In tests lasting ~28 months, drying rates under hot cell storage conditions (~34°C and 35% relative humidity) ranged from 1.5×10^{-4} to 6.9×10^{-3} cm (3.7×10^{-3} cm average) of water depth per day. At these rates, settled sludge solids originally covered by a 1-cm layer of water would be exposed to air after 5 months to 18 years (average 9 months).
- Water loss rates were found to be independent of the sample composition/radioactivity.
- Drying rates increased with time.
- Average drying rates were ~1800 times higher than could be explained by fluctuations in the atmospheric pressure and, in the capped graduated cylinders, were ~30-fold lower than were observed in a similar uncapped vessel stored under the same hot cell conditions.
- Drying rates apparently were most dependent on the cap seal integrity. The ambient radiation field in the hot cell likely contributed to cap liner degradation.

Sludge Compaction Tests:

- Settled sludge density for all materials, except SNF Comp (which contained significant uranium metal), changed little from the initial measurements after 7 days of settling to the end of the test (28-month period).
- The settled sludge densities in the present, extended settling-time, study were near, but generally higher than, densities found for the same sludges in prior shorter-duration testing.
- Settled sludge density increased with uranium concentration in the sludge solids.
- Agitation by the vortex mixer compacted the KB Sim, 96-13, KC-4, and FE-5 sludge to increase sludge density by ~1.3 to 2%. Agitation did not change the densities of KC-2/3 and KC-5 sludge.

- Sludge sample SNF Comp evidently still contained reactive uranium metal as shown by marked discernible gas voids. The density increase from 1.80 g/ml just before agitation to 2.20 g/ml just after agitation indicates that the gas void volume before agitation was about 18%.
- The SNF Comp sludge showed an "incubation time" of about 78 days before onset of gas generation at hot cell storage conditions.
- Gas bubbles in the supernatant liquid and apparent voids in the settled 96-13 sludge suggest that gas generation also occurred for this sludge.
- Most sludge samples were readily re-suspended after the 28-month settling time. The exception was sludge 96-13, which self-cemented over the test interval to a rigid block that could be fractured only with considerable pressure from the hot cell manipulators. Note that the 96-13 sample had dried out during prior storage, but had been reconstituted with water (dry sludge granules soaked in water for a week, then mixed aggressively) ~6 months before the present testing began.
- The sludge pH was found to be inversely proportional to uranium content (i.e., pH decreased from ~8 to ~5 with a dry-basis uranium concentration increase from ~2.5 to 82 wt%). Also, there was very little pH change during the 28-month test period.

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1.0 Introduction

The Hanford K Basins, K East (KE) and K West (KW), were built in the early 1950s for underwater storage of spent nuclear fuel (SNF) from the KE and KW Reactors. Since the 1980s, however, their principal use has been to store N Reactor SNF. The 1992 decision to deactivate the PUREX plant left approximately 2100 metric tons of irradiated fuel in the basins, with no means for near-term disposition. With time, much of this SNF degraded due to cladding breaches that occurred during reactor discharge and subsequent corrosion during storage. Ultimately these corrosion products, along with other materials, formed a sludge that now totals approximately 50 m^3 in both basins combined.

In addition to the products of the fracture and subsequent corrosion of irradiated uranium metal fuel, the sludge contains sand (from sand filters); infiltrated soils; steel, aluminum, and concrete structural component corrosion products; organic and inorganic ion exchange materials; and miscellaneous debris. Characterization studies have shown that the K Basin sludge composition and quantity varies according to its source location (Makenas et al. 1996-1999a,b; Pearce 2001; Mellinger et al. 2004). However, subsequent K Basin process activities (including the recently completed retrieval, washing, and packaging of the fuel) have further contributed to the sludge burden and altered the distribution reported in these prior characterizations.

While the SNF has been removed from the K Basins and placed into dry storage, the sludge remains under about 5 m^3 of water. Consequently, the focus is now on managing the sludge and decontaminating and decommissioning (D&D) the basins. The work discussed in this report was conducted by Pacific Northwest National Laboratory (PNNL), under contract to the Fluor Hanford K Basins Closure Project, to identify potential dryout mechanisms that may affect sludge behavior during storage and processing.

At the time testing was initiated, the plan for sludge management was to collect the sludge in large diameter containers (LDC) and store the containers at T Plant for a number of years before final processing and offsite disposal. The plan was revised in FY 2004 so that only ~6 m^3 of the less-radioactive sludge from the KE Basin will be transported in LDCs to T Plant. The remaining KE Basin sludge will be collected from the floor and pits into large freestanding containers located in the KE Basin pool. The sludge will settle and consolidate in these containers for a number of months before being transferred to the KW Basin and consolidated into large freestanding containers in the KW pool. The KW floor and pit sludge will also be consolidated into large containers. The sludge will be staged in these containers for up to 2 years before being removed for final treatment.

K Basin sludge samples sealed in screw-capped vessels (e.g., vials, jars) and stored in shielded hot cell facilities (hot cells) have been observed to lose supernatant liquid at unexpectedly high rates, causing a number of sludge samples to dry out. Re-hydrating the dried residual solid samples does not effectively restore the original physical properties of the sludge. Typically, the re-hydrated sludge maintains the hard clod consistency it acquires in drying and does not regain its finely particulate consistency. Consequently, concerns were raised about the ability to recover bulk quantities of sludge from containers if the sludge is allowed to dry out.

The laboratory tests described in this report were performed to obtain quantitative data on how the mass and volume of K Basin sludge, and its associated supernatant liquid, change with respect to storage time. For these tests, samples of K Basin sludge (all from the KE Basin) and a simulated (non-radioactive) K Basin sludge were loaded into glass graduated cylinders that were fitted with threaded plastic caps.

The loaded vessels were weighed and allowed to settle. The total volumes, settled sludge volumes, and weights of the vessels were recorded, with measurements taken more frequently during the initial periods when sludge volume change rates would be greater. At an intermediate time during compaction, the graduated cylinders and their contents were subjected to vibration using a laboratory vortex mixer. Measurements taken of the settled sludge volume before and after vibration (as might occur during overland transportation of containerized sludge) provided quantitative data of its effect on sludge consolidation. The laboratory testing followed the general requirements provide by the current Sampling and Analysis Plan (Baker et al. 2000) for K Basin sludge.

The sludge materials and experimental methods used in the dryout and compaction studies are described in Section 2.0. The results of these studies, including the effects of vibration, are presented in Section 3.0. Post-test evaluations of the sludge also are explained in Section 3.0. Appendix A presents the sample mass and volume data, and hot cell temperature and humidity values. Photographs of sludge samples before and after vibratory compaction are given in Appendix B.

2.0 Experimental Materials and Methods

The compaction and supernatant liquid evaporation rates and the resultant settled sludge densities for six K Basin sludge samples were determined over a 28-month period (May 14, 2002 to September 22, 2004). The samples were prepared from sludge materials taken from the KE Basin in prior sampling campaigns. A parallel sludge compaction and evaporation test with a simulated, non-radioactive, K Basin sludge (KB Sim) and an evaporation test with a KE Basin water sample (KB Sup) also were conducted over the same period. The KB Sim and KE Sup tests served as experimental controls for the KE Basin sludge testing. Previously unreported results from a separate 2-week test conducted in January 2004 on a related KE Basin sludge sample, taken from the KE North Loadout Pit (KE NLOP), are provided here for comparison and completeness.

All experiments, except the test with the KE NLOP sludge, were performed in the High Level Radiation Facility (HLRF) of the Radiochemical Processing Laboratory (RPL) in the Hanford 300 Area. The KE NLOP compaction test was conducted in an RPL glovebox. The experimental steps, defined in three Test Instructions approved by Fluor Hanford engineering and project personnel and cognizant scientists and managers at PNNL,(a) are discussed here. The KE Basin sludge and simulated sludge materials are detailed in Section 2.1, and experimental equipment and methods used in the compaction and evaporation tests are presented in Section 2.2.

2.1 Sludge Materials

Information on the sludge and supernatant liquids (KE Basin) used in this testing is provided in Table 2.1.(b) The sludge and supernatant liquid pH values given in Table 2.1 were measured with a calibrated wand-type pH meter in the hot cell (or glovebox in the case of KE NLOP) at the time the materials were loaded into the graduated cylinders for testing.

Table 2.2 shows the chemical and radionuclide concentrations of the sludge solids based on prior characterization testing. These data are expressed on a dry basis, because the total water quantities associated with the sludge samples in the present testing were not measured. Crystallographic phases in the sludge solids, identified by X-ray diffractometry (XRD), also are listed.

The pH of the sludge samples was measured both before and after testing. Although the pH changed little during testing, it is seen that the sludge samples with higher (≥59 wt%) uranium concentration are at pH ~5, while the samples with less uranium (<17 wt%) are at pH ~7-8. Figure 2.1 illustrates the correlation of pH with uranium concentration. The pH data, while not related to the compaction and evaporation testing, are useful in designing sludge handling, transport, and storage processes.

(a) Most experiments were conducted according to the Test Instructions, "K Basin Sludge Dryout Testing," TI-RPP-43262-T03 (April 2002), A. J. Schmidt, and "Addendum to K Basin Sludge Dryout Testing, Vibratory Compaction of Settled Sludge," TI-RPP-43262, Add-1 (August 2002), A. J. Schmidt. The KE NLOP settling test was performed according to Test Instruction, "Preparation of KE NLOP Composites and Samples," 46857-TI02, (December 2003), C. H. Delegard.

(b) Further descriptions of the origin and nature of the radioactive sludge materials are given in the internal memorandum, "Summary of Initial Laboratory Data from the Consolidated and Single Pull Core Sampling Campaigns," 01-SNF/RBB-004, from R. B. Baker and T. L. Welsh to K. L. Pearce, May 10, 2001, Fluor Hanford, Richland, WA; and by Poloski et al. (2002) and Pitner (1999). Properties of the simulated sludge material, KB Sim, are described by Schmidt and Elmore (2002).

Table 2.1. Sludges and Supernatant Liquids Used in Testing

Test Name	Original Sludge Sample		Description	pH	Additional Supernatant Liquid
	Name	Collection Date			
KB Sup	KC 2/3 Sup-1	4 Mar to 8 Apr 1999	KC 2/3 Sup-1 KE supernatant liquid	5.60	–
KB Sim	–	–	3.2% W/Co, 32% SS, 29% Kleen Blast, 18% flyash, 18% Min-U-Sil 40 with deionized water	–	DI water (pH 7.01)
FE-5	FE-5	13 Jan & 26 Apr 1999	KE Weasel Pit sludge	7.96	FE-5 (pH 7.96)
KC-2/3	KC-2/3 (Whole)	4 Mar to 8 Apr 1999	KE canister sludge composite	5.38	KC 2/3 Sup-1 (pH 5.60)
KC-4	KC-4 Dup L PG	30-31 Mar 1999	KE floor sludge from between canisters	7.80	KC 2/3 Sup-1 (pH 5.60)
KC-5	KC-5 L PG	29-30 Mar 1999	KE floor sludge from main basin	7.25	KC-5 Sup (pH 7.25)
96-13	96-13 SSOL	18 Apr 1996	KE canister sludge sample; sample had dried out and was crushed and re-wetted for testing	4.92	KC 2/3 Sup-1 (pH 5.60)
SNF Comp	SNF-Comp 60S-PG	4 Mar to 12 Apr 1999; also see text and Table 2.2	~64 vol% KE floor sludge, ~26 vol% KE canister sludge, ~7 vol% fuel corrosion product (U_4O_9), and ~3 vol% uranium fuel fragments	5.42	KC 2/3 Sup-1 (pH 5.60) plus deionized water
KE NLOP	KENLOP-Comp	13 & 19 Dec 2003	Composite of top-to-bottom core sample of KE North Loadout Pit sludge (Mellinger et al. 2004)	8.31	KE NLOP supernatant (pH 7.52)

Correlating radionuclide concentration to uranium concentration also is useful for estimating radiolytic, fissile material, and TRU (primarily ^{241}Am and ^{238,239,240}Pu) concentrations for sludge storage or process evaluations. The correlations found for the tested sludges are depicted in Figure 2.2 and presented in Table 2.3. It is seen that ^{154}Eu, ^{241}Am, the Pu isotopes, and the TRU concentrations correlate well, while the ^{60}Co and ^{137}Cs concentrations do not correlate well, with uranium concentration. The failure of ^{60}Co and ^{137}Cs to follow uranium is likely because they are relatively soluble and can escape the low solubility uranium oxide matrix. In contrast, Eu, Am, Pu, and the combined Am and Pu, as TRU, are much less soluble and remain with the uranium oxide. Once dissolved, the ^{60}Co and ^{137}Cs also can be collected by the ion exchange modules and by fugitive ion exchange materials lost in the K Basin sludge. The ^{60}Co and ^{137}Cs solubility and their subsequent loading onto ion exchange materials thus serve to alter their concentrations with respect to uranium in sludge. The association of ^{154}Eu, ^{241}Am, and Pu with uranium confirms observations made in prior studies (e.g., Makenas et al. 1996).

The ratios of ^{154}Eu, ^{241}Am, and the Pu concentrations to uranium concentration were used here to estimate the amounts of ^{154}Eu, ^{241}Am, and the Pu isotopes that the crushed irradiated metallic fuel contributed to the total inventory of the SNF Comp sludge sample. The presence of residual unreacted uranium metal in the SNF Comp sample at the start of the testing was evaluated. The original SNF Comp materials came from two prior gas generation tests (SNF + Floor 60L and SNF + Can 60L; Schmidt et al. 2003). These two tests originally contained 51.88 g of 0- to 6350-μm-diameter crushed irradiated fuel particles. Because the N Reactor fuel contains 7 wt% cladding, 93% of the 51.88 g of crushed fuel (i.e., 48.2 g) was estimated to be uranium metal. The quantities of hydrogen (arising from the reaction of uranium metal

Table 2.2. Compositions of KE Basin Sludge Solids Used in Testing

	Concentration, dry basis						
Analyte, wt%	**FE-5**	**KC-2/3**	**KC-4**	**KC-5**	**96-13**	**SNF Comp**	**KE NLOP**
Al	2.65	5.16	6.82	15.3	1.45	4.62	3.93
Ca	1.20	0.134	1.04	0.481	0.698	0.234	0.937
Fe	30.6	1.84	24.3	16.1	0.281	5.76	6.83
Mg	0.146	0.0462	0.330	0.177	0.190	0.0800	0.122
Na	<0.03	0.240	0.360	0.374	0.0430	0.177	0.0404
Si	0.329	0.752	4.91	5.46	Not analy.	1.61	36.3
U	5.32	59.0	16.6	6.36	82.0	60.2	2.51
Initial pH	7.96	5.38	7.80	7.25	4.92	5.42	8.31
Final pH	7.87	5.28	7.41	Sample lost	5.25	5.23	–
Analyte, µCi/g							
^{60}Co	0.875	0.442	1.09	1.10	<0.9	–	0.280
^{137}Cs	170	860	1680	1320	648	–	34.6
^{154}Eu	0.985	8.13	2.60	1.11	9.12	~9.1	0.542
^{241}Am	10.2	90.5	29.3	13.1	72.0	~104	7.82
^{238}Pu	2.06	16.2	4.91	1.99	–	~17	~0.28
^{239,240}Pu	13.1	113.8	39.2	13.1	110	~128	9.00
TRU	25.4	220	73.4	28.2	182	~240	17.1
Phases(a)	Q, L, Go	Ba, Gi, N, Sc, Ur	Not determined	Ba, Be, Gi, Go, N, Q, Sc (KC-5 P250 only)	Ur	Ba, Be, Bo, Gi, Go, N, Sc, Ur	Q, Z
Reference/Basis	Bryan et al. (2004)	(b)	Delegard et al. (2000)	Delegard et al. (2000)	Makenas et al. (1997)	(c)	Mellinger et al. (2004); Shelor et al. (2004); (d)

(a) Ba – bayerite [α-Al$(OH)_3$], Be – becquerelite [$Ca(UO_2)_6O_4(OH)_6{\cdot}8H_2O$], Bo – boehmite [γ-AlOOH], Gi – gibbsite [γ-Al$(OH)_3$], Go – goethite [α-FeOOH], L – lepidocrocite (γ-FeOOH), N – nordstrandite [β-Al$(OH)_3$], Q – quartz (SiO_2), Sc – schoepite [$UO_3{\cdot}2.25H_2O$], Ur – uraninite [$UO_{2.x}$], Z – zirconia (ZrO_2).

(b) Based on analyses presented in Delegard et al. (2000) for the separate KC-2/3 P250 and M250 (plus and minus 250-µm particle size) fraction analyses and the weight fraction of solids reporting to the two fractions (28% and 72%, respectively; Bredt et al. 1999).

(c) SNF Comp came from residues of gas generation tests “SNF + Can 60L” and “SNF + Floor 60L” reported in Schmidt et al. (2003). The “SNF + Can 60L” composite sludge was prepared from KC-1 (10.6 wt%), KC-2/3 M250 (69.8 wt%), and KC-2/3 P250 (19.6 wt%), settled sludge basis, and contained 66.98 g of sludge blend plus 26.36 g of crushed fuel particles. The “SNF + Floor 60S” composite sludge was prepared from KC-4 (41.1 wt%) and KC-5 (58.9 wt%) settled sludge, and contained 98.87 g of sludge blend plus 25.52 g of crushed fuel particles. The SNF Comp composition is based on analyses presented in Poloski et al. (2002) and Schmidt et al. (2003) and these assumptions:

- The fuel particles are 93 wt% uranium metal (based on results in Schmidt et al. 2003).
- ^{154}Eu, ^{241}Am, ^{238}Pu, and ^{239,240}Pu concentrations in the fuel (per mass of uranium) are the same as found in sludge because of the low solubility of these elements and their capture in the preponderant low-solubility uranium phases. The uranium-based concentrations of ^{60}Co and ^{137}Cs in the sludge and fuel may not be the same, however, because of the high water solubility of cobalt and cesium and the confounding effects of basin water ion exchange purification and losses of ion exchange media to the basin sludge. See text and Figure 2.2.

The phase assignments are based on analyses presented in Table 2.7 of Delegard et al. (2000) for post-test samples of KC-2/3 P250 and KC-5 P250 only, and thus may omit phases from other contributing sludge.

(d) ZrO_2 likely is present, based on its reported presence in the related sludge sample FE-3; “Summary of Initial Laboratory Data from the Consolidated and Single Pull Core Sampling Campaigns,” 01-SNF/RBB-004, R. B. Baker and T. L. Welsh to K. L. Pearce, May 10, 2001, Fluor Hanford, Richland, WA.

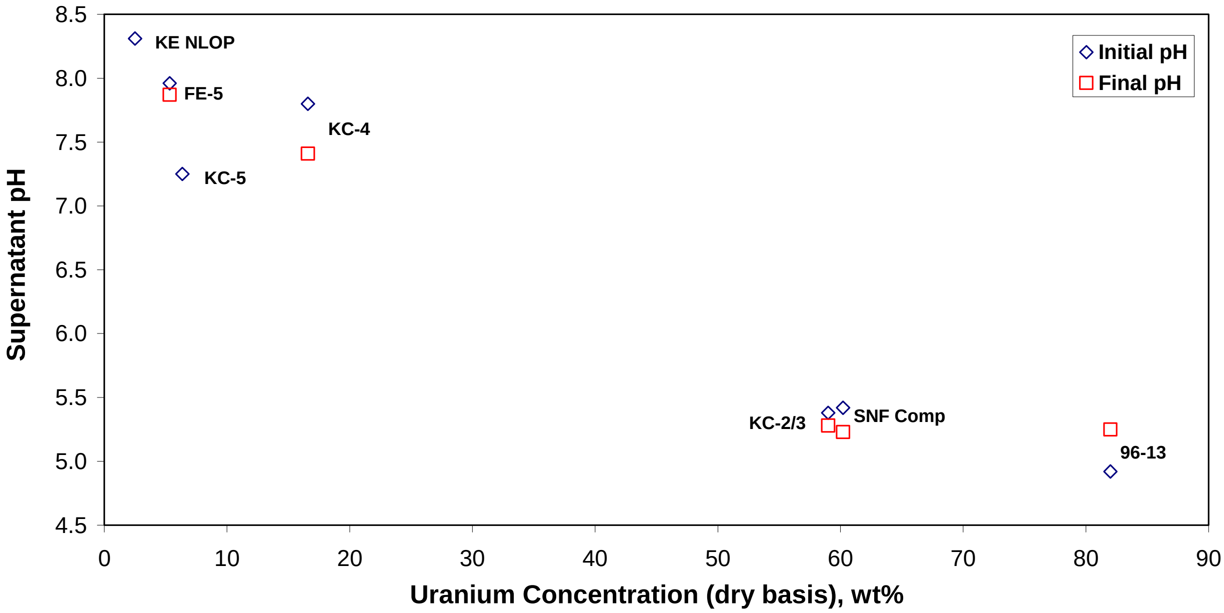

Figure 2.1. Correlation of Supernatant Liquid pH with Uranium Concentration in the Sludge

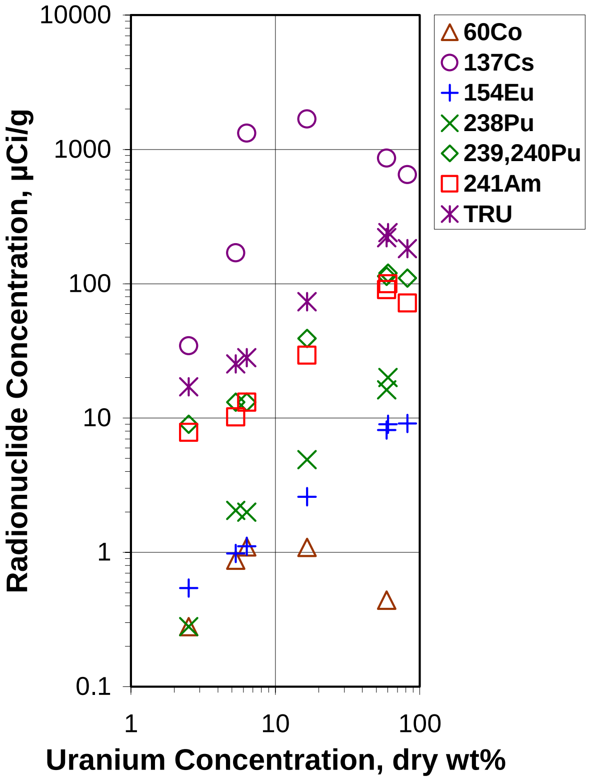

Figure 2.2. ^{60}Co, ^{137}Cs, ^{154}Eu, ^{241}Am, ^{238}Pu, ^{239,240}Pu, and TRU Concentrations as a Function of Uranium Concentration in the Sludge Solids (left to right, KE-NLOP, FE-5, KC-5, KC-4, KC-2/3, SNF-Comp, and 96-13)

Table 2.3. Radionuclide Concentrations with Respect to Uranium

Isotope	**µCi/g U**									
	FE-5	**KC-2/3**	**KC-4**	**KC-5**	**96-13**	**SNF Comp**	**KE NLOP**	**Average**	**Std. Dev.**	**Rel. Std. Dev., %**
^{60}Co	16.4	0.75	6.6	17.3	–	–	11.2	10.4	6.9	66
^{137}Cs	3200	1460	10120	20800	790	–	1380	6280	7890	126
^{154}Eu	18.5	13.9	15.7	17.5	11.1	15.0	21.6	16.2	3.4	21
^{241}Am	192	153	177	206	87.8	166	312	185	68	37
^{238}Pu	38.7	27.5	30.0	31.3	–	33.2	11.2	28.6	9.4	33
^{239,240}Pu	246	193	236	206	134	199	359	225	69	31
TRU	477	374	442	443	222	399	682	434	137	32

with water; $U + 2 H_2O \rightarrow UO_2 + 2 H_2$) and the gaseous krypton and xenon fission product isotopes (released when uranium metal corrodes) were measured during the two tests to determine the uranium metal quantities remaining in the sludge. The gas analyses showed that from 60% to 70% of the initial 48.2 g of uranium metal remained at the completion of the experiments in January 2001. According to SNF Databook kinetics (Duncan 2001) and the test conditions, the uranium metal particles to that point would have lost ~470 µm of their original diameter through corrosion. This means that particles originally smaller than ~470 µm would have been completely consumed.

The sludge/fuel mixtures from the gas generation testing were stored in the hot cell at ~33°C for the 15 months after the testing was completed until the present tests commenced in early May 2002. The uranium metal remaining in the sludge/fuel mixture would have continued to corrode to further decrease the residual particle diameters by an additional ~400 µm under anoxic conditions. However, under oxic conditions, uranium corrosion would have been about 1/30 to 1/50 as fast, and only ~10 µm of particle diameter would have been lost (Johnson et al. 1994; Hilton 2000). Therefore, it is certain uranium metal still remained in the sample SNF Comp by May 2002. The gas bubbles and voids subsequently observed provided evidence of residual uranium metal.

Similar evaluations can be made for the other KE Basin sludge samples used in the present tests, for example, KE canister sludge sample 96-13, which was retrieved in April 1996. While no assessment of uranium metal concentration was made, its high uranium concentration (82 wt%, dry basis) and origin indicate that significant metallic uranium may have originally been present. Sludge samples KC-2/3, KC-4, KC-5, and FE-5 were retrieved from the KE Basin early in 1999. Of these, gas generation testing showed that only KC-2/3 contained significant uranium metal (Delegard et al. 2000; Bryan et al. 2004). The KC-2/3 settled sludge sample contained ~1.9 wt% uranium metal when tested (as separate >500-µm and <500-µm fractions) from October 1999 until May 2000, and the uranium corrosion could be modeled as arising from nominal 800-µm-diameter uranium metal particles. The KE NLOP sludge, sampled in December 2003, contained negligible uranium metal according to gas generation testing (Mellinger et al. 2004).

The KC-2/3, KC-4, KC-5, and FE-5 samples were stored continuously underwater in the hot cell at ~33°C for the 38- to 41-month period between sampling and the present tests. The 96-13 sample was kept underwater for most of its 73-month hot cell storage period, but was found in a dry condition prior to the present testing. The corrosion rate of uranium at 33°C in anoxic water is 0.0237 µm/hour based on SNF Databook kinetics (Duncan 2001). The corrosion depth at this rate would be ~690 µm in 40 months (i.e., uranium metal particles smaller than ~1380 µm in diameter should have been completely consumed after the anoxic hot cell storage) and ~1250 µm in 73 months. No uranium metal would be expected to remain in the original uranium metal-bearing KC-2/3 sample or in the older 96-13 sample if anoxic corrosion had occurred over the entire storage interval. As noted above, corrosion rates are 1/30 to 1/50 as rapid if oxic conditions prevail. Under such conditions, as little as 14-μm to 25-μm depth of corrosion might have occurred, with nearly all of the original "nominal" 800-μm-diameter uranium metal surviving.

The presence of uranium metal in the KC/4, KC-5, FE-5, and KE NLOP sludge at the commencement of the present testing is unlikely; if present, the concentration was negligibly small. The presence of residual uranium metal in the KC-2/3 and 96-13 sludge cannot be confirmed or excluded. However, the only evidence of uranium metal in these samples in the present tests was a single observation of bubbles in the 96-13 sludge.

2.2 Experimental Methods

Experiments were performed to determine the settling and long-term compaction behavior of various KE Basin sludges, the corresponding rates of the settled sludge density increase, and the rates of water loss. All tests were performed in the HLRF hot cell facilities, except the tests with the KE NLOP sludge, which were conducted in a glovebox. The methods used in the two experiment types are described. Most of the sludge samples had previously undergone short-term settling tests during initial characterization and thus provide some comparative settled sludge density data.

2.2.1 Tests in the Hot Cells

The tests conducted in the hot cell used Kimble/Kontes class B glass 250-ml ("to contain") glass graduated cylinders, model number 482600-0250 (Figure 2.3). The cylinders were fitted with phenolic threaded plastic caps having flat polytetrafluoroethylene (PTFE) linings. The approximate dimensions of the screw-top graduated cylinders were 32.5 cm high, 3.8-cm outer diameter, and 3.5-cm inner diameter.

Sludge samples stored in hot cell facilities are kept in glass vials and jars with screw-top closures. The vials generally are 20- or 35-ml capacity and have polyethylene cone-shaped cap liners. Jars (250- or 500-ml) are used for larger-volume samples and have wide-mouth caps with flat PTFE cap liners.

Figure 2.3. Graduated Cylinder and Black Phenolic Cap Used in Evaporation and Compaction Tests. Note: for comparison, white cap and conical liner used to seal 20-ml and 35-ml sample vials.

The 250-ml graduated cylinders were loaded with 100±20 ml of settled sludge, and the cylinder walls were washed with the selected supernatant liquid to remove any coating of sludge solids (see Table 2.1). The KB Sup test had supernatant liquid only (no sludge solids). The sludge solids in the remaining tests were allowed to settle for 7 days beginning May 7, 2002. Excess supernatant liquid was withdrawn by suction on May 14, 2002, to leave approximately 20 ml (ranging from 17 to 21 ml) of clear supernatant liquid in each cylinder. As shown in Table 2.4, at the time of supernatant liquid removal, the settled sludge volumes ranged from 79 to 120 ml (i.e., 8.2- to 12.5-cm depth). The KB Sup test had 117 ml of liquid with no sludge. After the supernatant liquid was withdrawn, the samples were tightly capped, the sludge and solution volume levels recorded, and the closed graduated cylinders weighed. Lead-rings were placed around the bases of the graduated cylinders to help keep them upright during hot cell operations. The cylinders were re-weighed and the sludge/liquid mixtures allowed to settle.

The samples were carefully re-weighed (minimizing undue agitation), and the levels of the settled sludge and supernatant liquid measured over the 28-month test period. The values were recorded in the test instructions. Measurements were taken more frequently in the first few months when sludge volume change rates would be greater. Readings of the hot cell temperature and relative humidity also were recorded. As shown in Figure 2.4, temperatures ranged from 31°C to 38°C, and the relative humidity ranged from 30% to 41%; the average observed temperature was 34°C, and the average relative humidity was 35%. These temperatures are typical of hot cells where the worker operating gallery air is drawn into the hot cells and warmed by the strong in-cell lighting.

Table 2.4. Sludge and Total Volumes for Compaction Tests in the Hot Cells

Sludge	Sludge Volume, ml	Total Volume, ml
KB Sup	0	117
KB Sim	120	140
FE-5	95	116
KC-2/3	97	116
KC-4	103	121
KC-5	107	128
96-13	101	118
SNF Comp	79	100

The effect of vibration on sludge compaction was tested on August 21, 2002, after 112 days of settling (105 days of observed water loss). On that date, the sludge-bearing samples in their graduated cylinders were agitated on a laboratory vortex mixer.[a] The graduated cylinders were held upright in a ring stand

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[a] To simulate the vibrations induced during transportation, agitation that provided vertical action at 30 Hertz would have been ideal. However, no such mixer was readily available. Instead, a laboratory vortex mixer (VWR brand Mini Vortexer, Variable Speed Control, VWR catalog no. 58816-121) was used. The mixer provides vigorous swirling action, but with little vertical vibration, and much higher oscillatory frequency than produced by a bouncing truck-bed. To determine the suitability of the vortex mixer, preliminary cold testing with a K Basin sludge simulant was performed. The simulant was the same as used for a large-scale settling test by Schmidt and Elmore (2002). In the cold testing performed here, a graduated cylinder containing settled simulant was placed on the vortex mixer for 10 min (speed set at 5, or roughly 1800 rpm). After mixing, the sludge was allowed to settle overnight. The consolidated sludge volume was found to be 15% less than the original settled sludge. This degree of settling is approximately the same as was observed by Schmidt and Elmore (2002) after overland transport of a ~1-ft-tall and 1-ft-diameter column of settled sludge in the back of a truck. The tests performed here indicated that the vortex mixer would provide a suitable level of agitation to compact/consolidate the sludge samples.

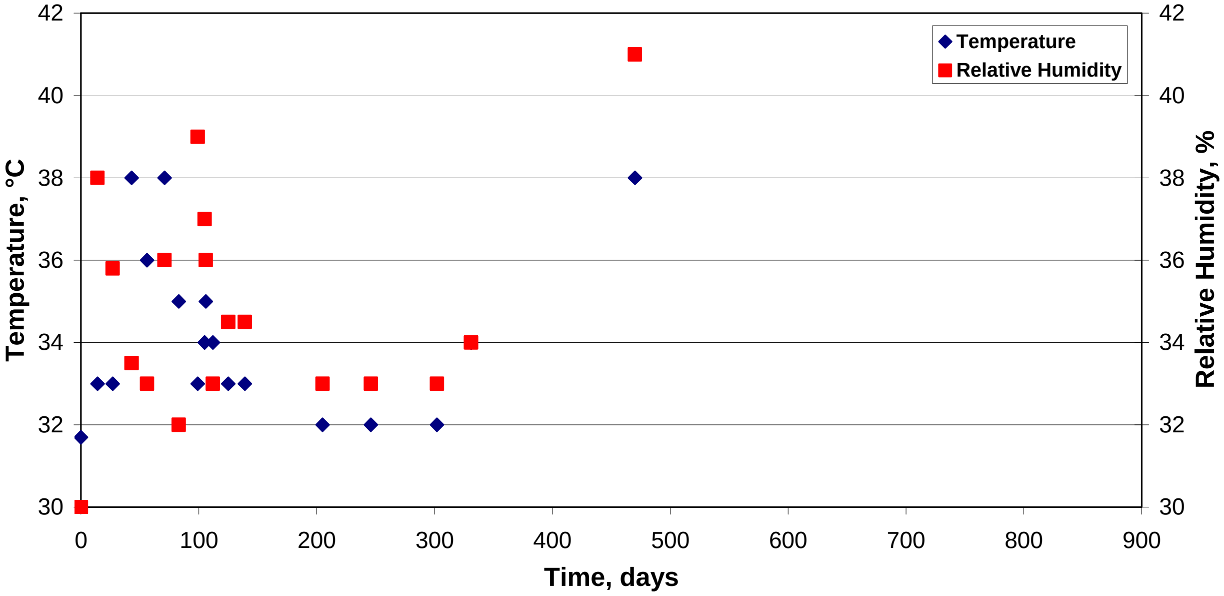


Figure 2.4. Temperature and Relative Humidity in Hot Cell Tests

with the base resting on the mixer. Each sample was agitated for 12 min, but at intensities that varied from sample to sample according to their resistance to movement; agitation vigor was increased until the sludge became visibly suspended. Observations were made during the agitation and afterwards while the sludge re-settled.

About 1 month later (September 30, 2002; i.e., after 146 days of settling and 139 days of observed water loss), sample SNF Comp was opened so that about 3-5 ml of supernatant liquid could be removed for a chemical analysis in a different study. To minimize agitation, the clear liquid was withdrawn by suction using a transfer pipet. K Basin supernatant liquid was added to replace the water taken by sampling, and the cap was re-attached. At this time, it also was noted that the graduated cylinder for KC-5 had broken, forcing the experiment with that sample to be terminated.

At 338 days of settling (331 days of water loss), new caps were placed on the graduated cylinders for all samples. The caps were exchanged to determine if new caps, or their installation on the graduated cylinders, would improve the integrity of the seal as measured by water loss rates. At the same time, water was added to all samples, except FE-5 and KE Sup, to replace water lost by evaporation and to keep the sludge completely wetted for the balance of testing. Because sample FE-5 had lost little water and sample KB Sup contained water but no sludge, they did not need additional water.

Settling continued undisturbed, except for occasional weight and associated water and sludge level measurements, until September 22, 2004, or for 869 days total of settling (862 days of water loss). Twenty observations of sludge and total (sludge plus supernatant liquid) level and total mass were made for most cylinders over the entire test period. The evaporation rate of water from an uncapped 250-ml graduated cylinder subsequently was measured in a separate week-long hot cell test. The summary data for all tests are provided in Appendix A.

2.2.2 Sample KE NLOP in the Glovebox

For the test with the KE NLOP sludge sample, 50 ml of well-stirred, settled composite KE NLOP sludge were added to a tare-weighed 250-ml glass graduated cylinder, and the cylinder was re-weighed. This graduated cylinder had a pour-spout top instead of a threaded cap top as used in the hot cell tests. The cylinder was filled to the 250-ml mark with supernatant liquid taken from the contributing KE NLOP samples and again weighed. The cylinder top then was sealed with paraffin film; the cylinder and contents inverted and shaken; and the cylinder set on its base to allow the solids to settle.

The levels of settled solids were recorded periodically from January 6 to 21, 2004 (15 days total), but with greater frequency at first when settling was most rapid. The relatively cool glovebox temperature during testing (9°C) corresponds to typical K Basin temperatures. The summary level and mass data are given in Appendix A.

3.0 Results and Discussion

The compaction rate, evaporation rate, and settled sludge density results for the hot cell tests are presented in Section 3.1. The KE NLOP sample test results are discussed in Section 3.2.

3.1 Hot Cell Evaporation and Compaction Tests

Tests conducted for the six KE Basin sludge materials and the simulated sludge were monitored for weight, settled sludge, and total (sludge plus supernatant liquid) volumes. The weight loss data show the evaporation rate of the supernatant liquid under hot cell storage conditions, while the settling rate data provide the sludge compaction kinetics. The settled sludge density was calculated based on the settled sludge volume data, the associated weight data, and the fact that the supernatant liquid has negligible dissolved salt and, thus, has the density of water.

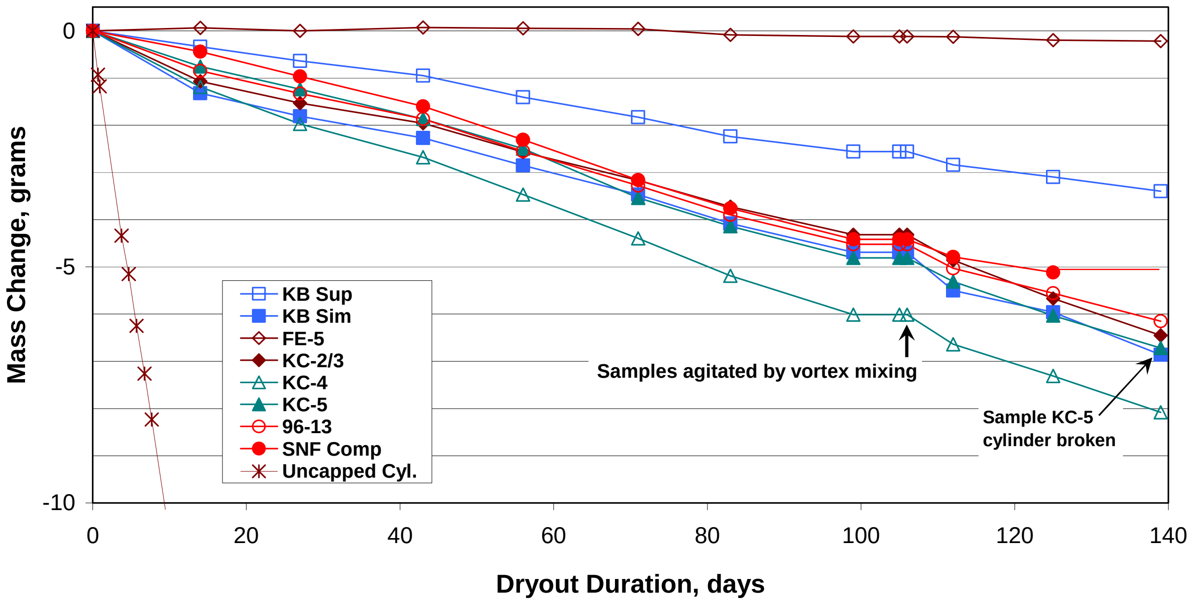
3.1.1 Water Evaporation Observations

The purpose of the evaporation rate testing was to help determine the cause of the seemingly high water loss rates observed for sludge samples held in the laboratory hot cells. Causes of high evaporation rates might include the nature of the material (e.g., radioactive or non-radioactive materials, sludge or liquid), atmospheric pressure fluctuations, or poor sealing of the sample vessels (glass graduated cylinders) by the screw-caps. The effects of these potential causes of high water loss were examined. To understand the benefit gained by capping, the evaporation rate from an uncapped graduated cylinder also was measured and compared with rates that would be expected under temperature and humidity similar to those found in the hot cell.

The test results showed that water losses from K Basin sludge samples being kept in capped graduated cylinders under hot cell conditions (about 34°C and 35% relative humidity) appear to be most affected by the integrity of the seal. Little difference in water loss rates was noted between graduated cylinders containing water or sludge plus water; or between radioactive and non-radioactive sludge. The water loss rates ranged by a factor of ~50, from about 1.5×10^{-4} to 6.9×10^{-3} cm of water depth per day (average 3.7×10^{-3} cm/day). At these rates, a sludge sample covered by a 1-cm layer of water would expose settled solids to air after 5 months to 18 years (9-month average). In comparison, water loss from a similar, but uncapped, graduated cylinder was $\sim1.1\times10^{-1}$ cm of water depth per day; 1-cm depth would be lost in 9 days. Capping lowered the water evaporation rates under still-air conditions by about 30-fold. However, the average water loss rates observed for the capped graduated cylinders was about 1800 times greater than would be caused solely by fluctuations in atmospheric pressure. Water loss rates increase with storage time, serving to diminish the projected time to the onset of drying of the settled sample solids. Irradiation in the hot cell field or vibrations may have caused the sealing degradation.

The results of water loss testing are discussed below.

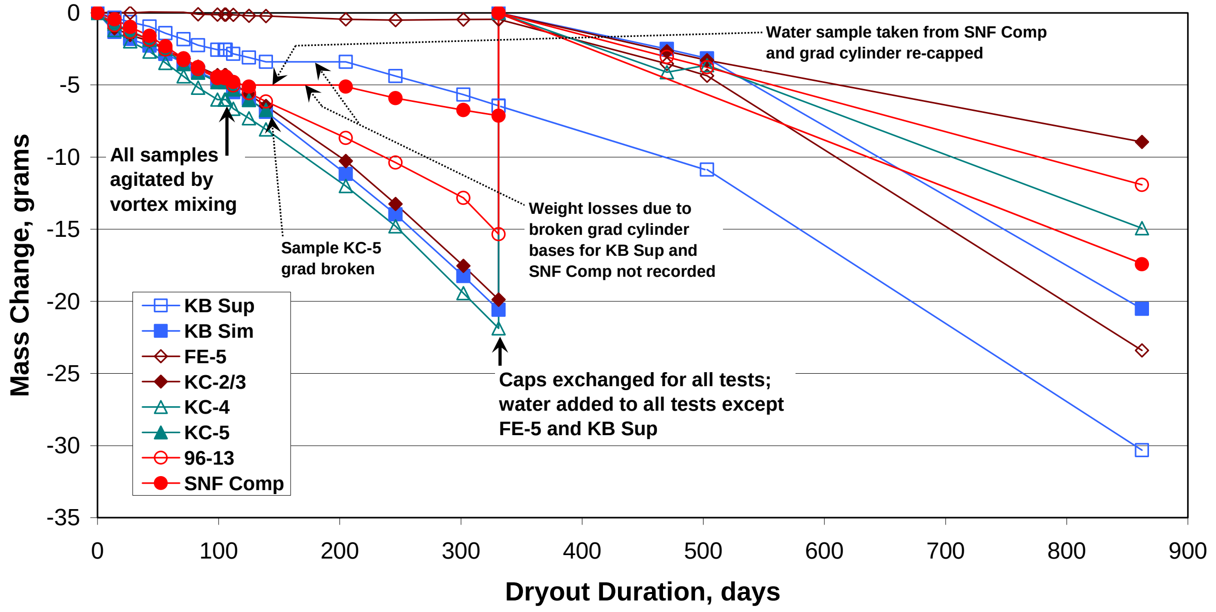
The rates of water loss of the sludge samples, the supernatant liquid, and the simulated sludge are compared in Figure 3.1 for the first 139 days of testing (where similar weight loss data were observed for six of the eight samples). Results for water loss from the uncapped graduated cylinder also are shown in Figure 3.1. Results over the entire test interval are given in Figure 3.2. Water loss rates in the first 331 days ranged from 0.0014 g/day (for sample FE-5) to 0.066 g/day (for sample KC-4), a difference of a



Samples agitated by vortex mixing

Sample KC-5 cylinder broken

Figure 3.1. Water Loss for KE Basin Sludge and Supernatant Liquid Over the Initial 139 Days



Water sample taken from SNF Comp and grad cylinder re-capped

All samples agitated by vortex mixing

Sample KC-5 grad broken

Weight losses due to broken grad cylinder bases for KB Sup and SNF Comp not recorded

Caps exchanged for all tests; water added to all tests except FE-5 and KB Sup

Figure 3.2. Water Loss for KE Basin Sludge and Supernatant Liquid Over the Entire Test Interval

factor of ~50. Based on the ~9.6-cm^2 water surface area in each graduated cylinder, these rates correspond to the loss of ~1.5×10^{-4} to 6.9×10^{-3} cm of water depth per day. Overall, 218.99 g of water were lost from all eight samples (note that the graduated cylinder for KC-5 was broken after 139 days, and this experiment had to be terminated early). Therefore, the average weight loss was 0.035 g/day or 3.7×10^{-3} cm of water depth per day; the average water loss over the entire 862-day test interval for a single experiment was 30.6 g. The average water loss rate observed from the capped graduated cylinders was about 30 times lower than the 1.07 g/day water loss rate (~1.1×10^{-1} cm/day) observed for an uncapped graduated cylinder under otherwise similar hot cell storage conditions.

The water loss rates may be compared with water evaporation rates that would be expected at the existing hot cell temperature and humidity in quiescent air. The following empirical relationship (Equation 42 from Penman 1946) is valid for water evaporation into air from open water vessels with low freeboard walls:

$$E, \text{mm/day} = 0.44\,(1 + 0.27\,u_0)\,(e_s - e_d) \tag{1}$$

In the above equation, E is the evaporation rate in mm/day; u_0 is the air velocity in miles per hour (assumed to be zero at the water surface in the graduated cylinder); e_s is the vapor pressure of water, in Torr, at saturation (e.g., 40 Torr for the 34°C hot cell temperature); and e_d is the observed vapor pressure of water (35% of 40 Torr or 14 Torr in the hot cell). According to this equation and at the conditions maintained in the hot cell, the water evaporation rates from an open low-walled vessel would be about 1.1 cm/day. The calculated water loss rate due to simple evaporation thus is about a factor of 300 greater than is observed, on average, for the capped graduated cylinders.

At the same time, the calculated water evaporation rate is 10 times greater than is observed for the uncapped cylinder. A decreased rate is observed even though the high air currents in the hot cell would be expected to aid water evaporation. The ~15-cm height of the graduated cylinder above the water level likely helps decrease the water evaporation by preserving a zone of relatively water-saturated air above the water surface. The caps, therefore, decrease the water evaporation rates by a factor of ~300 compared with what would be expected for uncapped cylinders filled to the brim. On average, however, capping the graduated cylinders decreased water loss rates by a factor of 30.

Natural atmospheric pressure fluctuations and gas leakage past the cap seals also were considered as possible mechanisms for water loss. The fluctuations would cause water-saturated air (100% relative humidity) to leave the graduated cylinder vapor space when atmospheric pressure dropped and to be replaced by air at the 35% relative humidity of the hot cell when the pressure increased. Because pressure fluctuations within the hot cell were not recorded, atmospheric pressure changes recorded at the Hanford weather station were examined for the period of May 2002 to September 2004.[a]

The atmospheric pressure values registered hourly at the Hanford weather station are plotted in Figure 3.3. The total hour-to-hour pressure drops and gains were summed separately for the 862-day test interval and were found to be 114.9 inches of mercury (1 atm = 29.92 inches of mercury). The vapor head space in the graduated cylinders is about 180 ml (total volume about 300 ml with about 120 ml occupied by sludge and supernatant liquid as shown in Table 2.1). Thus, about (114.9/29.92 × 180 ml =) 691 ml of water-saturated air, with water vapor pressure of 40 Torr, would be vented from each graduated cylinder, on average, over the test period and be replaced with 34°C air at 35% relative humidity (14 Torr water vapor pressure). Over the 862 days of the testing, about 0.00094 moles or 0.017 g of water would be lost in this manner.

$$\Delta n(H_2O) = \frac{\Delta PV}{RT} = \frac{\dfrac{40-14\ \text{Torr}\ H_2O}{760\ \text{Torr/atm}} \times 0.691\,\text{l}}{\dfrac{0.08206\ \text{l atm}}{\text{mol K}} \times 307\ \text{K}} = 9.39 \times 10^{-4}\ \text{moles}\ H_2O \tag{2}$$

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[a] Hanford weather station atmospheric pressure records were provided by Kenneth Burk of PNNL. Actual pressure fluctuations within the hot cell likely would track the observed atmospheric pressure fluctuations with additional small perturbations due to ventilation air balance spikes (e.g., doors opening and closing) in the RPL.

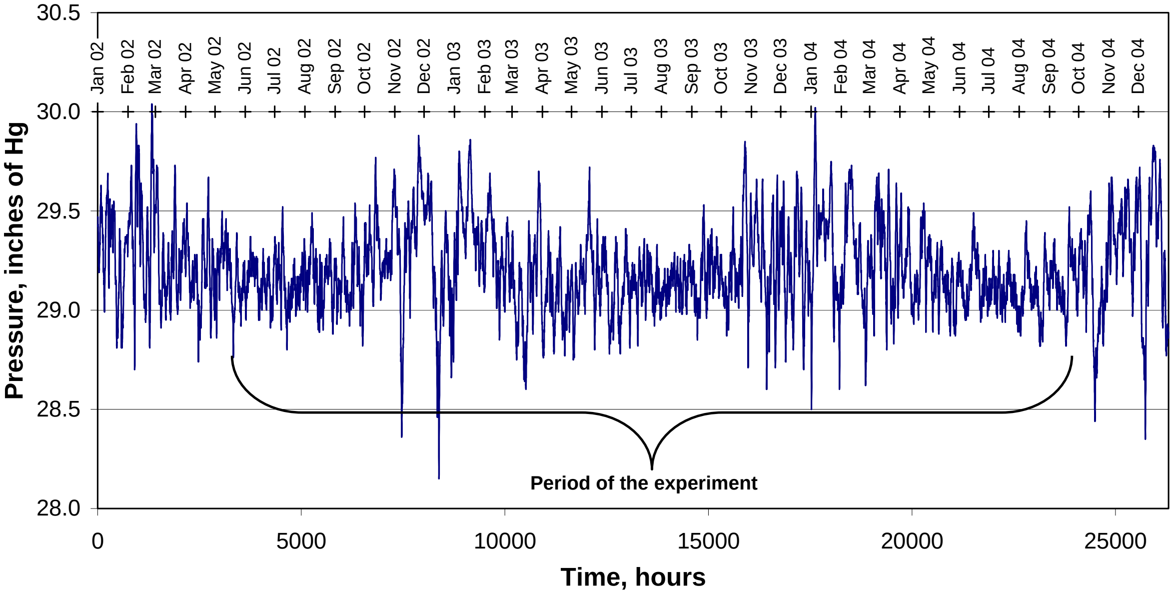

Figure 3.3. Atmospheric Pressure Registered at the Hanford Weather Station, 2002-2004

This amount is far less (by a factor of 1800) than the 30.6-g average water amounts lost in testing and is even less than the lowest observed loss rates for the initial period of test FE-5, when about 0.45 g of water was lost in the first 331 days. Therefore, the atmospheric "breathing" phenomenon produces an effect far too small to explain the entire amount of water loss observed in the present tests.

The effect of the sample materials on water loss rate also was examined. The water loss rates, however, did not appear to depend on whether the material was radioactive (i.e., actual sludge) or a non-radioactive simulant, or even if sludge was present. Thus, it is seen that the KB Sim sludge sample, which contained no radioactive constituents, lost water at a rate near those observed for the KC-4 and KC-2/3, which are among the more radioactive sludges tested (see Table 2.2). In addition, the KB Sup sample, which contained no sludge, lost water at a rate that was bracketed by rates observed for tests of samples containing sludge.

As shown in Figure 3.2, the water loss rates increased for nearly all of the tests as time proceeded. For example, the water loss rate for sample KC-4 was 0.063 g/day in the first 83 days of the test and was 0.083 g/day in the 84-day interval (from 247 to 331 days) just before the caps were exchanged. Loss of sealing integrity with time, perhaps due to radiolytic degradation of the PTFE cap insert or to vibrational loosening of the cap, would explain the increased water loss rate. Although no influence of material (and its contributed radiation) on water loss rates was found, the experiments were conducted in hot cell facilities having ~5 Rad/hour of background radiation. However, no definitive explanation for the generally increasing rate of water loss with time can be offered based on the experiments with capped graduated cylinders.

In contrast to the water loss rate observations of most of the other test cylinders, the water loss rate for the SNF Comp sample decreased with time. The rate was 0.041 g/day over the first 125 days of testing, but decreased to 0.016 g/day in the 126-day period from 205 to 331 days. However, in this test the cap was removed at 139 days, a water sample withdrawn, and the cap re-attached. The lower water loss rate after sampling likely was because the cap was attached more securely after sampling than before sampling.

The variable influence of the sealing integrity on water evaporation rates explains the observation that evaporation rates changed for most tests following the cap replacements at 331 days. The evaporation rates increased for some samples (e.g., FE-5) and decreased for others (e.g., KB Sim). Finally, the variable sealing integrity explains the widely varying (factor of 50) water loss rates observed for the various test cylinders and test intervals.

3.1.2 Sludge Compaction Rate and Density Observations

Settled sludge and total (sludge plus supernatant liquid) volume and sludge mass data were recorded over the 862-day hot cell tests. These data showed that the densities of the one simulated sludge and the six KE Basin sludge types varied little as a function of time, that most of the sludges compacted little after the initial 7 days of settling, and that most sludge densities were little affected by agitation at an intermediate (112-day) settling time. The SNF Comp sample was the exception and showed considerable evolution of gas during the compaction tests such that a gas void, with an overlying plug of settled SNF Comp sludge that rose with time, was observed in the graduated cylinder. The 96-13 sample showed one instance of gas bubbles on the liquid surface during the present testing.

The gas bubbles and gas void formation, and their effects on settled sludge density, are explained by the presence of residual uranium metal in the SNF Comp, and perhaps 96-13, sludge samples. Uranium metal corrodes in water to produce hydrogen gas ($U + 2\ H_2O \rightarrow UO_2 + 2\ H_2$). As discussed in Section 2.1, the uranium metal particles in the SNF Comp sludge, originally ranging in particle size from 0 to 6350-μm, would have been expected to lose no more than 900 μm of diameter during prior corrosion testing and wet storage in the hot cell. Therefore, particles originally greater than about 900 μm in diameter would have remained in the SNF Comp sludge and thus be available to react with water to produce hydrogen gas. As has been observed in prior sludge settling tests (see, for example, Figures 3.10 and 3.11 of Makenas et al. 1997), hydrogen gas generated by uranium corrosion can lift settled sludge plugs upward in 2-liter graduated cylinders of even greater diameter (~8 cm) than the 250-ml cylinders (~3.5-cm diameter) used in the present testing.

The results of the sludge compaction testing are discussed below.

The densities of sludge samples in the 28-month hot cell compaction tests are presented in Figure 3.4. The times reflect the initial 7-day settling period in which no sludge level data were recorded. It is seen that, for most sludges, the density changed little from the initial measurements after 7 days of settling until the final measurements at 869 days. Settled sludge densities generally were found to increase with increased uranium concentration in the dry solids (Figure 3.5). It also was noted that agitating the sludge with the vortex mixer had only small to negligible effects on most settled sludge densities. However, as seen in Figure 3.4, sharp changes in settled sludge level and density were found for the SNF Comp sample after agitation. As discussed later, these changes are attributed to gas bubbles released by the agitation.

The maximum densities of the settled sludge are given in Table 3.1 and compared with values obtained from prior testing of the same sludge materials. This comparison shows that most densities found in the present study were near or greater than those observed in earlier tests. The exception is SNF Comp, which had a markedly lower density. The greater settled sludge densities found in the present tests may be a result of the longer settling times and the greater depth of sludge solids. The greater sludge depth (8.2 to 12.5 cm in the present tests compared with 2 to 5 cm in prior tests) would compact the underlying sludge and lead to higher density.

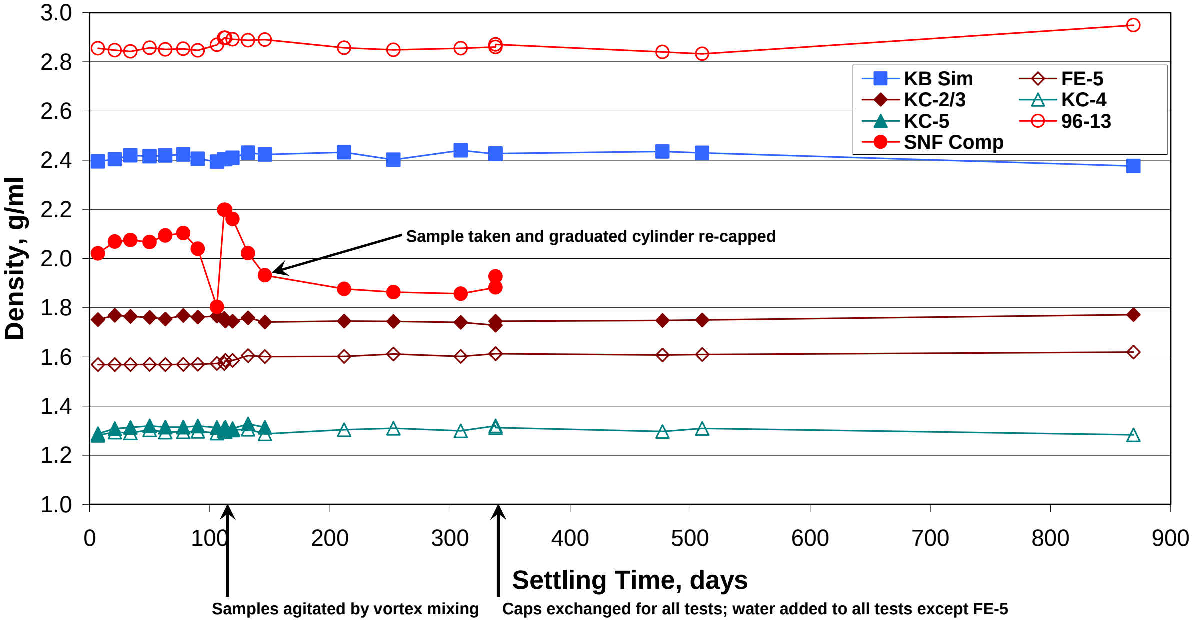

Figure 3.4. Sludge Density as a Function of Settling Time

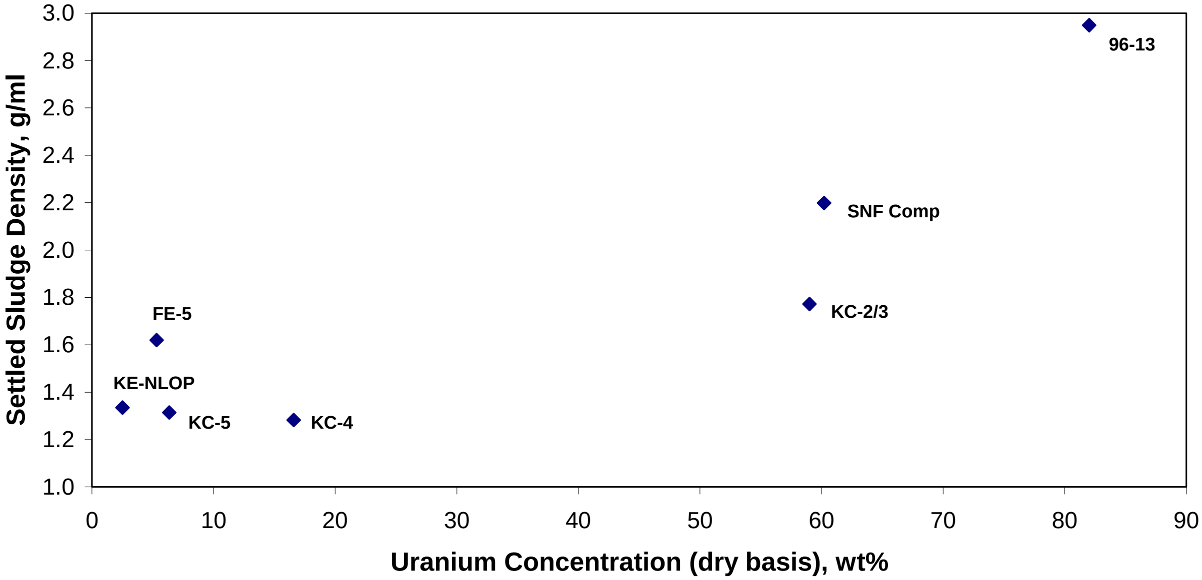

Figure 3.5. Final Settled Sludge Density as a Function of Uranium Concentration

During testing with the vortex mixer, each sample was agitated for 12 min, but different speed settings were used according to the resistance of the sludge to mixing. Observations of the contents during this mixing are given in Table 3.2. Samples were observed 4 to 6 hr after agitation and found to have clear supernatant liquids. See Appendix B for images of the sludge samples before and after agitation. Sludge and supernatant liquid volumes were recorded 4 to 6 hr after agitation with subsequent readings taken ~23 hr and ~94 hr after agitation. The settled sludge volumes before mixing and for the 94 hr after mixing are shown in Figure 3.6.

Table 3.1. Settled Sludge Densities

Sludge	KE Sludge Origin	U, wt% (dry)	Density, g/ml			% Difference(b)
			Prior Studies(a)	This Study		
				1 Week	Final	
KB Sim	Simulated sludge	0.0	2.24	2.39	2.38	+6.2
FE-5	Weasel Pit	5.32	1.47	1.57	1.62	+10.2
KC-2/3	Canister sludge composite	59.0	1.30	1.75	1.77	+36.2
KC-4	Floor sludge between canisters	16.6	1.26	1.28	1.28	+1.6
KC-5	Floor sludge from main basin	6.36	1.28	1.29	1.31	+2.3
96-13	Canister sludge that had dried in prior storage	82.0	2.67	2.86	2.95	+10.5
SNF Comp	Floor, canister, and crushed fuel mix	60.2	2.32	2.02	2.20	-5.2
KE NLOP	North Loadout Pit	2.51	1.35	1.33	1.34 2 weeks	-0.7

(a) Data for KB Sim are from Schmidt and Elmore (2002). Data for all other sludges are from Poloski et al. (2002). Note that the value used for KE NLOP density, from Table S.1 (Poloski et al. 2002), is for FE-3, a composite taken from the KE North Loadout Pit main pit and channel; the KE NLOP sludge sample was taken from the main pit only (Mellinger et al. 2004). Note also that densities for KC-2/3 fractions less-than and greater-than 250 μm were 2.13 and 2.11 g/ml, respectively (Bredt et al. 1999).

(b) % Difference in the final settled sludge density in the present tests compared with the density found in prior testing; $\% \text{ Difference} = \dfrac{\text{Final density (this study)} - \text{Density (prior studies)}}{\text{Density (prior studies)}} \times 100\%$

Table 3.2. Final Settled Sludge Densities and Observations After Agitation

Sludge	Vortex Mixer Speed Setting and Mixing Time	Observations
KB Sim	2 (0-1 min); 3 (2-4 min); 3.5 (4-12 min)	Supernatant liquid layer became slightly cloudy and individual particles observed to vibrate above the settled white solids
FE-5	1 (0-12 min)	Light brown milkshake color and consistency; upper third appeared slightly darker brown
KC-2/3	3 (0-12 min)	Difficult to mix; supernatant liquid layer turned cloudy
KC-4	2 (0-12 min)	Good mixing to get light brown color
KC-5	1 (0-12 min)	Light brown milkshake color and consistency
96-13	1 (0-1 min); 2 (3-7 min); 5 (7-12 min)	Lumpy sludge with light and dark bits against cylinder wall; good mixing did not occur even with mixer setting of 5
SNF Comp	1 (0-~6 min); 1.2 (~6-12 min)	"Aqueous void" seen at 10-20 ml volume level (sludge above and below the void); void disappeared after mixing

Changes in settled volumes for most of the sludge samples were 2 ml or less following the agitation and mixing. Samples KB Sim, 96-13, KC-4, and FE-5 compacted by 1.5 to 2.0 ml in the first 4 days after agitation. This additional compaction is equivalent, at the total ~100-ml settled sludge volume, to a ~1.3-2% increase in sludge density. Samples KC-2/3 and KC-5 showed no change in settled volume after agitation, and KC-5 apparently had higher settled volume (lower density) just after agitation before

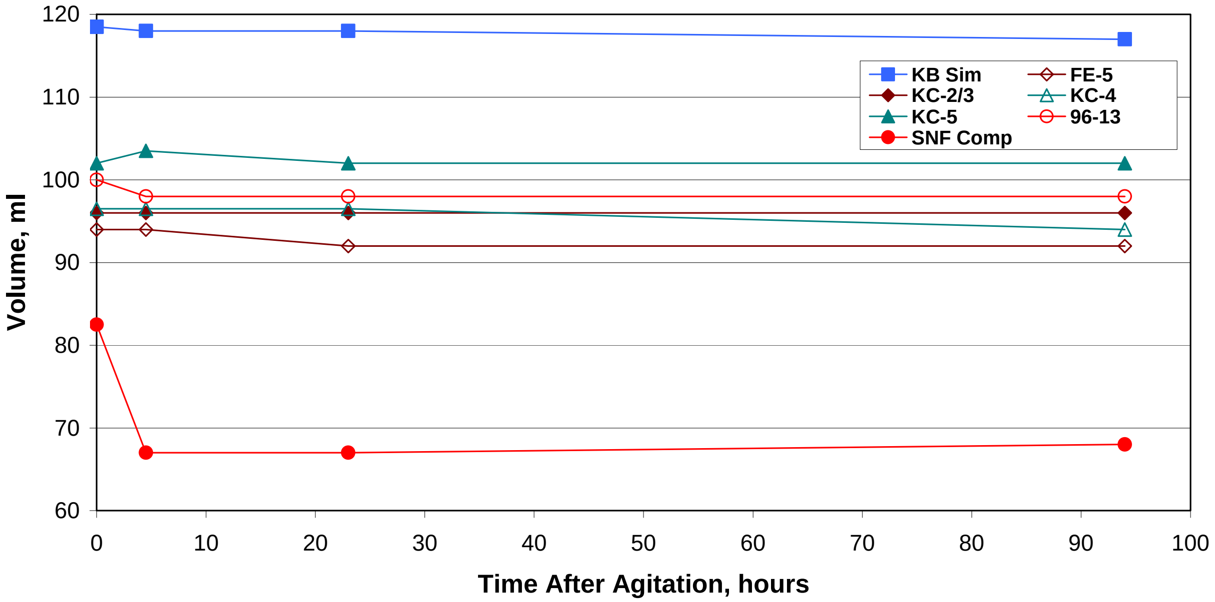

Figure 3.6. Settled Sludge Volume Before and After Agitation

settling to its original volume. However, agitation instigated an abrupt 15.5 ml (19%) drop in settled sludge volume for the SNF Comp test. This drop in sludge volume suggested that the "aqueous void" seen between the 10-ml and 20-ml level before agitation (as noted in Table 3.1 and shown in Appendix B) actually was a gas bubble and that the gas bubble was released by the agitation.

Evidence for gas formation within the SNF Comp sample is found in its anomalous changes in settled sludge density when compared with the other sludge samples. The change in sludge densities with time can be seen in Figure 3.4. It is seen that the density of SNF Comp increased up to 78 days settling time (i.e., until July 24, 2002). On this date of maximum sludge density, bubbles were observed on the surface of the supernatant liquid. From 78 days until the samples were agitated at 113 days (August 27, 2002), the SNF Comp sludge density decreased from about 2.10 g/ml to 1.80 g/ml. The settled sludge density peaked at 2.20 g/ml when measured 4.5 hr after agitation, but gas production evidently continued such that the apparent sludge density quickly decreased, ultimately reaching about 1.86 g/ml on March 12, 2003, and 1.90 g/ml on April 10, 2003.

A void within the SNF Comp settled sludge sample again was observed beginning August 27, 2003, after 477 days of settling. The void existed until the end of testing, preventing meaningful measurements of sludge volume and density from August 27, 2003, onward. The void observed on August 27, 2003, spanned the 30-ml to the 110-ml level of the graduated cylinder (see Figure 3.7 for the appearance on the following day).

The void grew in volume as observed during the next measurement on September 29, 2003. By the final observation on September 22, 2004, the supernatant liquid had penetrated the upper sludge plug and had fallen into the void without collapsing much, if any, of the upper sludge plug. Some of the gas that had been in the void likely was vented through the overlying sludge as the gas volume decreased from ~120 ml to ~75 ml. The movement of the sludge, void, and liquid levels over the interval from April 10, 2003 (the last measurement date before the void was observed) through September 22, 2004, can be seen in Figure 3.8.

Figure 3.7. SNF Comp Sludge Compaction Test on August 28, 2003

Only one other instance of gas evolution was observed during the compaction tests. In sludge sample 96-13, after 253 days of settling (January 15, 2003), some bubbles were seen on the liquid surface.

3.1.3 Post-Test Examination of Settled Sludge

After the tests were completed on September 22, 2004, the settled sludge was removed from the graduated cylinders and returned to storage jars for further characterization and experiments if needed. Most of the sludge materials re-suspended readily and could be transferred to new vessels by repeated slurrying and decantation steps, except the 96-13 sludge.

The 96-13 settled sludge remained tightly held in the graduated cylinder and little of the sludge solids became mobile, even when the cylinder was inverted and shaken. Ultimately, the graduated cylinder had to be broken apart. Even then the sludge retained its cylindrical shape. The large self-cemented sludge pieces then were crumbled using a steel spatula to form smaller agglomerated lumps that could be put into the storage jar. Figure 3.9 shows the sludge concretion before it was completely broken away from the graduated cylinder and again just after the glass was broken away.

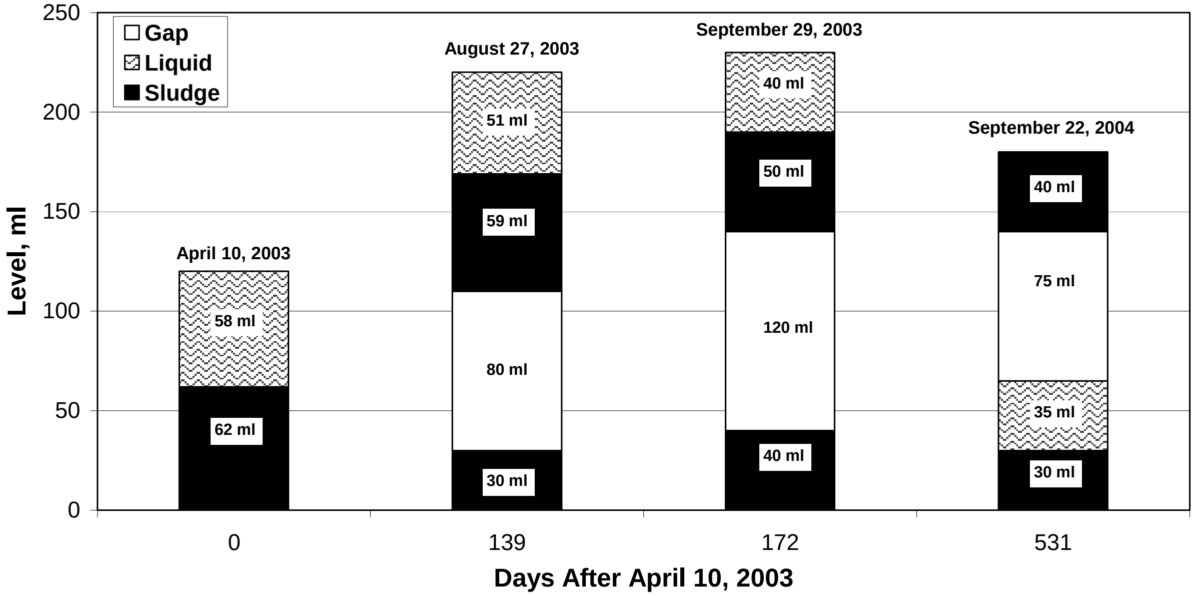

Figure 3.8. Settled Sludge, Supernatant Liquid, and Gas Gap Layer Development for Sample SNF Comp

As seen in Figure 3.9, the 96-13 sludge solids also contained rounded voids, some of which seemed to be partially merged with adjacent voids. The voids give evidence that gas formed during the compaction tests. However, bubbles were observed in the supernatant liquid only once during the compaction tests (see Section 3.1.2). Agitation also was found to compact the sludge by about 2 vol%, possibly by removing some of the voids. A mottled appearance for this sample, which may have been the same voids, also was noted after the vortex agitation testing (see Table 3.2).

Figure 3.9. Sample 96-13 During Removal (left) and After Removal (right) from the Glass Graduated Cylinder

The self-agglomeration of 96-13, although more pronounced, was similar to the physical form of fuel pieces and fuel piece plus KE Basin sludge at the conclusion of gas generation testing conducted at 40°C to 95°C (Schmidt et al. 2003). During recovery of sludge from reactor vessels at the conclusion of the tests, hard pan and sludge cake were encountered that required moderate effort to break up and mobilize. It is important to note that some of the gas generation test sample materials were readily re-suspended during recovery. However, self-cementation has not been observed after sludge storage in the hot cell, provided that the samples are kept immersed in water. This 96-13 sample had been allowed to dry during storage (see Table 2.1), which may have contributed to its subsequent anomalous behavior to form voids and to strongly self-cement.

3.2 Glovebox Testing of KE NLOP Sludge

Results of tests with KE NLOP sludge showed that, in general, the sludge volume stabilizes after about 1 week of settling time (Mellinger et al. 2004). The same data were reassessed to determine settled sludge density as a function of settling time. The results are plotted in Figure 3.10. The density observed after about 15 days of settling to a 61-ml volume approaches 1.34 g/ml. Based on the experience with the other sludge samples reported in Section 3.1.2, little further consolidation or density increase would have been expected to occur with more extended settling time. However, slightly greater density might have been observed for the KE NLOP sludge if a larger, 100-ml sample, as described in Section 3.1 for the hot cell sludge compaction tests, were used.

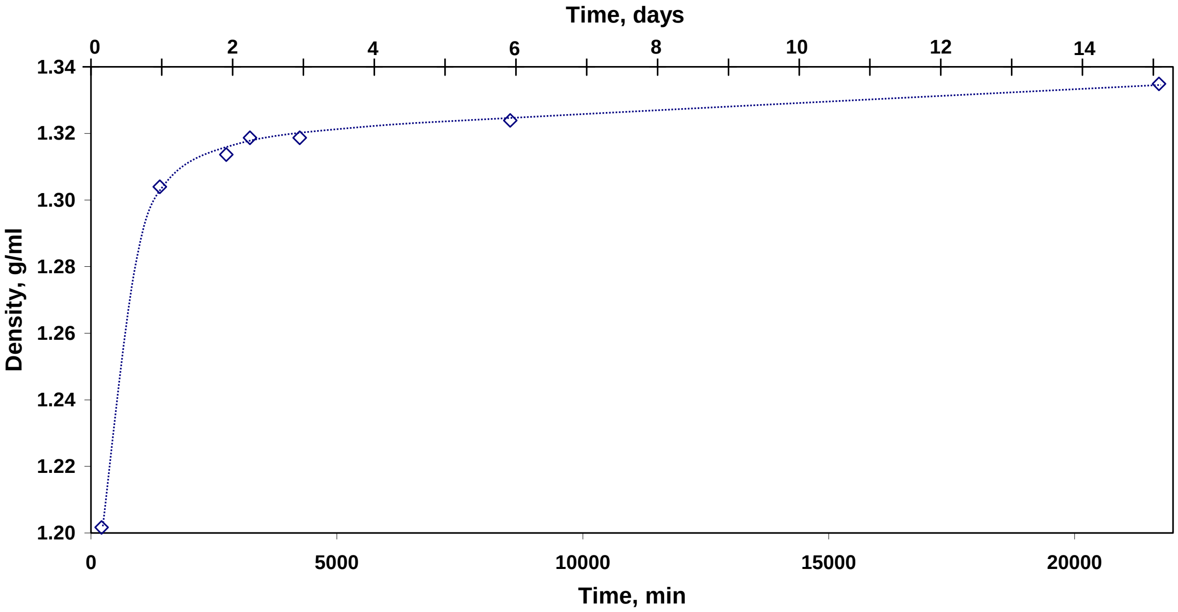

Figure 3.10. KE NLOP Sludge Density as a Function of Settling Time

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Appendix A

Sample Mass and Volume Data, Hot Cell Temperature and Humidity Values

Table A.1. Total Sample Mass Data

Test	Total Mass, g																			
	2002														2003					2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	1054.64	1053.79	1053.31	1052.77	1052.09	1051.36	1050.74	1050.12	1050.12	1050.12	1049.61	1049.08	1048.49	1045.96	1044.26	1041.81	1039.29	1058.27	1055.21	1054.50	1046.35
FE-5	915.48	915.54	915.48	915.55	915.53	915.52	915.39	915.36	915.36	915.36	915.35	915.28	915.26	915.04	914.98	915.02	915.03	915.03	911.95	911.12	892.08
KB Sim	1077.27	1075.95	1075.46	1075.00	1074.42	1073.80	1073.19	1072.58	1072.58	1072.58	1071.77	1071.31	1070.41	1066.11	1063.30	1059.03	1056.69	1074.95	1072.45	1071.80	1054.43
KB Sup	862.43	862.09	861.79	861.48	861.02	860.60	860.19	859.87	859.87	859.87	859.59	859.33	859.03	838.72	837.74	836.45	835.68	835.68	(a)	831.25	811.80
KC-2/3	947.80	946.73	946.27	945.84	945.23	944.63	944.07	943.48	943.48	943.48	942.94	942.13	941.35	937.53	934.55	930.26	927.92	956.36	953.70	953.07	947.42
KC-4	906.53	905.34	904.56	903.85	903.06	902.13	901.34	900.52	900.52	900.52	899.89	899.22	898.45	894.53	891.74	887.09	884.66	906.88	902.77	903.25	891.95
KC-5	917.58	916.82	916.34	915.71	915.08	914.04	913.44	912.77	912.77	912.77	912.27	911.55	910.86	Graduated cylinder broken							
SNF Comp	929.43	928.99	928.46	927.83	927.12	926.27	925.67	925.01	925.01	925.01	924.64	924.31	933.67	909.42	908.62	907.80	907.41	926.06	(a)	(a)	908.63
Dry Time, days	0	14	27	43	56	71	83	99	105	106	112	125	139	205	246	302	331	331	470	503	862

(a) Note: 8/27/2003 KB Sup volume not measured to avoid disturbing the bubble that was forming in SNF Comp.

Table A.2. Vessel and Lead Donut Tare Weight Data

Test	Tare Weight, g
96-13	749.32
FE-5	745.58
KB Sim	770.03
KB Sup	745.59
KC-2/3	758.97
KC-4	756.60
KC-5	758.90
SNF Comp	748.88

Table A.3. Open Graduated Cylinder Water Evaporation Data

Value	Open Graduated Cylinder Water Evaporation Data										
Date & Time	1/13/05 15:50	1/14/05 8:30	1/14/05 14:00	1/17/05 9:38	1/18/2005 8:22	1/19/2005 9:05	1/20/2005 9:32	1/21/2005 8:18	1/24/2005 9:20	1/25/2005 16:20	1/26/2005 6:50
Dry Time, days	0.00	0.69	0.92	3.74	4.69	5.72	6.74	7.69	10.73	12.02	12.63
Total Mass, g	376.43	375.50	375.25	372.09	371.28	370.18	369.17	368.20	364.93	363.70	363.08

Table A.4. Net Sludge Plus Supernatant Liquid Mass Data

Test	Sample Sludge plus Supernatant Liquid Mass, g																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	305.32	304.47	303.99	303.45	302.77	302.04	301.42	300.80	300.80	300.80	300.29	299.76	299.17	296.64	294.94	292.49	289.97	308.95	305.89	305.18	297.03
FE-5	169.90	169.96	169.90	169.97	169.95	169.94	169.81	169.78	169.78	169.78	169.77	169.70	169.68	169.46	169.40	169.44	169.45	169.45	166.37	165.54	146.50
KB Sim	307.24	305.92	305.43	304.97	304.39	303.77	303.16	302.55	302.55	302.55	301.74	301.28	300.38	296.08	293.27	289.00	286.66	304.92	302.42	301.77	284.40
KB Sup	116.84	116.50	116.20	115.89	115.43	115.01	114.60	114.28	114.28	114.28	114.00	113.74	113.44	93.13	92.15	90.86	90.09	90.09	(a)	85.66	66.21
KC-2/3	188.83	187.76	187.30	186.87	186.26	185.66	185.10	184.51	184.51	184.51	183.97	183.16	182.38	178.56	175.58	171.29	168.95	197.39	194.73	194.10	188.45
KC-4	149.93	148.74	147.96	147.25	146.46	145.53	144.74	143.92	143.92	143.92	143.29	142.62	141.85	137.93	135.14	130.49	128.06	150.28	146.17	146.65	135.35
KC-5	158.68	157.92	157.44	156.81	156.18	155.14	154.54	153.87	153.87	153.87	153.37	152.65	151.96	Graduated cylinder broken							
SNF Comp	180.55	180.11	179.58	178.95	178.24	177.39	176.79	176.13	176.13	176.13	175.76	175.43	184.79	160.54	159.74	158.92	158.53	177.18	(a)	(a)	159.75
Dry Time, days	0	14	27	43	56	71	83	99	105	106	112	125	139	205	246	302	331	331	470	503	862

(a) Note: 8/27/2003 KB Sup volume not measured to avoid disturbing the bubble that was forming in SNF Comp.

Table A.5. Sample Mass Change Data

Test	**Mass Change, g** (note caps replaced for all samples and water added to all but FE-5 and KB Sup on 4/10/2003)																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	0.00	-0.85	-1.33	-1.87	-2.55	-3.28	-3.90	-4.52	-4.52	-4.52	-5.03	-5.56	-6.15	-8.68	-10.38	-12.83	-15.35	0.00	-3.06	-3.77	-11.92
FE-5	0.00	0.06	0.00	0.07	0.05	0.04	-0.09	-0.12	-0.12	-0.12	-0.13	-0.20	-0.22	-0.44	-0.50	-0.46	-0.45	-0.45	-3.53	-4.36	-23.40
KB Sim	0.00	-1.32	-1.81	-2.27	-2.85	-3.47	-4.08	-4.69	-4.69	-4.69	-5.50	-5.96	-6.86	-11.16	-13.97	-18.24	-20.58	0.00	-2.50	-3.15	-20.52
KB Sup	0.00	-0.34	-0.64	-0.95	-1.41	-1.83	-2.24	-2.56	-2.56	-2.56	-2.84	-3.10	-3.40	-3.40	-4.38	-5.67	-6.44	-6.44	(a)	-10.87	-30.32
KC-2/3	0.00	-1.07	-1.53	-1.96	-2.57	-3.17	-3.73	-4.32	-4.32	-4.32	-4.86	-5.67	-6.45	-10.27	-13.25	-17.54	-19.88	0.00	-2.66	-3.29	-8.94
KC-4	0.00	-1.19	-1.97	-2.68	-3.47	-4.40	-5.19	-6.01	-6.01	-6.01	-6.64	-7.31	-8.08	-12.00	-14.79	-19.44	-21.87	0.00	-4.11	-3.63	-14.93
KC-5	0.00	-0.76	-1.24	-1.87	-2.50	-3.54	-4.14	-4.81	-4.81	-4.81	-5.31	-6.03	-6.72	Graduated cylinder broken							
SNF Comp	0.00	-0.44	-0.97	-1.60	-2.31	-3.16	-3.76	-4.42	-4.42	-4.42	-4.79	-5.12		-5.12	-5.92	-6.74	-7.13	0.00	(a)	(a)	-17.43
Dry Time, days	0	14	27	43	56	71	83	99	105	106	112	125	139	205	246	302	331	331	470	503	862

(a) Note: 8/27/2003 KB Sup volume not measured to avoid disturbing the bubble that was forming in SNF Comp.

Table A.6. Settled Sludge Volume Data

Test	Settled Sludge Volume, ml																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	101.0	101.0	101.0	101.0	101.0	101.0	101.0	100.0	98.0	98.0	98.0	99.0	98.0	100.0	99.0	100.0	100.0	100.0	100.0	100.0	96.0
FE-5	95.0	95.0	95.0	95.0	95.0	95.0	94.5	94.0	94.0	92.0	92.0	89.0	89.5	89.0	87.5	89.0	89.0	89.0	88.0	88.0	88.0
KB Sim	120.0	119.0	118.0	118.0	118.0	118.0	119.0	118.5	118.0	118.0	117.0	117.0	117.0	116.0	118.0	116.0	117.0	117.0	116.0	116.0	118.0
KB Sup	Not applicable																				
KC-2/3	97.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.0	96.5	95.0	96.0	97.5	95.0	96.0	96.0	96.0	95.0	94.0
KC-4	103.0	98.0	98.0	97.0	97.0	97.0	97.0	96.5	96.5	96.5	94.0	94.0	94.0	92.0	88.0	92.0	91.0	91.0	92.0	90.0	90.0
KC-5	107.0	104.0	104.0	103.0	102.5	102.5	102.5	102.0	103.5	102.0	102.0	100.0	99.0	Graduated cylinder broken							
SNF Comp	79.0	76.0	76.0	76.0	73.5	73.0	74.0	82.5	67.0	67.0	68.0	72.0	73.0	62.5	62.5	62.0	62.0	62.0	59.0	Gas pocket formed	
Settle Time, days	7	21	34	50	63	78	90	106	112	113	119	132	146	212	253	309	338	338	477	510	869

Table A.7. Total Sludge Plus Supernatant Liquid Volume Data

Test	Total (Sludge plus Supernatant Liquid) Volume, ml																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	118.0	118.0	118.0	116.0	116.0	115.0	115.0	114.0	115.0	115.0	115.0	113.0	114.0	111.0	112.0	107.0	104.0	122.0	122.0	122.0	110.0
FE-5	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	116.0	115.0	115.0	113.0	112.0	92.0
KB Sim	140.0	139.0	138.0	138.0	137.0	136.0	136.0	137.5	137.0	137.0	137.0	134.0	134.0	130.0	128.0	122.0	120.0	138.0	136.0	136.0	122.0
KB Sup	117.0	117.0	117.0	117.0	116.0	116.0	116.0	115.0	116.0	116.0	116.0	114.0	116.0	112.0	113.0	108.0	108.0	108.0	(a)	108.0	90.0
KC-2/3	116.0	114.0	114.0	114.0	114.0	112.0	112.0	111.0	112.0	113.0	112.5	110.0	112.0	107.0	103.0	101.0	99.0	126.0	123.0	123.0	116.0
KC-4	121.0	120.0	119.5	118.0	118.0	117.0	116.0	116.0	115.0	115.5	115.0	114.0	115.0	110.0	108.0	103.0	99.0	122.0	119.0	119.0	110.0
KC-5	128.0	126.0	125.0	124.0	124.0	123.0	122.0	122.0	122.0	122.0	122.0	120.0	121.0	Graduated cylinder broken							
SNF Comp	100.0	99.0	98.0	98.0	98.0	97.0	100.0	110.0	96.0	96.0	97.0	102.0	117.0	106.0	106.0	106.0	104.0	120.0	220.0	230.0	180.0
Settle Time, days	7	21	34	50	63	78	90	106	112	113	119	132	146	212	253	309	338	338	477	510	869

(a) Note: 8/27/2003 KB Sup volume not measured to avoid disturbing the bubble that was forming in SNF Comp.

Table A.8. Settled Sludge Density

Test	Settled Sludge Density, g/ml																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
96-13	2.855	2.847	2.842	2.857	2.850	2.853	2.847	2.869	2.897	2.897	2.892	2.887	2.890	2.857	2.849	2.855	2.860	2.871	2.840	2.833	2.949
FE-5	1.568	1.569	1.569	1.570	1.569	1.569	1.571	1.573	1.573	1.586	1.586	1.605	1.601	1.602	1.612	1.602	1.613	1.613	1.608	1.610	1.620
KB Sim	2.394	2.404	2.420	2.416	2.419	2.423	2.406	2.394	2.404	2.404	2.409	2.431	2.423	2.432	2.401	2.440	2.425	2.428	2.436	2.430	2.376
KB Sup	Not applicable																				
KC-2/3	1.752	1.769	1.765	1.760	1.754	1.768	1.762	1.767	1.756	1.746	1.745	1.759	1.742	1.746	1.745	1.740	1.729	1.745	1.749	1.750	1.772
KC-4	1.282	1.294	1.292	1.303	1.295	1.296	1.297	1.290	1.301	1.296	1.302	1.306	1.287	1.305	1.310	1.299	1.320	1.312	1.297	1.309	1.283
KC-5	1.288	1.308	1.313	1.320	1.315	1.315	1.319	1.313	1.309	1.314	1.309	1.328	1.314	Graduated cylinder broken							
SNF Comp	2.021	2.069	2.075	2.067	2.093	2.104	2.040	1.803	2.198	2.199	2.161	2.022	1.932	1.876	1.863	1.857	1.883	1.927	Gas pocket formed		
Settle Time, days	7	21	34	50	63	78	90	106	112	113	119	132	146	212	253	309	338	338	477	510	869

Table A.9. Hot Cell Temperature and Relative Humidity Data

Value	Hot Cell Temperature and Relative Humidity																				
	2002														2003						2004
	5/14	5/28	6/10	6/26	7/9	7/24	8/5	8/21	8/27	8/28	9/3	9/16	9/30	12/5	1/15	3/12	4/10	4/10	8/27	9/29	9/22
T, °C	31.7	33.0	33.0	38.0	36.0	38.0	35.0	33.0	34.0	35.0	34.0	33.0	33.0	32.0	32.0	32.0	34.0	34.0	38.0	Not recorded	
Relative Hum. %	30.0	38.0	35.8	33.5	33.0	36.0	32.0	39.0	37.0	36.0	33.0	34.5	34.5	33.0	33.0	33.0	34.0	34.0	41.0		
Elapsed Time, days	0	14	27	43	56	71	83	99	105	106	112	125	139	205	246	302	331	331	470	503	862

Table A.10. KE NLOP Sludge Settling Data

Value	KE NLOP Sludge Settling Data															
Settle Time, min	0	2	15	30	45	60	75	90	105	215	1402	2750	3234	4245	8524	21715
Settle Time, days	0.000	0.001	0.010	0.021	0.031	0.042	0.052	0.063	0.073	0.149	0.974	1.910	2.246	2.948	5.919	15.080
Sludge Level, ml	250	248	236	222	208	195	181	168	156	98	65	63	62	62	61	59
Sludge Mass, g	269.76	267.76	255.76	241.76	227.76	214.76	200.76	187.76	175.76	117.76	84.76	82.76	81.76	81.76	80.76	78.76
Density, g/ml	–	–	–	–	–	–	–	1.118	1.127	1.202	1.304	1.314	1.319	1.319	1.324	1.335
Note that the density of the initial settled KE NLOP sludge used in this test was 1.395 g/ml (i.e., mass and initial volume were 69.76 g and 50 ml, respectively).																

Appendix B

Photographs of Sludge Samples Before and After Compaction by Agitation on a Vortex Mixer

Figure B.1. Sludge KC-5 Before (left) and 4-6 Hours After (right) Vortex Mixing

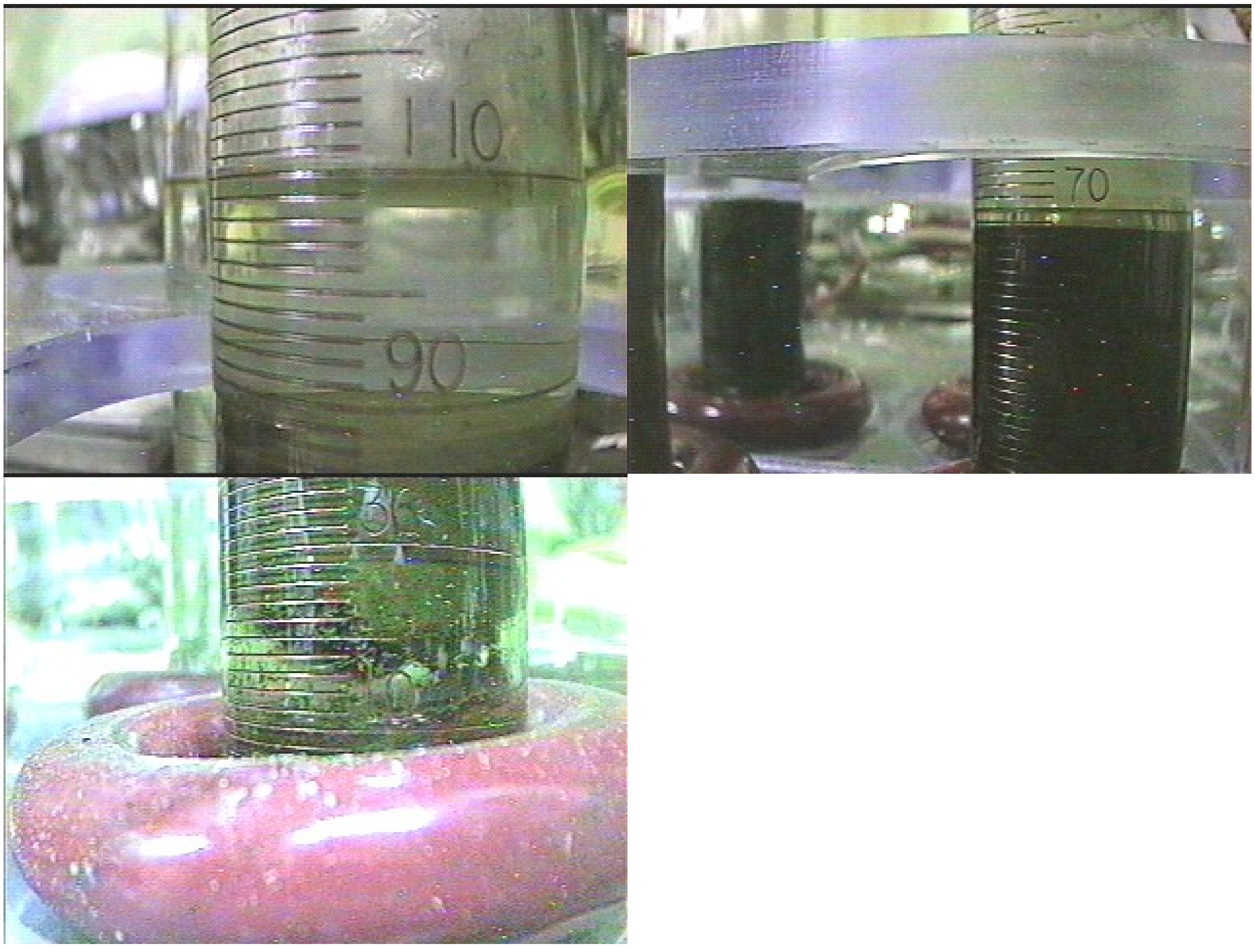

Figure B.2. Sludge SNF-Comp Before (top and bottom left) and 4-6 Hours After (right) Vortex Mixing. Note the irregular "aqueous void" above the pink-colored lead donut in the bottom left image. This void, which is pale green with the darker green sludge plug present above it in the cylinder, disappeared after agitation on the vortex mixer.

Figure B.3. Sludge 96-13 Before (left) and 4-6 Hours After (right) Vortex Mixing

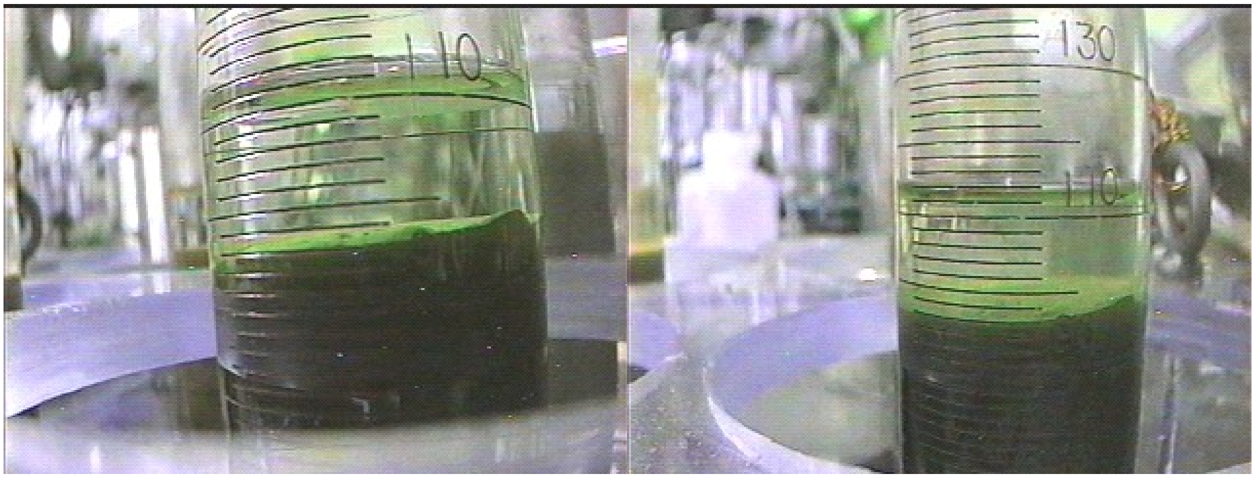

Figure B.4. Sludge KC-2/3 Before (left) and 4-6 Hours After (right) Vortex Mixing

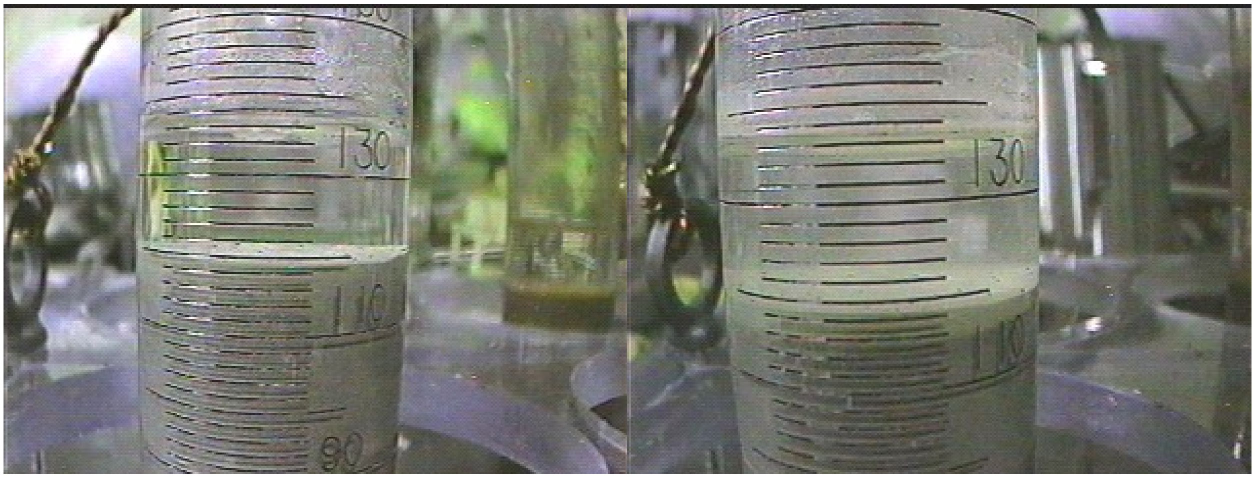

Figure B.5. Sludge KB Sim Before (left) and 4-6 Hours After (right) Vortex Mixing

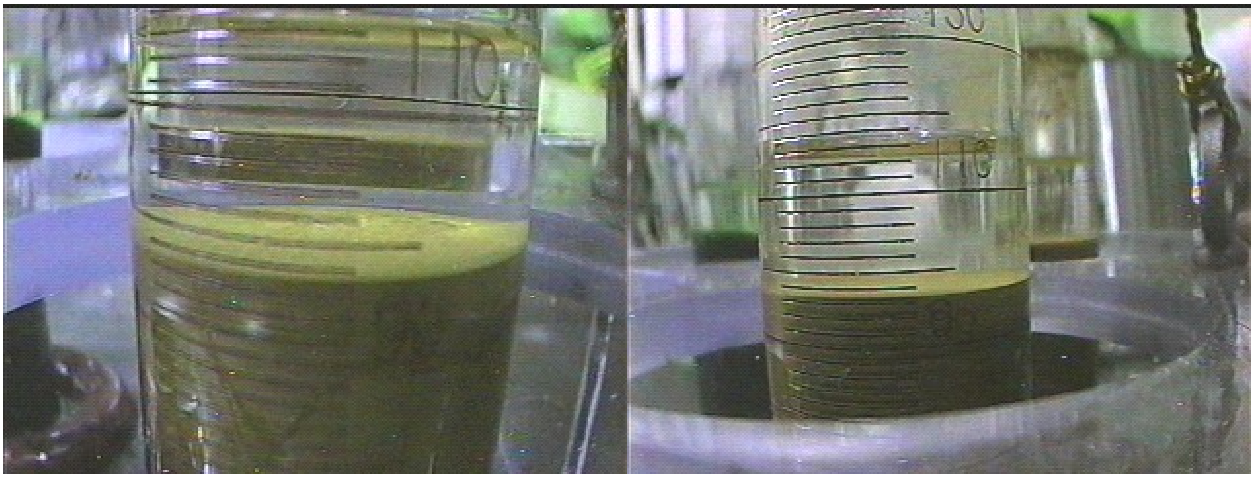

Figure B.6. Sludge KC-4 Before (left) and 4-6 Hours After (right) Vortex Mixing

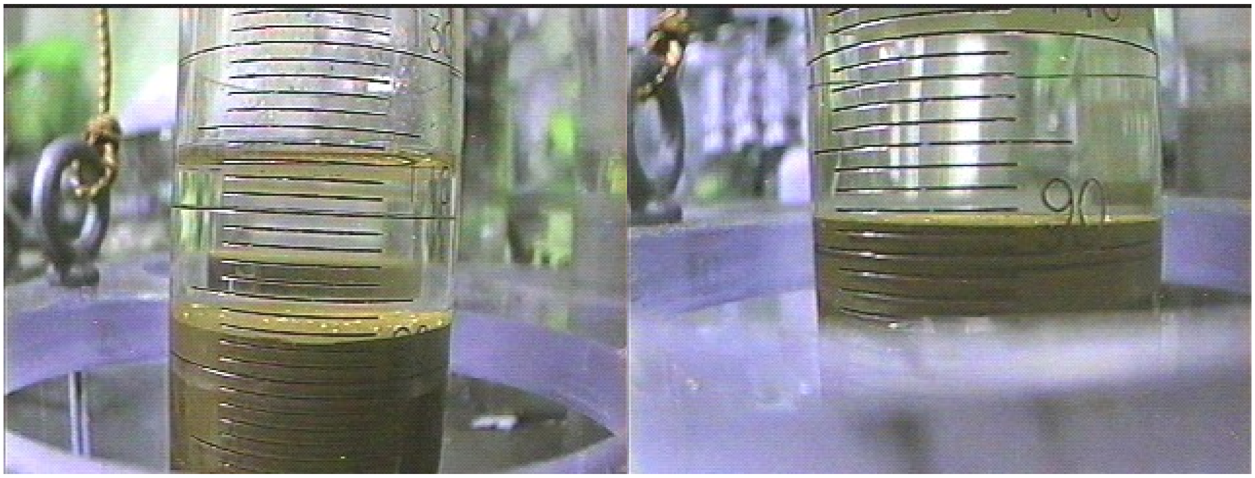

Figure B.7. Sludge FE-5 Before (left) and 4-6 Hours After (right) Vortex Mixing