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STP K Basin Sludge Sample Archive at the Pacific Northwest National Laboratory FY2014

June 2014

SK Fiskum
MR Smoot
AJ Schmidt

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Pacific Northwest National Laboratory
Richland, Washington 99352

Summary and Recommendations

The Pacific Northwest National Laboratory (PNNL) currently houses 88 samples (~10.5 kg) of K Basin sludge (81 wet and seven dry samples) on behalf of the Sludge Treatment Project (STP), which is managed for the U.S. Department of Energy (DOE) by the CH2M Hill Plateau Remediation Company (CHPRC). Selected samples are intended to serve, in part, as sentinels to enhance understanding of sludge properties after long-term storage, and thus enhance understanding of sludge behavior following transfer to sludge transfer and storage containers (STSCs) and storage at the Hanford 200 Area central plateau. In addition, remaining samples serve in contingency for future testing requirements.

At PNNL, the samples are tracked and maintained under a prescriptive and disciplined monthly sample-monitoring program implemented by PNNL staff. This report updates the status of the K Basin archive sludge sample inventory to April 2014. The previous inventory status report, PNNL-22245 (Fiskum et al. 2013, limited distribution report), was issued in February of 2013. This update incorporates changes in the inventory related to repackaging of 17 samples under test instructions 52578-TI052, *K Basin Sludge Sample Repackaging for Continued Long Term Storage,* and 52578-TI053, *K Basin Sludge Sample Repackaging Post-2014 Shear Strength Measurements*. Note that shear strength measurement results acquired in 2014 are provided separately.[1]

Specifically, this report provides the following:

- a description of the K Basin sludge sample archive program and the sample inventory
- a summary and images of the samples that were repackaged in April 2014
- up-to-date images and plots of the settled density and water loss from all applicable samples in the inventory
- updated sample pedigree charts, which provide a roadmap of the genesis and processing history of each sample in the inventory
- occurrence and deficiency reports associated with sample storage and repackaging

Key Observations and Recommendations

The archive sample program includes monthly monitoring of all samples. This frequency has been effective in maintaining the integrity of the sample inventory (i.e., trending of evaporation losses and assessment of container conditions). With this program, in the past year, one sample slurry out of 81 total lost cover water. Monthly sample monitoring should be continued to maintain a viable set of test materials for Phase 2 of the STP and to continue the long-term study.

Sample repackaging was executed consistently with the test instructions. Very little material was lost during the transfer of samples to new containers. The suspected brittleness of the old containers was confirmed during waste packaging operations.

[1] Letter 52578-2014-L07 from SK Fiskum to DB Engelman, June 20, 2014.

The densities of sludge samples increased moderately during the initial month of settling, and then remained essentially constant or underwent very gradual increases during the year. There are only a few isolated samples from which settled density trended toward decreasing.

In general, sludge samples originating from the K East Basin (SCS-CON-240, -250, and -260) and K West Basin (SCS-CON-210 and -220) appear to reach a constant density in three to six months. Several sludge samples originating from the K West Basin settler tubes (SCS-CON-230) appeared to increase in density (via increasing compaction) through 12 months of storage. Sludge samples from the flocculated settling study provide mixed results.

Acronyms and Abbreviations

CHPRC	CH2M Hill Plateau Remediation Company
DOE	U.S. Department of Energy
DI	deionized (water)
ID	identification
NLOP	North Loadout Pit
PNNL	Pacific Northwest National Laboratory
RPL	Radiochemical Processing Laboratory
SAL	Shielded Analytical Laboratory
STP	Sludge Treatment Project
STSC	sludge transfer and storage container
TI	test instruction

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1.0 Introduction

Pacific Northwest National Laboratory (PNNL) staff maintains 88 K Basin sludge samples (wet and dry samples) at the Radiochemical Processing Laboratory (RPL) in accordance with the PNNL Sludge Treatment Project (STP) data quality objective documents and sampling and analysis plans (Baker et al. 2009a, 2009b; Westcott et al. 2009). Sample maintenance activities are performed under contract with the CH2M Hill Plateau Remediation Company (CHPRC) under PNNL project 52578. In turn, CHPRC manages the STP for the U.S. Department of Energy (DOE).

The K Basin samples in the PNNL inventory were collected from a series of sampling events at various positions in the East and West K Basins (see Appendix C for pre-2004 descriptions)[1] and from six large engineered containers (SCS-CON-210 through SCS-CON-260) currently located in the K West Basin (post-2009 sampling). Many of the samples have been processed to accommodate testing directed by the CHPRC. K Basin sludge core samples collected from the large engineered containers have been processed at PNNL according to many specific test instructions (TIs) that included one or more of the following scopes:

- core reconstitution and subsampling
- container composite preparation, sieving, and subsampling
- settling tests (30-minute settling and treat-and-transfer settling)
- long-term storage study
- additional studies on selected materials.

Over time, several samples have been removed from the active inventory because 1) they were combined with other materials to make a larger stock, 2) they were no longer needed and sufficient stock of other sludge samples was available, or 3) they had dried out and were no longer useful. Those samples that have been consolidated, split, or removed were documented as delineated in completed TIs 52578-TI051,[2] 52578-TI052,[3] and 52578-TI053.[4] Processing from the latter two TIs is included in this updated status summary report (work associated with 52578-TI051 was included in the 2013 sample status summary).

Selected samples are intended to serve, in part, as sentinels to enhance understanding of sludge properties after long-term storage and thus enhance understanding of sludge behavior following transfer to sludge transfer and storage containers (STSCs) and storage at the Hanford 200 Area central plateau. As input to this objective, the settled density and water loss from all applicable samples in the inventory are monitored monthly as a function of time and sample handling activities.

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[1] The samples collected before 2004 were added to the K Basin sample archive in April 2012 for active monitoring and maintenance. They are intended to support STP Phase 2 testing per Letter 46498-L15 from AJ Schmidt to JP Sloughter, June 30, 2010.
[2] Fiskum, SK. 2012. *K Basin Sludge Sample Consolidation for Continued Long Term Storage*. Test Instruction 52578-TI051, Pacific Northwest National Laboratory, Richland, Washington.
[3] Edwards, MK. 2013. *K Basin Sludge Sample Repackaging for Continued Long Term Storage*. Test Instruction 52578-TI052, Pacific Northwest National Laboratory, Richland, Washington.
[4] Fiskum, SK. 2014. *K Basin Sludge Sample Repackaging Post-2014 Shear Strength Measurements*. Test Instruction 52578-TI053, Pacific Northwest National Laboratory, Richland, Washington.

In addition, remaining samples serve in contingency for future testing requirements. Archived sample materials have proven very useful for the resolution of emerging issues as the sludge disposition plan has evolved and as project-design activities have been implemented. Further, obtaining, transporting, analyzing, and testing new sludge samples from the K Basin remains a major investment for the K Basin project, both in time and funds (e.g., typical rough order-of-magnitude estimates are years and millions of dollars).

Samples are maintained in hot cells due to their highly radioactive nature. Because of the high ventilation requirement of the hot cells, wet samples may be subject to evaporative water loss; thus, samples are monitored monthly to assess water content, and water is added as needed. Once the wet sludge samples become dry, chemical changes (e.g., oxidation reactions) may accelerate and physical properties (e.g., shear strength and particle size) are permanently altered.

Sample maintenance also requires keeping the sludge samples in sturdy containers. The high radiation fields of these samples and the hot cell cause container darkening and embrittlement over extended exposure; thus, sludge samples periodically need to be transferred to new containers.

This report updates the status of the K Basin sludge inventory following shear strength measurements conducted on selected samples described in TI 52578-TI027 Addendum B[5] and the repackaging efforts described in TI 52578-TI052 and TI 52578-TI053. Nine samples tested for shear strength were removed from the long-term storage study and forwarded to the general sample archive. Two samples, TI001-B3-M and TI002-B1-M, were also reincorporated into the sample archive because they were found to have remained intact within their 50-mL plastic centrifuge tubes; these two samples were repackaged according to TI 52578-TI053.

Table 1 summarizes the repackaging efforts conducted per TI 52578-TI052 and TI 52578-TI053 (new sample processing since description in PNNL-22245 [Fiskum et al. 2013]). The sample volumes and masses were determined from the monthly monitoring log sheets except as noted.

Table 1. K Basin Repackaging Strategy

Sample Description	Sample Identification (ID)	Processing in April 2013			
		Settled Mass, g (Dec. 2012)	Settled Volume, mL (Dec. 2012)	Prior Package Date	April 2013 Repackaging Activity
Minus 500 micron sieve fraction, for rheology, from SCS-CON-220	TI009-SO	~20 to 30[(a)]	10 to 15[(a)]	August 2009	Transfer to a new 120-mL jar
Remaining KW250-B2 in a 120-mL Qorpak® from SCS-CON-250	KW250-B2-Settling	207	117	July 2010	Split between two new 120-mL Qorpak jars
Top-to-bottom sample composite from the KE North Loadout Pit (NLOP) (See PNNL-21836)	KE NLOP #2	271	196	March 2012	Split between two 240-mL low-form Qorpak jars

[5] Burns, CA. 2014. *FY14 Work Scope for Test Instruction 52578-TI027*. Test Instruction 52578-TI027 Addendum B, Pacific Northwest National Laboratory, Richland, Washington.

Table 1. K Basin Repackaging Strategy (Continued)

Sample Description	Sample Identification (ID)	Processing in April 2013			
		Settled Mass, g (Dec. 2012)	Settled Volume, mL (Dec. 2012)	Prior Package Date	April 2013 Repackaging Activity
Composite of SCS-CON-240, -250, and -260	52578-TI027-KEComp-1[(b)]	100	58	July 2010	Transfer to a new 120-mL jar
Composite of SCS-CON-240, -250, and -260	52578-TI027-KEComp-2[(b)]	103	5	December 2010	Transfer to a new 120-mL jar
Composite of SCS-CON-240, -250, and -260	52578-TI027-KEComp-3[(b)]	92	53	July 2010	Transfer to a new 120-mL jar
SCS-CON-220 core A1	52578-TI027-KW220A1[(b)]	112	70	July 2010	Transfer to a new 120-mL jar
SCS-CON-220 core B3	52578-TI027-KW220B3[(b)]	99	60	July 2010	Transfer to a new 120-mL jar
Flocculated SCS-CON-220 core B4	KW220-B4-Settling Study (Floc 3)[(b)]	100	60	Jan 2012	Transfer to a new 120-mL jar
SCS-CON-230 container composite	TI051-230B4-2[(b)]	162	63	October 2012	Transfer to a new 120-mL jar
SCS-CON-230 container composite	52578-TI027-KW230-2[(b)]	210	80	May 2011	Transfer to a new 120-mL jar
SCS-CON-210 core B1	KW210-B1-Archive-1[(b)]	97	83	February 2011	Transfer to a new 120-mL jar
SCS-CON-240 core A2	KW240-A2 Split	80	50	July 2010	Transfer to a new 120-mL jar
SCS-CON-250 core B2	KW250-B2 Split	75	43	July 2010	Transfer to a new 120-mL jar
Combined and flocculated SCS-CON-220-B4 and SCS-CON-250-A4	52578-TI027-Floc-1	101	55	July 2010	Transfer to a new 120-mL jar
Reserve physical property sample from SCS-CON-210 core B3	TI001-B3-M	27	17	February 2011	Combine and transfer to new 120-mL jar
SCS-CON-210 core B3	KW210-B3-Archive-3	90	68	February 2011	
Reserve physical property sample from SCS-CON-210 core B1	TI002-B1-M	21	17	February 2011	Transfer to a 120-mL jar

(a) Sample volume obtained from the monthly monitoring log sheet as of December 4, 2012; mass estimated from density of 2.0 g/mL reported in Fiskum et al. 2011 for the −500 micron container composite.

(b) Samples tested for shear strength in March 2014, before repackaging.

2.0 Sample Handling

Samples are maintained in hot cells at the PNNL RPL Shielded Analytical Laboratory (SAL). Currently, sludge samples are contained in 120-mL Qorpak jars or 240-mL low-form Qorpak jars. Samples are handled on a monthly basis to evaluate sample condition and top off with deionized (DI) water as needed. Samples are periodically repackaged into new jars.

2.1 Monthly Sample Handling

Samples are examined on a monthly basis to assess water content in the headspace, sludge height (for calculating volume), slurry height (for calculating volume), and container integrity. Most samples are stored in lidded metal bins, which are kept in a wall-mounted rack holder for secure storage in Cell 4 of the SAL. This practice clears the cell deck such that it is available for experiments and protects the samples from being tipped over or misplaced. Several samples (from the pre-2004 collection) containing accountable quantities of nuclear material (i.e., enough fissionable material to be tracked by PNNL Safeguards and Criticality Safety) are maintained in Cell 3.

Samples are handled carefully at all times (e.g., removing the bins from support hooks, placing the bins on the cell deck, transferring samples from Cell 4 [storage location] to Cell 2 [balance location]); however, inadvertent jostling is assumed to occur with each manipulation. Depending on the specific circumstances, the intensity and type (e.g., drops, knocks, vibration) of jostling varies considerably.[a]

Once samples are unloaded from the bins, they are rotated and examined for sludge and slurry height. Height measurements are determined from adhesive centimeter scales affixed to the outside of each container. Next, the samples are weighed. Each movement associated with these activities results in further unavoidable jostling.

If it is deemed that additional water is needed, the jar lid is removed, additional DI water is added, the jar threads are rinsed/cleaned if needed, and then the jar is recapped. Such manipulations are conducted in a secondary containment. The adjusted total fluid height and gross mass are measured and recorded. Often the water addition results in disturbance of the top layer of sludge. This disturbance obscures the sludge height; the sludge height cannot be accurately measured until a settling period has passed. Typically, this measurement is delayed until the next monthly evaluation. After the required measurements are taken, the samples are returned to the bins, moved back to Cell 4, and returned to the wall-mounted rack holders. Each of these manipulations results in further unavoidable jostling.

In some cases, the jar lids are very tightly sealed to the jar. This situation is generally the result of a small amount of sludge caught in the threads that has dried over time. The manipulator fingers are not particularly strong and it is difficult to get a tight grip on the lids. Each lid edge is wrapped with duct tape to minimize the slip and maximize manipulator finger grip; this is usually enough to allow lid loosening with repeated attempts. One sample jar lid was especially tight (51578-TI027-KEComp2, December

[a] Similar to the sludge samples at the RPL, sludge contained within STSCs would experience vibrations and accelerations likely to result in some sludge consolidation. Vibrations would likely be experienced as STSCs are loaded with sludge and prepared for transport in the K West Basin Annex, during transport of the STSCs from the 100 K Area to the 200 West Area, and during off-loading and placement of the STSCs into a cell in T-Plant.

2010); in the process of opening this jar, the glass cracked. The sludge was quickly transferred to a new jar.[a]

Note that the sludge and slurry heights can only be read to the nearest 0.2 cm, and therefore volumes and densities are accurate only to two significant figures (or one significant figure if the sludge level is <1.0 cm). The relative uncertainty of 0.2 cm applies to all height measures and impacts the sample volume calculation and dominates the uncertainty (not calculated) in sludge density. Sludge density is estimated from the mass and volume data as defined in the following equations.

settled sludge mass (m_S) $$m_S = m_G - m_{CT} - \left[(v_T - v_S) \times D_L\right]$$

settled sludge density (D_S) $$D_S = \frac{m_S}{v_S}$$

where m_{CT} = container tare mass (g)

m_G = gross container mass (m_{CT} + slurry) (g)

v_T = total slurry volume (liquid plus settled sludge) (mL)

v_S = volume of settled sludge (mL)

D_L = liquid density (g/mL), 1.00 g/mL for K Basin and deionized water

Monthly monitoring processes are conducted to check that sludge is maintained in a wetted and safe configuration. However, the determination of sludge volume increase (i.e., from potential gas production/retention) is confounded by required sample handling activities (i.e., accelerations incurred while the bins are moved could result in an unobserved release of an entrained gas).

2.2 Sample Repackaging

Sample repackaging was conducted in March and April 2014 and entailed the transfer of sludge slurry into new, clear, graduated jars. The preferred jar is one that easily supports shear strength testing (small diameter to accommodate sludge heights up to 4 cm). Qorpak jars that have caps lined with green thermoset F217 (a phenolic plastic) and polytetrafluoroethylene lined caps are preferred for glass wall thickness (ruggedness) and high radiation tolerance, respectively. Each new jar was marked with the vertical-up adhesive centimeter scale. The baseline or zero-point of the scale was positioned at the 5-mL volume mark (120-mL Qorpak jar); at 5 mL volume, fluid was just beyond the internal upward curvature of the jar base.

Some of the headspace water was decanted and then the slurry was mixed with a stainless steel spoon to dislodge and suspend solids. With the aid of a funnel, the slurry was poured into the new jar. A portion of the decanted water was returned to the parent jar, mixed with the remaining sludge, and again poured into the receiving jar. Rinsing was repeated as needed. Finally, the old jar was inverted over the funnel and rinse water was squirted into it to flush out heavy particles. These measures were used to

[a] An occurrence report, OR-52578-12-1-10, was written to address this issue; the issue was disclosed to the Buyer's Technical Representative (BTR) on 12/16/10.

enhance the transfer of dark, dense particles, possibly containing uranium, into the new jar. In some instances, a transfer pipet and water were used to dislodge the dark and dense particles.

During all sludge handling, some sludge is lost. Typically, a small film of sludge clings to the inner jar walls and neck. Other losses stem from sludge adhering to the mixing spoon, slurry splashing out during the mixing process, or slurry dripping during the transfer process. After sludge transfer, the emptied jar and lid are weighed to assess how much sludge slurry remains in the emptied jar. Other loss sources cannot be well estimated and are ignored. The net masses of the emptied jars representing residual sludge and flush water were calculated, and ranged from 0.2 g to 2.21 g. A 0.947-g glass fragment was recovered from sample 52578-TI027-KEComp-2 during the repackaging process; see the associated figure in Table 2. This fragment had the look and feel of Qorpak glass and likely was a fragment from the earlier jar breakage in 2010 associated with this sample.

The transferred sludge samples were resuspended and allowed to settle for 24 hours. Images of the 52578-TI053 initial sample (before transfer), emptied container, and final sample after the 24-hr settling period are provided in Table 2. Also shown is an image of the MacBeth Color Checker Chart taken with the in-cell camera; it is juxtaposed next to an image of the chart taken from the manufacturer’s web page. Colors with a red hue are generally washed out when photographed; other colors photograph more closely to the true color.

After repackaging, sample maintenance was relinquished to the monthly monitoring program.

Table 2. Sample Repackaging Photographs, March/April 2014

Sample Description	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
Composite of KW240, KW250, and KW260	52578-TI027-KEComp-1 to TI053-KEComp-1			
Composite of KW240, KW250, and KW260	52578-TI027-KEComp-2 to TI053-KEComp-2 (and recovered glass shard)			
				

Table 2. (Continued)

Sample Description.	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
Composite of KW240, KW250, and KW260	52578-TI027-KEComp-3 to TI053-KEComp-3			
KW220 core A1	52578-TI027-KW220A1 to TI053-KW220A1			
KW220 core B3	52578-TI027-KW220B3 to TI053-KW220B3			

Table 2. (Continued)

Sample Description.	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
Flocculated KW220 core B4	KW220-B4-Settling Study (Floc 3) to TI053-KW220B4-Settling			
KW230 container composite	TI051-230B4-2 to TI053-KW230B4-2			
KW230 container composite	52578-TI027-KW230-2 to TI053-KW230-2			

Table 2. (Continued)

Sample Description.	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
KW210 core B1	KW210-B1-Archive-1 to TI053-KW210B1			
KW240 Core A2	KW240-A2 Split to TI053-KW240A2			
KW250 Core B2	KW250-B2 Split to TI053-KW250B2			

Table 2. (Continued)

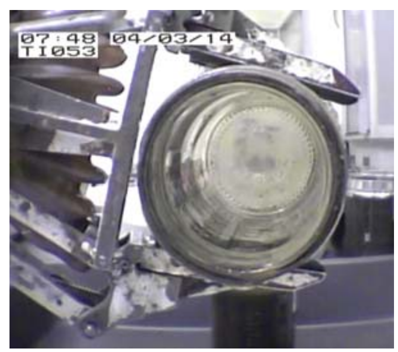
Sample Description.	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
Combined flocculated KW220-B4 and KW250-A4 samples	52578-TI027-Floc-1 to TI053-FlocMix			
Reserve physical property sample from KW210-B3	TI001-B3-M to TI053-KW210B3		No image collected	N/A Added to TI053-KW210B3
KW210-B3 core composite	KW210-B3-Archive-3 to TI053-KW210B3			

Table 2. (Continued)

Sample Description.	Sample ID	Initial Sample Condition	Emptied Container	Final Sample, after 24-hr Settling
Reserve physical property sample from KW210-B1	TI002-B1-M to TI053-KW210B1			
MacBeth Color Checker Chart		Original 	Typical In-Cell 	

After processing samples, the spent Qorpak jars were crushed to reduce overall waste volume. The spent jars broke relatively easily, requiring minor tapping with a steel block to fracture. In contrast, a new jar was similarly crushed (in the hot cell); it required significantly more effort to break into pieces. The ease of breaking the spent jars verifies that the glass had become brittle with the radiation exposure. It is recommended that the sample jar lifetime for hot cell use be limited to nominally four years. Many of the samples in the long-term storage study are approaching the four-year recommended limit (see Table 3).

Table 3. Package Age of Remaining Samples in Long-Term Storage Study

SCS-CON-	Sample ID	Packing Date	Storage Time, years
210 Long-term	KW210-B1-Archive-2	2/2011	3
210 Long-term	KW210-B3-Archive-1	2/2011	3
210 Long-term	KW210-B3-Archive-2	2/2011	3
210 Long-term with floc	KW210-CCA-Settling Study	1/2012	2
240/250/260 Long-term	52578-TI027 KECOMP-4	7/2010	4
240/250/260 Long-term	52578-TI027 KECOMP-5	7/2010	4
240/250/260 Long-term	52578-TI027 KECOMP-6	7/2010	4
220 Long-term	52578-TI027 KW220-B4	7/2010	4
230 Long-term	52578-TI027-KW230-1	5/2011	3
230 Long-term	52578-TI027-KW230-3	5/2011	3
230 Long-term	52578-TI027-KW230-4	5/2011	3

3.0 Sample Summary

As of April 2014, PNNL is tracking 88 sludge samples (~10.5 kg) that originated from the K East and West Basins. A total of 59 samples are associated with the processing of subsamples collected from the large engineered containers (K West Basin) in 2009 and 2010. The sample evolution and processing is summarized in a series of flowcharts (Appendix A). The flowcharts include the recent subsampling/handling activities for sample repackaging (TI 52578-TI052 and TI 52578-TI053). From these flowcharts, the pedigrees of the tracked samples can be readily understood.

The other 29 tracked samples are from pre-2004 sludge collection activities from the K East and West Basins (22 samples stored wet and seven samples stored dry). These samples were placed into new containers around April 2012 as described by Sinkov et al. (2012) and incorporated into the K Basin PNNL project 52578 monthly monitoring program in November 2012 (see Appendix C). The only change to this current inventory is KENLOP #2, which was split into two jars per TI 52578-TI052.

Table 4 shows nominal wet settled sludge sample volumes and masses associated with the grouped sample inventory as of April 8, 2014. The masses, and sludge and slurry heights, are traceable to the monthly sample inventory log sheets; the wet settled sludge volumes, slurry volumes, and densities were calculated. Volumes and densities should be considered gross estimates because individual sample volumes are calculated to a maximum of two significant figures (from reading the centimeter scale affixed to the container). Large rock-like features present in several pre-2004 samples confounded the measure of the total volume.

Three groups of sludge are shown in Table 4: 1) post-2009 collected samples, 2) samples remaining in the long-term storage study, and 3) pre-2004 collected samples. Parenthetical values in Table 4 provide the total number of sludge containers relevant to a specific sludge category. Masses, volumes, and total sample numbers differ slightly from those reported previously (Fiskum et al. 2013) for the following reasons:

- Additional settling likely occurred that reduced total sludge volume and mass.
- Some samples were split and others combined.
- Two samples were added back to the archive.
- Several samples were removed from the long-term storage study and placed into the regular sample archive.
- Sludge sample height reading varied relative to large rock-like structures (i.e., irregular topography).

Table 4. Summary of Sludge Samples in the SAL, April 8, 2014

Engineered Container	Sludge Classification	Collection Date at K Basin	Mass, g	Volume, mL	Average Density, g/mL	Max. Density, g/mL	Min. Density, g/mL	Sample Count
Post-2009 Sludge Sample Collection								
SCS-CON-210	KW Originating	2010	724	565	1.3	1.7	1.1	9
SCS-CON-220	KW Originating	2009	338	228	1.5	1.7	1.4	4
SCS-CON-220-B4	KW Originating	2009	60	35	1.7	1.7	1.7	1
SCS-CON-230	KW Settler	2010	1,261	540	2.3	2.7	1.9	11
SCS-CON-240	KE Originating	2009	310	175	1.8	1.9	1.6	3
SCS-CON-250	KE Originating	2009	445	265	1.7	1.9	1.5	5
SCS-CON-260	KE Originating	2009	607	352	1.7	1.9	1.6	8
SCS-CON-240/250/260	KE Originating	2009	324	202	1.6	1.6	1.6	3
Sample Material Participating in Long-Term Storage Study								
Unflocculated								
SCS-CON-210	KW Originating	2010	253	203	1.2	1.3	1.2	3
SCS-CON-220-B4	KW Originating	2009	102	58	1.8	1.8	1.8	1
SCS-CON-230	KW Originating	2010	658	248	2.6	2.7	2.6	3
SCS-CON-240/250/260	KE Originating	2009	312	175	1.8	1.8	1.8	3
Flocculated								
SCS-CON-210	KW Originating	2010	109	80	1.4	1.4	1.4	1
SCS-CON-220/250	KE/KW Originating	2009	117	72	1.6	1.6	1.6	1
SCS-CON-220-B4	KW Originating	2009	110	72	1.5	1.5	1.5	1
SCS-CON-230	KW Settler	2010	162	73	2.2	2.2	2.2	1
SCS-CON-250-A4	KE Originating	2009	101	55	1.8	1.8	1.8	1
Pre-2004 Sludge Sample Collection[(a)]								
Samples defined by Sinkov et al. 2012, Tables 3.1 and 3.2 (wet samples)			3,970	2,210	1.1 to 2.5	2.4	1.1	22
Samples defined by Sinkov et al. 2012, Table S.2 (dry samples)			541	N/A	N/A	N/A	N/A	7
(a) Volume and mass confounded by large monolithic rock-like structures in several samples.								

Table 5 shows the total sample inventory status as of April 8, 2014. Again, volumes and densities should be considered gross estimates because the individual sample volumes are calculated to a maximum of two significant figures (from reading the centimeter scale affixed to the container). The third significant figure in the density result is provided for information only.

No samples in the current monthly monitoring inventory have dried out. One sample was found with no headspace water (i.e., KW210-B1-Archive-4). However, it was still moist and not considered to be compromised.

Table 5. K Basin Sample Archive Summary, April 8, 2014

SCS-CON-	Sample ID	Net Sludge Level, cm	Net Slurry Level, cm	Sludge Volume, mL	Sludge Mass, g	Sludge Density, g/mL[(a)]
210	KW210-A1-Archive-A	2.1	5.6	40	61	1.53
210	KW210-A4-Archive-A	2.3	6.3	43	73	1.69
210	TI053-KW210B1	5.1	6.8	90	106	1.18
210 Long-term	KW210-B1-Archive-2	4.0	6.1	72	83	1.16
210	KW210-B1-Archive-4	4.9	6.2	87	100	1.15
210	KW210-B1-Archive-7	4.5	6.0	80	92	1.15
210	TI053-KW210B3	5.5	6.7	97	124	1.28
210	TI053-KW210B1B	0.7	5.1	17	18	1.10
210 Long-term	KW210-B3-Archive-1	3.5	4.9	63	84	1.32
210 Long-term	KW210-B3-Archive-2	3.8	5.9	68	86	1.26
210	KW210-B3-Archive-4	4.0	5.8	72	87	1.22
210	KW210-CC-A (New)	2.1	6.0	40	62	1.56
210 Long-term with floc	KW210-CCA-Settling Study	4.5	6.1	80	109	1.37
220	TI053-KW220A1	5.3	6.7	93	136	1.45
220	TI051-220A3	2.0	6.3	38	65	1.69
220	TI053-KW220B3	4.6	6.8	82	117	1.43
220	TI052-220SO-2	0.6	5.8	15	21	1.39
220	TI051-220 B4	1.8	6.0	35	60	1.70
220 Long-term	52578-TI027 KW220-B4	2.9	5.4	58	102	1.75
220 With floc	TI053-KW220B4-Settling	4.0	6.7	72	110	1.54
230	KW230-CC-420	0.1	6.2	7[(e)]	15[(e)]	2.31[(e)]
230	TI051-230A2-1	0.2	6.2	8[(e)]	19[(e)]	2.31[(e)]
230	TI051-230A2-2	2.4	6.4	45	88	1.96
230	TI051-230A3-1	1.9	6.4	37	92	2.52
230	TI051-230A3-2	2.9	6.0	53	135	2.54

Table 5. (Continued)

SCS-CON-	Sample ID	Net Sludge Level, cm	Net Slurry Level, cm	Sludge Volume, mL	Sludge Mass, g	Sludge Density, g/mL[a]
230	TI051-230B2-1	1.8	6.3	35	69	1.98
230	TI051-230B2-2	3.7	6.3	67	130	1.94
230	TI051-230B4-1	3.8	6.3	68	155	2.27
230	TI051-230B4-3	3.3	5.1	60	160	2.67
230	TI053-KW230B4-2[b]	3.9	6.8	70	172	2.46
230	TI053-KW230-2	5.1	6.7	90	224	2.49
230 Long-term	52578-TI027-KW230-1	5.1	6.5	90	230	2.56
230 Long-term	52578-TI027-KW230-3	4.4	5.7	78	211	2.69
230 Long-term	52578-TI027-KW230-4	4.5	5.9	80	217	2.71
230 With floc	TI047-KW230-CC Floc Study	4.1	6.1	73	162	2.21
240	TI051-240B1	3.2	6.1	58	109	1.86
240, -500 microns	TI051-240SCC-1	3.0	5.0	55	106	1.93
240	TI053-KW240A2	3.4	6.9	62	96	1.55
250	TI051-250A4	1.7	6.4	33	59	1.77
250	TI051-250SCC-1	3.2	5.3	58	108	1.85
250	TI052-250B2-2	3.1	6.3	57	94	1.65
250	TI052-250B2-3	3.1	6.4	57	97	1.70
250	TI053-KW250B2	3.3	6.8	60	87	1.46
260	TI051-260A1-1	2.9	6.0	53	90	1.69
260	TI051-260A1-2	2.2	6.4	42	72	1.73
260	TI051-260A1-3	2.4	6.3	45	76	1.69
260	TI051-260B3-1	2.8	6.1	52	83	1.61
260	TI051-260B3-2	2.8	5.5	52	97	1.88
260	TI051-260B3-3	2.7	4.9	50	94	1.88
260, -500 microns	TI051-260SCC-1	1.6	6.1	32	53	1.66
260, -500 microns	TI051-260SCC-2	1.3	6.0	27	42	1.59
240/250/260	TI053-KEComp-1	3.8	6.7	68	109	1.60
240/250/260	TI053-KEComp-2	3.7	6.7	67	107	1.61
240/250/260	TI053-KEComp-3	3.7	6.6	67	107	1.61
240/250/260 Long-term	52578-TI027 KECOMP-4	2.8	5.1	57	100	1.77
240/250/260 Long-term	52578-TI027 KECOMP-5	2.7	6.1	55	99	1.80
240/250/260 Long-term	52578-TI027 KECOMP-6	3.2	5.5	63	113	1.78
220/250 With floc	TI053-FlocMix	4.0	6.8	72	117	1.63
250 With floc	KW250-A4-Settling Study	3.0	6.0	55	101	1.84
Pre-2004	KC-2/3 Comp X	4.2	5.1	160	350	2.18

Table 5. (Continued)

SCS-CON-	Sample ID	Net Sludge Level, cm	Net Slurry Level, cm	Sludge Volume, mL	Sludge Mass, g	Sludge Density, g/mL[(a)]
Pre-2004	KC-2/3 Comp Y	4.6	5.5	175	422	2.42
Pre-2004	96-05	2.3	5.0	92	205	2.22
Pre-2004	96-13 Comp A	3.4	5.4	132	292	2.22
Pre-2004	TI052-KENLOP#2-2	2.3	5.2	92	134	1.45
Pre-2004	TI052-KENLOP#2-3	2.3	5.3	92	130	1.40
Pre-2004	KC-4-2 X	3.3	5.1	128	203	1.58
Pre-2004	KC-4-2 Y	0.0	3.4	14[(d)]	16[(d)]	1.2[(d)]
Pre-2004	KC-6	3.2	5.0	125	158	1.26
Pre-2004	FE-5 Comp 1X	3.7	4.9	142	255	1.79
Pre-2004	FE-5 Comp 1Y	0.5	2.8	28	53	1.89
Pre-2004	KE Floc Comp	2.4	4.5	96	148	1.54
Pre-2004	96-13 Solids Grad	0.5+[(c)]	5.3	67[(c)]	150[(c)]	2.23[(c)]
Pre-2004	96-13 SSOL	2.0-4.8[(c)]	5.0	135[(c)]	321[(c)]	1.73[(c)]
Pre-2004	KC-4 Whole	2.0	5.3	82	149	1.83
Pre-2004	KE Pit	2.4	4.3	96	200	2.08
Pre-2004	KE NLOP #1	2.3	5.8	92	100	1.09
Pre-2004	KE NLOP #3	4.0	5.5	153	264	1.72
Pre-2004	KC-4 P250	0.7	5.1	35	77	2.20
Pre-2004	KC-1 M500	0.1	3.1	21[(d)]	48[(d)]	2.3[(d)]
Pre-2004	KC-6 New X	3.5	5.0	135	158	1.16
Pre-2004	KC-6 New Y	3.0	5.7	117	140	1.20
Pre-2004	K Basin Fuel Fines M500	Dry sample			154.2	
Pre-2004	K Basin Fuel Fines P500	Dry sample			38.3	
Pre-2004	Fe-3 Comp 1	Dry sample			121.3	
Pre-2004	SNF Comp Settling Study	Dry sample			52.6	
Pre-2004	Test 3 Residue	Dry sample			30.6	
Pre-2004	96-13 Settling Study	Dry sample			77.6	
Pre-2004	SFEC 9604	Dry sample			66.16	

(a) Sludge densities are accurate to two significant figures; the third figure is provided for information and to inform rounding decisions.

(b) The sample ID TI053-230B42 is misnamed; it is the daughter sample of TI025-B-Rh which is a container composite sample, not a specific B4 core sample.

(c) The sludge sample contained a large monolithic structure in addition to settled sludge that confounded calculations of settled sludge volume and mass. Therefore, sludge volume and mass were copied from original data collection, Sinkov et al. (2012), to provide a sense of the sludge mass and volume these samples represent.

(d) The volume and mass were small and were copied from data reported by Sinkov et al. (2012).

(e) The volumes are low and thus have high relative uncertainties. The weighted average sample densities were applied to the volume to estimate the sludge mass.

Starting in Fiscal Year 2013, photographic documentation of every sample was collected with the goal of obtaining one photograph per sample per year. For samples repackaged according to TI 52578-TI053, photographs are shown in Table 2. Other samples are documented in Table 6 through Table 8; the most recent photographing year is also provided. In some cases, the jar opacity in the dry pre-2004 collected samples is so high that the contents cannot be photographed well.

Also shown in Table 6 and Table 7 are histories of calculated sludge density and sample water loss as functions of time. Sludge sample water loss was calculated from changes in the gross container mass. Densities were calculated from the net slurry masses (gross mass minus the tare), slurry heights, and sludge heights. Data gaps indicate a break in the sequence associated with one or more of the following activities:

1. Sample removed from inventory for other use (e.g., shear strength measurements),

2. Balance did not meet calibration requirements and was not used (occurrence report [OR] OR-52578-11-29-12; see Appendix D),

3. The gross mass, sludge height, or slurry height was in obvious error (incorrectly read or recorded) based on examination of pre- and post-dated measurements (OR-52578-01-23-13; see Appendix D).

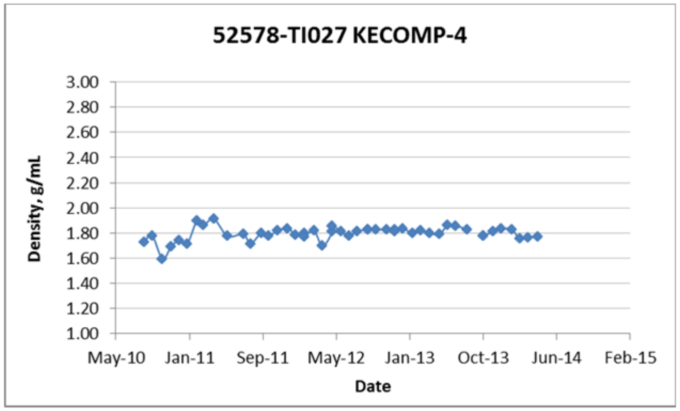
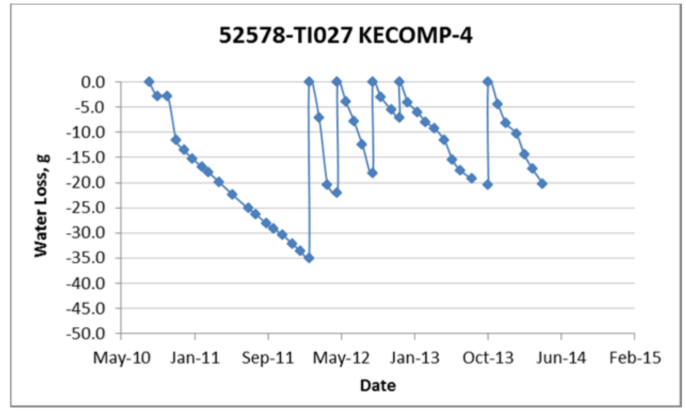
Density generally reached steady state within several (3 to 12) months after repackaging. Water loss rates were variable from sample to sample and were likely dependent on how well each jar lid was sealed rather than on sample-specific chemical reactions.

Periodically, water was added to the containers and the relative zero-point was reset. Thus, the water loss profiles generally appear as a sawtooth pattern. For all samples, a visible layer of water was maintained above the samples in all cases, except KW210-B1-Archive-4, which remained wetted, but was not saturated for a brief period of time. Appendix B provides graphs of water losses as functions of time for grouped items to allow general comparisons of water loss rates.

Ambient cell temperature, recorded in 2012 and 2013, was generally between 20 and 35°C. This storage temperature is higher than the K Basin temperature of 15°C. The cell temperature variation is also shown in Appendix B.

Table 6. Post-2009 Collected Samples

<table>
<tr><th>Sample ID</th><th>Settled Sludge Density History</th><th>Water Loss History</th></tr>
<tr><td>52578-TI027 KECOMP-4

2014</td><td>
</td><td>
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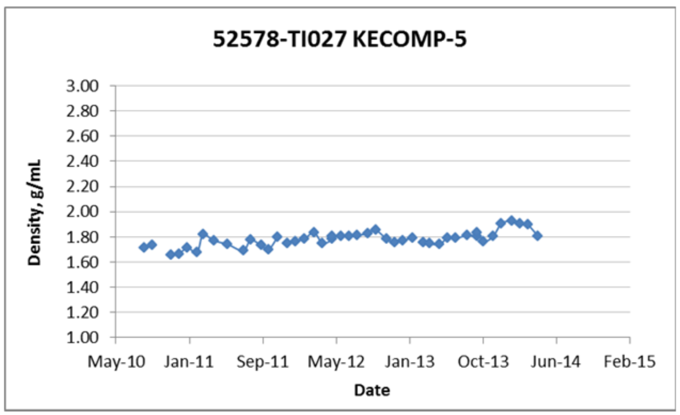
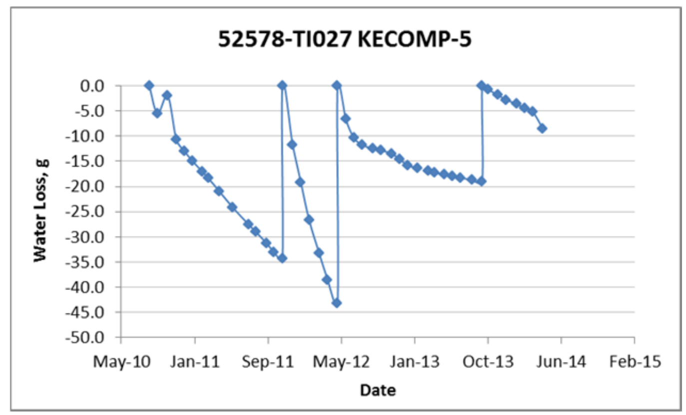
2014</td><td>
</td><td>
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Table 6. (Continued)

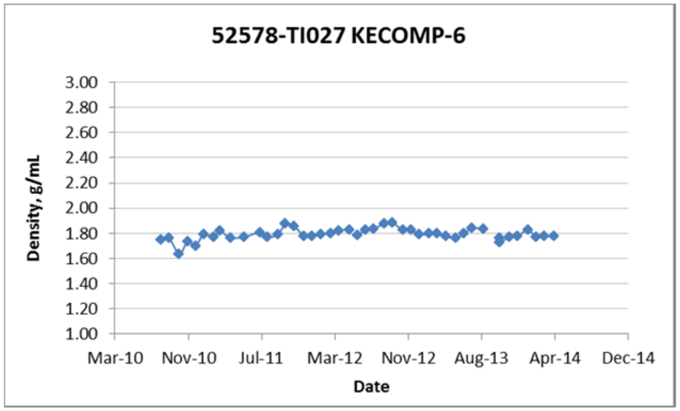
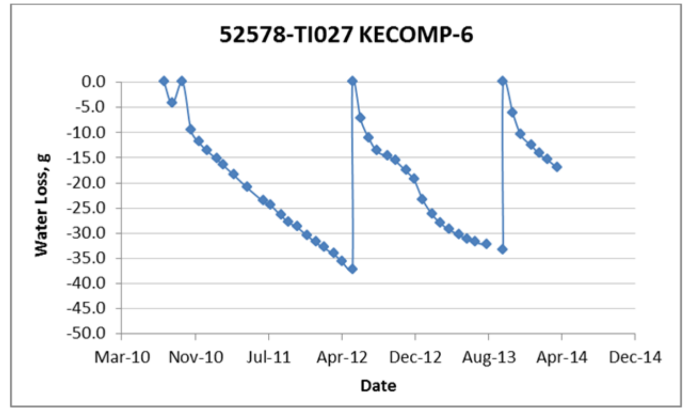
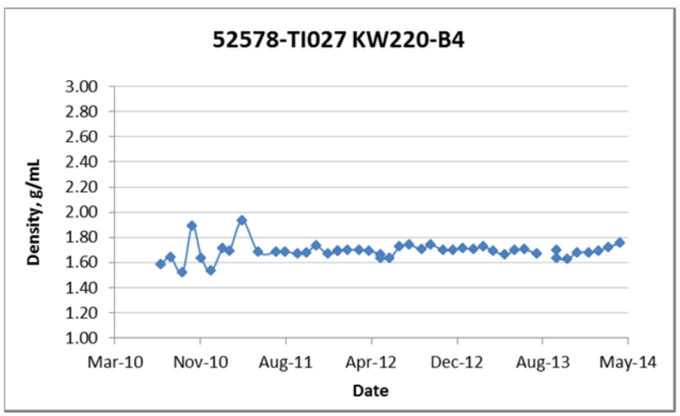
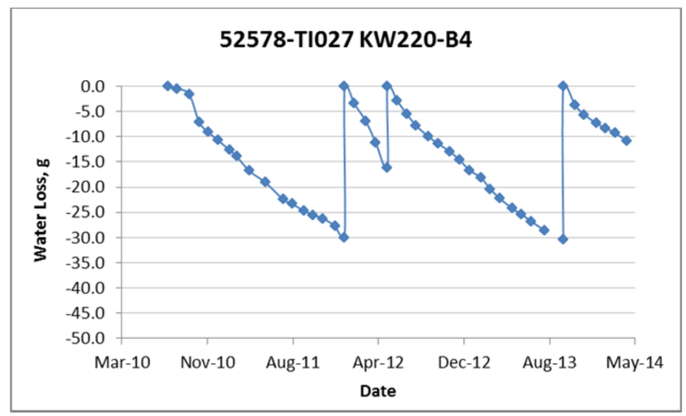
Sample ID	Settled Sludge Density History	Water Loss History
52578-TI027 KECOMP-6  2014		
52578-TI027 KW220-B4  2014		

Table 6. (Continued)

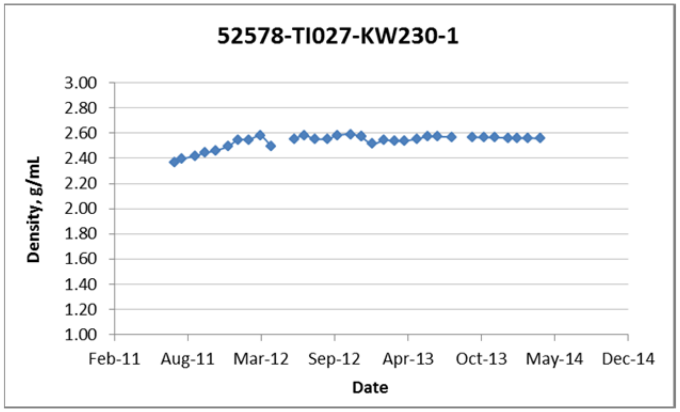
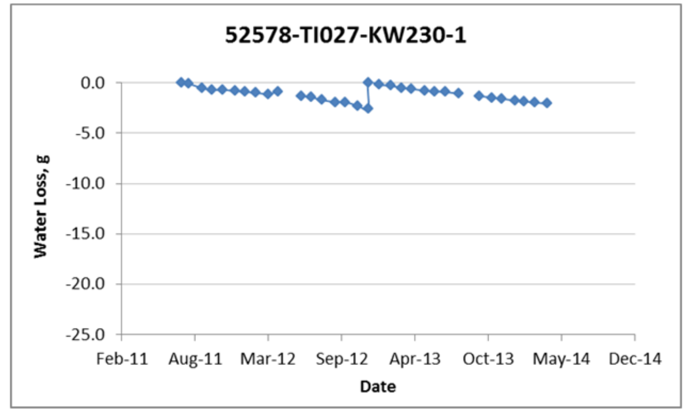
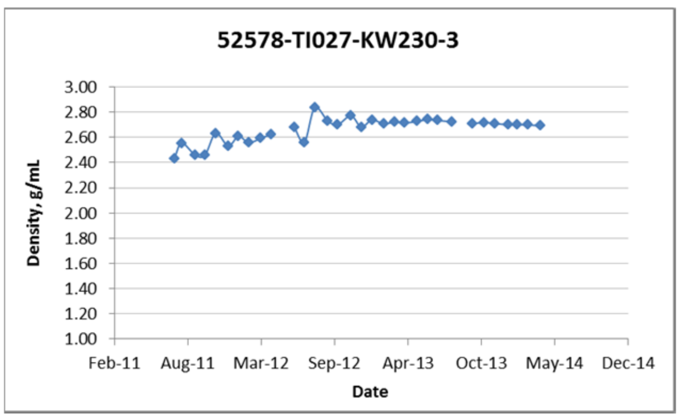
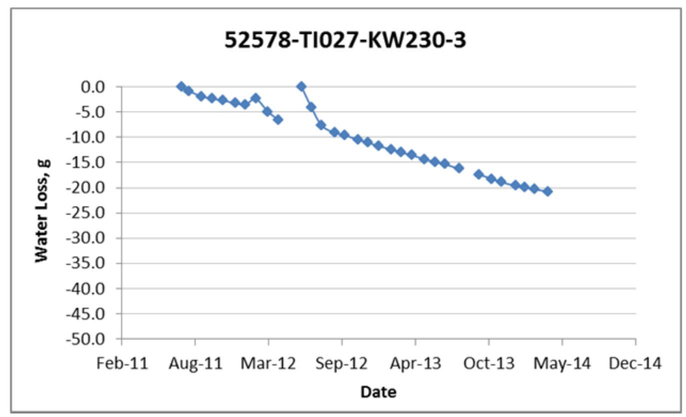
Sample ID	Settled Sludge Density History	Water Loss History
52578-TI027-KW230-1  2013		
52578-TI027-KW230-3  2013		

Table 6. (Continued)

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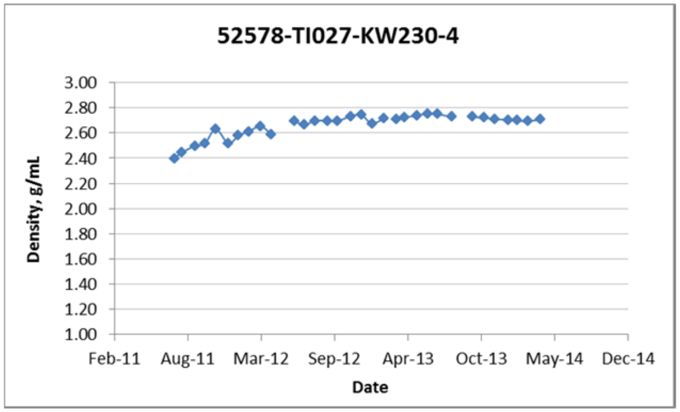
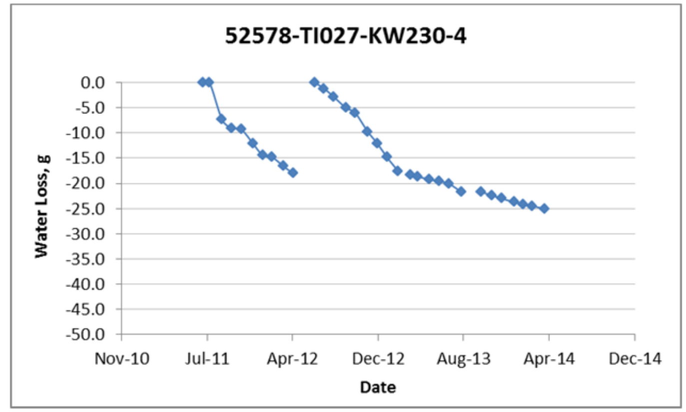
2013</td><td>
</td><td>
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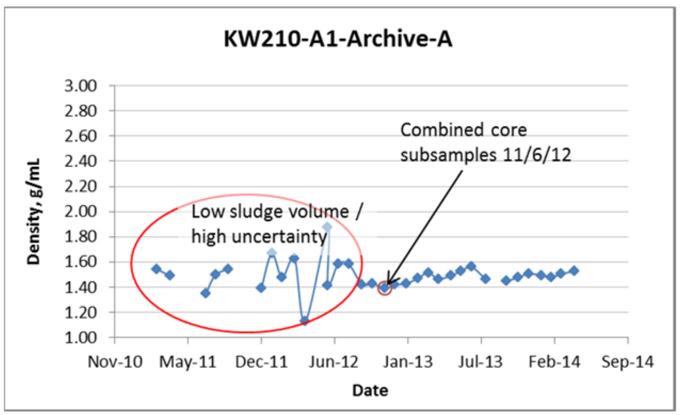
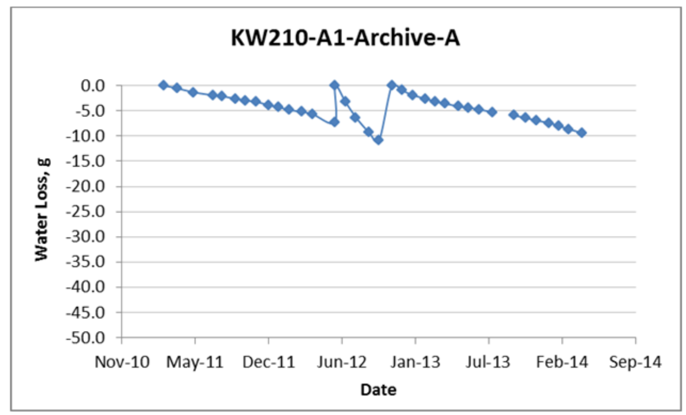
2013</td><td>
</td><td>
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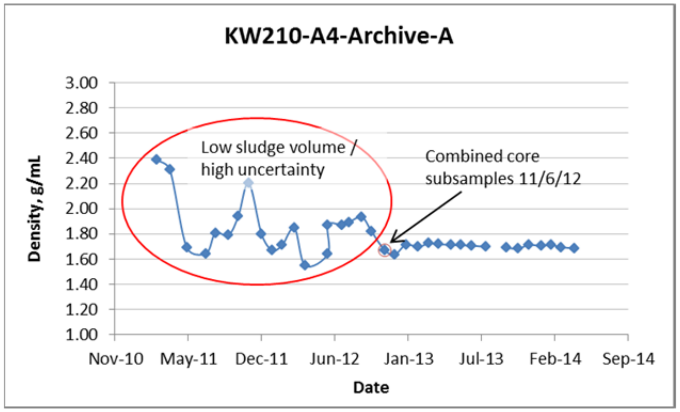
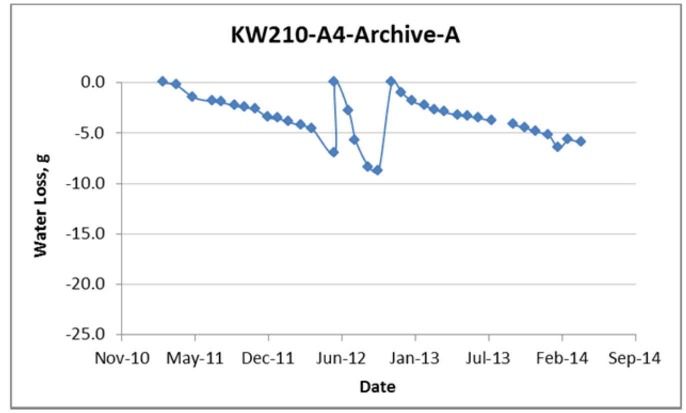
2013</td><td>
</td><td>
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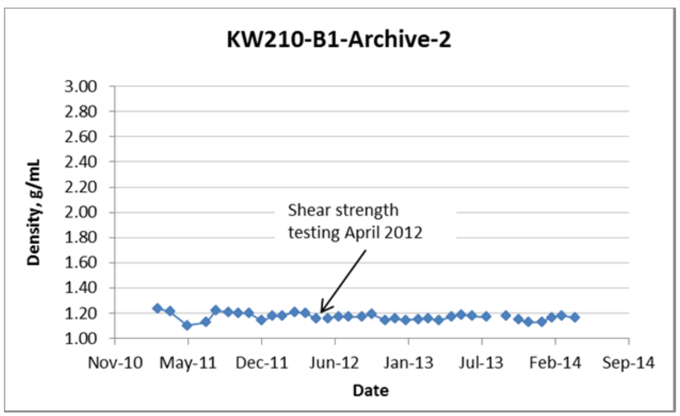
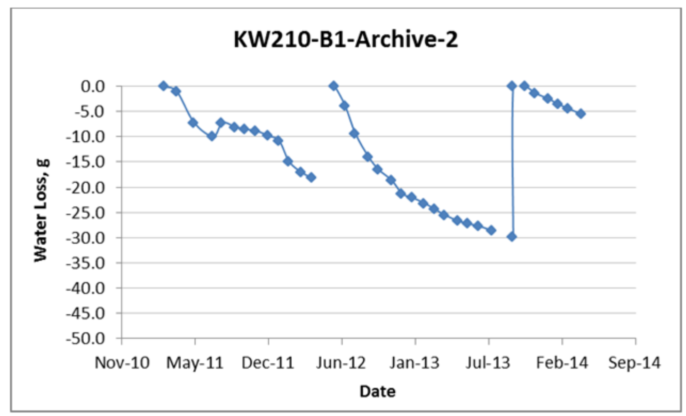
2014</td><td>
</td><td>
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Table 6. (Continued)

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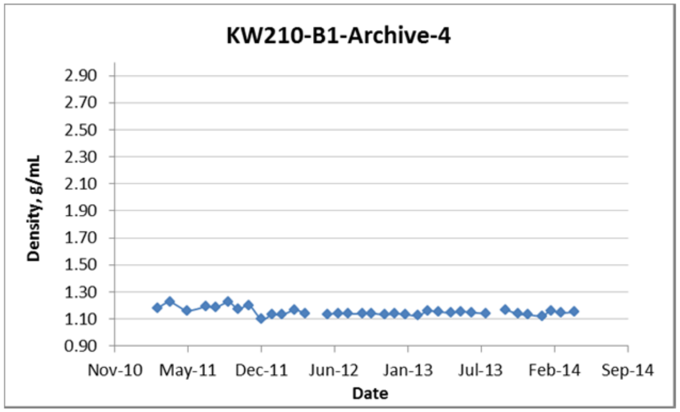
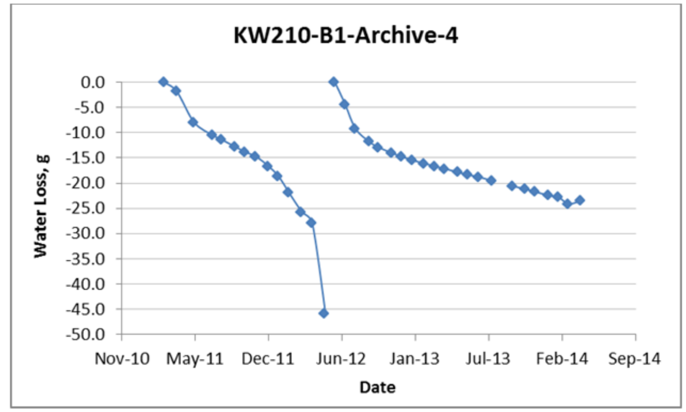
2013</td><td>
</td><td>
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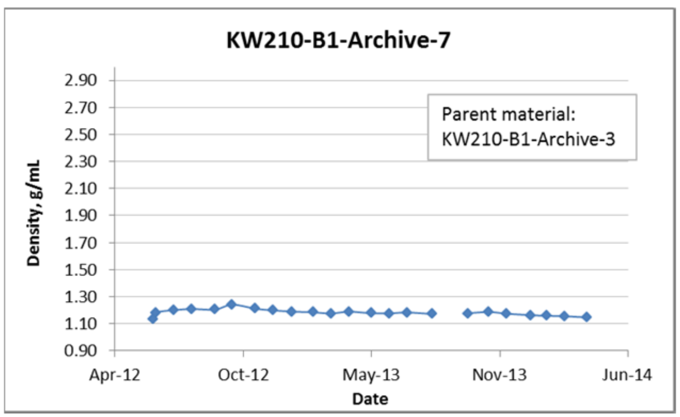
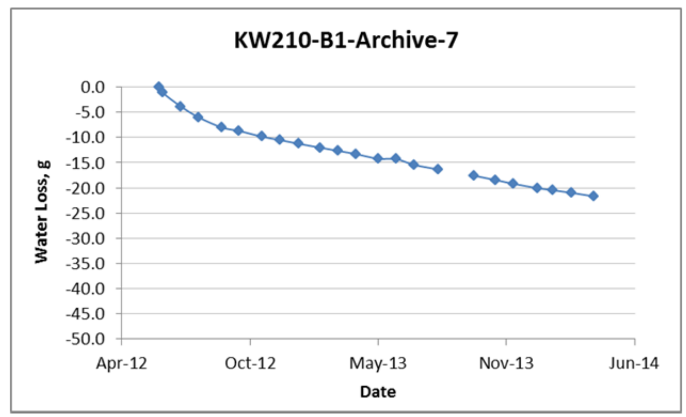
2013</td><td>
</td><td>
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Table 6. (Continued)

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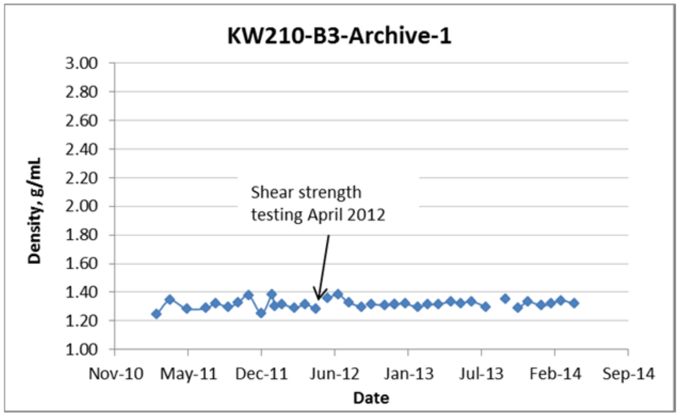
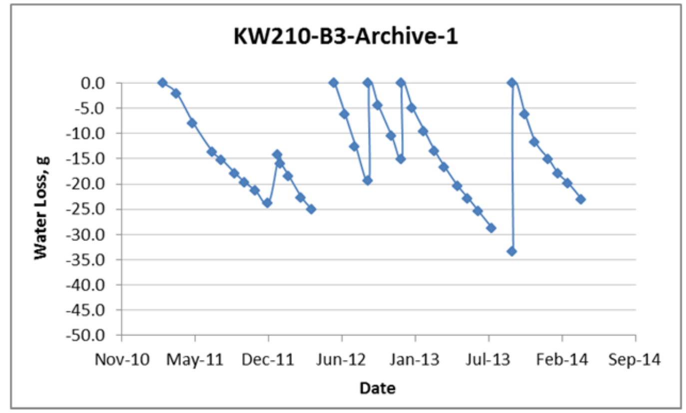
2014</td><td>
</td><td>
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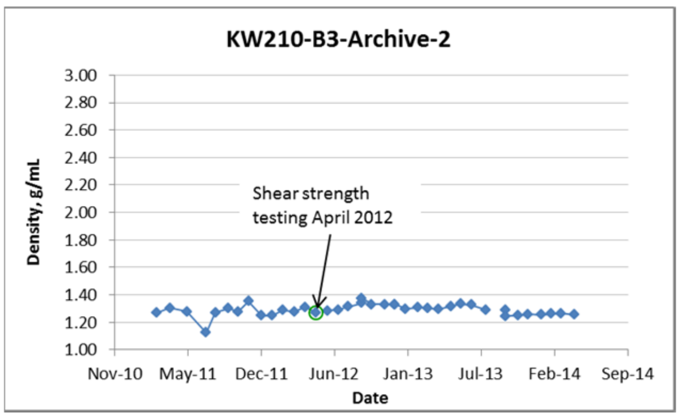
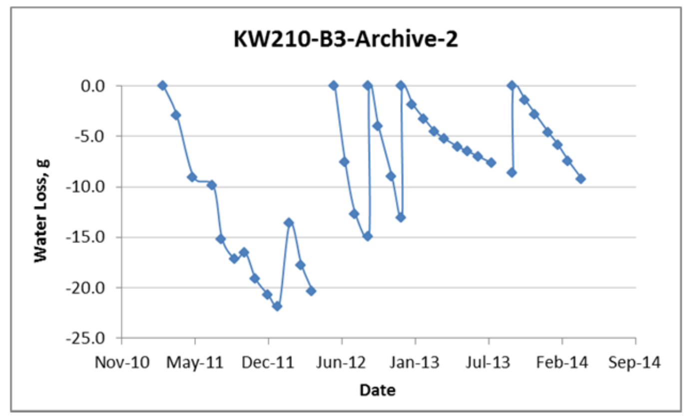
2014</td><td>
</td><td>
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Table 6. (Continued)

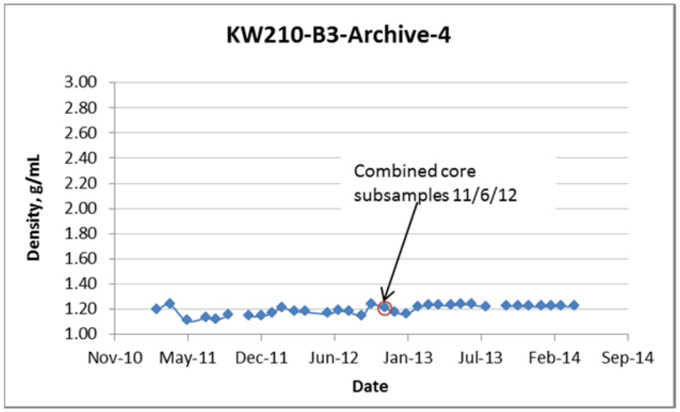
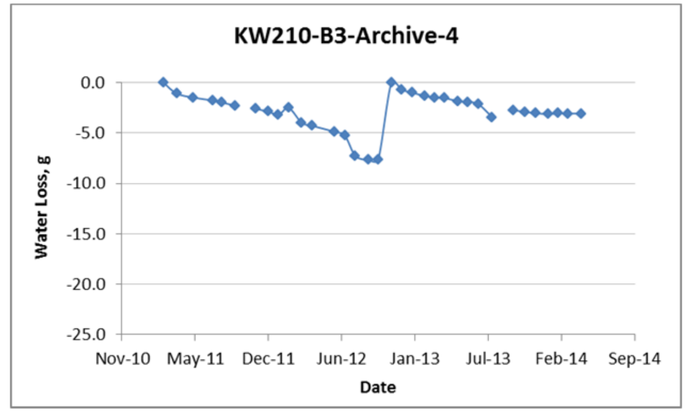
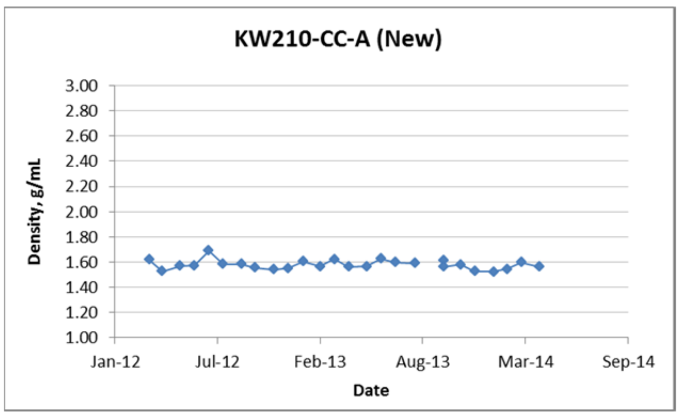
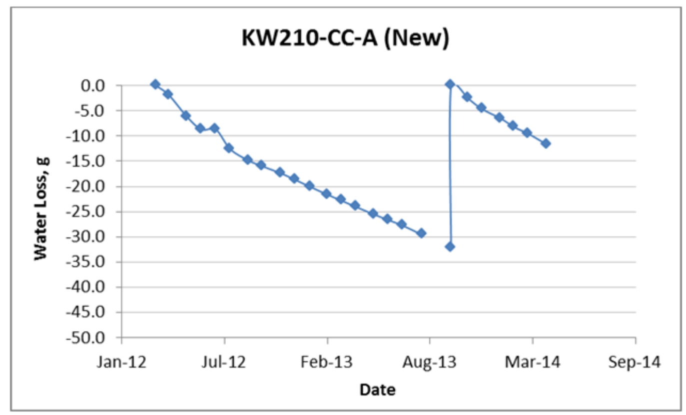
Sample ID	Settled Sludge Density History	Water Loss History
KW210-B3-Archive-4  2013	 	
KW210-CC-A (New)  2014	 	

Table 6. (Continued)

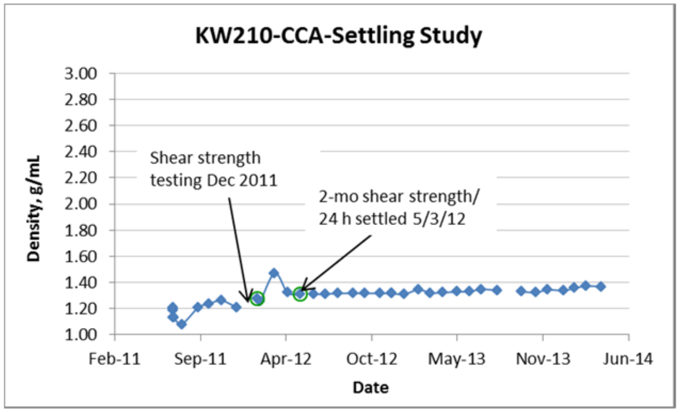
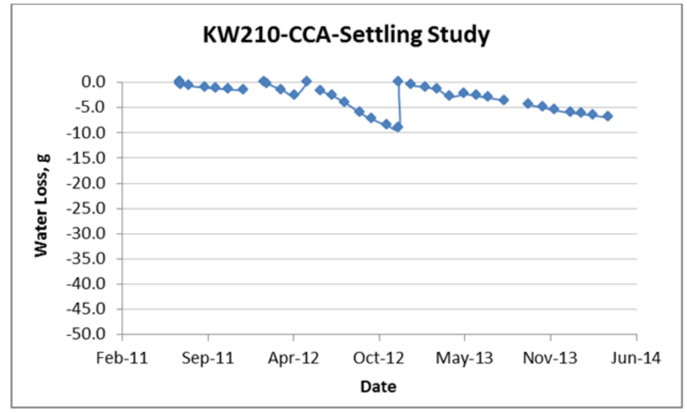
Sample ID	Settled Sludge Density History	Water Loss History
KW210-CCA-Settling Study  2014		
KW230-CC-420  2014	N/A, volume too small to measure accurately	

Table 6. (Continued)

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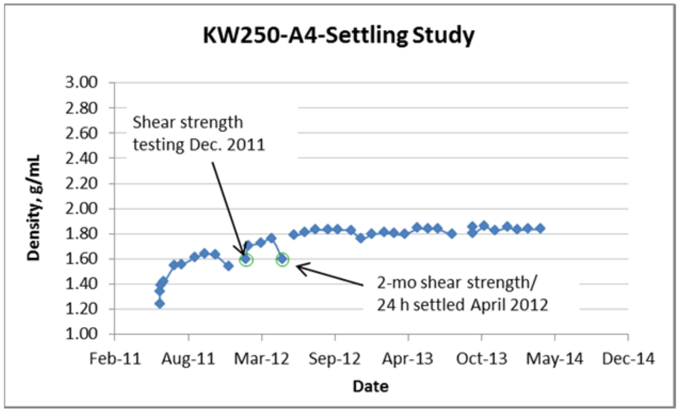
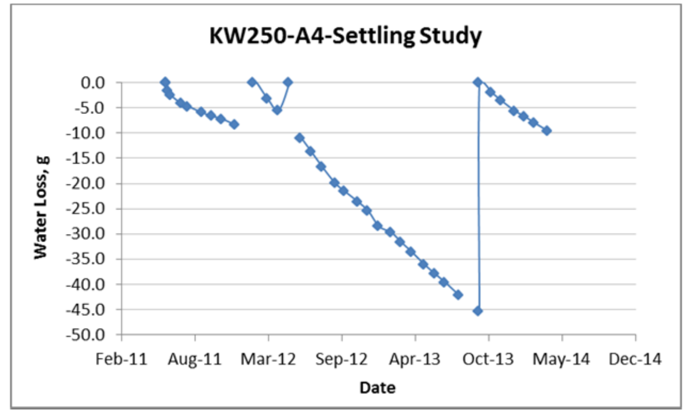
<tr><td>KW250-A4-Settling Study

2014</td><td>
</td><td>
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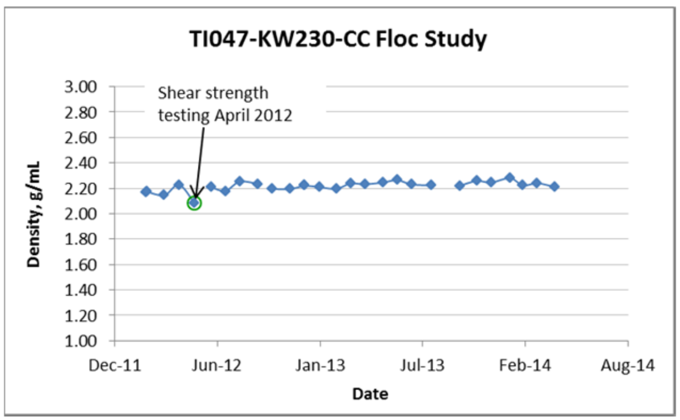
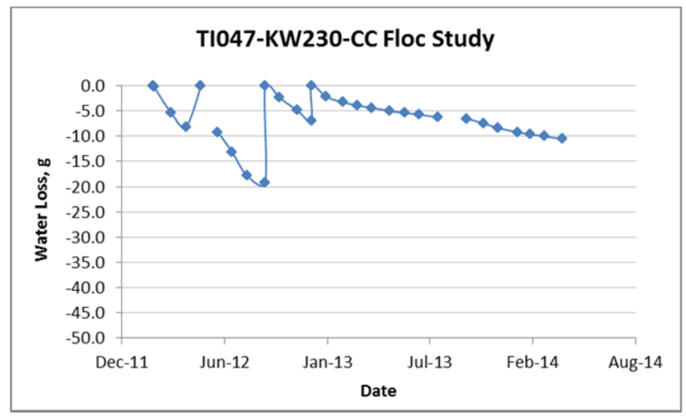
2013</td><td>
</td><td>
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Table 6. (Continued)

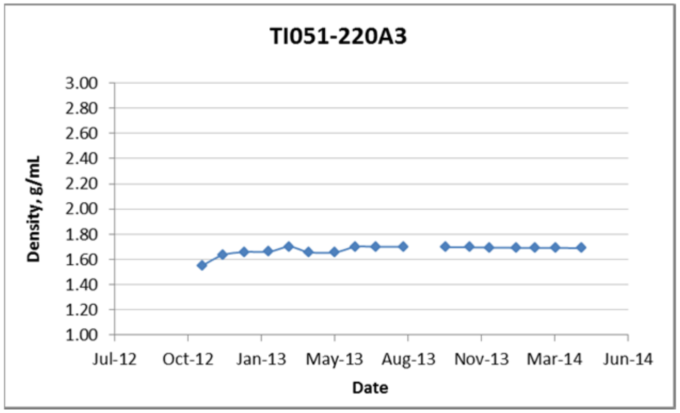
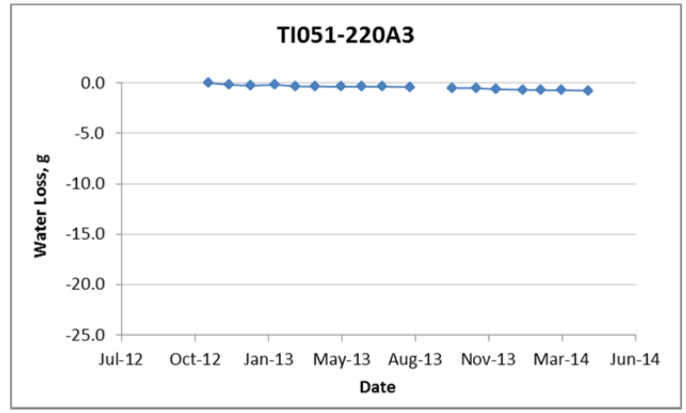
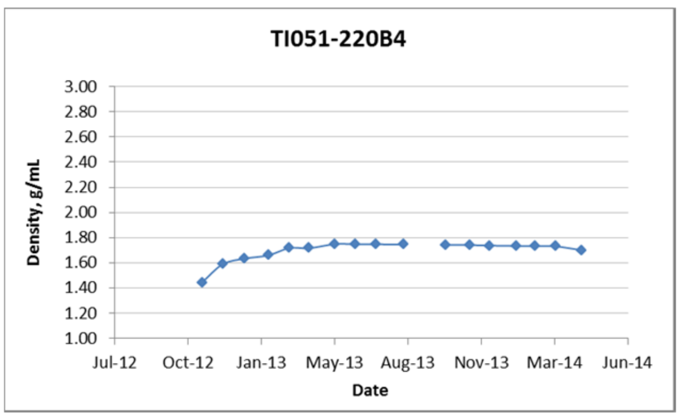
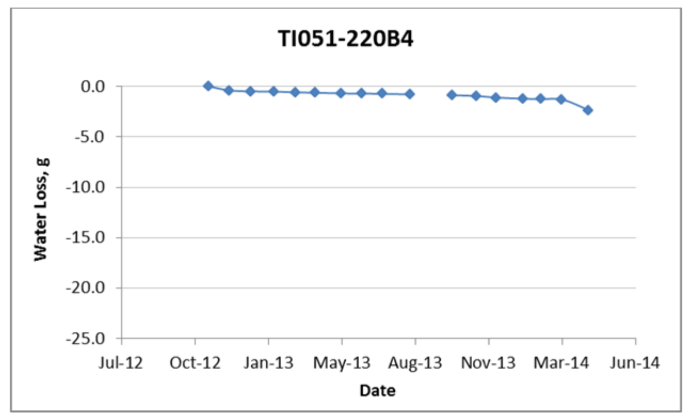
Sample ID	Settled Sludge Density History	Water Loss History
TI051-220A3  2013		
TI051-220 B4  2013		

Table 6. (Continued)

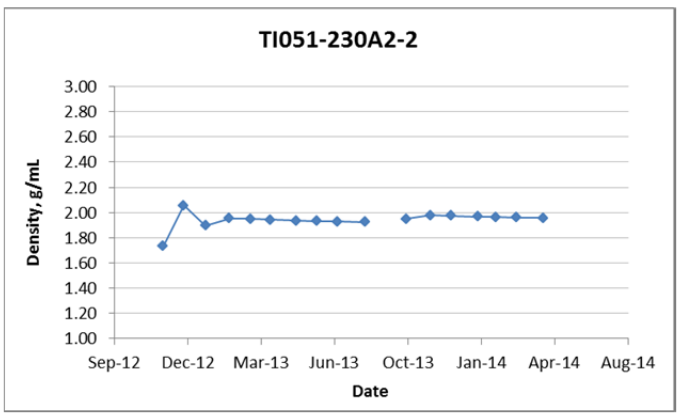
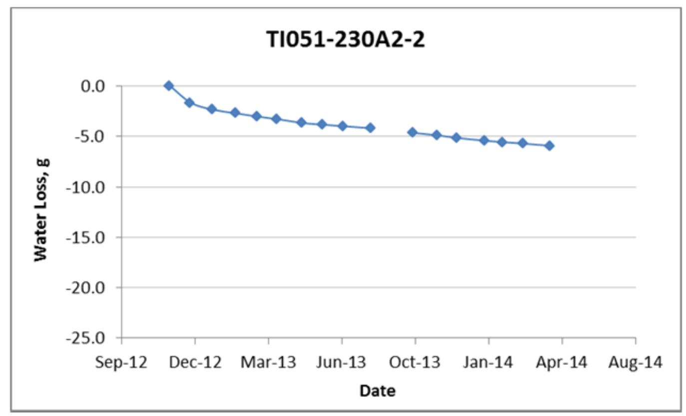
Sample ID	Settled Sludge Density History	Water Loss History
TI051-230A2-1  2013	N/A, volume too small to measure accurately	
TI051-230A2-2  2013		

Table 6. (Continued)

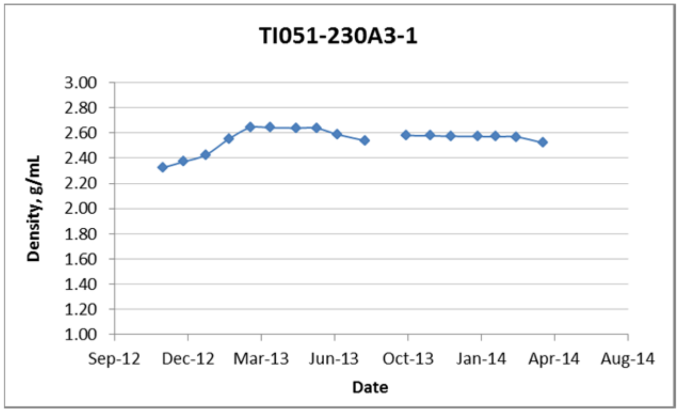
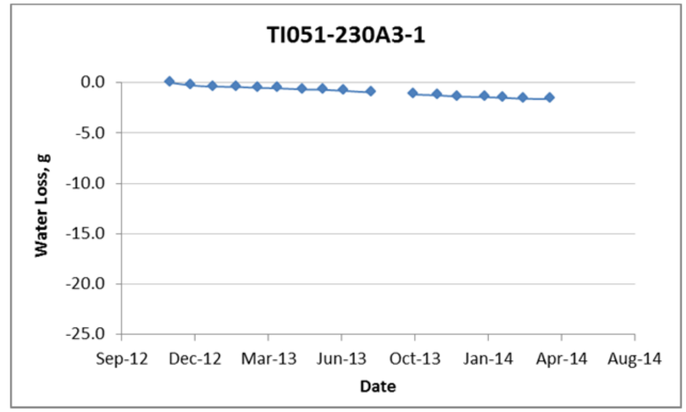
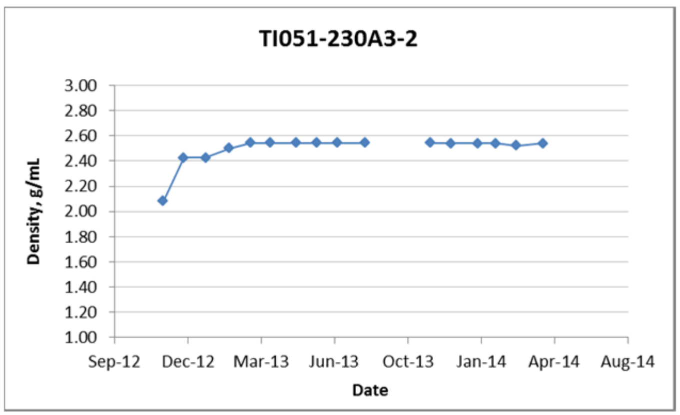
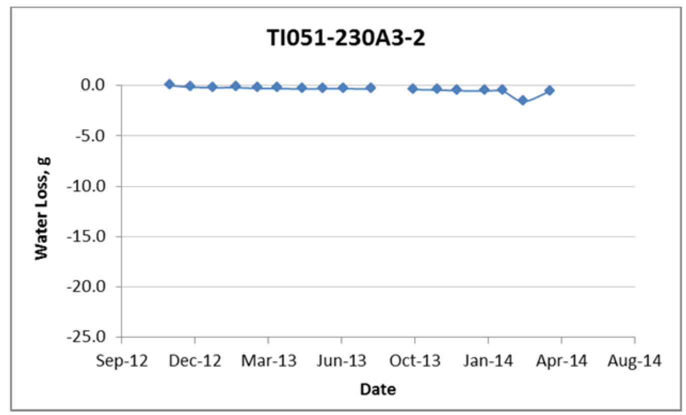
Sample ID	Settled Sludge Density History	Water Loss History
TI051-230A3-1  2013		
TI051-230A3-2  2014		

Table 6. (Continued)

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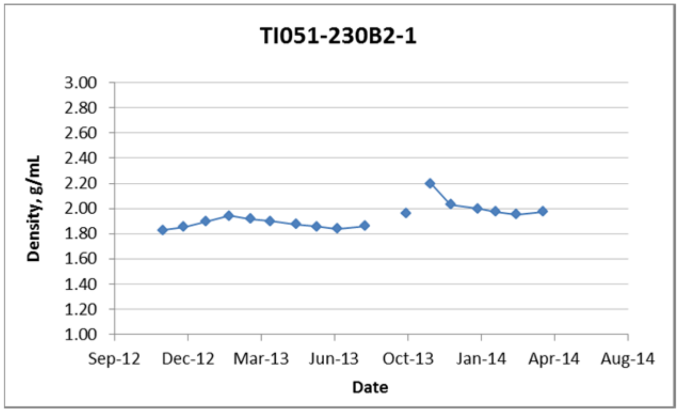
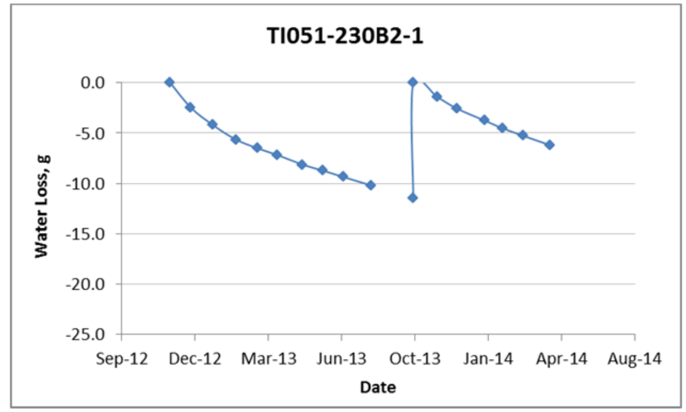
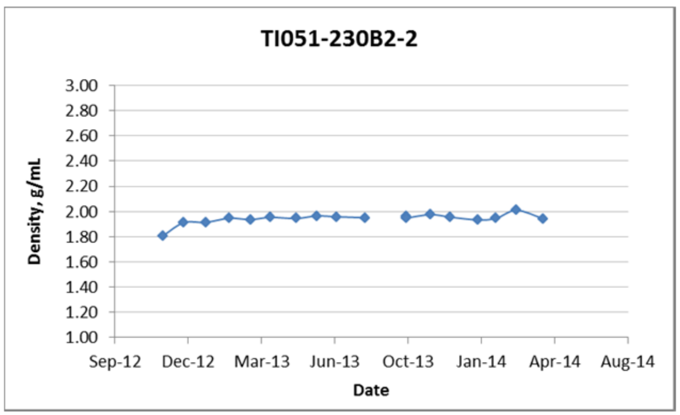
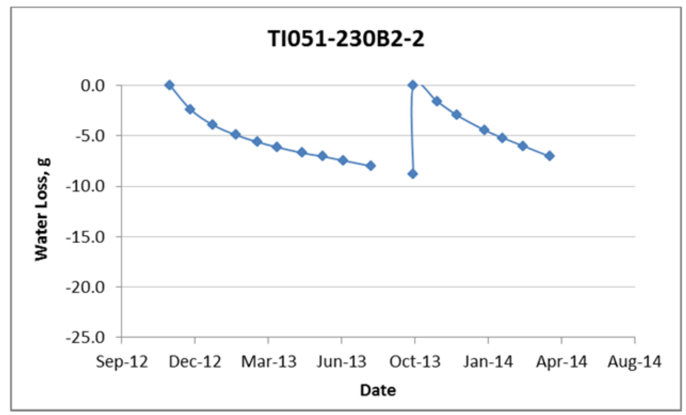
TI051-230B2-1  2013		
TI051-230B2-2  2013		

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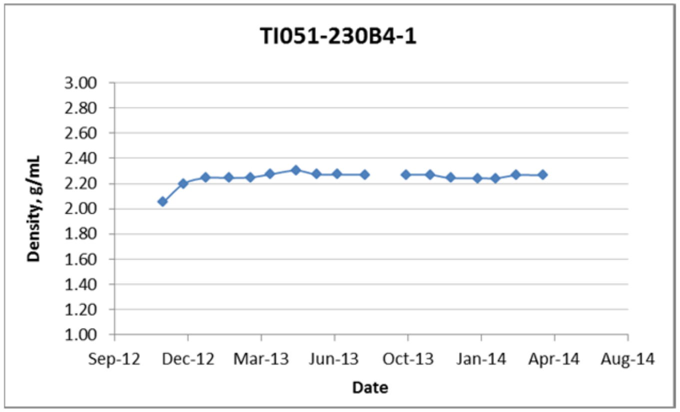
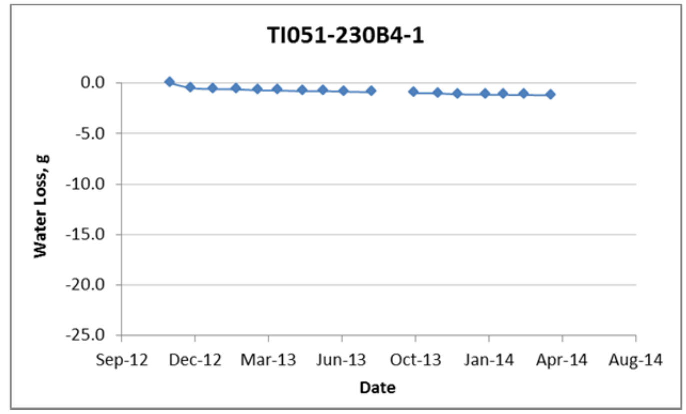
<td></td>
<td></td>
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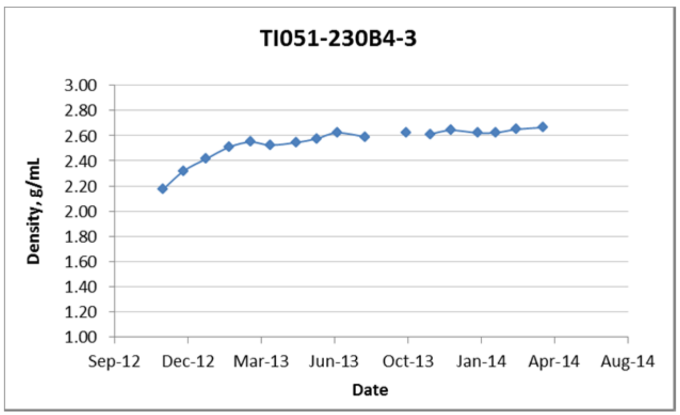
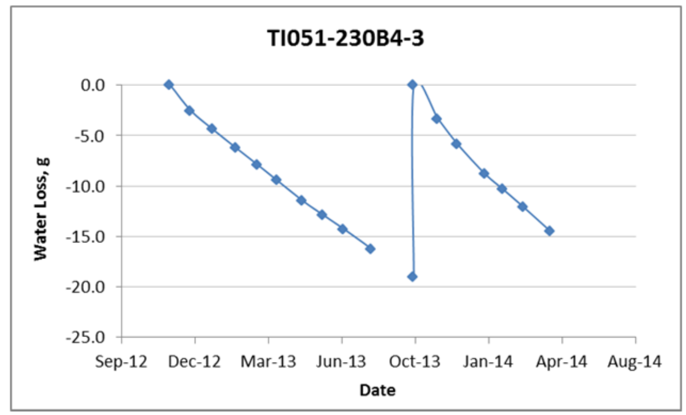
2013</td>
<td></td>
<td></td>
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Table 6. (Continued)

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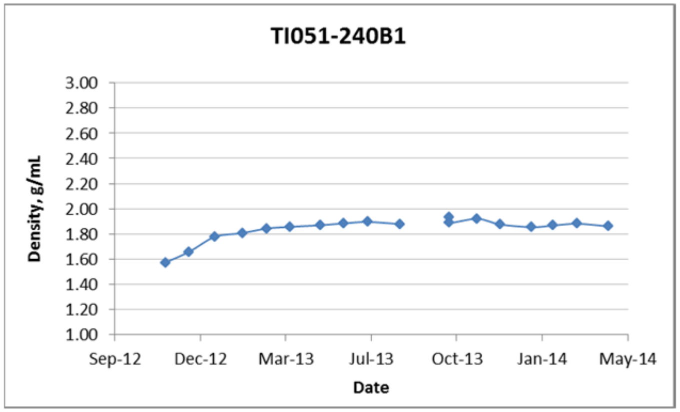
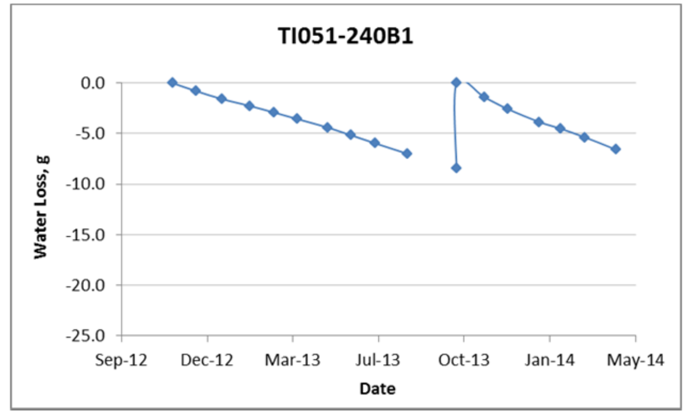
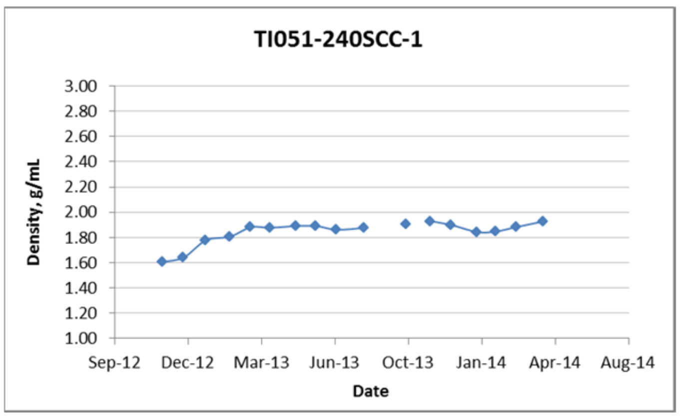
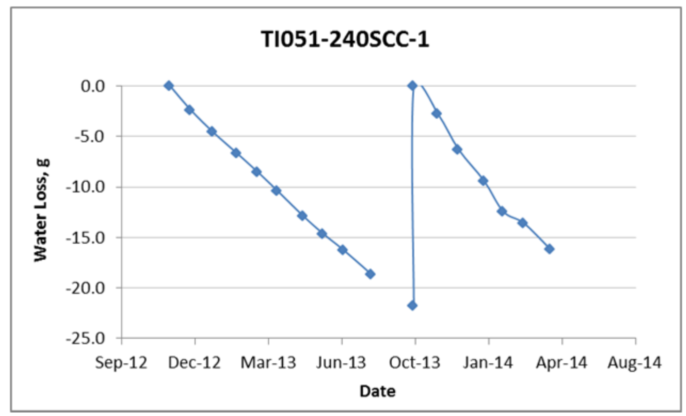
TI051-240B1  2013		
TI051-240SCC-1  2013		

Table 6. (Continued)

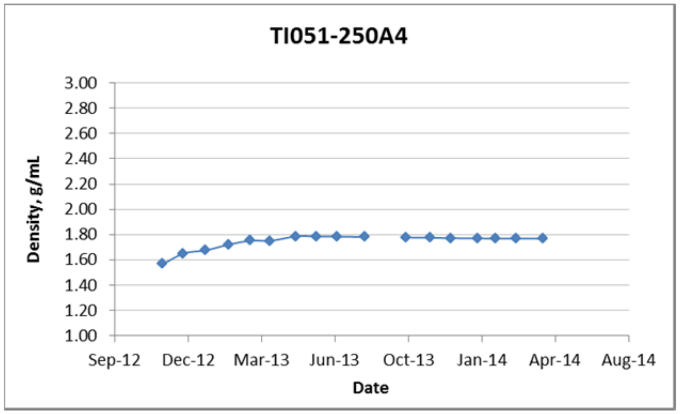
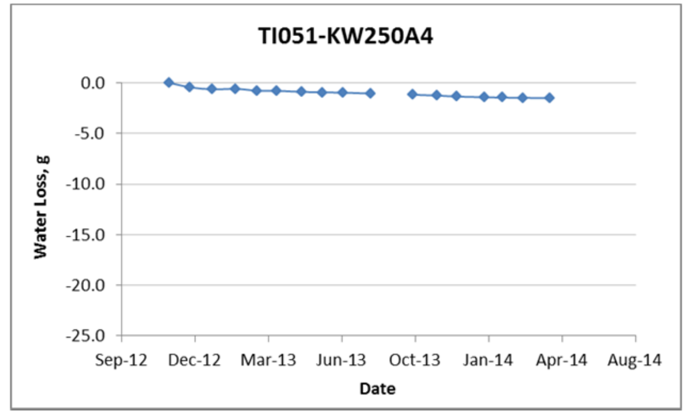
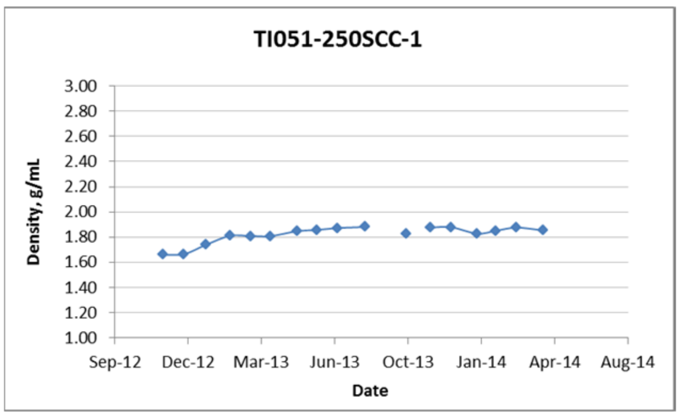
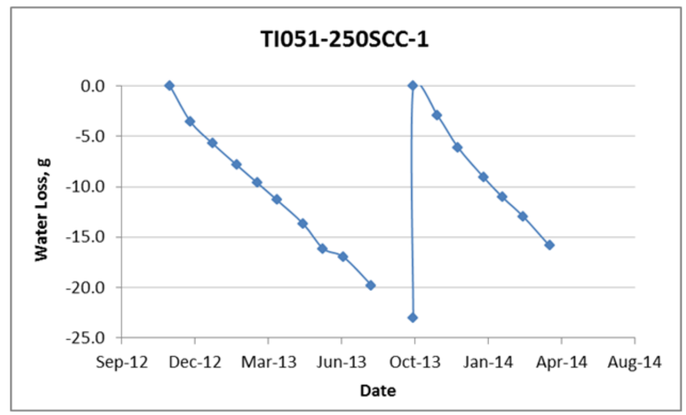
Sample ID	Settled Sludge Density History	Water Loss History
TI051-250A4  2013		
TI051-250SCC-1  2013		

Table 6. (Continued)

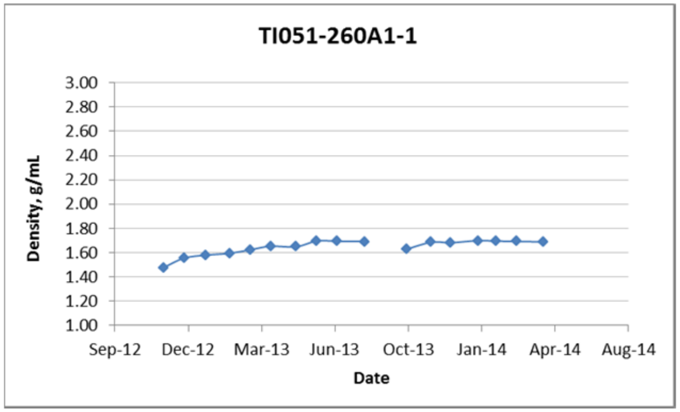
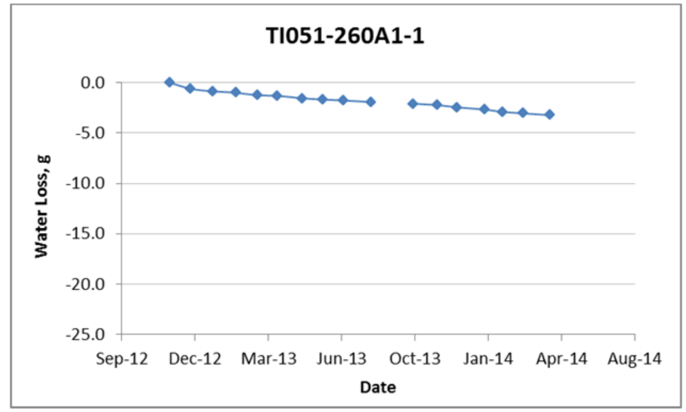
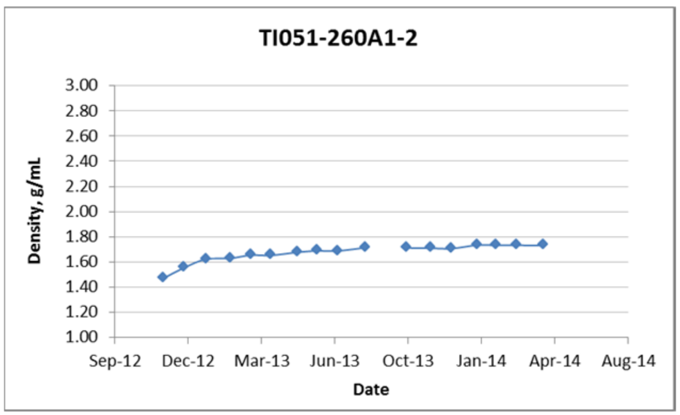
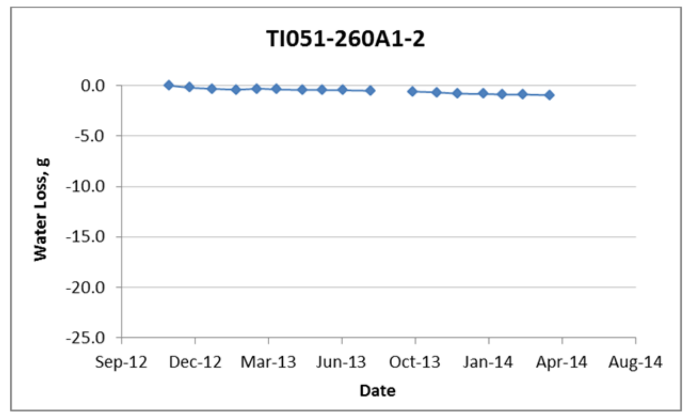
Sample ID	Settled Sludge Density History	Water Loss History
TI051-260A1-1  2013	 	
TI051-260A1-2  2013	 	

Table 6. (Continued)

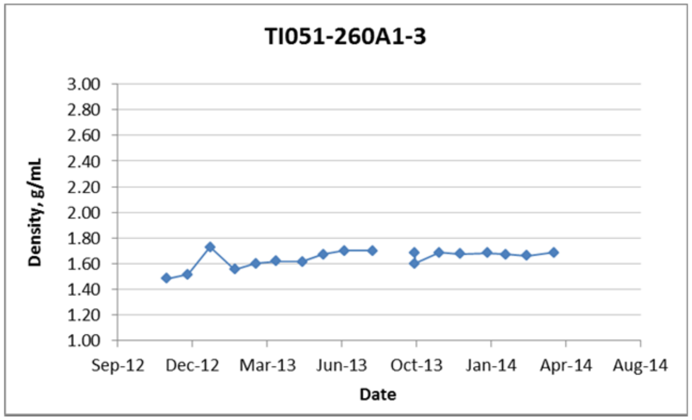
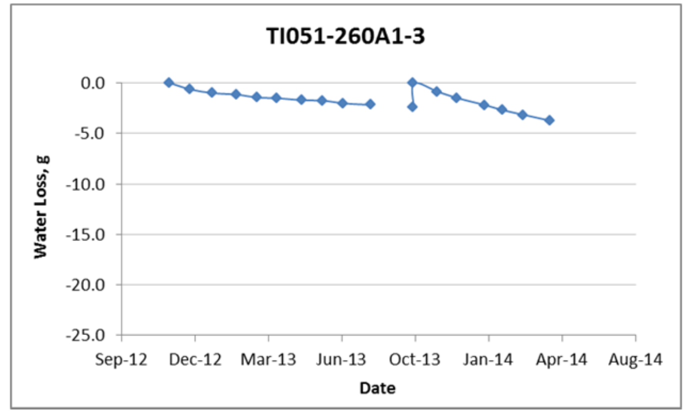
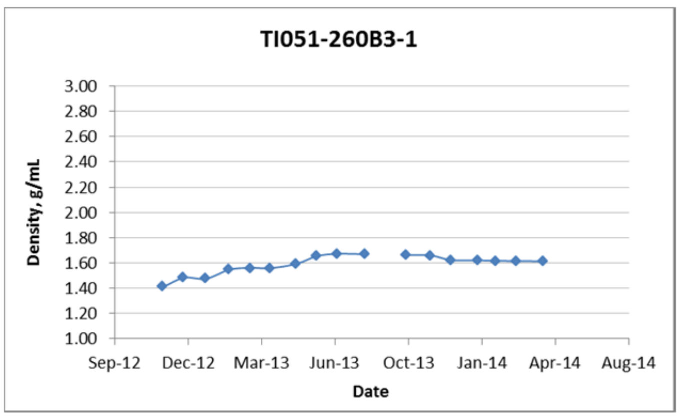
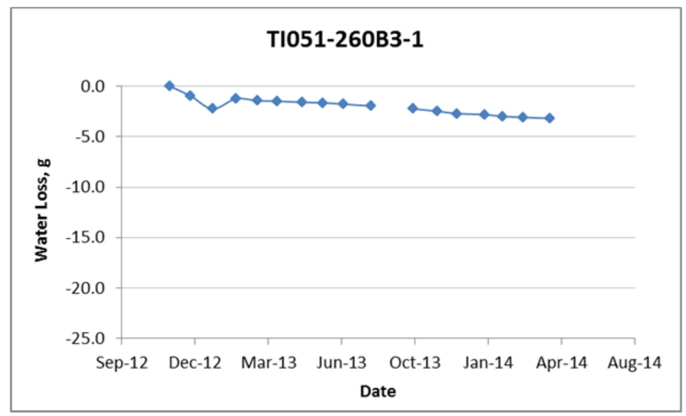
Sample ID	Settled Sludge Density History	Water Loss History
TI051-260A1-3  2013		
TI051-260B3-1  2013		

Table 6. (Continued)

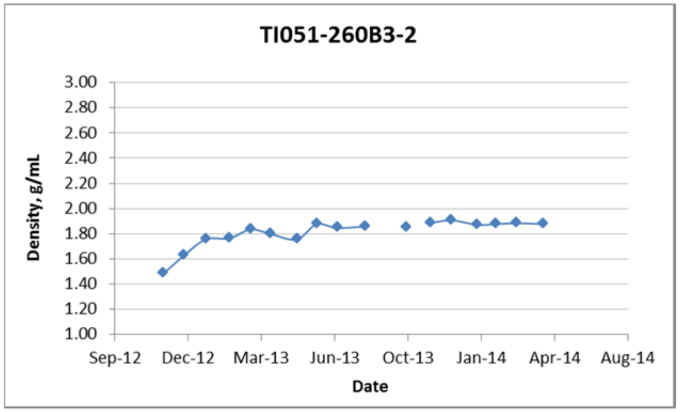
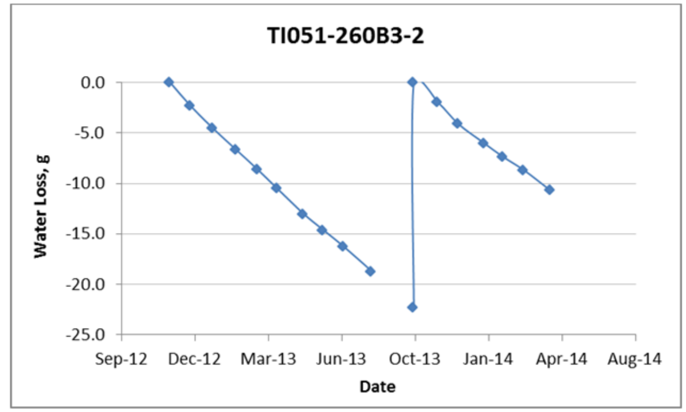
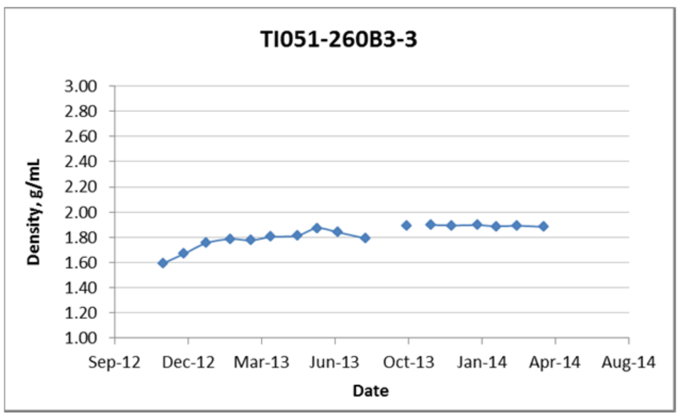
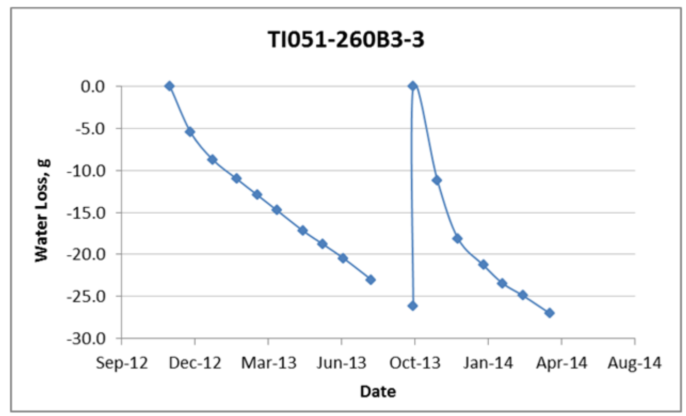
Sample ID	Settled Sludge Density History	Water Loss History
TI051-260B3-2  2013		
TI051-260B3-3  2013		

Table 6. (Continued)

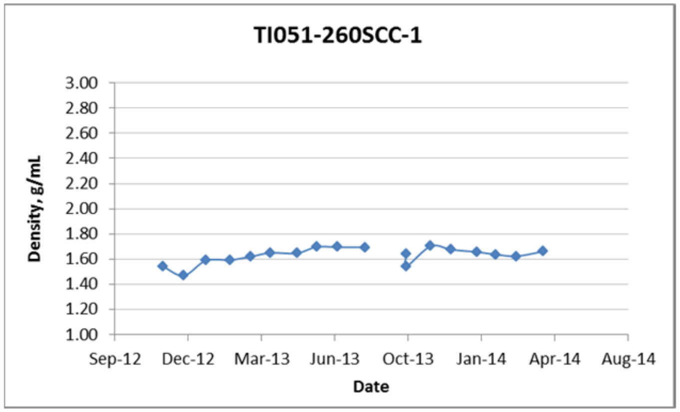
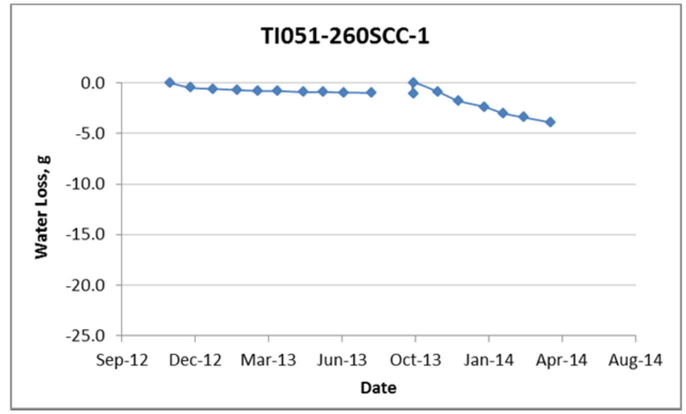
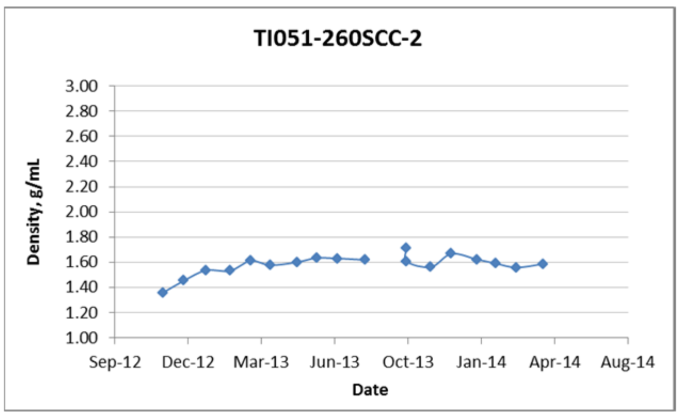
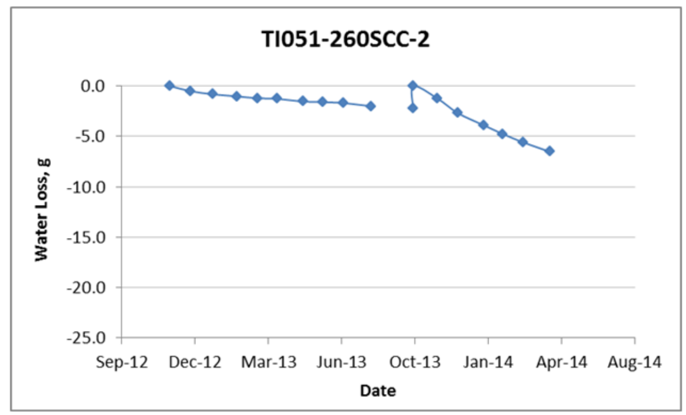
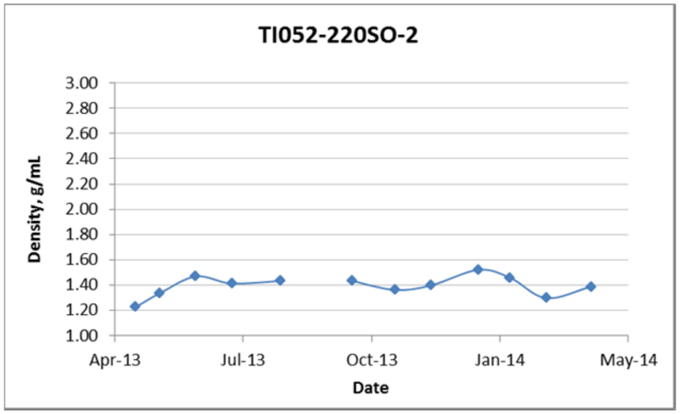
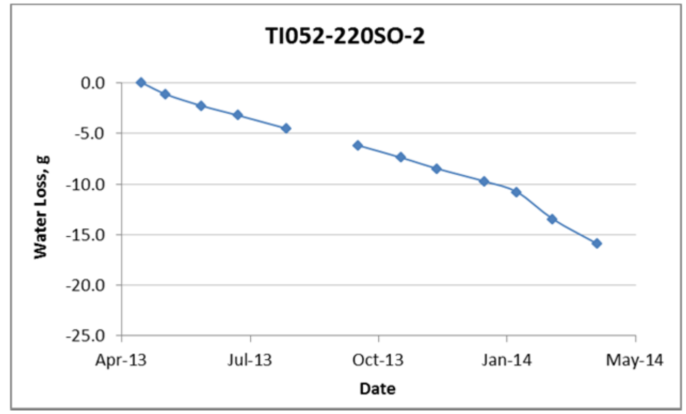
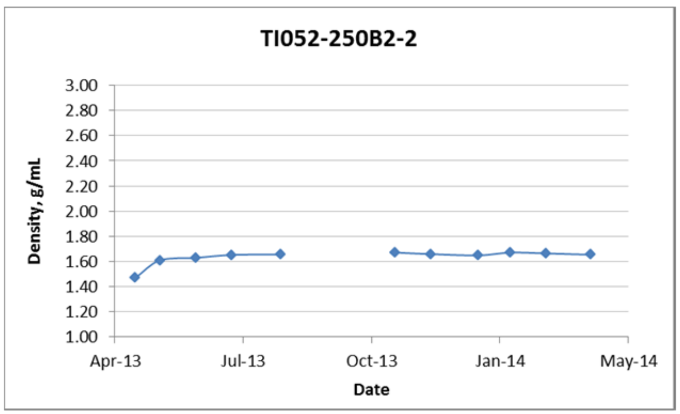
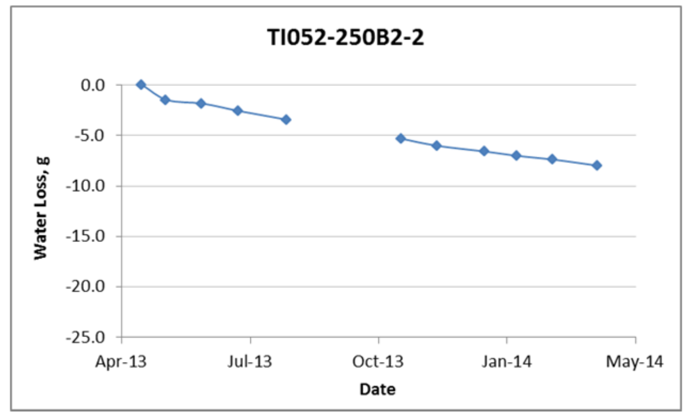
Sample ID	Settled Sludge Density History	Water Loss History
TI051-260SCC-1  2013		
TI051-260SCC-2  2013		

Table 6. (Continued)

Sample ID	Settled Sludge Density History	Water Loss History
TI052-220SO-2  2013		
TI052-250B2-2  2013		

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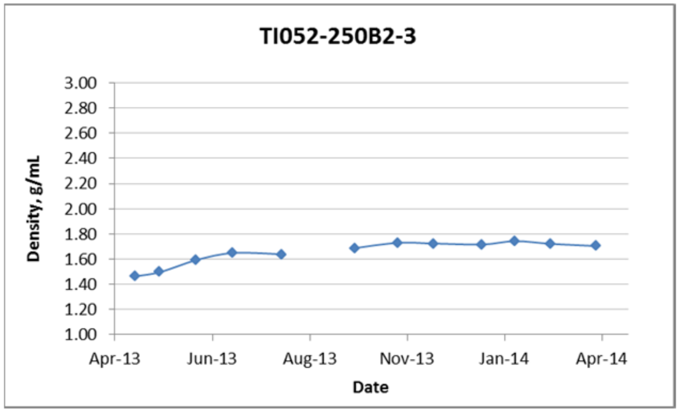
<td>
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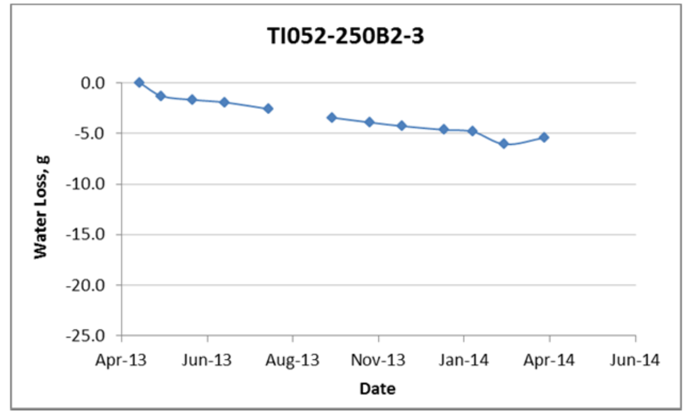
<td>
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Table 7. Pre-2004 Collected Samples Maintained Wet

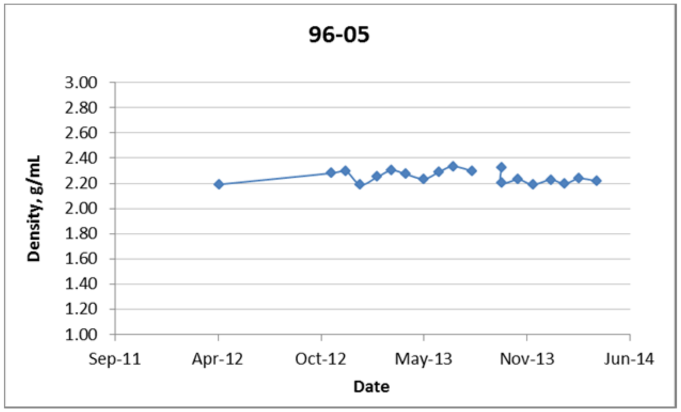
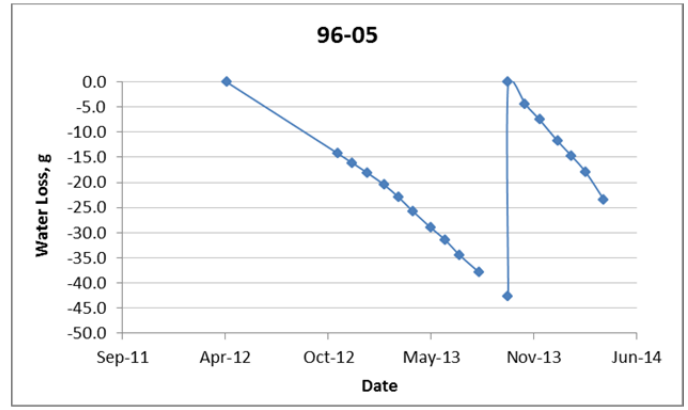
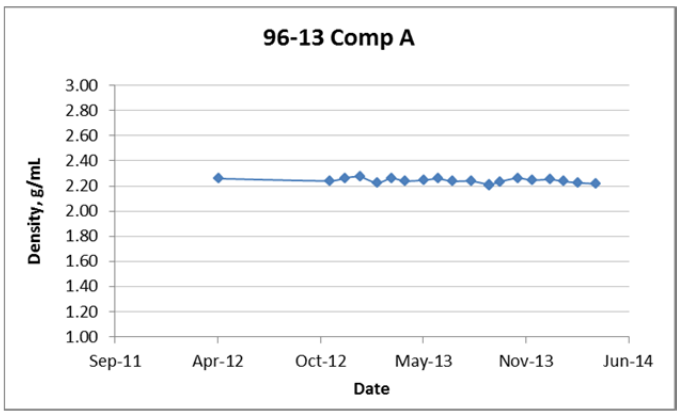
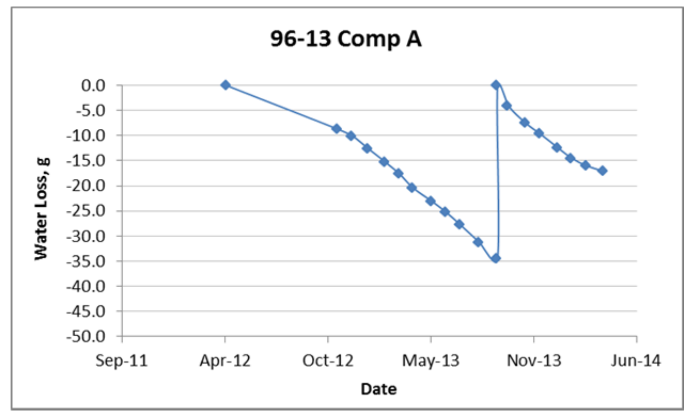
Sample ID	Settled Sludge Density History	Water Loss History
96-05  2014		
96-13 Comp A  2014		

Table 7. (Continued)

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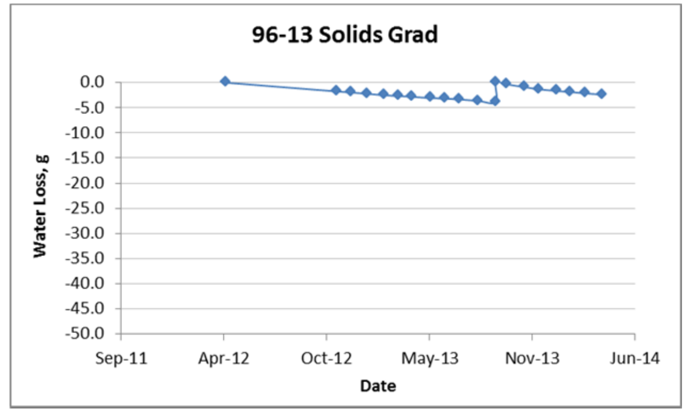
2014</td><td>N/A, note the large monolithic structure; volume cannot be calculated.</td><td>
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<tr><td>96-13 SSOL

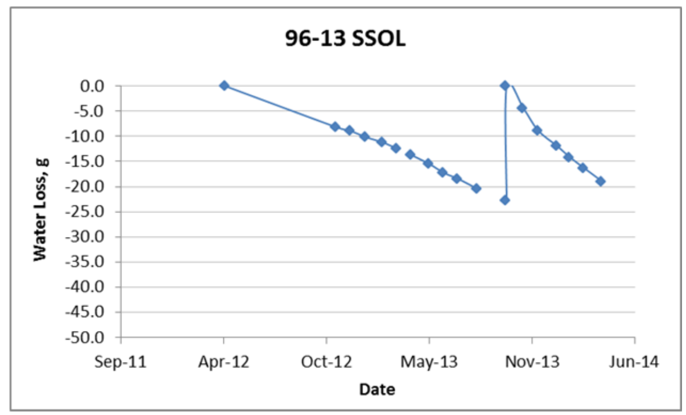

2013</td><td>N/A, note the large monolithic structure; volume cannot be calculated</td><td>
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Table 7. (Continued)

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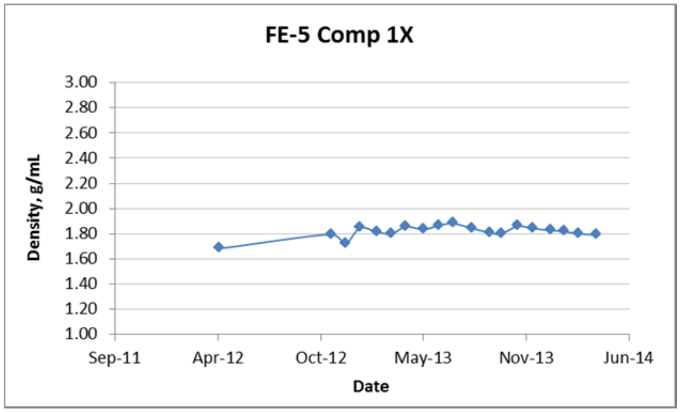
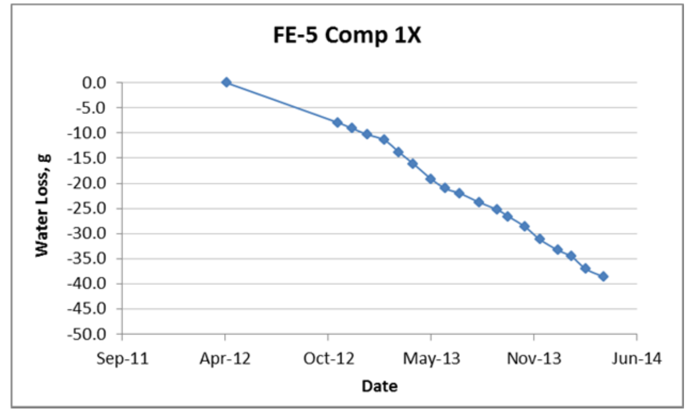
2014</td><td>
</td><td>
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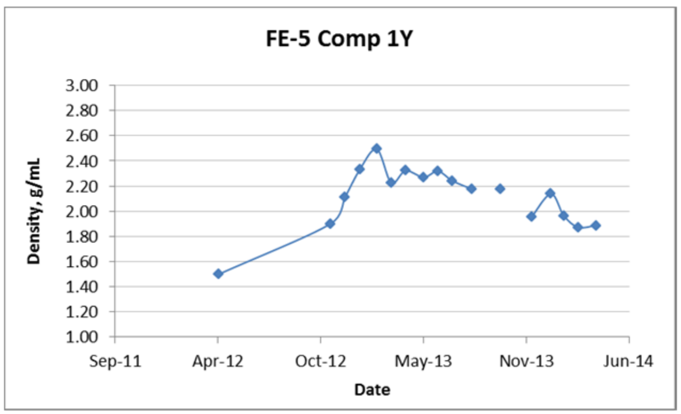
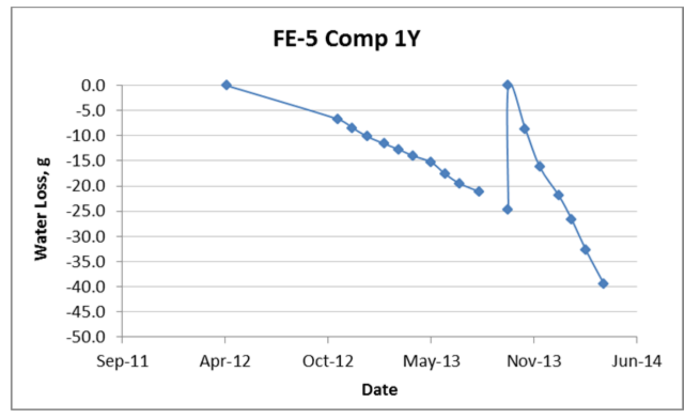
2013</td><td>
</td><td>
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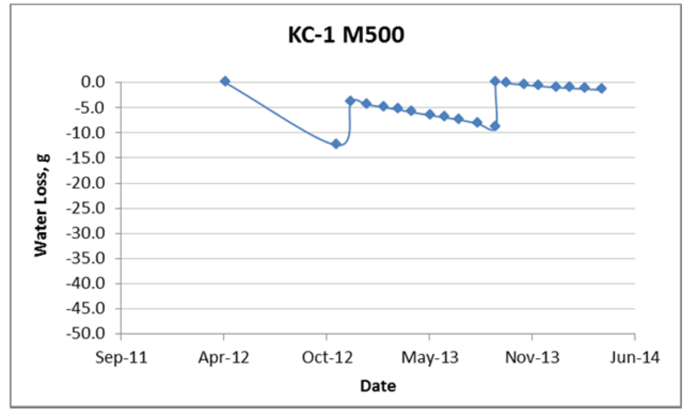
2014</td><td>N/A, volume too low to calculate</td><td>
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<tr><td>KC-2/3 Comp X

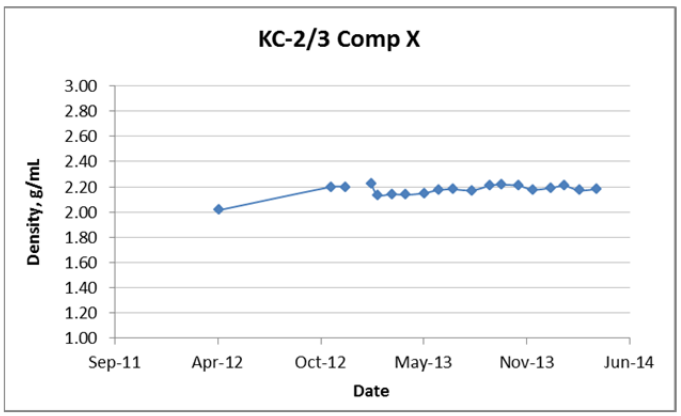
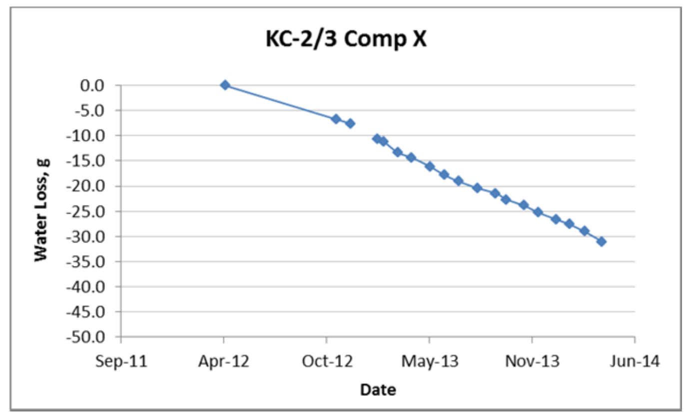
2014</td><td>
</td><td>
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Table 7. (Continued)

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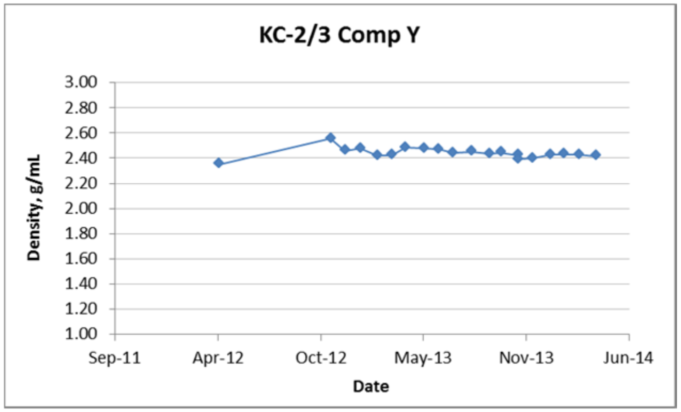
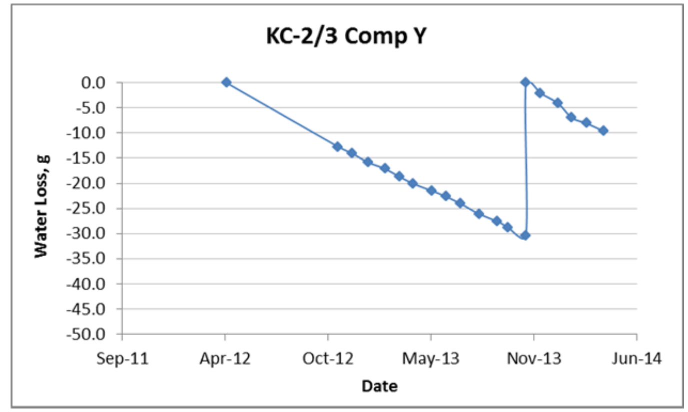
2014</td><td></td><td></td></tr>
<tr><td>KC-4-2 X

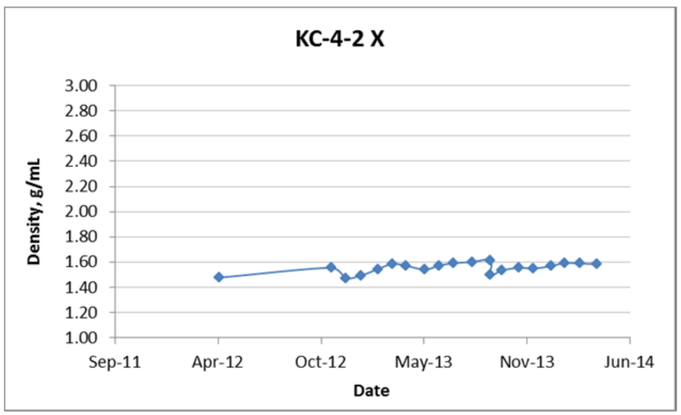
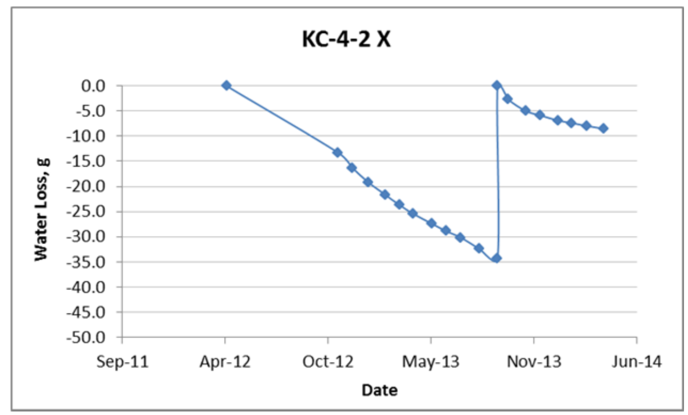
2014</td><td></td><td></td></tr>
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Table 7. (Continued)

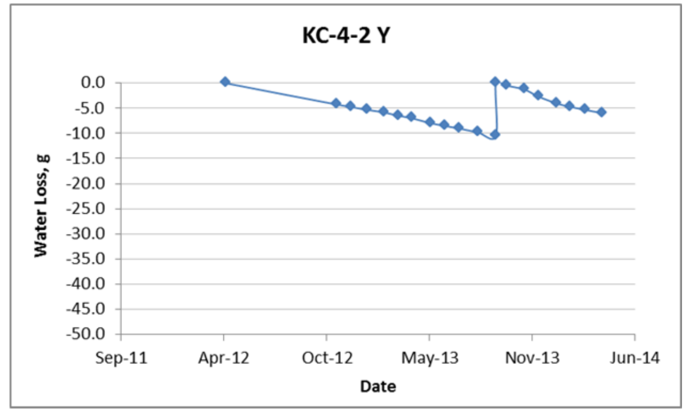
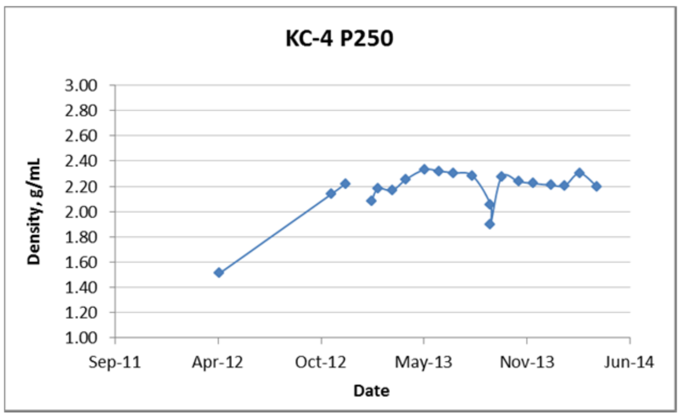
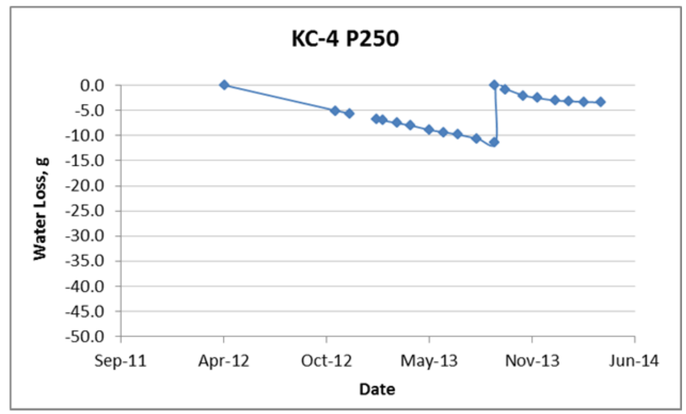
Sample ID	Settled Sludge Density History	Water Loss History
KC-4-2 Y  2014	N/A, volume too low to calculate. (Note the small blade-like structure above settled solids in foreground)	
KC-4 P250  2014		

Table 7. (Continued)

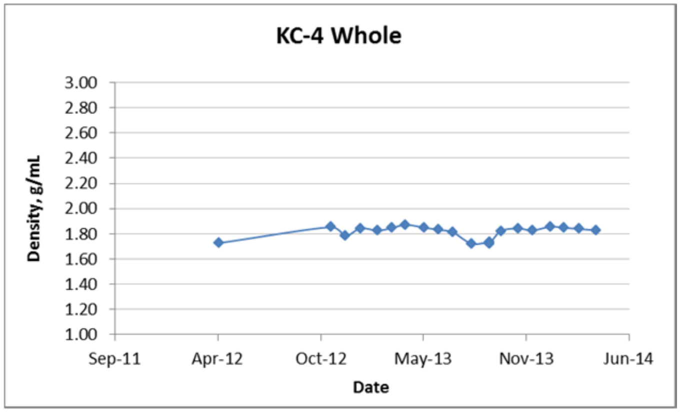
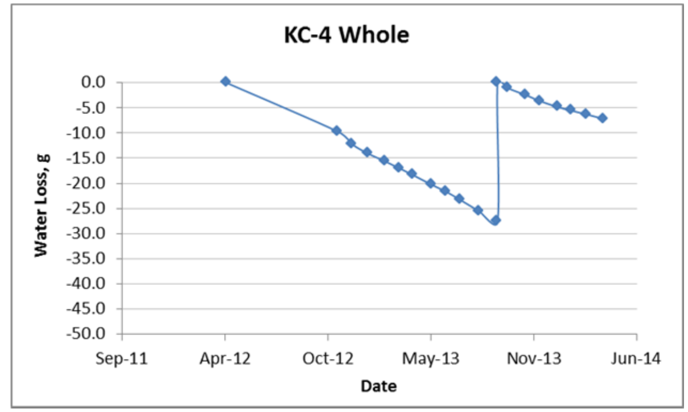
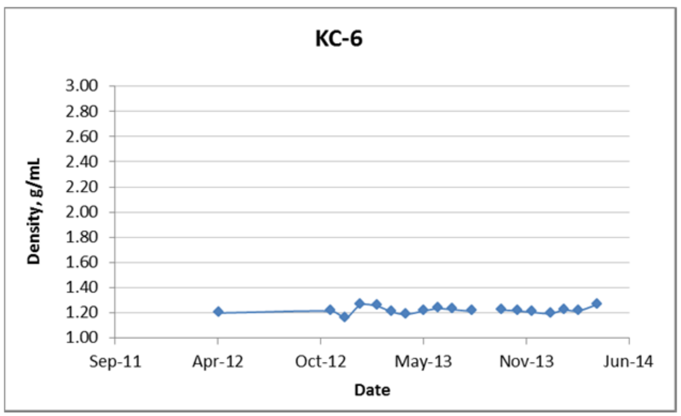
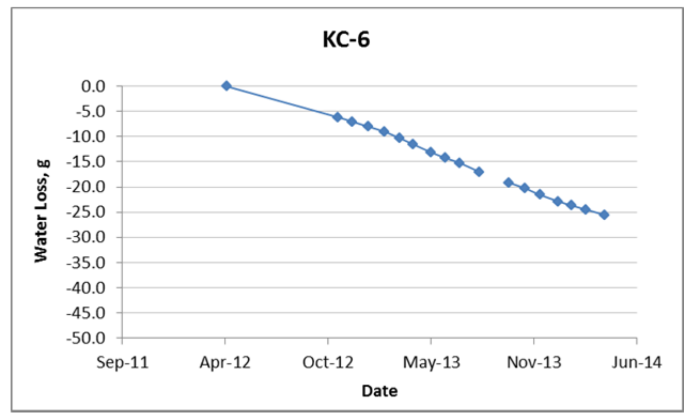
Sample ID	Settled Sludge Density History	Water Loss History
KC-4 Whole  2014		
KC-6  2013		

Table 7. (Continued)

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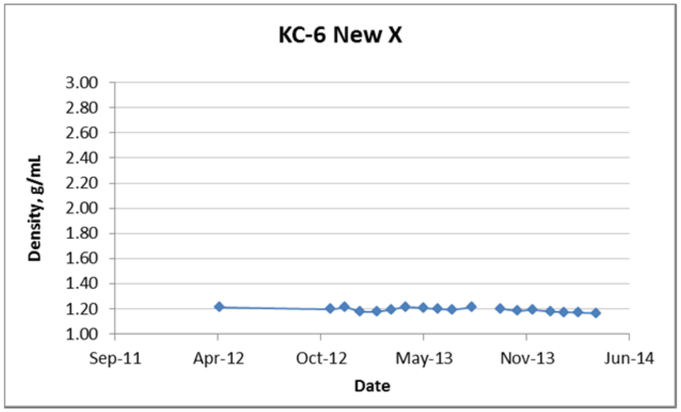
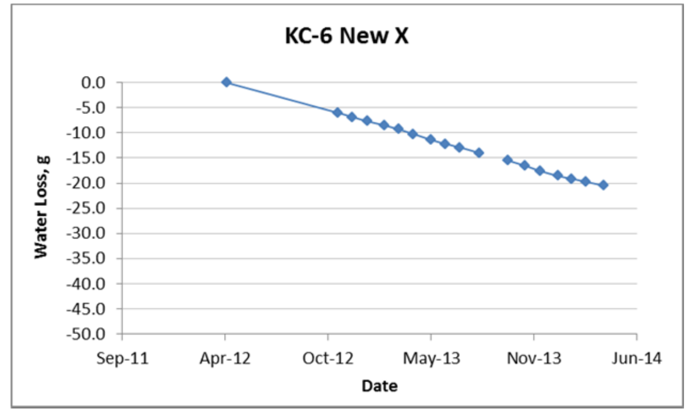
2014</td><td>
</td><td>
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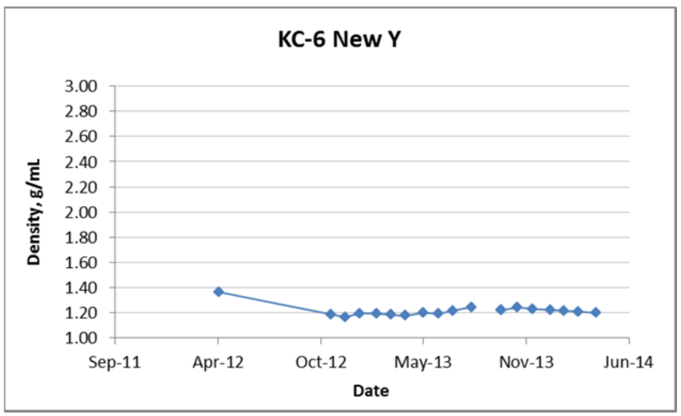
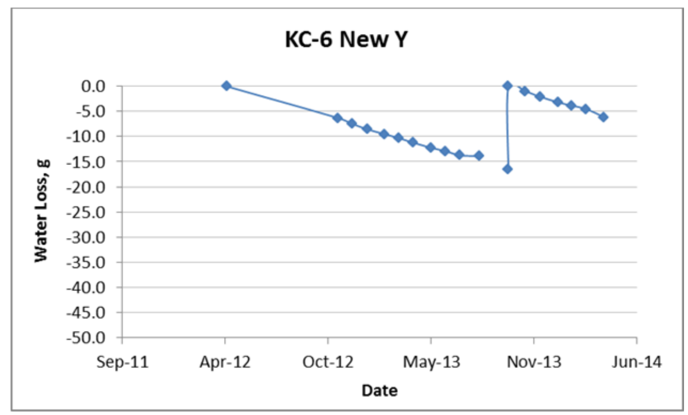
2014</td><td>
</td><td>
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Table 7. (Continued)

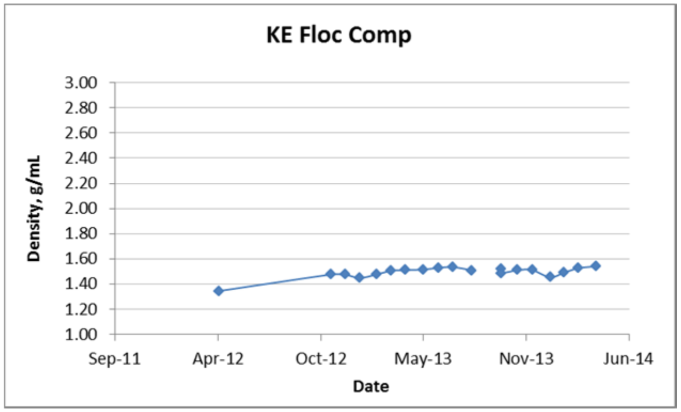
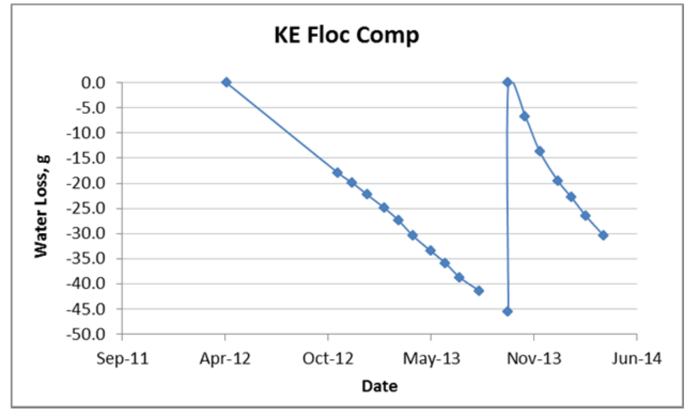
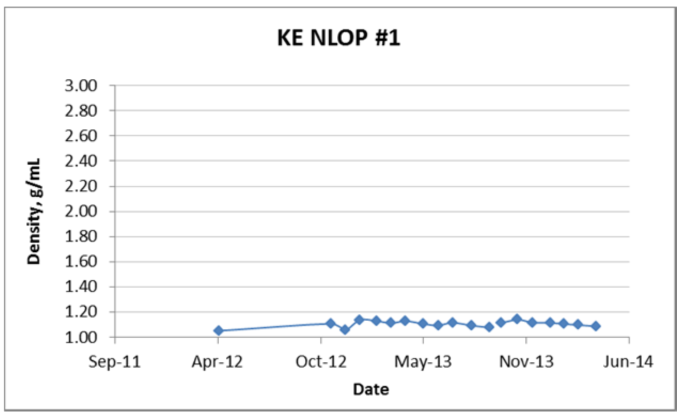
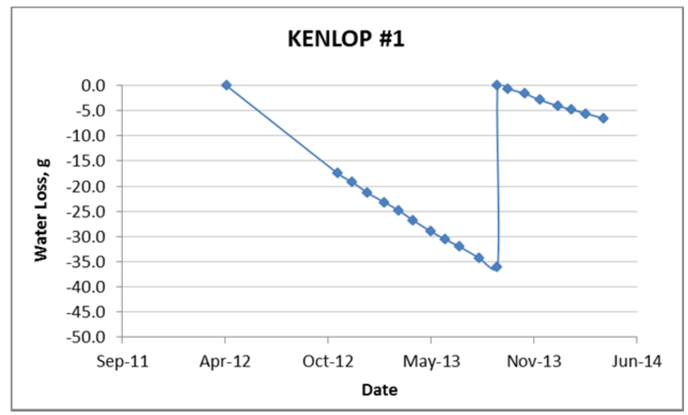
Sample ID	Settled Sludge Density History	Water Loss History
KE Floc Comp  2014		
KE NLOP #1  2013		

Table 7. (Continued)

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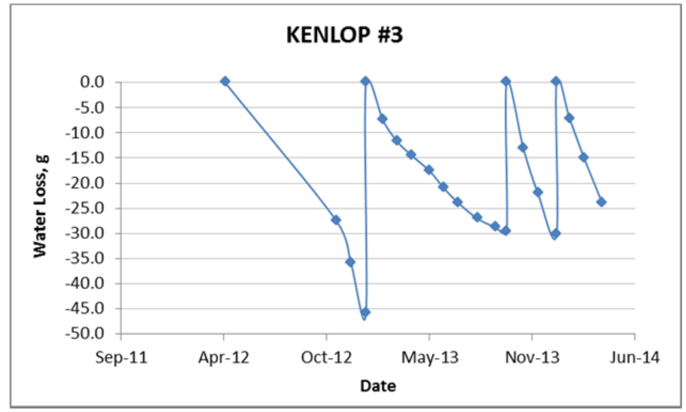
<tr><th>Sample ID</th><th>Settled Sludge Density History</th><th>Water Loss History</th></tr>
<tr>
<td>

KE NLOP #3

2013

</td>
<td>This is a unique sludge type in that it has an extremely turbid suspended solids layer that does not settle out. Either the settled sludge height only was recorded or the sludge plus suspended solids height was recorded—depending on the lighting in the cell.</td>
<td>



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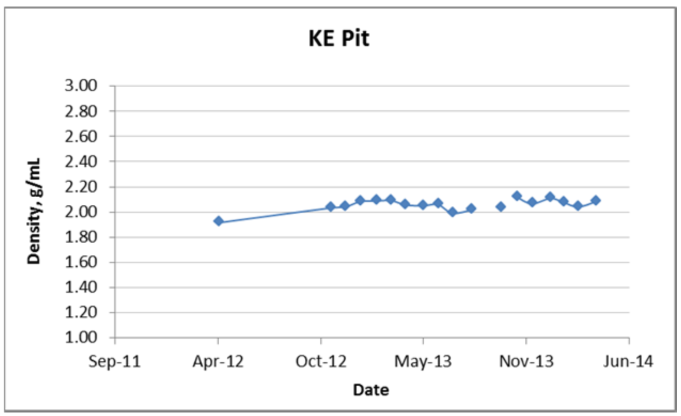
<tr>
<td>

KE Pit

2013

</td>
<td>



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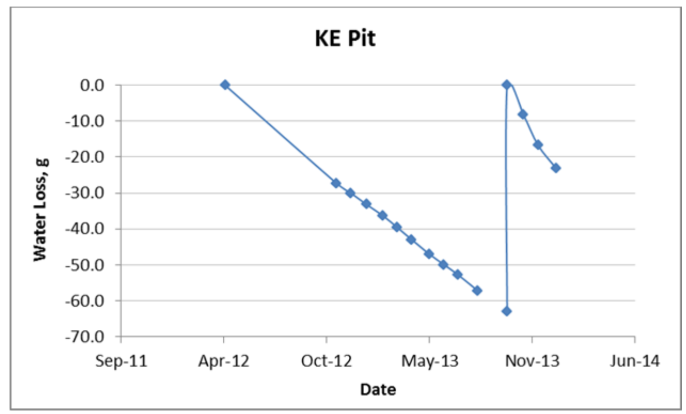

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Table 7. (Continued)

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<tr>
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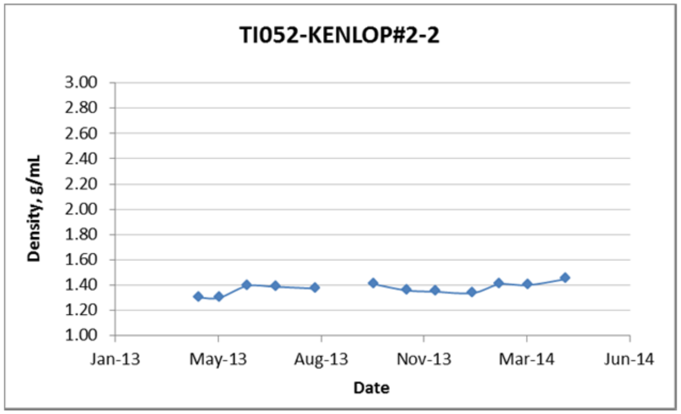
2013</td>
<td>
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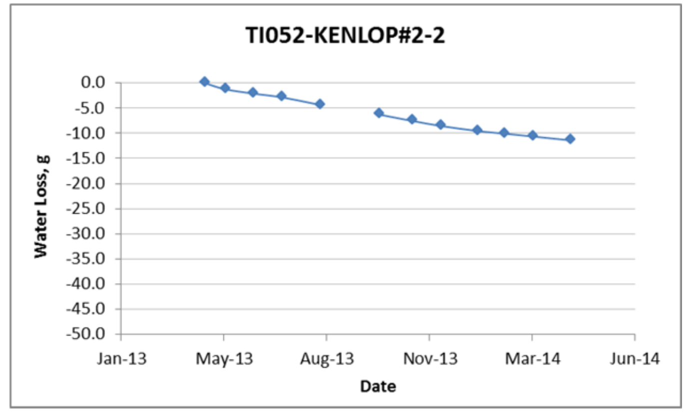
<td>
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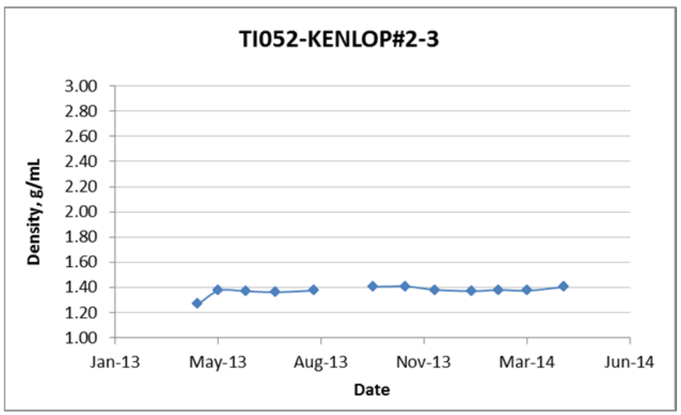
<tr>
<td>TI052-KENLOP#2-3


2013</td>
<td>
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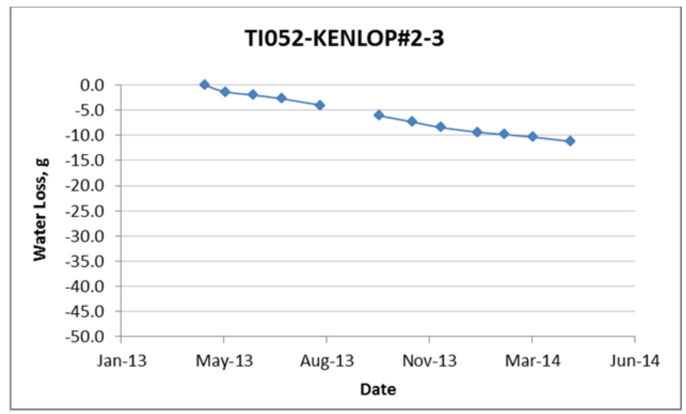
<td>
</td>
</tr>
</table>

Table 8. Pre-2004 Collected Samples Maintained Dry

Sample ID	Year	Most Recent Image	Sample ID	Year	Most Recent Image
FE-3 Comp-1 (dry)	2014		96-13 Settling Study	2013	
Basin Fuel Fines P500 (dry)	2014		K Basin Fuel Fines M500 (dry)	2014	

Table 8. (Continued)

Sample ID	Year	Most Recent Image	Sample ID	Year	Most Recent Image
SNF Comp Settling Study	2014		SFEC 96-04 (dry)	2014	
Test 3 Residue (dry)	2014				

4.0 Summary

The K Basin sludge sample archive in the RPL SAL is maintained in good condition for use in Phase 2 or other K Basin sludge process testing. Most sludge samples (81) are maintained in a wetted condition to minimize physical changes. Seven sludge samples were received and are maintained in a dry condition. Samples are monitored monthly to determine sample condition and container integrity. DI water is added as necessary to maintain a wetted condition.

The storage temperature is higher than the ambient K Basins temperature. Any potential changes in the sludge physical properties are expected to be similar to those in the main sludge material in the K Basins (such as chemical composition and particle size distribution).

Container replacement is recommended on four-year intervals to better provide for overall storage integrity. The glass sustains radiation damage over time. After four years in the hot-cell environment, the glass containers become weakened and brittle.

5.0 References

Baker RB, JL Westcott, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009a. *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*. KBC-33786, Rev. 2, CH2M Hill Plateau Remediation, Company, Richland, Washington.

Baker RB, JL Westcott, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009b. *Quality Assurance Project Plan / Sampling and Analysis Plan for Containerized KW Settler Sludge*. KBC-40467, Rev. 1, CH2M Hill Plateau Remediation, Company, Richland, Washington.

Delegard CH, AJ Schmidt, and JW Chenault. 2011. *Characteristics of KE Basin Sludge Samples Archived in the RPL – 2007*. PNNL-17078, Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

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Fiskum SK, JM Billing, SJ Bos, CA Burns, CD Carlson, DS Coffey, JV Crum, RC Daniel, CH Delegard, MK Edwards, OT Farmer, LR Greenwood, SA Jones, D Neiner, BM Oliver, KN Pool, AJ Schmidt, RW Shimskey, SI Sinkov, SZ Soderquist, CJ Thompson, ML Thomas, T Trang-Le, and MW Urie. 2011. *Characterization Data Package for Containerized Sludge Samples Collected from Engineered Containers SCS-CON-240, 250, 260, and 220*. PNNL-19035, Rev. 1 (limited distribution), Pacific Northwest National Laboratory, Richland, Washington.

Makenas BJ, TL Welsh, RB Baker, EW Hoppe, AJ Schmidt, J Abrefah, JM Tingey, PR Bredt, and GR Golcar. 1997. *Analysis of Sludge from Hanford K East Basin Canisters.* HNF-SP-1201, DE&S Hanford, Inc., Richland, Washington.

Schmidt AJ, RT Hallen, DS Muzatko, and SR Gano. 2004. *KE Basin Sludge Flocculant Testing*. PNNL-14730, Pacific Northwest National Laboratory, Richland, Washington.

Sinkov, SI, CH Delegard, AJ Schmidt, JW Chenault. 2012. *Characteristics of STP Pre-2004 Archived KE Basin Sludge Samples Before and After Re-Jarring in the RPL – April 2012*. PNNL-21836 (35451-RPT20), Battelle Pacific Northwest National Laboratory, Richland, Washington.

Westcott JL, BJ Makenas, TL Welsh, JA Pottmeyer, and AJ Schmidt. 2009. *Data Quality Objectives for Sampling and Analysis of K Basin Sludge (DQO)*, HNF-36985, Rev. 3, CH2M Hill Plateau Remediation Company, Richland, Washington.

Appendix A

Post-2009 Collected K Basin Sludge Sample Pedigree

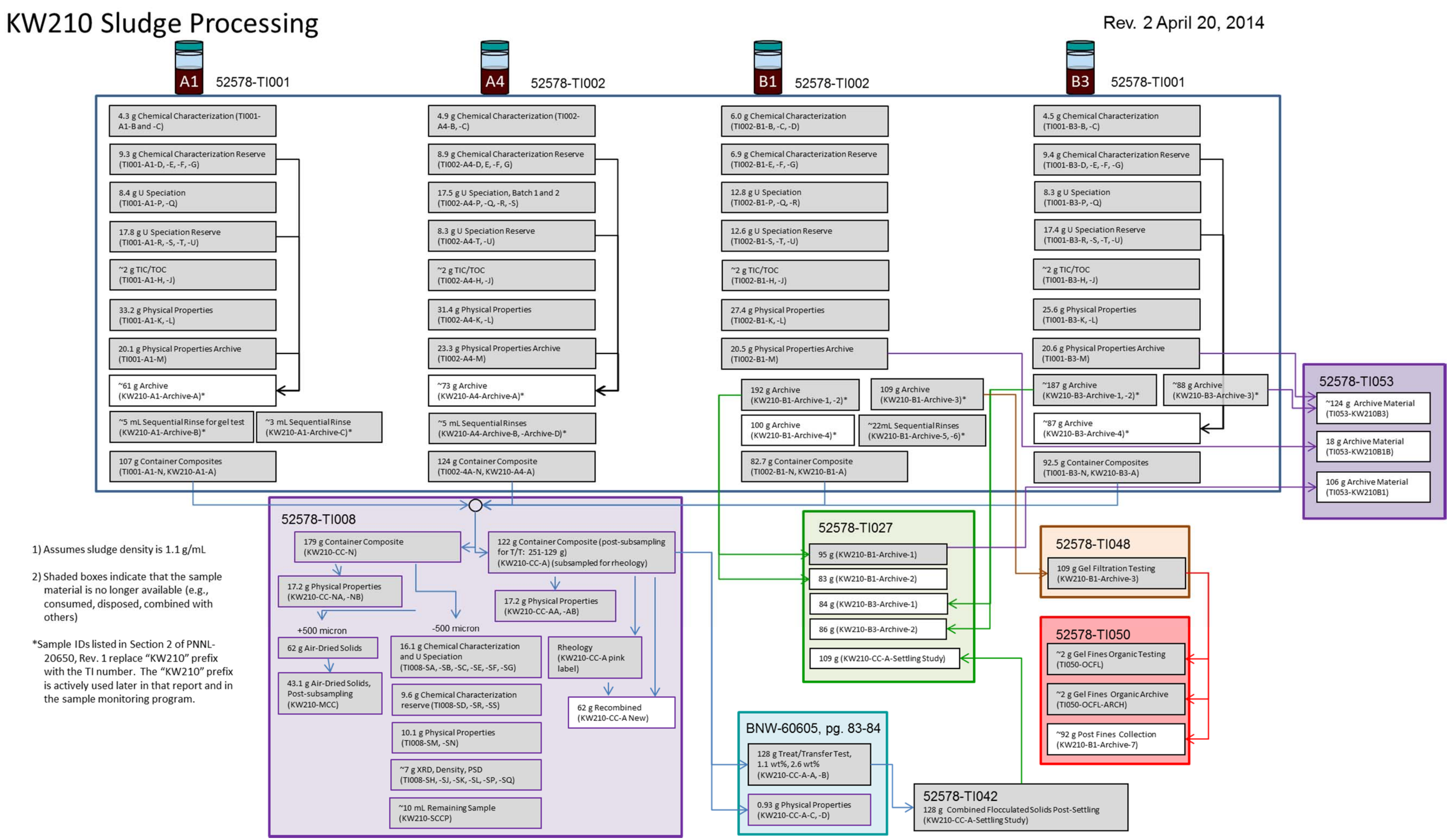
Appendix A

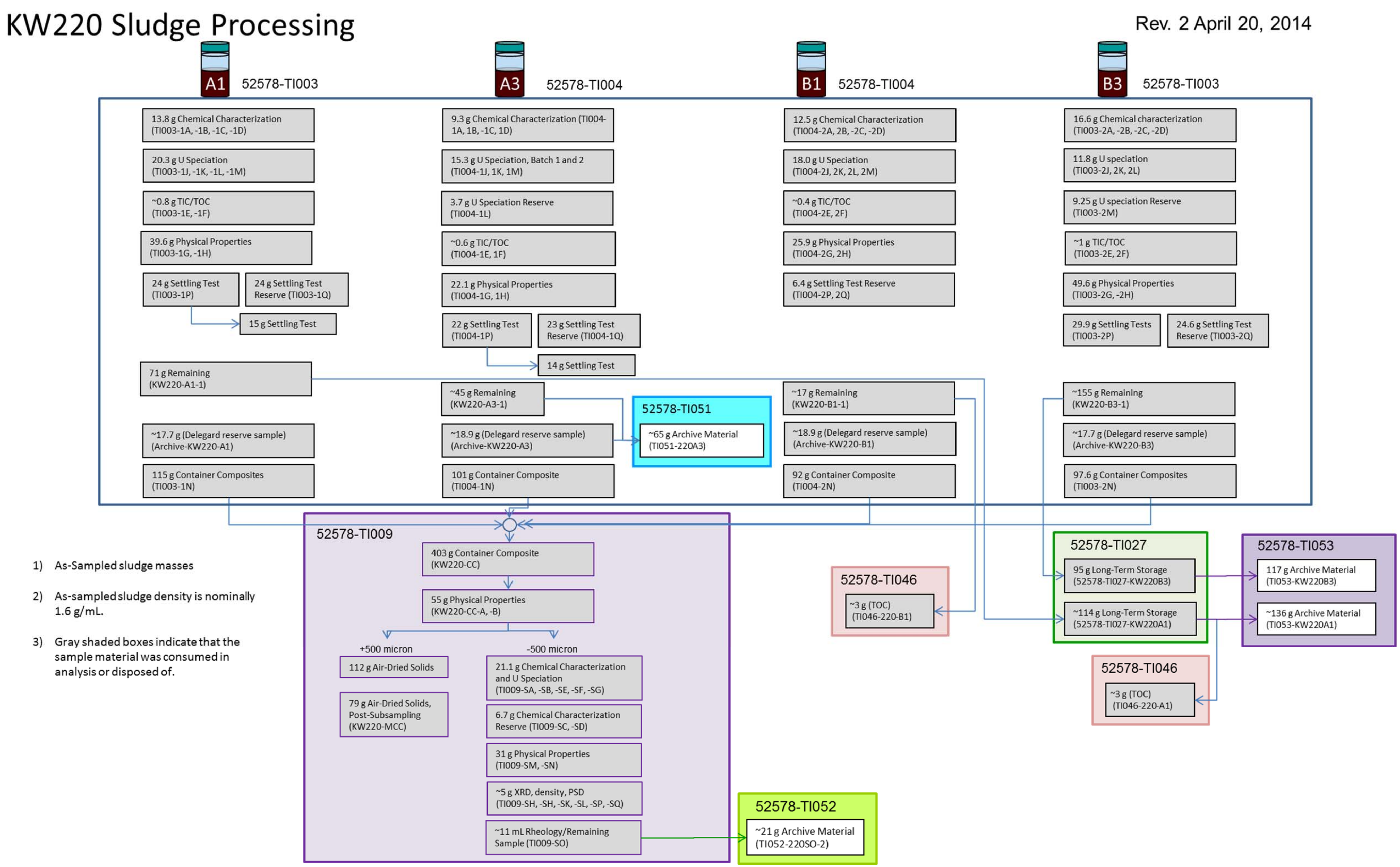
Post-2009 Collected K Basin Sludge Sample Pedigree

The pedigrees of many samples collected during post-2009 sample collection processing activities are illustrated in the following flowcharts. The charts present the engineered container ID (e.g., SCS-CON-210), the core or container composite sample ID (e.g., A1), the associated test instruction ID (e.g., 52578-TI001), and the sample mass (as-sampled condition)[1] with associated test scope and specific sample ID (shown in boxed text) (e.g., 4.3 g Chemical Characterization [TI001-A1-B, and -C]). Gray-shaded sample boxes indicate whether a sample was consumed or forwarded to another test scope, recombined, or discarded. Colored blocks help delineate the test instruction under which the sample was generated/handled. Unshaded sample boxes indicate the sample is still available for testing and include the wet settled sludge mass based on April 2014 measurements.

It is noted that in several cases the estimated mass is higher than that of the parent sample or previously reported mass. For example, sample TI053-KW220-A1 is shown at 136 g, which represents settled solids mass shortly after repackaging. This mass is higher than that of the parent sample 52578-TI027-KW220-A1 with last reported mass of 114 g; the 114 g represents sludge that had settled for approximately two years and is thus more compact and contains less interstitial water. Other samples show lower solids mass than previously reported. For example, KW210-B3-Archive-4 sludge was previously reported to contain 100 g sludge and in the current report is measured at 87 g. In this case, the sludge sample has had another 1.2 years settling time in which additional compaction has occurred and less water is incorporated with the settled sludge mass.

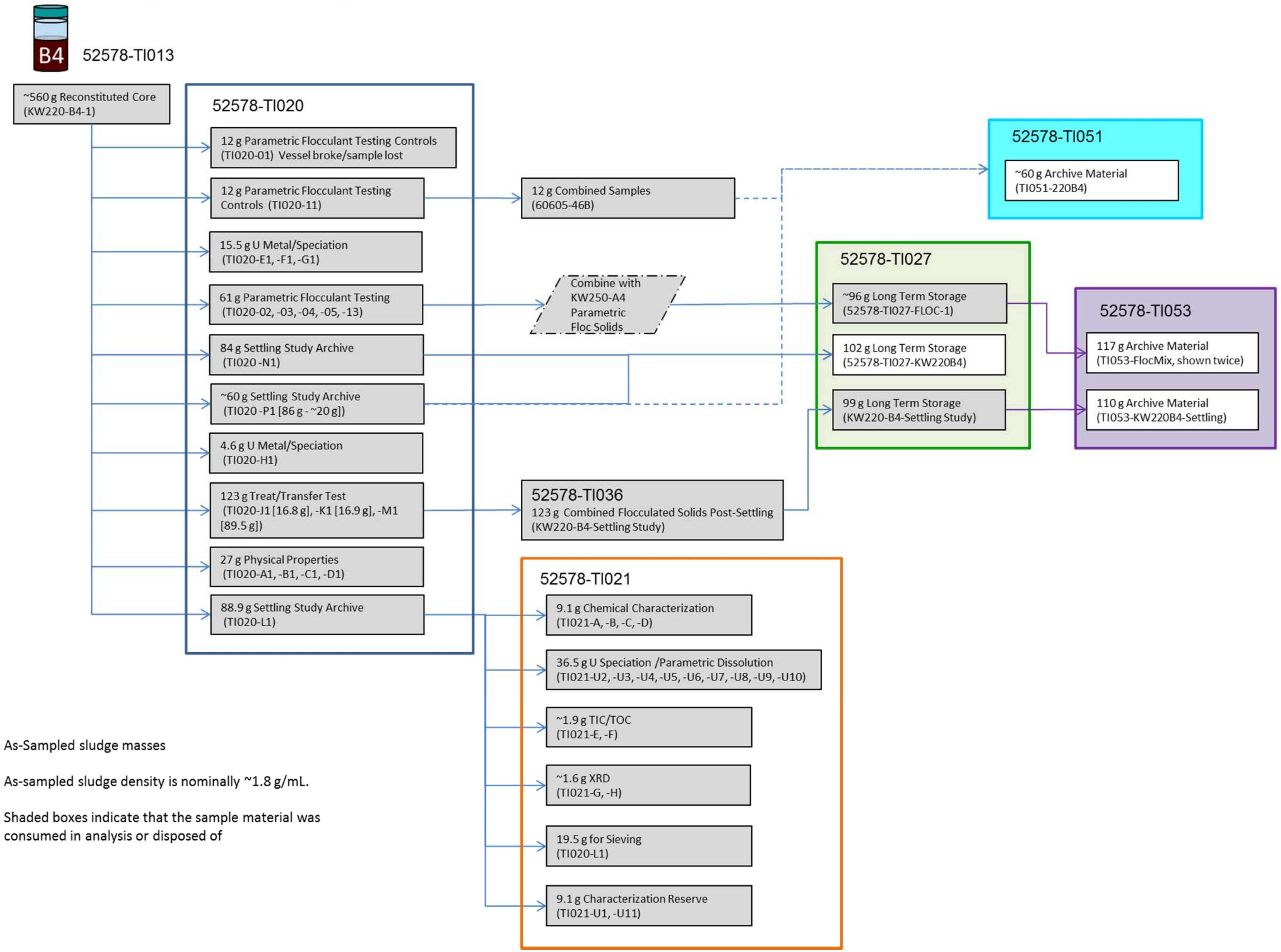
[1] The as-sampled sludge has been thickened after "batch settling" followed by mixing / homogenizing the as-settled sludge and removing any resulting displaced interstitial water. Based on past experience with sludge, in the dewatered state, sludge possesses sufficient shear strength to suspend / uniformly disperse the dense uranium metal particles. Consequently, it is in this dewatered state that subsamples were collected from the parent sample. The "dewatered" state is an arbitrary measure since the water entrainment condition in the solids will range somewhere between fully watered in a free-flowing condition (as-settled sludge) and mixed and centrifuged, where the maximum water is squeezed out of solids interstices. The time it takes to reach this state can be highly variable, as it is affected by the sludge composition and settling rates. In testing with simulants, the dewatered condition exhibited significantly more variability in density relative to "as-settled" sludge.


KW210 Sludge Processing
Rev. 2 April 20, 2014
A1 52578-TI001
A4 52578-TI002
B1 52578-TI002
B3 52578-TI001
4.3 g Chemical Characterization (TI001-A1-B and -C)
9.3 g Chemical Characterization Reserve (TI001-A1-D, -E, -F, -G)
8.4 g U Speciation (TI001-A1-P, -Q)
17.8 g U Speciation Reserve (TI001-A1-R, -S, -T, -U)
~2 g TIC/TOC (TI001-A1-H, -J)
33.2 g Physical Properties (TI001-A1-K, -L)
20.1 g Physical Properties Archive (TI001-A1-M)
~61 g Archive (KW210-A1-Archive-A)*
~5 mL Sequential Rinse for gel test (KW210-A1-Archive-B)*
~3 mL Sequential Rinse (KW210-A1-Archive-C)*
107 g Container Composites (TI001-A1-N, KW210-A1-A)
4.9 g Chemical Characterization (TI002-A4-B, -C)
8.9 g Chemical Characterization Reserve (TI002-A4-D, E, -F, G)
17.5 g U Speciation, Batch 1 and 2 (TI002-A4-P, -Q, -R, -S)
8.3 g U Speciation Reserve (TI002-A4-T, -U)
~2 g TIC/TOC (TI002-A4-H, -J)
31.4 g Physical Properties (TI002-A4-K, -L)
23.3 g Physical Properties Archive (TI002-A4-M)
~73 g Archive (KW210-A4-Archive-A)*
~5 mL Sequential Rinses (KW210-A4-Archive-B, -Archive-D)*
124 g Container Composite (TI002-4A-N, KW210-A4-A)
6.0 g Chemical Characterization (TI002-B1-B, -C, -D)
6.9 g Chemical Characterization Reserve (TI002-B1-E, -F, -G)
12.8 g U Speciation (TI002-B1-P, -Q, -R)
12.6 g U Speciation Reserve (TI002-B1-S, -T, -U)
~2 g TIC/TOC (TI002-B1-H, -J)
27.4 g Physical Properties (TI002-B1-K, -L)
20.5 g Physical Properties Archive (TI002-B1-M)
192 g Archive (KW210-B1-Archive-1, -2)*
109 g Archive (KW210-B1-Archive-3)*
100 g Archive (KW210-B1-Archive-4)*
~22mL Sequential Rinses (KW210-B1-Archive-5, -6)*
82.7 g Container Composite (TI002-B1-N, KW210-B1-A)
4.5 g Chemical Characterization (TI001-B3-B, -C)
9.4 g Chemical Characterization Reserve (TI001-B3-D, -E, -F, -G)
8.3 g U Speciation (TI001-B3-P, -Q)
17.4 g U Speciation Reserve (TI001-B3-R, -S, -T, -U)
~2 g TIC/TOC (TI001-B3-H, -J)
25.6 g Physical Properties (TI001-B3-K, -L)
20.6 g Physical Properties Archive (TI001-B3-M)
~187 g Archive (KW210-B3-Archive-1, -2)*
~88 g Archive (KW210-B3-Archive-3)*
~87 g Archive (KW210-B3-Archive-4)*
92.5 g Container Composites (TI001-B3-N, KW210-B3-A)
52578-TI053
~124 g Archive Material (TI053-KW210B3)
18 g Archive Material (TI053-KW210B1B)
106 g Archive Material (TI053-KW210B1)
52578-TI008
179 g Container Composite (KW210-CC-N)
122 g Container Composite (post-subsampling for T/T: 251-129 g) (KW210-CC-A) (subsampled for rheology)
17.2 g Physical Properties (KW210-CC-NA, -NB)
17.2 g Physical Properties (KW210-CC-AA, -AB)
+500 micron
-500 micron
62 g Air-Dried Solids
43.1 g Air-Dried Solids, Post-subsampling (KW210-MCC)
16.1 g Chemical Characterization and U Speciation (TI008-SA, -SB, -SC, -SE, -SF, -SG)
Rheology (KW210-CC-A pink label)
9.6 g Chemical Characterization reserve (TI008-SD, -SR, -SS)
62 g Recombined (KW210-CC-A New)
10.1 g Physical Properties (TI008-SM, -SN)
~7 g XRD, Density, PSD (TI008-SH, -SJ, -SK, -SL, -SP, -SQ)
~10 mL Remaining Sample (KW210-SCCP)
52578-TI027
95 g (KW210-B1-Archive-1)
83 g (KW210-B1-Archive-2)
84 g (KW210-B3-Archive-1)
86 g (KW210-B3-Archive-2)
109 g (KW210-CC-A-Settling Study)
52578-TI048
109 g Gel Filtration Testing (KW210-B1-Archive-3)
52578-TI050
~2 g Gel Fines Organic Testing (TI050-OCFL)
~2 g Gel Fines Organic Archive (TI050-OCFL-ARCH)
~92 g Post Fines Collection (KW210-B1-Archive-7)
BNW-60605, pg. 83-84
128 g Treat/Transfer Test, 1.1 wt%, 2.6 wt% (KW210-CC-A-A, -B)
0.93 g Physical Properties (KW210-CC-A-C, -D)
52578-TI042
128 g Combined Flocculated Solids Post-Settling (KW210-CC-A-Settling Study)
1) Assumes sludge density is 1.1 g/mL
2) Shaded boxes indicate that the sample material is no longer available (e.g., consumed, disposed, combined with others)
*Sample IDs listed in Section 2 of PNNL-20650, Rev. 1 replace "KW210" prefix with the TI number. The "KW210" prefix is actively used later in that report and in the sample monitoring program.

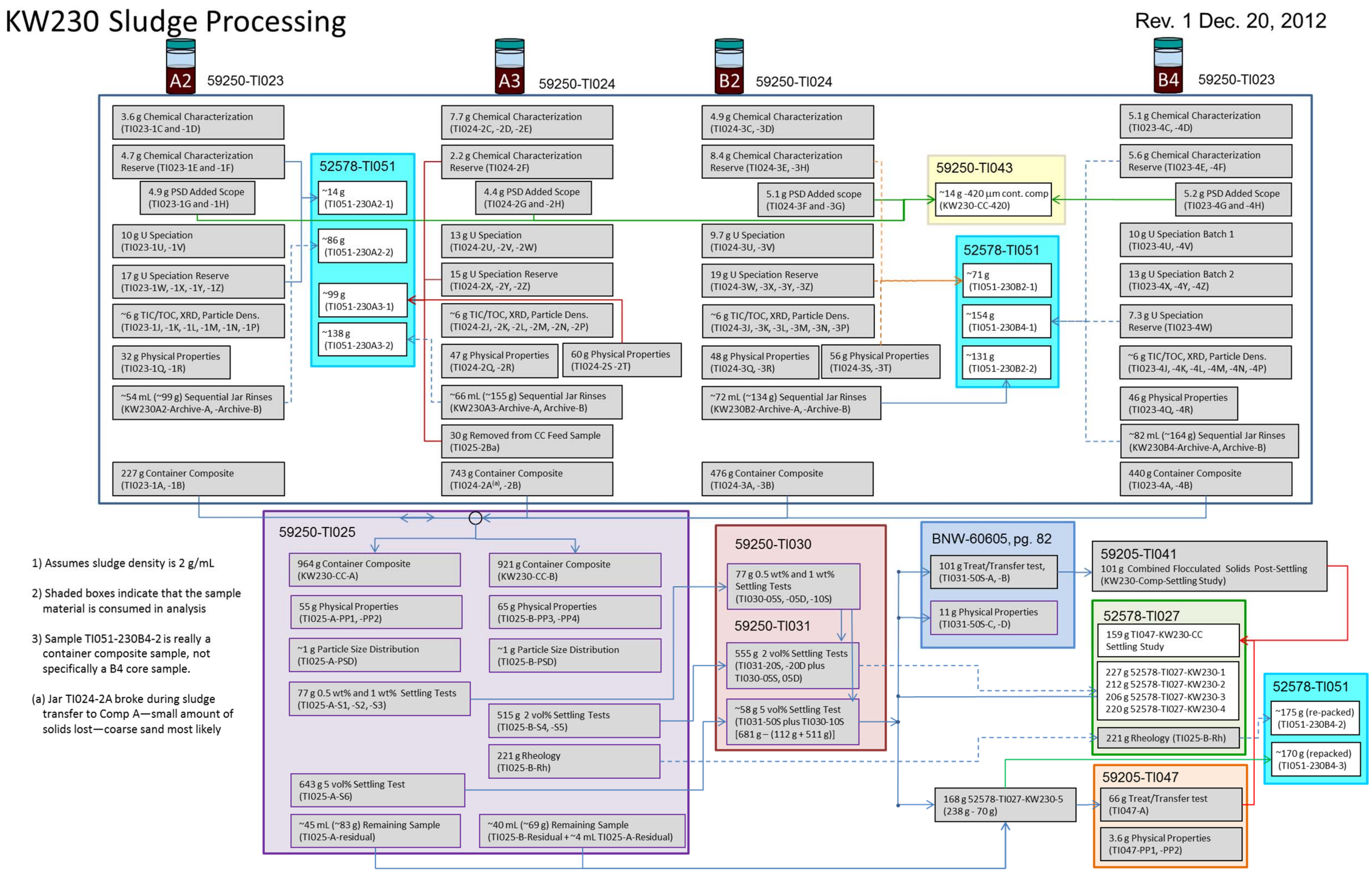

KW220 Sludge Processing
Rev. 2 April 20, 2014
A1
52578-TI003
A3
52578-TI004
B1
52578-TI004
B3
52578-TI003
13.8 g Chemical Characterization (TI003-1A, -1B, -1C, -1D)
20.3 g U Speciation (TI003-1J, -1K, -1L, -1M)
~0.8 g TIC/TOC (TI003-1E, -1F)
39.6 g Physical Properties (TI003-1G, -1H)
24 g Settling Test (TI003-1P)
24 g Settling Test Reserve (TI003-1Q)
15 g Settling Test
71 g Remaining (KW220-A1-1)
~17.7 g (Delegard reserve sample) (Archive-KW220-A1)
115 g Container Composites (TI003-1N)
9.3 g Chemical Characterization (TI004-1A, 1B, -1C, 1D)
15.3 g U Speciation, Batch 1 and 2 (TI004-1J, 1K, 1M)
3.7 g U Speciation Reserve (TI004-1L)
~0.6 g TIC/TOC (TI004-1E, 1F)
22.1 g Physical Properties (TI004-1G, 1H)
22 g Settling Test (TI004-1P)
23 g Settling Test Reserve (TI004-1Q)
14 g Settling Test
~45 g Remaining (KW220-A3-1)
~18.9 g (Delegard reserve sample) (Archive-KW220-A3)
101 g Container Composite (TI004-1N)
52578-TI051
~65 g Archive Material (TI051-220A3)
12.5 g Chemical Characterization (TI004-2A, 2B, -2C, -2D)
18.0 g U Speciation (TI004-2J, 2K, 2L, 2M)
~0.4 g TIC/TOC (TI004-2E, 2F)
25.9 g Physical Properties (TI004-2G, 2H)
6.4 g Settling Test Reserve (TI004-2P, 2Q)
~17 g Remaining (KW220-B1-1)
~18.9 g (Delegard reserve sample) (Archive-KW220-B1)
92 g Container Composite (TI004-2N)
16.6 g Chemical characterization (TI003-2A, -2B, -2C, -2D)
11.8 g U speciation (TI003-2J, 2K, 2L)
9.25 g U speciation Reserve (TI003-2M)
~1 g TIC/TOC (TI003-2E, 2F)
49.6 g Physical Properties (TI003-2G, -2H)
29.9 g Settling Tests (TI003-2P)
24.6 g Settling Test Reserve (TI003-2Q)
~155 g Remaining (KW220-B3-1)
~17.7 g (Delegard reserve sample) (Archive-KW220-B3)
97.6 g Container Composites (TI003-2N)
52578-TI009
403 g Container Composite (KW220-CC)
55 g Physical Properties (KW220-CC-A, -B)
+500 micron
-500 micron
112 g Air-Dried Solids
79 g Air-Dried Solids, Post-Subsampling (KW220-MCC)
21.1 g Chemical Characterization and U Speciation (TI009-SA, -SB, -SE, -SF, -SG)
6.7 g Chemical Characterization Reserve (TI009-SC, -SD)
31 g Physical Properties (TI009-SM, -SN)
~5 g XRD, density, PSD (TI009-SH, -SH, -SK, -SL, -SP, -SQ)
~11 mL Rheology/Remaining Sample (TI009-SO)
52578-TI052
~21 g Archive Material (TI052-220SO-2)
52578-TI046
~3 g (TOC) (TI046-220-B1)
52578-TI027
95 g Long-Term Storage (52578-TI027-KW220B3)
~114 g Long-Term Storage (52578-TI027-KW220A1)
52578-TI053
117 g Archive Material (TI053-KW220B3)
~136 g Archive Material (TI053-KW220A1)
52578-TI046
~3 g (TOC) (TI046-220-A1)
1) As-Sampled sludge masses
2) As-sampled sludge density is nominally 1.6 g/mL.
3) Gray shaded boxes indicate that the sample material was consumed in analysis or disposed of.

KW220-B4 Sludge Processing

Rev. 2 April 20, 2014

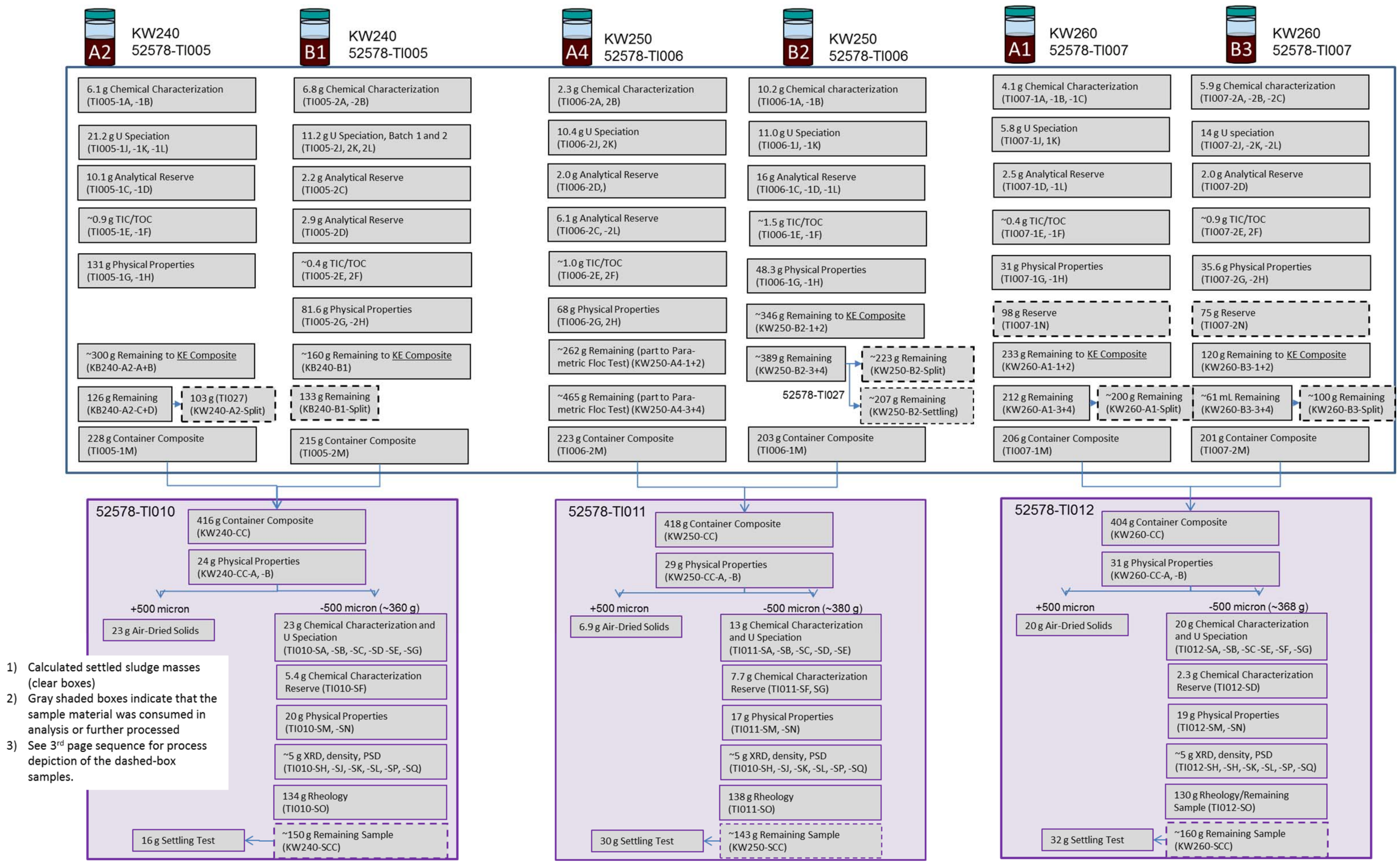


1) As-Sampled sludge masses
2) As-sampled sludge density is nominally ~1.8 g/mL.
3) Shaded boxes indicate that the sample material was consumed in analysis or disposed of


KW230 Sludge Processing
Rev. 1 Dec. 20, 2012
A2 59250-TI023
A3 59250-TI024
B2 59250-TI024
B4 59250-TI023
3.6 g Chemical Characterization (TI023-1C and -1D)
4.7 g Chemical Characterization Reserve (TI023-1E and -1F)
4.9 g PSD Added Scope (TI023-1G and -1H)
10 g U Speciation (TI023-1U, -1V)
17 g U Speciation Reserve (TI023-1W, -1X, -1Y, -1Z)
~6 g TIC/TOC, XRD, Particle Dens. (TI023-1J, -1K, -1L, -1M, -1N, -1P)
32 g Physical Properties (TI023-1Q, -1R)
~54 mL (~99 g) Sequential Jar Rinses (KW230A2-Archive-A, -Archive-B)
227 g Container Composite (TI023-1A, -1B)
52578-TI051
~14 g (TI051-230A2-1)
~86 g (TI051-230A2-2)
~99 g (TI051-230A3-1)
~138 g (TI051-230A3-2)
7.7 g Chemical Characterization (TI024-2C, -2D, -2E)
2.2 g Chemical Characterization Reserve (TI024-2F)
4.4 g PSD Added Scope (TI024-2G and -2H)
13 g U Speciation (TI024-2U, -2V, -2W)
15 g U Speciation Reserve (TI024-2X, -2Y, -2Z)
~6 g TIC/TOC, XRD, Particle Dens. (TI024-2J, -2K, -2L, -2M, -2N, -2P)
47 g Physical Properties (TI024-2Q, -2R)
60 g Physical Properties (TI024-2S -2T)
~66 mL (~155 g) Sequential Jar Rinses (KW230A3-Archive-A, Archive-B)
30 g Removed from CC Feed Sample (TI025-2Ba)
743 g Container Composite (TI024-2A(a), -2B)
4.9 g Chemical Characterization (TI024-3C, -3D)
8.4 g Chemical Characterization Reserve (TI024-3E, -3H)
5.1 g PSD Added scope (TI024-3F and -3G)
9.7 g U Speciation (TI024-3U, -3V)
19 g U Speciation Reserve (TI024-3W, -3X, -3Y, -3Z)
~6 g TIC/TOC, XRD, Particle Dens. (TI024-3J, -3K, -3L, -3M, -3N, -3P)
48 g Physical Properties (TI024-3Q, -3R)
56 g Physical Properties (TI024-3S, -3T)
~72 mL (~134 g) Sequential Jar Rinses (KW230B2-Archive-A, -Archive-B)
476 g Container Composite (TI024-3A, -3B)
59250-TI043
~14 g -420 µm cont. comp (KW230-CC-420)
52578-TI051
~71 g (TI051-230B2-1)
~154 g (TI051-230B4-1)
~131 g (TI051-230B2-2)
5.1 g Chemical Characterization (TI023-4C, -4D)
5.6 g Chemical Characterization Reserve (TI023-4E, -4F)
5.2 g PSD Added Scope (TI023-4G and -4H)
10 g U Speciation Batch 1 (TI023-4U, -4V)
13 g U Speciation Batch 2 (TI023-4X, -4Y, -4Z)
7.3 g U Speciation Reserve (TI023-4W)
~6 g TIC/TOC, XRD, Particle Dens. (TI023-4J, -4K, -4L, -4M, -4N, -4P)
46 g Physical Properties (TI023-4Q, -4R)
~82 mL (~164 g) Sequential Jar Rinses (KW230B4-Archive-A, Archive-B)
440 g Container Composite (TI023-4A, -4B)
1) Assumes sludge density is 2 g/mL
2) Shaded boxes indicate that the sample material is consumed in analysis
3) Sample TI051-230B4-2 is really a container composite sample, not specifically a B4 core sample.
(a) Jar TI024-2A broke during sludge transfer to Comp A—small amount of solids lost—coarse sand most likely
59250-TI025
964 g Container Composite (KW230-CC-A)
921 g Container Composite (KW230-CC-B)
55 g Physical Properties (TI025-A-PP1, -PP2)
65 g Physical Properties (TI025-B-PP3, -PP4)
~1 g Particle Size Distribution (TI025-A-PSD)
~1 g Particle Size Distribution (TI025-B-PSD)
77 g 0.5 wt% and 1 wt% Settling Tests (TI025-A-S1, -S2, -S3)
515 g 2 vol% Settling Tests (TI025-B-S4, -S5)
221 g Rheology (TI025-B-Rh)
643 g 5 vol% Settling Test (TI025-A-S6)
~45 mL (~83 g) Remaining Sample (TI025-A-residual)
~40 mL (~69 g) Remaining Sample (TI025-B-Residual + ~4 mL TI025-A-Residual)
59250-TI030
77 g 0.5 wt% and 1 wt% Settling Tests (TI030-05S, -05D, -10S)
59250-TI031
555 g 2 vol% Settling Tests (TI031-20S, -20D plus TI030-05S, 05D)
~58 g 5 vol% Settling Test (TI031-50S plus TI030-10S [681 g – (112 g + 511 g)]
BNW-60605, pg. 82
101 g Treat/Transfer test, (TI031-50S-A, -B)
11 g Physical Properties (TI031-50S-C, -D)
59205-TI041
101 g Combined Flocculated Solids Post-Settling (KW230-Comp-Settling Study)
52578-TI027
159 g TI047-KW230-CC Settling Study
227 g 52578-TI027-KW230-1
212 g 52578-TI027-KW230-2
206 g 52578-TI027-KW230-3
220 g 52578-TI027-KW230-4
221 g Rheology (TI025-B-Rh)
52578-TI051
~175 g (re-packed) (TI051-230B4-2)
~170 g (repacked) (TI051-230B4-3)
59205-TI047
168 g 52578-TI027-KW230-5 (238 g - 70 g)
66 g Treat/Transfer test (TI047-A)
3.6 g Physical Properties (TI047-PP1, -PP2)

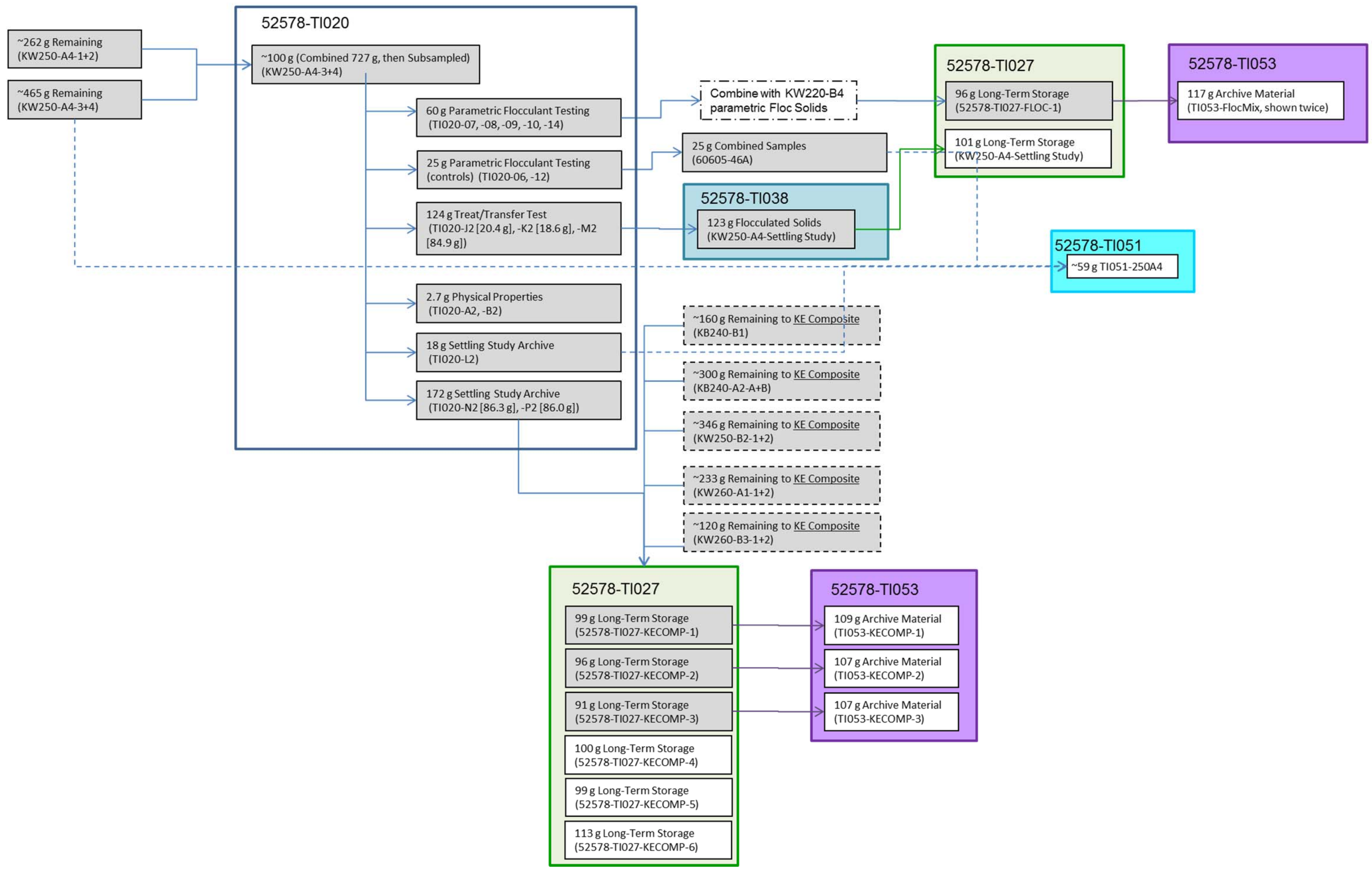
KW240, KW250, KW260 Sludge Processing

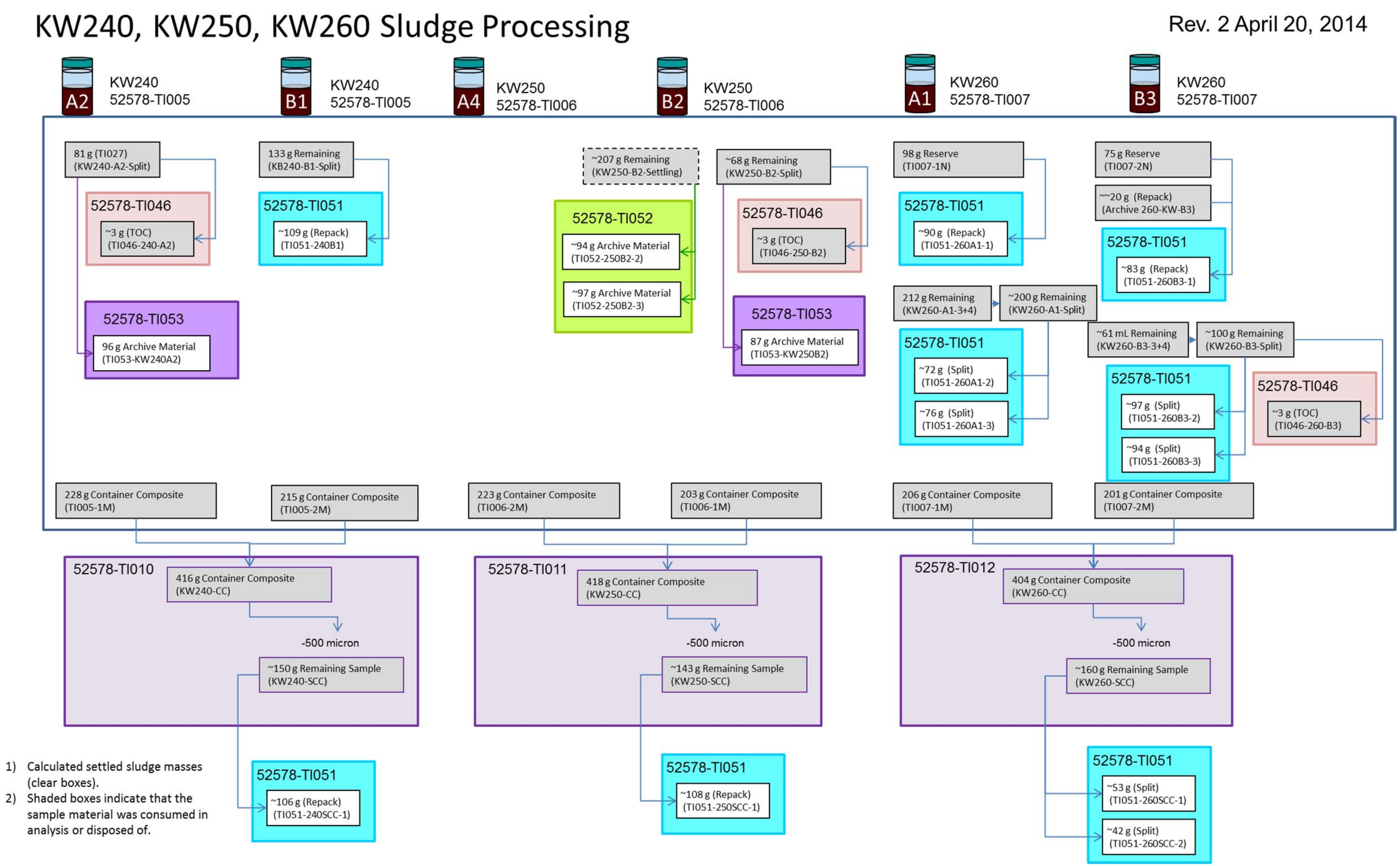
Rev. 2 April 20, 2014



KW240, KW250, KW260 Sludge Processing

Rev. 2 April 20, 2014




KW240, KW250, KW260 Sludge Processing
Rev. 2 April 20, 2014
A2
KW240
52578-TI005
B1
KW240
52578-TI005
A4
KW250
52578-TI006
B2
KW250
52578-TI006
A1
KW260
52578-TI007
B3
KW260
52578-TI007
81 g (TI027)
(KW240-A2-Split)
52578-TI046
~3 g (TOC)
(TI046-240-A2)
52578-TI053
96 g Archive Material
(TI053-KW240A2)
133 g Remaining
(KB240-B1-Split)
52578-TI051
~109 g (Repack)
(TI051-240B1)
~207 g Remaining
(KW250-B2-Settling)
52578-TI052
~94 g Archive Material
(TI052-250B2-2)
~97 g Archive Material
(TI052-250B2-3)
~68 g Remaining
(KW250-B2-Split)
52578-TI046
~3 g (TOC)
(TI046-250-B2)
52578-TI053
87 g Archive Material
(TI053-KW250B2)
98 g Reserve
(TI007-1N)
52578-TI051
~90 g (Repack)
(TI051-260A1-1)
212 g Remaining
(KW260-A1-3+4)
~200 g Remaining
(KW260-A1-Split)
52578-TI051
~72 g (Split)
(TI051-260A1-2)
~76 g (Split)
(TI051-260A1-3)
75 g Reserve
(TI007-2N)
~~20 g (Repack)
(Archive 260-KW-B3)
52578-TI051
~83 g (Repack)
(TI051-260B3-1)
~61 mL Remaining
(KW260-B3-3+4)
~100 g Remaining
(KW260-B3-Split)
52578-TI051
~97 g (Split)
(TI051-260B3-2)
~94 g (Split)
(TI051-260B3-3)
52578-TI046
~3 g (TOC)
(TI046-260-B3)
228 g Container Composite
(TI005-1M)
215 g Container Composite
(TI005-2M)
223 g Container Composite
(TI006-2M)
203 g Container Composite
(TI006-1M)
206 g Container Composite
(TI007-1M)
201 g Container Composite
(TI007-2M)
52578-TI010
416 g Container Composite
(KW240-CC)
-500 micron
~150 g Remaining Sample
(KW240-SCC)
52578-TI011
418 g Container Composite
(KW250-CC)
-500 micron
~143 g Remaining Sample
(KW250-SCC)
52578-TI012
404 g Container Composite
(KW260-CC)
-500 micron
~160 g Remaining Sample
(KW260-SCC)
52578-TI051
~106 g (Repack)
(TI051-240SCC-1)
52578-TI051
~108 g (Repack)
(TI051-250SCC-1)
52578-TI051
~53 g (Split)
(TI051-260SCC-1)
~42 g (Split)
(TI051-260SCC-2)
1) Calculated settled sludge masses (clear boxes).
2) Shaded boxes indicate that the sample material was consumed in analysis or disposed of.

Appendix B

Water Loss from K Basin Sludge

Appendix B

Water Loss from K Basin Sludge

Sludge sample water loss, as a function of time (date), was calculated from changes in the gross container mass. The following charts (Figure B.1 through Figure B.4) depict water mass loss as a function of date for selected representative samples stored for an extended period. The samples are grouped according to their genesis (e.g., large engineered container at the KW Basin). Periodically, water was added to the containers and the relative zero-point was reset. Thus, the water loss profiles generally appear as a sawtooth pattern. For the samples shown in Figure B.1 through Figure B.4, a visible layer of water was maintained above the samples in all cases, except KW210-B1-Archive-4, which remained wetted, but was not saturated for a brief period of time (See Section 3.0).

Water evaporation rates varied significantly between samples and among individual sample measurements. There was no consistent water loss rate within any waste group. The observed variations are likely attributable to differences in container lid seal integrity.

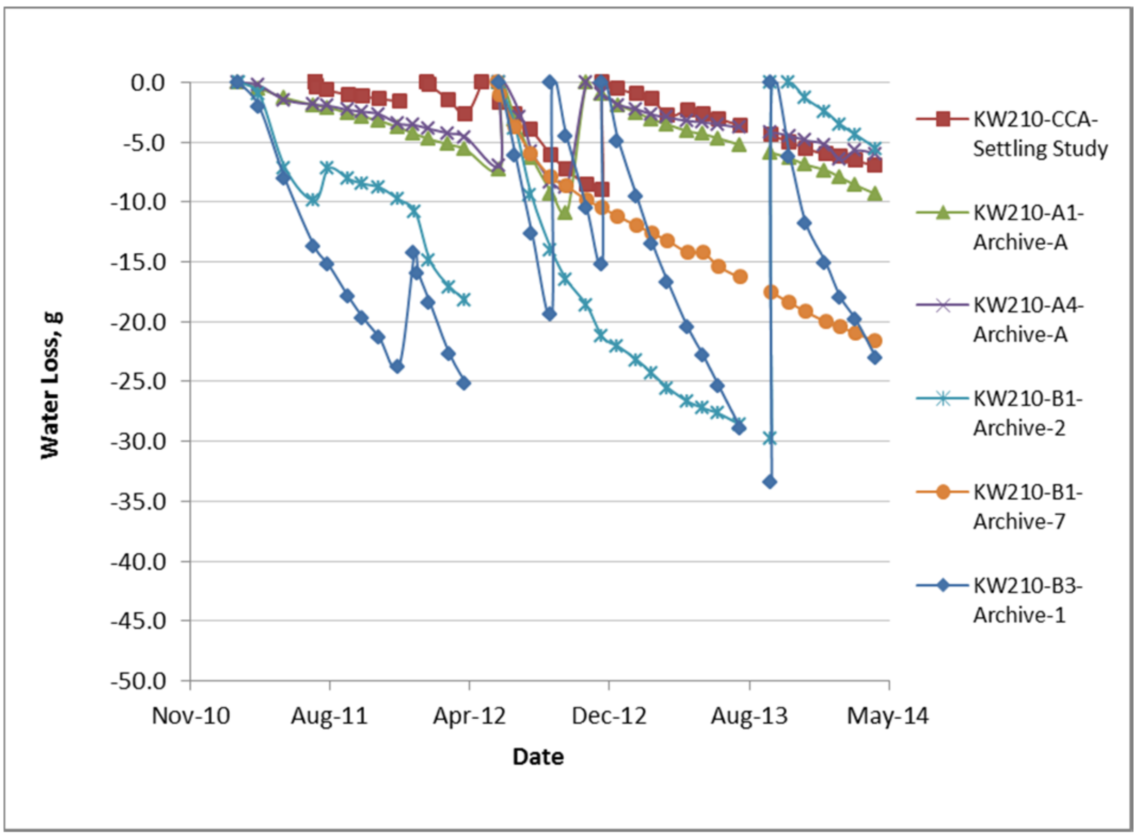


Figure B.1. Selected SCS-CON-210 Samples

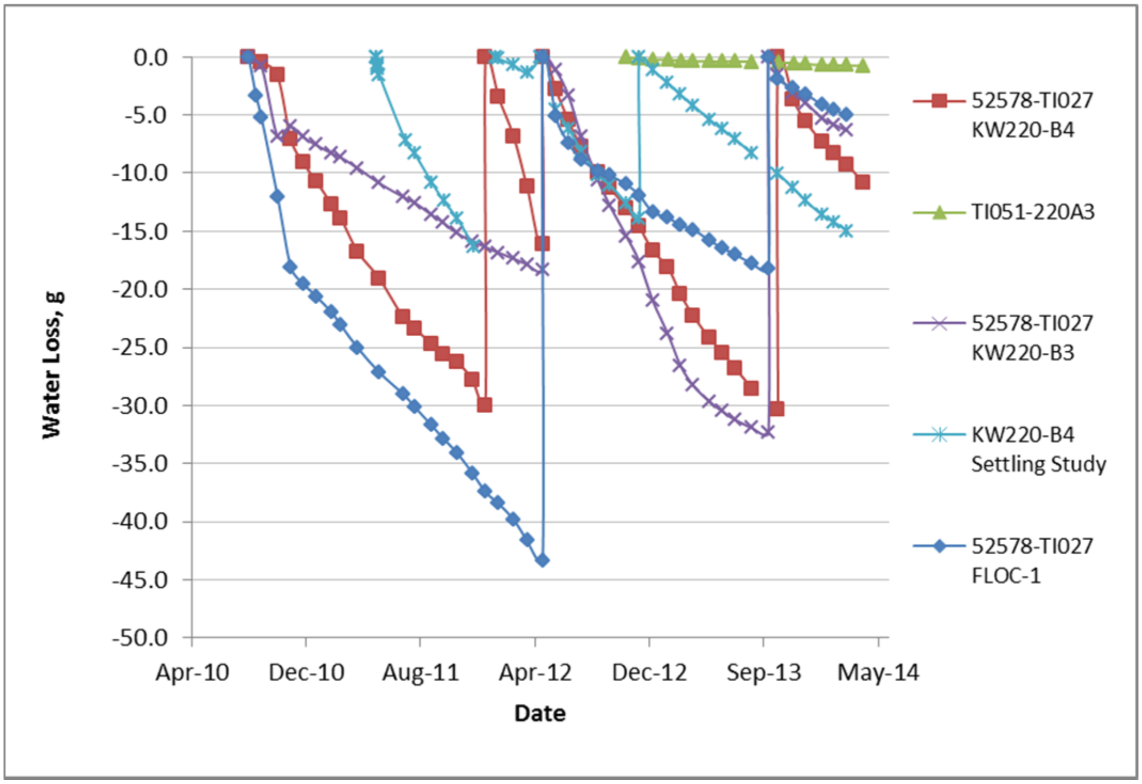


Figure B.2. Selected SCS-CON-220 Samples

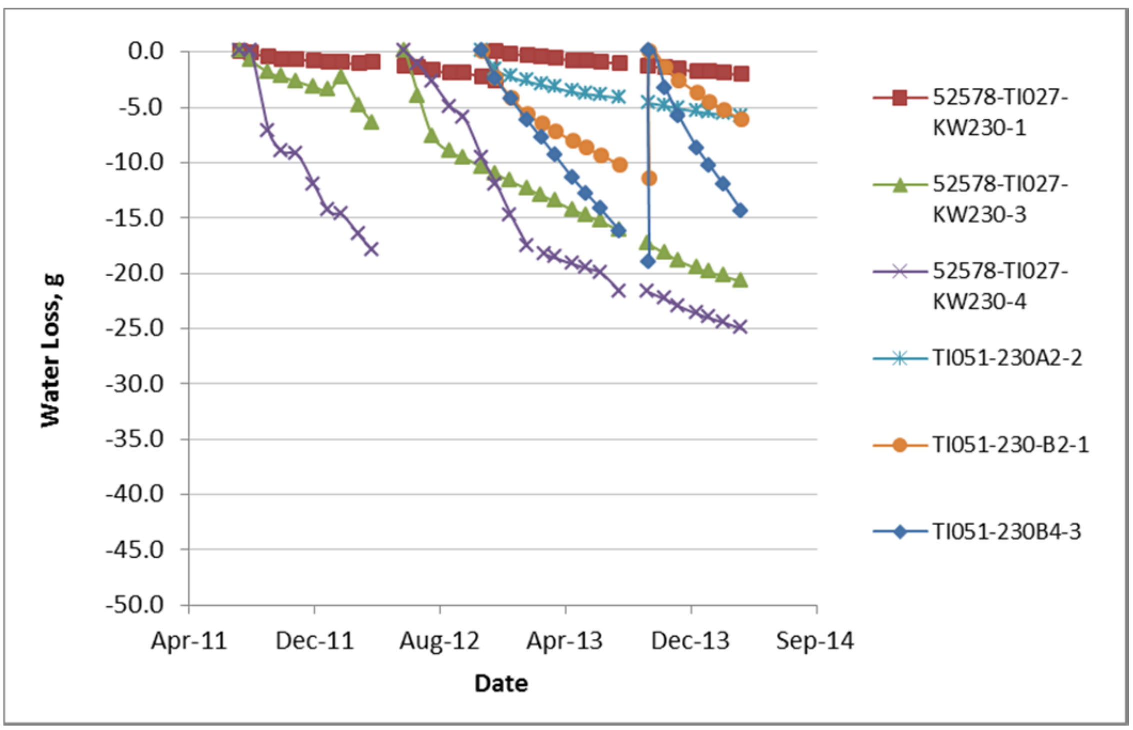


Figure B.3. Selected SCS-CON-230 Samples

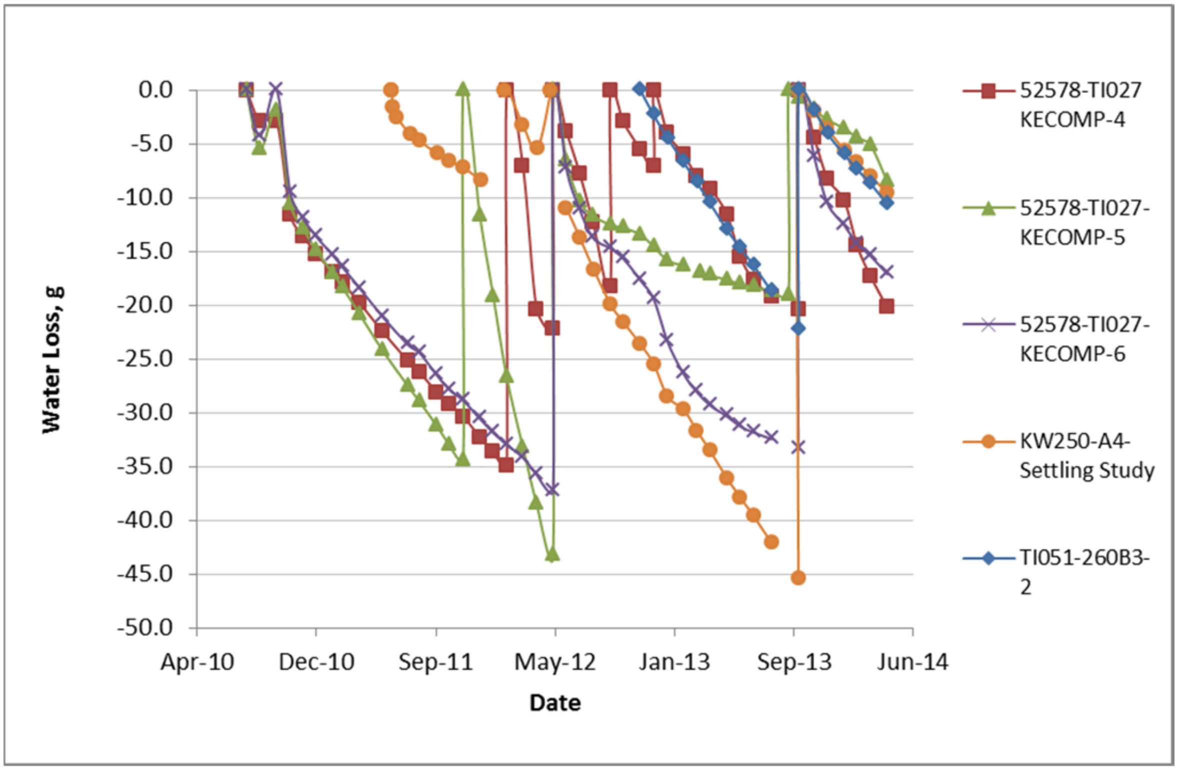


Figure B.4. Selected SCS-CON-240, -250, -260 Samples

The ambient cell temperature was also monitored as a function of time using a Type K thermocouple connected to a Dickson FT530 Dual Channel Temperature Display unit (barcode 28535, serial number: 10217201). The ambient cell temperature was downloaded from the unit into an Excel® format. Graphs of the cell temperature as a function of date for 2011 and 2012 are shown in Figure B.5 and Figure B.6. The temperature profile for 2013 was confounded with technical issues and is not provided.

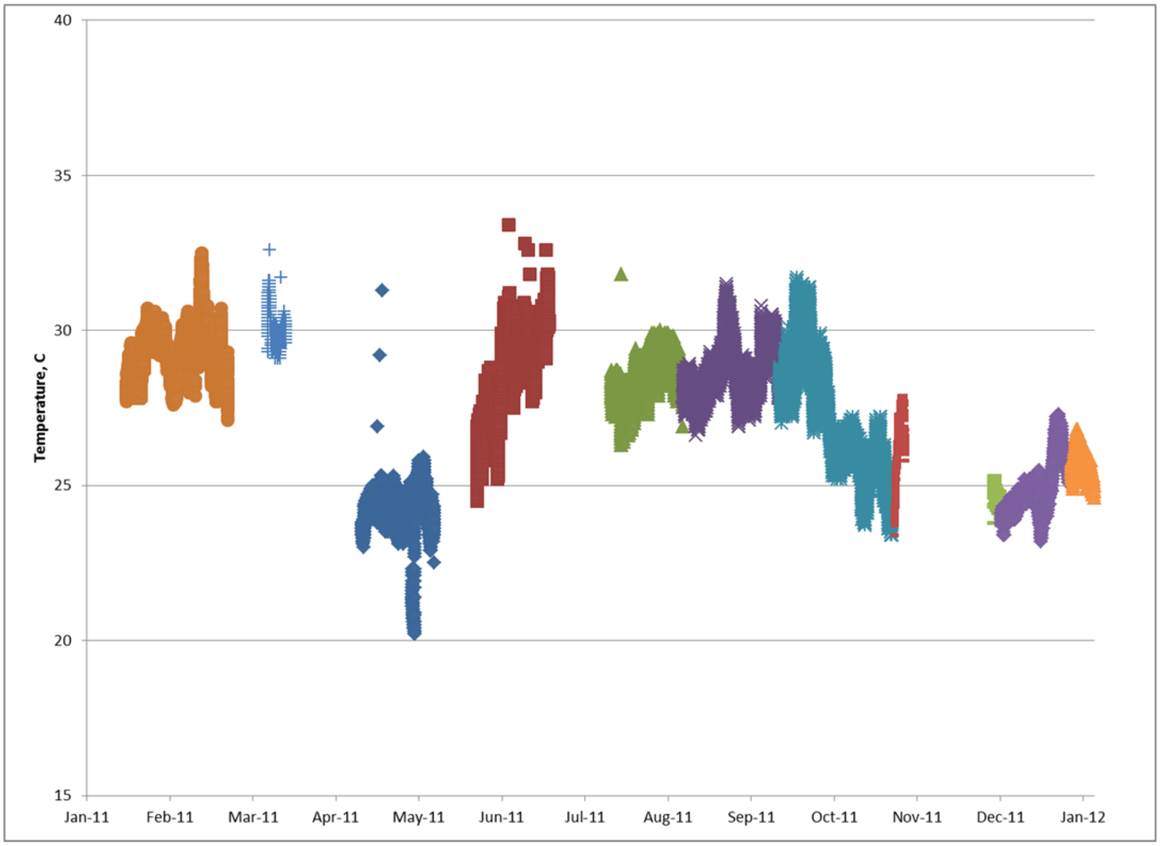


Figure B.5. Ambient Temperature in SAL, 2011. (Coloration indicates different data download dates.)

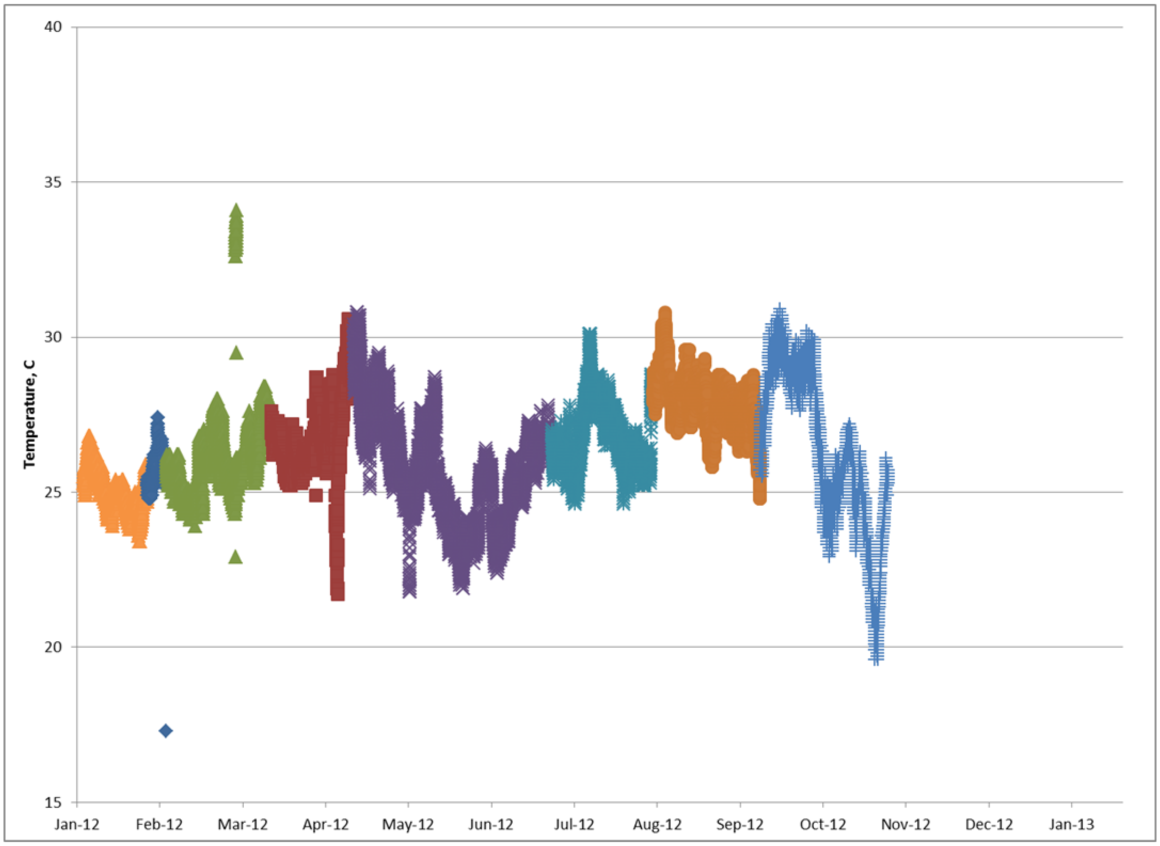


Figure B.6. Ambient Temperature in SAL, 2012. (Coloration indicates different data download dates.)

Appendix C

Summary of Pre-2004 Samples

Appendix C

Summary of Pre-2004 Samples

Summaries of the samples collected prior to 2004 are provided in Table C.1 and Table C.2. These tables were originally published in Sinkov et al. 2012 (Table S.1 and S.2) and are provided here to show sample genesis and measured sludge volumes and densities prior to the current monthly sample-monitoring effort.

Table C.1. Sources and Properties of Pre-2004 Re-Jarred KE Basin Sludge Samples (adapted from PNNL-21836,* Table S.1)

Sample ID	Source (based on Table 9.1 comments from the Delegard et al. 2011 report)	Re-Jarring Date	Settled Volume, ml	U Dry Wt%	Sludge Density, g/cm³ Present Study April 2012	Sludge Density, g/cm³ Previously Determined[(a)]
KC 2/3 Comp	Mixture of KC-2 and KC-3 consolidated sample with high to moderate damaged fuel from all three bays	28 Mar 2012	185 182	59.0	2.02 (X jar) 2.35 (Y jar)	2.03 (cone) 2.14 (jar)
96-05	Single closed-bottom canister with very poor condition fuel	27 Mar 2012	92	58.5	2.19	1.77 (cone) 2.34 (1997)[(b)]
96-13 Comp A	Composite of samples 96-01, -05, -06, -08,-13, and -15 from single canisters with good to very poor condition fuel	27 Mar 2012	132	52.1	2.29	1.90 (cone)
KE NLOP #1	KE North Load Out Pit; top-to bottom sample composite present in three jars (#1, #2, #3)	29 Mar 2012	142	2.51	1.05	1.06 (cone) 1.23 (2004)[(c)]
KE NLOP #2		28 Mar 2012	200		1.49	
KE NLOP #3		29 Mar 2012	203		1.72	
KC-4-2	Consol. sample from floor between barrels of open bot. can w/highly damaged fuel from all three bays	28 Mar 2012	142 14	16.6	1.48 (X jar) 1.19 (Y jar)	1.53 (cone) 1.60 (jar)
FE-5 Comp 1	Weasel pit including South Loadout Pit Sludge	28 Mar 2012	164 49	5.32	1.68 (X jar) 1.50 (Y jar)	1.66 (cone) 1.68 (jar)
KE Floc Comp	Composite of KC-4 M250, KC-5, FE-5 and KC Can Comp	28 Mar 2012	132	10.3	1.35	1.25 (cone) 1.30 (jar)
96-13 Solids Grad	Single canister with poor condition fuel	28 Mar 2012	67	74.0	2.23	2.458 (1997)[(b)]
96-13 SSOL	Single canister with poor condition fuel	05 Apr 2012	135	74.0	2.37	2.458 (1997)[(b)]
KC-4 Whole	Consol. sample from floor between barrels of open bot. can w/highly damaged fuel from all three bays	28 Mar 2012	89	16.6	1.73	1.60 (cone) 1.56 (jar)
KE Pit	Weasel Pit Composite of KES-P-16, -Q-17, -R-18, -S-19, & -T-20	29 Mar 2012	107	7.99	1.92	1.77 (cone) 1.92 (jar)
KC-4 P250	>250 µm particle size fraction for wet sieved KC-4 sludge	29 Mar 12	53	No data	1.51	No data
KC-1 M500	Canister sludge from highly damaged fuel collected from one sampling location	29 Mar 2012	21	68.6	2.31	2.05 (cone) 2.66 (jar)
KC-6	Consol. sample from floor area in west bay known to be v. high in ion exchange beads	28 Mar 2012	114	0.314	1.20	1.31 (cone) 1.20 (jar)
KC-6 New	KC-6 Carboy material	27 Mar 2012	117 117	0.314	1.21 (X jar) 1.36 (Y jar)	No data

(a) All data (unless indicated otherwise) are from: Delegard CH, AJ Schmidt, and JW Chenault. 2011. *Characteristics of KE Basin Sludge Samples Archived in the RPL – 2007*. PNNL-17078, Rev. 1, Pacific Northwest National Laboratory, Richland, WA.
(b) Makenas BJ, TL Welsh, RB Baker, EW Hoppe, AJ Schmidt, J Abrefah, JM Tingey, PR Bredt, and GR Golcar. 1997. *Analysis of Sludge from Hanford K East Basin Canisters.* HNF-SP-1201, DE&S Hanford, Inc., Richland, WA.
(c) Schmidt AJ, RT Hallen, DS Muzatko, and SR Gano. 2004. *KE Basin Sludge Flocculant Testing*. PNNL-14730, Pacific Northwest National Laboratory, Richland, WA.

Table C.2. Sludge Samples Being Maintained as Dry Solids (adapted from PNNL-21836,* Table S.2)

Sample ID	Original Jar Gross Weight, g	Original Jar Tare, g	Net Amount in Original Jar, g	New Jar Tare, g	New Jar Gross Weight, g	Net Amount in New Jar,
K Basin Fuel Fines	282.7	89.59	193.11	-	-	-
K Basin Fuel Fines M500	-	-	-	89.2	243.4	154.2
K Basin Fuel Fines P500	-	-	-	89.2	127.5	38.3
FE-3 Comp 1	240.3	Not measured	Cannot be calculated	-	-	-
FE-3 Comp 1	-	-	-	233.6	354.9	121.3
SNF Comp Settling Study	274.6	Not measured	Cannot be calculated	-	-	-
SNF Comp Settling Study	-	-	-	89.3	141.9	52.6
Test 3 Residue	260.8	Not measured	Cannot be calculated	-	-	-
Test 3 Residue	-	-	-	89.2	119.8	30.6
96-13 Settling Study	299.9	Not measured	Cannot be calculated	-	-	-
96-13 Settling Study	-	-	-	89.2	167.6	77.6

* Sinkov, SI, CH Delegard, AJ Schmidt, JW Chenault. 2012. *Characteristics of STP Pre-2004 Archived KE Basin Sludge Samples Before and After Re-Jarring in the RPL – April 2012*. PNNL-21836 (35451-RPT20), Battelle Pacific Northwest National Laboratory, Richland, Washington.

Appendix D

Occurrence and Deficiency Reports

Appendix D

Occurrence and Deficiency Reports

The K Basin process for documenting issues of concern and determining whether significant conditions adverse to quality are indicated includes the use of occurrence forms, deficiency forms, and nonconformance forms as applicable. The project manager and Quality Engineer evaluate occurrences for conditions adverse to quality to determine whether an occurrence is a significant condition adverse to quality that requires elevation to a deficiency or nonconformance report. Occurrences and deficiencies are defined as follows:

- Occurrences are defined to be any issue affecting sample integrity or data quality. Occurrence Reports (ORs) are tracked to document any issue of concern that needs to be brought to the attention of the client, and when applicable, to define appropriate corrective actions or paths forward.

- Deficiencies are defined as failures to follow work-controlling documents such as test plans, test instructions, or procedures. Deficiency reports (DRs) may also result from occurrences that are determined by the responsible project manager to be a significant condition adverse to quality requiring corrective action, preventive action and tracking to closure.

Table D.1 provides a summary of the issued applicable OR forms.

Table D.1. OR and DR Form Summary

ID	Summary	Page
OR-52578-11-29-12	Balance used outside of the valid calibration period.	D.2
OR-52578-1-23-13	Discrepancies in the recorded gross masses during monthly monitoring.	D.7
DR-52578-1-23-13	Discrepancies in the recorded gross masses during monthly monitoring with proactive problem solving to improve observation and data collection for K Basin work.	D.9
OR-52578-5-15-13	Weight performance check was not recorded on the balance performance check log.	D.19
OR-52578-09-12-13	Broken right hand manipulator in Cell 2 limited the ability to collect gross sample masses.	D.20
OR-52578-4-8-14	The 1-kg balance performance check mass was mixed up with the certified 1-kg calibration mass.	D.21

K-Basin Occurrence Report

Title: Balance used outside valid calibration period (Ohaus AV4101C SN: 8031221019) 0 AK 2/13/13	**Date:** 11/29/12
Unique Identifier: 52578-11-29-12	
Primary Person Identifying Issue: Lisa Middleton	
Distribution: Email and posting on K-Basin Share-Point	

Deborah Coffey	Andy Schmidt
Sandy Fiskum	Rick Shimskey
Lisa Middleton	Margaret Smoot
Karl Pool	Rosie Garza

Date Submitted to K-Basin Records: 12-10-12

Occurrence Description:
Balance performance checks are addressed in the procedure, ADM-RSEG-Balances, *Balance Performance Checks*. The first step in the procedure is to verify the calibration expiration date from the posted sticker associated with the balance. The procedure also defines steps to be performed when performance checks do not fall within the upper and lower limits listed on the BPCL form.

Measurements for K Basin monthly sample monitoring were performed in September and October of 2012 using a balance without a valid calibration. The Ohaus AV4101C (SN 8031221019) did not have a valid calibration for balance use in September and October 2012, calibration expired 8/2012; however the BPCL for the balance indicated that the calibration was valid until 2/2013. The balance was calibrated on 7/3/12 but the calibration was only valid until August 2012, when it was due for its regularly scheduled annual calibration. The calibration sticker on the cell window for this balance showed an August 2012 expiration date. This issue will be brought to the attention of the RPL Balance Coordinator.

The first BPCL entries after the 7/3/12 calibration, on 7/30/12, were not initially acceptable; the entries on the BPCL say the balance needed to be re-calibrated and leveled before an acceptable performance check was obtained. On 7/31/12 the performance check was acceptable.

On 9/4/12, the next use, the performance check again was unacceptable; the BPCL says "Scraped paint off/leveled balance/1st try no intolerance [?]. Did not come in. Then an auto cal was performed and an acceptable performance check is documented on 9/4/12.

Subsequent performance checks on 9/5/12, 9/18/12, 10/1/12, 10/2/12 and 10/5/12 were documented as acceptable. See attached BPCLs.

Impact of Occurrence:
The sample masses obtained in September and October 2012 for K Basin monthly sample monitoring will not be traceable to a balance with a valid calibration.

Significant Condition Adverse to Quality? Yes ____ No X
The BPCL forms indicate that when used the balance was operating acceptably. What is of concern is that a systematic action to correct balance performance and re-weigh the check weights after each action is not documented on the BPCL. The initial action should be to assure that the balance is level, and if not to level it and then re-weigh. The next action is to re-zero the balance and reweigh, a weight is to be recorded after each step so it is clear which action led to an acceptable outcome.

52578-11-29-132 DK4 2/13/13 Page 2 of 5

Evaluated by: Sandra K. Fiskum **Date:** 12/10/12

Further Disposition: ☒ No ☐ Yes If yes, ☐ Nonconformance? ☐ Deficiency?

Date Occurrence Closed: 12-10-2012

Signature/Date of Responsible QE: Deborah Coffey 12/10/12

Signature/Date of Responsible K-Basin Manager: Sandra K. Fiskum 12/10/12

Comments:

Upcoming monthly sample monitoring measurements will be done using the Mettler PR5003DR SN:1120100979, located in SAL.

Recommended corrective actions are:

- Hold a briefing/retraining session with SFO staff & applicable RPL staff to review ADM-RSEG-Balances requirements and the steps to take when a balance performance check fails.
- Maintain a copy of ADM-RSEG-Balances in SAL to be used as a reference for corrective actions to take when a performance check fails and the proper order to perform the corrective actions in.

Attachments:

- BPCLs (2) Ohaus AV4101C (SN 8031221019)
- Certificate of Calibration for Ohaus AV4101C (SN 8031221019)

Balance Performance Check Log ✓XAP 7/3/12

Pacific Northwest National Laboratory — **Ohaus AV4101C** — Analytical Support Operations

Balance Information	Check Weight (CW) Information	#1	CW Units	
Identificaiton (e.g. WD34567) 8031221019	CW Identification	Buddy& Holly		This Balance Requires the use of
	CW Assigned Mass (+Units):	3050.0	g	External, Certified weights for Calibration
Location (e.g. Building/Room) SAL / Cell 5	Method for Assigning Diff/Range[a]:	Option 1		Use Calibration weight set:
	CW Difference ± Mass (+Units):			S/N = 10241
Calibration Due Date Feb-13	Or Range Low Mass (+Units) &:	3049.1	g	Expires 5/15/15
	Range High Mass (+Units):	3050.9	g	

(a) Enter "Option #1" or "Option #2" from Procedure PNL-ASO-052, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
7/3/2012	NG	Buddy & Holly	3050.0		Weights taken to establish acceptance criteria.
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
7/30/12	BP	" "	3051.5	N	Recal & centered bubble
7/30/12	BP	" "	3051.5	N	still out of tol
7/30/12	BP	" "	3050.1	Y	Auto recal, passed.
7/31/12	BP	" "	3050.1	Y	
~~9/4/12~~	~~NG~~	~~" "~~	~~3052.1~~	~~NO~~	Scrubbed paint off / leveled Balance / 1st try No in tolerance Did Not Come in
9-4-12	NG	" "	3050.0	Y	Did auto Cal
9-5-12	BP	" "	3049.9	Y	
9-18-12	JWC	Buddy&Holly	3050.5	Y	

NG 9-19-12

> **Complete all column entries.**
> For corrections, place single line through incorrect entry, enter correction and initial/date.
> When full, forward to the ASO M&TE Records Custodian; replace with new Log.
> Following recalibration, lineout/ initial/date unused rows and forward to ASO M&TE Records Custodian; replace with new Log.
> To calibrate or recalibrate a balance contact the ASO M&TE Records Custodian.
> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature [signature] **Date** 9/19/12 **Date to ASO Records** ______

8031221019_BPCL_7-3-12_Buddy&Holly_SAL_Cell 5.xls

52578-11-29-132 DK+ 2/13/13 Page 4 of 5

Balance Performance Check Log ✓ XNP 9/18/12

Pacific Northwest National Laboratory **Ohaus AV4101C** Analytical Support Operations

Balance Information	Check Weight (CW) Information	#1	CW Units	Serial No.	Associated Weight(s)	Exp. Date
Identificaiton (e.g. WD34567)	CW Identification	Buddy& Holly		10241	2kg, 2kg 1 dot	5/15/2015
8031221019	CW Assigned Mass (+Units):	3050.0	g	10241	1kg, 500g, 100g	5/15/2015
Location (e.g. Building/Room)	Method for Assigning Diff/Range[a]:	Option 1		10241	50g	5/15/2015
SAL / Cell 5	CW Difference ± Mass (+Units):					
Calibration Due Date	Or Range Low Mass (+Units) &:	3049.1	g			
Feb-13	Range High Mass (+Units):	3050.9	g			

(a) Enter "Option #1" or "Option #2" from Procedure PNL-ASO-052, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
7/3/2012	NG	Buddy & Holly	3050.0		Weights taken to establish acceptance criteria.
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
7/3/2012	NG	Buddy & Holly	3050.0		
10/1/12	BP	Buddy & Holly	3050.8	Y	
10/2/12	BP	" "	3050.8	Y	
10/5/12	BG	" "	3050.1	Y	

> Complete all column entries.

> For corrections, place single line through incorrect entry, enter correction and initial/date.

> When full, forward to the ASO M&TE Records Custodian; replace with new Log.

> Following recalibration, lineout/ initial/date unused rows and forward to ASO M&TE Records Custodian; replace with new Log.

> To calibrate or recalibrate a balance contact the ASO M&TE Records Custodian.

> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature ________________ **Date** ________ **Date to ASO Records** __________

8031221019_BPCL_7-3-12_Buddy&Holly_SAL_Cell 5.xls

52578-11-29-1~~3~~2 AK 7 2/13/13 Page 5 of 5

Supplement to Certificate of Calibration Issued 11/29/2012

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902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN038031221019120703

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Balance	Ohaus	AV4101C	8031221019	8031221019	325 201
Units	**Readability**	**SOP**	**Cal Date**	**Last Cal Date**	**Cal Due Date**
g	0.1	QC012 8/11	7/3/12	5/25/12	8/2012

FUNCTIONAL CHECKS

ECCENTRICITY	LINEARITY	STANDARD DEVIATION	ENVIRONMENTAL CONDITIONS
Test Wt: 2000 Tol: 0.2	Test Wt: 1000 x 4 Tol: 0.2	Test Wt: 1000 Tol: 0.1	☐ Good ☑ Fair ☐ Poor
As-Found: Pass: ☑ Fail: ☐	As-Found: Pass: ☑ Fail: ☐	1. 1000.0 5. 1000.0 9. 1000.0 2. 1000.0 6. 1000.0 10. 1000.0 3. 1000.0 7. 1000.0 4. 1000.0 8. 1000.0 Result 0.00	Temperature: 22.8°C
As-Left: Pass: ☑ Fail: ☐	As-Left: Pass: ☑ Fail: ☐		

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
4000	4003.3	4000.1	0.11
3000	3002.4	3000.0	0.11
2000	2001.6	2000.0	0.11
1000	1000.8	1000.0	0.11
500	500.4	500.0	0.11
100	100.0	100.0	0.11

☑ Accepted ☐ Rejected / Per Clause(s): 17.5 b

AQSS Reviewer [signature] 11-30-12

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set			CUSTOMER			

Permanent Information Concerning this Equipment:
UNIT IN HOT CELL

Comments/Info Concerning this Calibration:
7/12 log #11779467 WP #H18889 As found out of tolerance @ 4000, 3000, 2000, 1000 & 500g checks. Performed auto span adjustment. Discussed results w/Nicole Green. As left within tolerance.

Report prepared/reviewed by: M. Marcell **Date:** 11-29-12 **Technician:** R. Hintz
Report revised by: M. Marcell **Date:** 11-29-12 **Signature:** [signature]

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

Member: National Conference of Standards Laboratories and Weights & Measures

PT ID: BATN03

K-Basin Occurrence Report

Title: Monthly Monitoring Data Discrepancies	Date: 01/23/13

Unique Identifier: OR 52578-01-23-13

Primary Person Identifying Issue: Margaret Smoot

Distribution: Email and posting on K-Basin Share-Point

Sandy Fiskum	Andy Schmidt	Ben Palma
Deborah Coffey	Rick Shimskey	Margaret Smoot

Date Submitted to K-Basin Records:

Occurrence Description (1):

Gross mass discrepancies recorded on the monthly monitoring log sheets were found during technical review and data reduction as shown in gray highlight in the table below.

Date	Sample ID	Tare Weight		Gross Weight		Solid's Level (cm or ml)		Fluid's Level (cm or ml)		Supernate Added
2/21/2011	52578-TI027 KW220-A1	134.0	g	279.333	g	93.8	cm	95.0	cm	NO
3/30/2011	52578-TI027 KW220-A1	134.0	g	217.7	g	93.2	cm	95.0	cm	NO
5/16/2011	52578-TI027 KW220-A1	134.0	g	215.9	g	93.8	cm	95.1	cm	NO
7/8/2011	52578-TI027 KW220-A1	134.0	g	215.1	g	94.5	cm	95.2	cm	NO
8/2/2011	52578-TI027 KW220-A1	134.0	g	215.2	g	94.2	cm	94.8	cm	NO
9/7/2011	52578-TI027 KW220-A1	134.0	g	270.932	g	94.3	cm	95	cm	YES
9/7/2011	52578-TI027 KW220-A1	134.0	g	287.354	g	94.5	cm	95.5	cm	YES
4/3/2012	KC-2/3 Comp X	236.7	g	649.8	g	4.9	cm	6.0	cm	NO
11/6/2012	KC-2/3 Comp X	236.7	g	643.065	g	4.1	cm	>6	cm	NO
12/4/2012	KC-2/3 Comp X	236.7	g	642.236	g	4.1	cm	5.8	cm	NO
1/3/2013	KC-2/3 Comp X	236.7	g	514.469	g	4.1	cm	5.6	cm	NO
1/24/2013	Kc-2/3 Comp X	236.7	g	639.1	g	4.1	cm	5.6	cm	NO
4/3/2012	KC-4 P250	236.8	g	413.4	g	1.2	cm	3.9	cm	NO
11/6/2012	KC-4 P250	236.8	g	408.352	g	0.7	cm	3.4	cm	NO
12/4/2012	KC-4 P250	236.8	g	407.734	g	0.7	cm	3.3	cm	NO
1/3/2013	KC-4 P250	236.8	g	487.072	g	0.8	cm	3.3	cm	NO
1/24/2013	KC-4 P250	236.8	g	406.7	g	0.8	cm	3.3	cm	NO
5/16/2011	KW230 A3-Archive B	133.67	g	273.1	g	30	ml	95	ml	NO
7/8/2011	KW230 A3-Archive B	133.67	g	267.7	g	1.2	cm	5.0	cm	NO
8/1/2011	KW230 A3-Archive B	133.67	g	241.2	g	0.8	cm	4.5	cm	NO
9/7/2011	KW230 A3-Archive B	133.67	g	260.805	g	1.3	cm	4.5	cm	NO
10/3/2011	KW230 A3-Archive B	133.67	g	257.6	g	1.8	cm	4.2	cm	NO

- Sample 52578-TI027 KW220-A1: The gross mass decreased ~60 g and later increased by ~ 60g (see 2/21/11-9/7/11 data) with no record of water removal or addition.
- Sample KC 2/3 Comp X: gross mass shows a 127.767 g loss (1/3/13) not commensurate with the slight change in slurry volume. Sample mass and heights were re-measured later in January as a result of the technical review.
- Sample KC-4-P250: gross mass shows an 80-g mass gain (1/3/12) with no noted water addition or slurry volume change. Sample mass and heights were re-measured later in January as a result of the technical review.
- Sample KW230-A3-Archive B shows a 26 g mass loss then 20 g mass gain (relative to the 8/1/11) reading.

Occurrence Description (2):
When reviewing the Sample Monitoring Log Instructions it was found that a "data evaluation" was to be performed "~ every 3 months". This task was not achieved and to common knowledge has not been done in over 2 years.

Impact of Occurrence (1):

Probable explanation for sample 52578-TI027 KW220-A1 discrepancies: because of the time crunch on reading over 100 samples on a non-remoted balance, the Hot Cell Technician assisting relayed the mass reading to the supervising technician. The glare on the hot cell windows made the reading of certain numbers quite difficult, more specifically differentiating between 1's & 7's, 0's & 8's on the balances digital read out.

No probable explanation for the KC 2/3 Comp X, KW230 A3-Archive B, and KC-4-P250 mass discrepancies can be inferred other than transcription errors.

Samples 52578-TI027 KW220-A1 and KW230 A3-Archive B: density cannot be estimated from the discrepant data.

Samples KC 2/3 Comp X and KC-4-P250: densities cannot be determined from the early January data, but can be assessed from data collected later in January 2013.

Impact of Occurrence (2):

Copies of the log sheet were incorporated into 52578-TI027 Fall of 2012 as part of the TI close-out process before a thorough technical review was performed. These copies would not contain the minor corrections applied as a course of the log review. Most review comments requested clarifications and initial and date of error corrections and cross-outs.

Significant Condition Adverse to Quality? Yes X No ____

Evaluated by: Sandra K Fiskum **Date:** 1/31/13

Further Disposition: ☐ No ☒ Yes **If yes,** ☐ **Nonconformance?** ☒ **Deficiency?**

Date Occurrence Closed:

Signature/Date of Responsible QE: Deborah Coffey 1/31/13

Signature/Date of Responsible K-Basin Manager: Sandra K Fiskum 1/31/13

Comments:

K- Basin Deficiency Report

Assessment/Audit #: NA **Deficiency/Finding #:** DR-52578-1-31-13	
Associated Occurrence Form? Yes If yes, OR-52578-01-23-13	
Title: Proactive Problem Solving for K Basin Work	**Date: 1/31/13**
Responsible Person: Sandy Fiskum, Project Manager	
Finding Submitted To: Sandy Fiskum, Project Manager	
Response Due Date: 2/15/13	
Finding: Staff need to be empowered to evaluate and correct issues contributing to the collection of incorrect data.	
Requirement: It is an expectation that monthly monitoring measures collected for the K Basin project will result in accurate data being recorded. *Hanford Analytical Services Quality Assurance Requirements Document (HASQARD) SUMMARY of REQUIREMENTS Applicable to K Basin Sample Analysis Participants*, Revision 2 (page 1 paragraph 2) states the following: *"All work including initial R&D investigations (after exploratory research has been completed), permitting, waste characterization and treatment, and clean site closure and long-term monitoring, will have a measurable level of quality for data usage and technical defensibility.."* If the balance cannot be easily read (see Section below), then staff either need to correct the problem or report the problem to the Project Manager and/or Project Quality Engineer.	
Background/Condition Observed: Several gross mass discrepancies were recorded on the K Basin monthly monitoring log sheets; these discrepancies were found during technical review and data reduction. The reason given for these issues was that because of the time crunch on reading over 100 samples on a non-remoted balance, the Hot Cell Technician assisting relayed the mass reading to the supervising technician. The glare on the hot cell windows made the reading of certain numbers quite difficult, more specifically differentiating between 1's and 7's, 0's and 8's on the balances digital read out.	
Instructions for Response: Complete each section of Part A and forward your response to the Audit Team Leader or QE. You may use a separate sheet if needed. All corrective actions must have an associated proposed due date If an action has been completed, attach objective evidence showing completion of the action.	
Significant Condition Adverse to Quality? Yes _X___ No ___	

Incorrect data resulted in loss of information such that sample densities could not be calculated.

Evaluated by: Sandra K Fiskum **Date:** 1/31/13

Verification Actions/Comments:

Date Response Submitted: 1/31/13

Response Accepted: 2) Yes X No ______ **Date of Evaluation:** 1/31/13 OSCoffey 1/31/13

Corrective Action Verification:

CA 1 Yes ____ No ______ Date of Evaluation: ________________

PA 1 Yes ____ No ______ Date of Evaluation: ________________

Date Finding Closed:

Signature of Responsible QA Staff or Audit Team Member: ________________________

Date Closed: ________________________

Deficiency Form

Part A

Assessment/Audit #: NA **Deficiency/Finding #:** DR-52578-1-31-13	
Associated Occurrence Form? Yes If yes, OR#: OR-52578-01-23-13	
Title: Proactive Problem Solving for K Basin Work	**Date: 1/31/13**
Responsible Person: Sandy Fiskum, Project Manager	

Identify the cause of the deficiency/deficiencies.
Inattention to detail

1) **Determine the impact of the deficiency/deficiencies.**

1a) Has any completed work or data been affected? If so, describe.
Yes, several entries during the monthly monitoring activity were affected. These are described in OR-52578-01-23-13.

1b) Evaluate if similar problems exist in other areas of work. If so, describe
There are currently no other active tasks occurring. Other areas of previously-conducted work have been reviewed and any issues identified and resolved.

2) **Identify what actions have been or will be taken to correct the immediate problem(s);**
A staff meeting will be held where the root cause of the OR/DR will be discussed. Proactive approaches will be defined and discussed (such as use of in-cell cameras to read the balance) to addressing this type of problem.

Correction Action (CA)1: To be completed by February 7, 2013.

3) **Identify what actions have been or will be taken to prevent recurrence;**
The staff meeting described above will also focus on defining actions to prevent recurrence.

Preventive Action (PA)1: To be completed by February 7, 2013.

4) **Identify who has been assigned as the responsible person to address each corrective and preventive action by the K-Basin Project Manager and be sure that their concurrence has been obtained as to when these will be completed. Note that corrective and preventive action is expected to occur and be completed in a timely manner.**
CA1: Sandy Fiskum, 2/7/13
PA1: Sandy Fiskum, 27/13

5) Signature of Finding Owner: Sandra K. Fisk Date: 1/31/13
Sandy Fiskum, Project Manager

Final version of DR-52578-1-31-13

Previous pages capture version that was used to document corrective and preventive action response and Project Manager acceptance of responsibility for actions and due dates.

K- Basin Deficiency Report

Assessment/Audit #: NA **Deficiency/Finding #:** DR-52578-1-31-13
Associated Occurrence Form? Yes If yes, OR-52578-01-23-13
Title: Proactive Problem Solving for K Basin Work — **Date: 1/31/13**
Responsible Person: Sandy Fiskum, Project Manager
Finding Submitted To: Sandy Fiskum, Project Manager
Response Due Date: 2/15/13
Finding: Staff need to be empowered to evaluate and correct issues contributing to the collection of incorrect data.
Requirement: It is an expectation that monthly monitoring measures collected for the K Basin project will result in accurate data being recorded. *Hanford Analytical Services Quality Assurance Requirements Document (HASQARD) SUMMARY of REQUIREMENTS Applicable to K Basin Sample Analysis Participants*, Revision 2 (page 1 paragraph 2) states the following: *"All work including initial R&D investigations (after exploratory research has been completed), permitting, waste characterization and treatment, and clean site closure and long-term monitoring, will have a measurable level of quality for data usage and technical defensibility.."* If the balance cannot be easily read (see Section below), then staff either need to correct the problem or report the problem to the Project Manager and/or Project Quality Engineer.
Background/Condition Observed: Several gross mass discrepancies were recorded on the K Basin monthly monitoring log sheets; these discrepancies were found during technical review and data reduction. The reason given for these issues was that because of the time crunch on reading over 100 samples on a non-remoted balance, the Hot Cell Technician assisting relayed the mass reading to the supervising technician. The glare on the hot cell windows made the reading of certain numbers quite difficult, more specifically differentiating between 1's and 7's, 0's and 8's on the balances digital read out.
Instructions for Response: Complete each section of Part A and forward your response to the Audit Team Leader or QE. You may use a separate sheet if needed. All corrective actions must have an associated proposed due date If an action has been completed, attach objective evidence showing completion of the action.

Page 1

Significant Condition Adverse to Quality? Yes X No ___

Incorrect data resulted in loss of information such that sample densities could not be calculated.

Evaluated by: Sandra K Fiskum **Date:** 2/12/13

Verification Actions/Comments:

Date Response Submitted: 1/31/13

Response Accepted: 2) Yes X No _____ **Date of Evaluation:** 1/31/13 DS Coffey 1-31-13

Corrective Action Verification:

CA 1 Yes X No ___ Date of Evaluation: 2/8/12

PA 1 Yes X No ___ Date of Evaluation: 2/8/12

Date Finding Closed:

Signature of Responsible QA Staff or Audit Team Member: DS Coffey

Date Closed: 2/8/2013

Deficiency Form

Part A

Assessment/Audit #: NA **Deficiency/Finding #:** DR-52578-1-31-13	
Associated Occurrence Form? Yes If yes, OR#: OR-52578-01-23-13	
Title: Proactive Problem Solving for K Basin Work	**Date: 1/31/13**
Responsible Person: Sandy Fiskum, Project Manager	

Identify the cause of the deficiency/deficiencies.

Inattention to detail
Time crunch to complete a long work activity
Poor visibility and poor lighting for reading the in-cell balance read-out
K Basin staff did not confirm reading obtained by Hot Cell Technician

1) Determine the impact of the deficiency/deficiencies.

1a) Has any completed work or data been affected? If so, describe.

Yes, several entries during the monthly monitoring activity were affected. These are described in OR-52578-01-23-13.

1b) Evaluate if similar problems exist in other areas of work. If so, describe

There are currently no other active tasks occurring. Other areas of previously-conducted work have been reviewed and any issues identified and resolved.

2) Identify what actions have been or will be taken to correct the immediate problem(s);

A staff meeting will be held where the root cause of the OR/DR will be discussed. Proactive approaches will be defined and discussed (such as use of in-cell cameras to read the balance) to addressing this type of problem.

Correction Action (CA)1: To be completed by February 7, 2013.

The meeting on February 7, 2013 (see attached agenda and sign-in sheet) provided several suggestions to prevent recurrence of the issue including:

- Slowing down and doing the job right the first time—if there are issues, talk to PM.
- Using other tools to help staff be successful; be creative. In this case, an in-cell camera might have helped with the interpreting the readout. A different balance could have been used. Using a step-stool (higher angle of vision) may help analyst to better see the balance read-out.
- Place your nose to the window and record your own observations/data without reliance on a 3rd party.
- Load the data into the spreadsheet when collected; errors tend to show up immediately.
- Noted that the balance was recently moved to Cell 3 where there are overhead lamps to improve lighting/visibility.

3)	**Identify what actions have been or will be taken to prevent recurrence;** The staff meeting described above will also focus on defining actions to prevent recurrence. Preventive Action (PA) 1: To be completed by February 7, 2013. See above discussing meeting results.
4)	**Identify who has been assigned as the responsible person to address each corrective and preventive action by the K-Basin Project Manager and be sure that their concurrence has been obtained as to when these will be completed. Note that corrective and preventive action is expected to occur and be completed in a timely manner.** CA1: Sandy Fiskum, 2/7/13 PA1: Sandy Fiskum, 27/13
5)	Signature of Finding Owner: Sandra K. Fiskum Date: 2/12/13 Sandy Fiskum, Project Manager

2/7/13 K-Basin Project Staff Meeting

K-Basin Project Staff Meeting Agenda

1. Deliverables
    a. Sample Status Summary (Fiskum with help from Smoot) Final due to client Feb. 15
        i. Spreadsheet tech reviews (Fountain) need to finish/sign
        ii. Report tech review—Fountain
    b. Rheology summary (Burns) Final due April 26 to the client; draft due April 12.
        i. Tech reviewer—Tingey?
        ii. QE—Dey
2. Monthly monitoring (<u>Smoot</u>)
3. Sample consolidation
    a. TI051 (Snow)
        i. QE review comment resolution status
    b. TI052 (Edwards)
        i. Draft status
4. Waste
    a. TI050 sample residuals (Edwards)
    b. SFO waste processing (Steen)
5. DR D52578-1-31-13
    a. Expectations:
        i. Data collection activities shall be conducted with utmost care. Staff time to collect data is valuable; there is no point in collecting invalid data.
        ii. *"All work including initial R&D investigations (after exploratory research has been completed), permitting, waste characterization and treatment, and clean site closure and long-term monitoring, will have a measurable level of quality for data usage and technical defensibility"* from *Hanford Analytical Services Quality Assurance Requirements Document (HASQARD) SUMMARY of REQUIREMENTS Applicable to K Basin Sample Analysis Participants*, Revision 2 (page 1 paragraph 2)
    b. Issue: hot cell tech read masses to K Basin staff—recorded masses were in obvious error; collected data could not be used.
    c. Contributing causes:
        i. Time crunch to complete long activity
        ii. Poor visibility of the in-cell balance readout
        iii. K Basin staff did not confirm mass by directly reading it
    d. Empowerment
        i. Slow down and do the job right the first time—if there are issues, talk to me.
        ii. Use other tools to help you be successful—be creative. In this case, an in-cell camera might have helped with the interpreting the readout. A different balance could have been used.
        iii. Place your nose to the window and record your own observations/data without reliance on a 3rd party.
        iv. Other suggestions?
6. D52578-3-19-12 change control and penetrometer (Shimskey/Coffey/Fiskum) need to close.

K-Basin Project 52578 Staff Meeting Attendance Form

7 February 2013

Printed Name	Signature	Organization	Role in Project 52578
Sandra K. Fiskum	Sandra K. Fiskum	D9H63	Project Manager
Rick W Shimskey	[illegible]	D9H63	
Carolyn Burns	C Burns	D9H63	Rheologist
Margaret Smoot	M Smoot	D9H63	Lab tech
BILL DEY	William Dey	Q&A	QE
DEBORAH COFFEY	Deborah Coffey	Q&A	QE
Matthew Edwards	M Edwards	D9H63	Scientist

K-Basin Occurrence Report

Title: TI Weight Performance Check Recorded on TI, But Not on BPCL Form	**Date: 5/15/13**
Unique Identifier: 52578-05-15-13	
Primary Person Identifying Issue: D Coffey	
Distribution: Email and posting on K-Basin Share-Point: D Coffey, M Edwards, S Fiskum, A Schmidt, L Snow	
Date Submitted to K-Basin Records:	
Occurrence Description: During the QE review of completed 52578-TI052, it was noted that a balance performance check was performed on 4/12/13 (page 14 of the TI) using the Mettler AT400 balance in RPL/305 benchtop (SN: N04143 a.k.a. 360-06-01-048), the BPCL where the 4/12/13 check was entered was not the correct form and had expired in February 2013.	
Impact of Occurrence: As the incorrect BPCL form was used, the performance check recorded on the TI may or may not have been acceptable as the acceptance limits were not available to make this determination. The updated BPCL form for the August 2012 calibration was located and confirmed that the 4/12/13 performance check (299.9998) was acceptable as the limits were 299.9982 to 300.0024. The correct BPCL form was situated at the balance in RPL/305 on 5/15/13.	
Significant Condition Adverse to Quality? Yes _____ No X **Evaluated by:** SK Fiskum **Date:** 5/15/13	
Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**	
Date Occurrence Closed: **Signature/Date of Responsible QE:** Deborah Coffey 5/15/13 **Signature/Date of Responsible K-Basin Manager:** Sandra K Fiskum 5/15/13	
Comments:	

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K-Basin Occurrence Report

Title: Broken Right Hand Manipulator in Cell 2	**Date: 9/17/13**
Unique Identifier: 52578-09-12-13	
Primary Person Identifying Issue: M Smoot	

Distribution: Email and posting on K-Basin Share-Point

Deborah Coffey	Andy Schmidt
Sandy Fiskum	Rick Shimskey
Margaret Smoot	Ben Palma

Date Submitted to K-Basin Records: 9/18/13

Occurrence Description:

During monthly monitoring activities in SAL, the right-hand manipulator in Cell 2 broke, negatively impacting the project's access to the balance. The event was discussed with the Project Manager (Sandy Fiskum) shortly after occurring. In the discussion it was decided that monthly monitoring activities would continue without mass measurements, with a stipulation that if any samples were found to need water, water would be added after the manipulator was fixed. No such samples were found.

Impact of Occurrence:

The impact of the occurrence was determined to be minimal; all samples were monitored for sludge and water levels and the objective of monthly monitoring (to keep sludge samples wet) was achieved. However, mass data are missing on approximately 75% of the total samples monitored for the month of September 2013

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Significant Condition Adverse to Quality? Yes ______No X

Evaluated by: Sandra K. Fiskum **Date:** 9/18/13

Further Disposition: ☒ **No** ☐ **Yes** **If yes,** ☐ **Nonconformance?** ☐ **Deficiency?**

Date Occurrence Closed: 9-18-13

Signature/Date of Responsible QE: Deborah Coffey 9-18-13

Signature/Date of Responsible K-Basin Manager: Sandra K. Fiskum 9/18/13

Comments:

K-Basin Occurrence Report

Title: SAL Mix-Up of the 1-kg Calibration and Performance Check Weights	**Date:** 4/8/14
Unique Identifier: 52578-04-08-14	
Primary Person Identifying Issue: Margaret Smoot	
Distribution: Email and posting on K-Basin Share-Point	Deborah Coffey, K Basin QE; Andy Schmidt; Sandy Fiskum, Project Manager; Rick Shimskey; Margaret Smoot; Carolyne Burns
Date Submitted to K-Basin Records:	

Occurrence Description:

K Basin work conducted in March and April 2014, supporting shear strength testing, sample repackaging, and monthly monitoring, required use of the Mettler PR5003DR balance in the shielded analytical laboratory (SAL) hot Cell 2.

When conducting the performance check, the analyst raised a concern regarding the location of the1-kg weight to be used as the check weight for the balance; it appeared possible that it was mixed up with a different 1-kg weight.

Two 1-kg masses are present in Cell 2. One mass is used as a calibrated standard for annual calibration activities; the other mass is used as the performance check weight (in combination with a 2-kg mass) to be used for daily balance performance check log (BPCL) entries.

The suspected mix-up was reported to the K Basin Project Manager and Quality Engineer (QE). The QE provided guidance to weigh both the "calibration 1-kg weight" and the "balance performance 1-kg weight" in conjunction with the 2-kg weight and if both measures were within the acceptance criteria (2999.93 g to 2999.99 g), continue work and place the weight most likely to be the calibration weight back in the calibration weight container. The check weight measures were:

- 2999.96 g (1 kg assumed check weight plus 2 kg) and
- 2999.96 g (1 kg assumed calibration weight plus 2 kg).

Impact of Occurrence:

The balance was not recalibrated with the suspected mixed up weights; therefore masses collected with the balance since 3/14/14 were not compromised.

The 1-kg calibration weight (with known expiration date) can no longer be differentiated from the check weight. The balance internal re-calibration feature cannot be used without a certified calibration weight. If the balance performance check weight drifts out of the prescribed tolerance, use of the balance will be put on hold until a replacement weight is ordered.

Significant Condition Adverse to Quality? Yes ______No X

The impact to data is low, but the impact to the project budget and the effort needed to correct the problem is of concern. This is a recurrence of ASO 98620-7-13-2010, where it was found that performance checks were not passing and it that the calibration weight set used to perform manual calibrations was compromised; 1 g and 20 g weights were missing. Further, calibration weights were being used as check weights. A new weight set was purchased and received on 7/20/10 and QCS re-calibrated the balance on 7/21/10. Balance was tagged as out-of-service from 7/13/10 to 7/21/10. On 7/28/10 S Fiskum provided balance use training to SFO staff.

Evaluated by: SK. Fiske **Date:** 4/15/14

Further Disposition: ☐ **No** ☒ **Yes** **If yes,** ☐ **Nonconformance?** ☒ **Deficiency?** SK Fiskum 4/15/14 DR-52578-4-8-14
Date Occurrence Closed: 4-15-2014 **Signature/Date of Responsible QE:** Deborah Coffey 4-15-2014 **Signature/Date of Responsible K-Basin Manager:** SK Fiskum 4/15/2014
Comments:

Distribution

CHPRC

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