

Testing of Enhanced Chemical Cleaning of SRS Actual Waste Tank 5F and Tank 12H Sludges

C. J. Martino
W. D. King

August 2011

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
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EXECUTIVE SUMMARY

In support of Savannah River Site (SRS) tank closure efforts, the Savannah River National Laboratory (SRNL) conducted Real Waste Testing (RWT) to evaluate an alternative to the baseline 8 wt% oxalic acid (OA) chemical cleaning technology for tank sludge heel removal. The large quantities of sodium oxalate and other metal oxalates formed impact downstream processes by requiring additional washing during sludge batch preparation and increase the amount of material that must be processed in the tank farm evaporator systems and the Saltstone Processing Facility. Enhanced Chemical Cleaning (ECC) was identified as a potential method for greatly reducing the impact of oxalate additions to the SRS Tank Farms without adding additional components to the waste that would extend processing or increase waste form volumes.

ECC is a promising alternative to bulk OA cleaning, which utilizes a more dilute OA and an oxalate destruction technology. Sludge is dissolved in a Treatment Tank by adding 2 wt% OA and mixing the tank contents. The mixture of dissolved sludge and suspended insoluble solids is transferred to the ECC reactor, where the OA and other oxalates are decomposed. Concentrated ozone gas is added to the reactor, resulting in soluble ozone and hydroxyl radicals that aid the decomposition of oxalate ion to carbon dioxide gas and raise the pH of the liquid. The change in chemistry inside the ECC reactor causes the dissolved sludge to precipitate, mostly in the form of metal oxides. Ultraviolet (UV) light can also be used to augment the decomposition of OA.

The primary goals for SRNL RWT are as follows:

- to confirm ECC performance with real tank sludge samples,
- to determine the impact of ECC on fate of actinides and the other sludge metals, and
- to determine changes, if any, in solids flow and settling behavior.

SRNL conducted two tests using actual SRS waste material from Tanks 5F and 12H. Testing involved sludge dissolution with an initial 2 wt% OA, the decomposition of the oxalates by ozonolysis, the evaporation of a portion of the material for water removal, and tracking the concentrations of key components in the Deposition Tank. The UV light was not utilized in the decomposition testing. The following are results of the ECC RWT:

- Using three OA batches per test, Test 1 showed an approximately 73% removal of Tank 12H sludge and Test 2 showed an approximately 80% removal of Tank 5F sludge. Removal includes both dissolution of soluble components and partial transfer of insoluble solids. The dissolution process was constrained to eight hours per OA batch and thus did not precisely represent the in tank dissolution process.
- Residuals remaining in the dissolution vessel at the conclusion of Test 1 were primarily aluminum (accounts for over 90% of the mass assuming boehmite is the major form) with small amounts of iron and manganese. Residuals remaining in the dissolution vessel at the conclusion of Test 2 were primarily iron (accounts for 66% of the mass assuming hematite is the major form) with small amounts of manganese and nickel.
- Some minor metals were not removed effectively (<40% removal) from the dissolution vessel of the RWTE with 2 wt% OA. For Test 1, these metals included the lanthanides and actinides Ce, Gd, La, Th, Eu, Am, and possibly Np. For Test 2, OA was more effective at removing the minor metals and only thorium was removed at a level below 40%.

- Decomposition of 2 wt% OA to levels of <100 ppm using 2 L/min of 5 wt% ozone at 70 °C, a pressure of 8 psi, and a liquid recirculation rate of 1.8 gal/min required 8 to 14 hours for nominal 3.2L batches. The pH and oxidation/reduction potential (ORP) were tracked during decomposition testing.
- Sludge components were tracked during OA decomposition, showing that most components have the highest soluble levels in the initial dissolved sludge and early decomposition samples and exhibit lower soluble levels as OA decomposition progresses. Samples from the end of the decomposition process typically have the lowest soluble level of sludge components.
- Over the time period studied, the changes to the solubilities of important components in the Deposition Tank storage tests were insignificant. .
- Deposition Tank storage testing for Tank 12H ECC processing indicated higher solubilities for uranium and plutonium when compared with the control condition. Similar testing for Tank 5F indicated higher solubilities for uranium when compared with the control condition. The solubilities of uranium and plutonium remained within the levels expected for Tank Farm high pH supernatant liquids, which are relatively low compared to the levels in the sludge solids.
- Use of the ECC Evaporator generally led to higher soluble component concentrations in the Deposition Tank storage tests.
- Over the range of 25 to 70 °C, the storage temperature was not a major factor affecting soluble concentrations during the Deposition Tank storage tests.
- Tank 5F sludge/OA slurries, believed to representative of ECC Treatment Tank materials, were not suitable for rheology measurements, due to the fact that most of the sludge particles settled rapidly. Chemical dissolution of these materials is likely necessary for the removal these materials from the treatment tank. Since iron oxide and hydroxide materials are primary chemical constituents for Tank 5F sludge, chemical dissolution in OA should be effective.
- Tank 12H sludge/OA slurries, believed to representative of ECC Treatment Tank materials, formed suspensions which were suitable for rheology measurements. Stress versus strain curves observed for these materials revealed that the rheological properties were characteristic of Bingham Plastic materials. For sludge/acid slurries containing 10 weight percent total solids, the measured viscosity and yield stress values were 7 cP and 6 Pa, respectively. Only minor effects on the rheological properties were observed across the temperature range of 30 to 50 °C. Little difference was observed between the process midpoint and endpoint samples. Based on these results, the transfer of significant amounts of Tank 12H sludge materials from the treatment tank by suspension (rather than dissolution) appears possible.
- Rheology testing of the Tank 5F and 12H Deposition Tank samples revealed that these slurries were thin with little yield stress. Even the ECC product slurries that were evaporated to remove $\geq 85\%$ of the water had low enough insoluble solids content as to not exhibit a significant yield stress.

- Settling of the adjusted Deposition Tank solids was comparable to the control and is fast enough as to not likely delay processing. Only the Tank 5F product without pH adjustment exhibited settling significantly slower than the control sample of sludge that did not go through the ECC process.

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LIST OF ABBREVIATIONS

CP	Valve prefix
CV AA	Cold Vapor Atomic Adsorption
ECC	Enhanced Chemical Cleaning
FM	Flow Measurement prefix
HLW	High-Level Waste
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma -- Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma -- Mass Spectrometry
M&TE	Measurement and Test Equipment
NIST	National Institute for Standards and Technology
OA	oxalic acid
ODU	Ozone Destruction Unit
ORP	Oxidation / Reduction Potential
PI	Pressure Indicator prefix
PMP	Polymethylpentene
PTFE	Polytetrafluoroethalene
PuTTA	Plutonium separation by thenoyltrifluoracetone
RWT	Real Waste Testing
SC	Storage Condition
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
TI	Temperature Indicator prefix
TOC	Total Organic Carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
UV	Ultraviolet

1.0 Introduction

Forty three of the High Level Waste (HLW) tanks at the Savannah River Site (SRS) have internal structures that hinder removal of the last approximately five thousand gallons of waste sludge solely by mechanical means. Chemical cleaning can be utilized to dissolve the sludge heel with oxalic acid (OA) and pump the material to a separate waste tank in preparation for final disposition.^{1,2} This dissolved sludge material is pH adjusted downstream of the dissolution process, precipitating the sludge components along with sodium oxalate solids. The large quantities of sodium oxalate and other metal oxalates formed impact downstream processes by requiring additional washing during sludge batch preparation and increase the amount of material that must be processed in the tank farm evaporator systems and the Saltstone Processing Facility. Enhanced Chemical Cleaning (ECC) was identified as a potential method for greatly reducing the impact of oxalate additions to the SRS Tank Farms without adding additional components to the waste that would extend processing or increase waste form volumes.^{3,4}

In support of Savannah River Site (SRS) tank closure efforts, the Savannah River National Laboratory (SRNL) conducted Real Waste Testing (RWT) to evaluate an alternative to the baseline 8 wt. % OA chemical cleaning technology for tank sludge heel removal. The baseline OA technology results in the addition of significant volumes of oxalate salts to the SRS tank farm and there is insufficient space to accommodate the neutralized streams resulting from the treatment of the multiple remaining waste tanks requiring closure.

ECC is a promising alternative to bulk OA cleaning, which utilizes a more dilute OA (nominally 2 wt. % at a pH of around 2) and an oxalate destruction technology. The technology is being adapted by AREVA from their decontamination technology for Nuclear Power Plant secondary side scale removal.

This report contains results from the SRNL small scale testing of the ECC process using SRS sludge tank sample material. A Task Technical and Quality Assurance Plan (TTQAP)⁵ details the experimental plan as outlined by the Technical Task Request (TTR).⁶ The TTR identifies that the data produced by this testing and results included in this report will support the technical baseline with portions having a safety class functional classification. The primary goals for SRNL RWT are as follows:

- to confirm ECC performance with real tank sludge samples,
- to determine the impact of ECC on fate of actinides and the other sludge metals, and
- to determine changes, if any, in solids flow and settling behavior.

1.1 Background

The ECC process supports closure of non-compliant waste tanks at SRS by removing the residual sludge heels in the tanks. Sludge is dissolved in a Treatment Tank by adding 2 wt% OA and mixing the tank contents. This mixture of dissolved sludge and suspended insoluble solids is transferred to the ECC reactor, where the OA and other oxalates are decomposed. Concentrated ozone gas is added to the reactor, resulting in soluble ozone and hydroxyl radicals that aid the decomposition of oxalate ion to carbon dioxide gas and raise the pH of the liquid. The change in chemistry inside the ECC reactor causes the dissolved sludge to precipitate, mostly in the form of metal oxides/hydroxides. The supplemental use of UV light in the ECC reactor holds potential

for increasing the oxalate decomposition but it is not included in the current baseline process design.

For F-Area tanks, the treated waste is transferred to a Deposition Tank where the solids will be allowed to settle, followed by water removal via the 2F evaporator (Figure 1-1). The planned treatment of H-Area tanks includes a flash evaporator to recover and recycle water (Figure 1-2). Sodium hydroxide will be added in-line to both the F-Area and H-Area stream during the transfer to the Deposition Tank to ensure compliance with the Corrosion Control Program requirements.

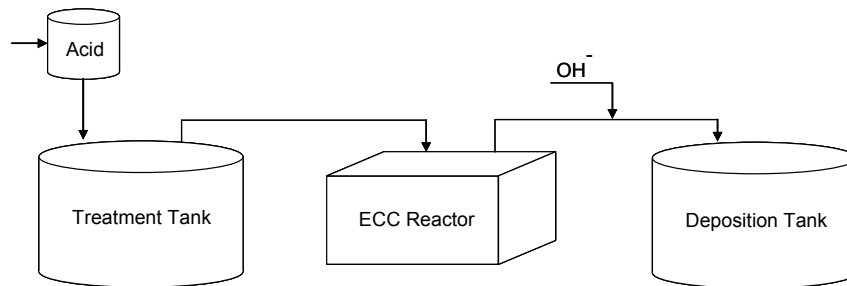


Figure 1-1: ECC Process in F-Area

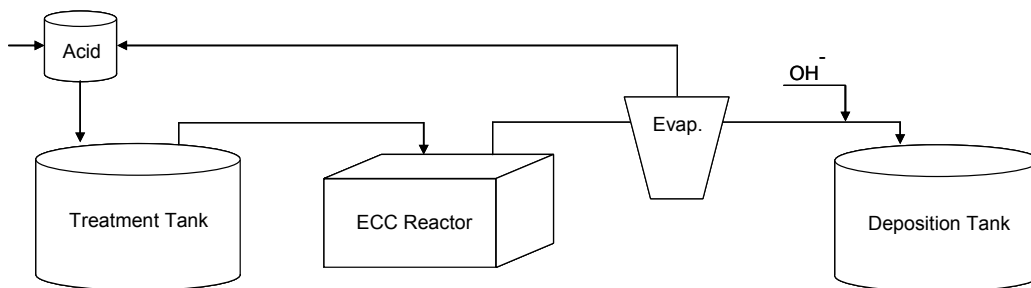


Figure 1-2: ECC Process in H-Area

2.0 Experimental Procedure

2.1 Testing Overview

The ECC RWT is designed to demonstrate the following:

- The use of ozone (with or without the use of UV light) will be effective for decomposing OA used for actual waste sludge dissolution.
- The solubility of some metals will change during the dissolution and the oxalate decomposition compared to nominal sludge storage conditions.
- The solubilities of metals and actinides in the ECC reactor product or evaporator product will return to levels comparable with the nominal sludge storage condition when brought back into Tank Farm corrosion control compliance (via contact with tank heel material and/or pH adjustment with sodium hydroxide) over a time period of hours to days.

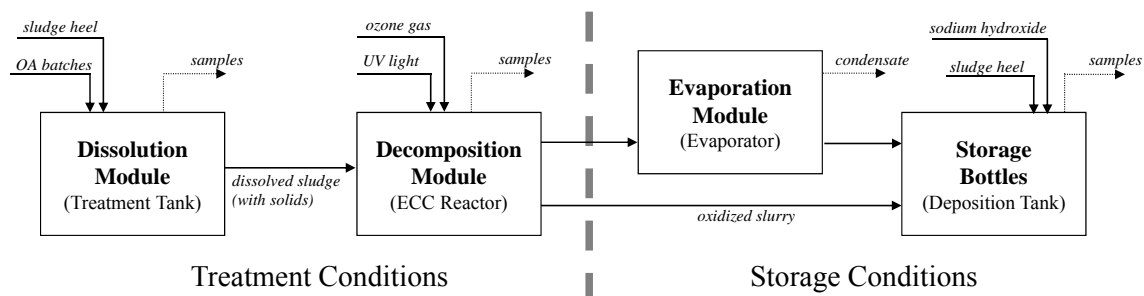


Figure 2-1: ECC Treatment and Storage Testing

The ECC RWT matrix is divided into two conceptual sections, treatment conditions and storage conditions. This distinction between the two types of testing is necessary because the tests will be performed concurrently, while involving two separate test matrices. Figure 2-1 illustrates the portions of the process affected by each matrix. The treatment conditions matrix pertains to the conditions and the samples from the Dissolution and Decomposition Modules. The storage conditions matrix pertains to the Deposition Tank conditions and related samples from the storage bottles.

2.1.1 Treatment Condition Parameters

Overall, four tests were planned at various treatment conditions, as shown in Table 2-1. Tests 1 and 2 are the process baseline cases, which do not use UV light to aid in the decomposition of oxalates. Tests 3 and 4, which use UV light to augment the decomposition of oxalate, are planned for a future report.

Each test was initiated by loading either 48.1 grams (Tank 12H) or 65.1 grams (Tank 5F) of actual waste sludge solids into the Dissolution Module basket. A series of nominally 3.2 L batches of 2 wt% OA were added to the Dissolution Module and heated to the dissolution temperature. The nominal dissolution batch duration was eight hours at temperature. Temperature and pH were recorded periodically. At the end of eight hours, a sample was taken and the material was transferred forward to the Decomposition Module.

Three OA batches were used for each test with the goal of 95% sludge removal from the Dissolution Module. Based on the equipment design, it was only possible to quantify the amount of undissolved sludge remaining in the Dissolution Module basket at the end of each OA batch. Approximations of sludge removal were made from the amount of material collected from the Dissolution Module. The dissolution batches were expected to transfer some undissolved sludge solids forward to the Decomposition Module. Thus, removal of sludge material from the Dissolution Module does not directly equate to dissolution of sludge materials.

The Decomposition Module was operated using the methodology recommended by AREVA and adapted as necessary by SRNL.⁷ The flow of ozone into the system through the eductor was balanced to the recirculation of the dissolved sludge material. The Decomposition Module operating temperature was 70 °C and the expected decomposition duration was 6 to 20 hours. The Decomposition Module vessel included measurement of temperature, pH, and ORP. Throughout Decomposition Module operations, several intermediate liquid or slurry samples were collected from the recirculation loop for preparation and analysis.

Table 2-1: Treatment Test Matrix

Test	Sludge Feed	Light?	OA Batches	Included in Report
1	Tank 12H	No	3	Yes
2	Tank 5F	No	3	Yes
3	Tank 12H	Yes	3	No
4	Tank 5F	Yes	3	No

The Decomposition Module was operated until the pH increased to above 7 and stabilized. At that point, a sample was tested by a permanganate method to confirm that the target endpoint of 100 ppm oxalate was reached. For the RWT, the target for OA decomposition of less than 100 ppm corresponds to 99.5% oxalate decomposition.

As evident in the treatment test matrix, the following parameters were varied:

- The source of the actual waste sludge, from Tank 5F or 12H.
- The use or non-use of UV light to aid in OA decomposition (this report only contains results without the use of UV light)

2.1.2 Storage Condition Parameters

At the conclusion of Decomposition Module operation, the material was emptied into an auxiliary vessel and was split into portions. The largest portion of the material was transferred to the Evaporation Module for further processing. The other portion was split among several storage bottles representing possible Deposition Tank conditions without the use of an ECC Evaporator. The Evaporator Module was operated under vacuum to effect an 84% volume reduction of the slurry through water removal at approximately 70 °C. At the conclusion of evaporation, the concentrated material was removed from the Evaporation Module and the concentrate slurry was split among several storage bottles representing possible Deposition Tank conditions after use of an ECC Evaporator.

The study of the interaction of components in the Deposition Tank was accomplished by monitoring the resultant ECC material in several storage bottles. Table 2-2 contains the storage test matrix. Each of the four tests utilized a separate set of nine storage bottles. The products from all three individual OA batches within each test utilized the same set of nine storage bottles. The storage bottles were held at the desired test conditions for 15 days (Tank 5F) or 35 days (Tank 12H) and sampled periodically. Additionally, pH and ORP probes were used to gather this data on the storage bottle samples at the time of sampling. Deposition Bottles were manually mixed periodically (at the time of sampling) but were not mixed continuously.

Table 2-2: Storage Test Matrix

Storage Condition	Temperature (°C)	Evaporation?	pH Adjust?	Sludge Heel?
1	50	No	Yes	No
2	50	No	Yes	Yes
3	50	No	No	No
4	50	Yes	Yes	No
5	50	Yes	Yes	Yes
6	50	Yes	No	No
7	70	*	Yes	Yes
8	~25 (ambient)	*	Yes	Yes
9 (control)	50	No	Yes	Yes

* nominal condition, Yes for Tank 12H, No for Tank 5F

For the planned F-area implementation of ECC, Storage Condition 2 (without evaporation, with pH adjustment and with tank heel material) is the nominal storage condition. For the planned H-area implementation of ECC, Storage Condition 5 (with evaporation, with pH adjustment and with tank heel material) is the nominal storage condition.

A single storage condition control was filled and sampled in parallel with each test. The control used a small amount of feed tank sludge, sodium hydroxide, and heel material. Multiple batches of deionized water were added to the control sample in parallel with the batches of decomposed tank cleaning solution.

As evident in the storage test matrix, the following parameters were varied for material leaving the Decomposition Module:

- Material was either be sent to the Evaporation Module or directly to storage bottles. A larger fraction of the material was sent to the Evaporation Module so that the volume of all storage bottles was about the same.
- Most of the Deposition Bottles was held at 50 °C, with additional bottles from the nominal test condition held at ambient cell temperature and 70 °C.
- A subset of the storage bottles initially had a small heel of dilute sludge slurry (from Tank 51H), while other storage bottles were initially be empty.
- A subset of the storage bottles were pH adjusted by addition of a small amount of 50 wt% NaOH targeting 1 M free hydroxide.

2.1.3 Actual Waste Sludge Materials

Two actual-waste sludge feeds from previous tank sampling are utilized as feeds for the RWT with an additional sludge material utilized for Deposition Tank sludge heel. Tank 5F sludge material is used to represent F-Area PUREX sludges and Tank 12H sludge material are used to represent H-Area HM sludges. Tank 51H sludge and supernate are used during testing as Deposition Tank heel material. Additional details and characterization of these materials are provided in Section 3.1.

2.2 Description of Equipment

The majority of the ECC RWT was performed in SRNL Shielded Cells B-Block. The equipment simulating the Treatment Tank, ECC Reactor, and Evaporator for the RWT was designed and provided by AREVA. The Deposition Tank and intermediate storage was provided by SRNL. Due to equipment limitations, the treatment tank rheology portion of the testing was accomplished without the use of the AREVA-supplied equipment. The treatment tank rheology testing was performed in SRNL Shielded Cells A-Block in a location close to the rheometer.

2.2.1 ECC Real Waste Test Equipment

The primary ECC RWT equipment was designed and fabricated by AREVA per a contract with SRR and through consultation with SRNL. The equipment design is based on the SRR Statement of Work,⁸ the AREVA RWT technical requirements and equipment design report,⁹ and the AREVA RWT project plan¹⁰ and is reflected in the Piping and Instrumentation Diagram¹¹. Configuration and operating conditions are outlined in the Process Flow Diagram.⁷

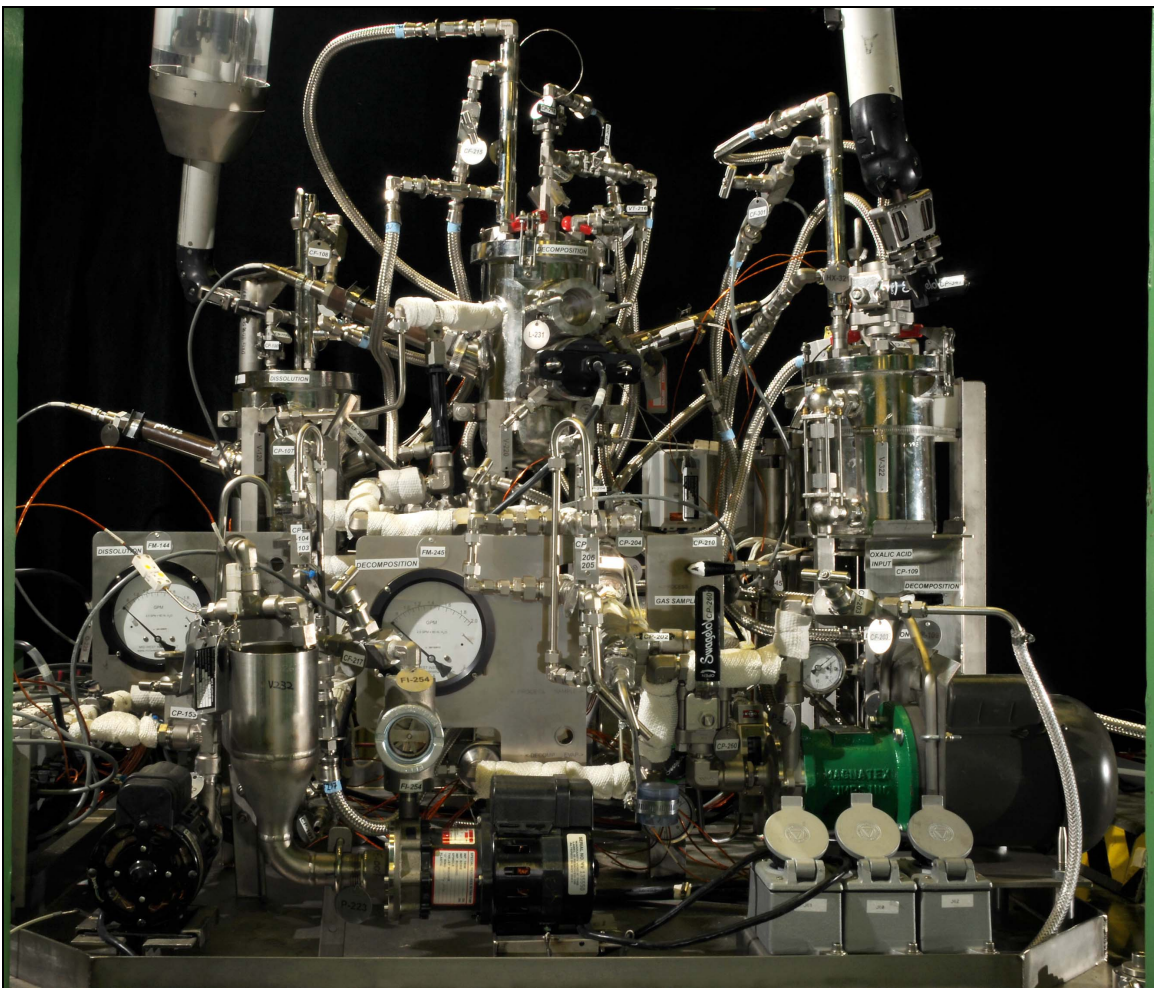


Figure 2-2: ECC RWT Equipment in Mockup Cell

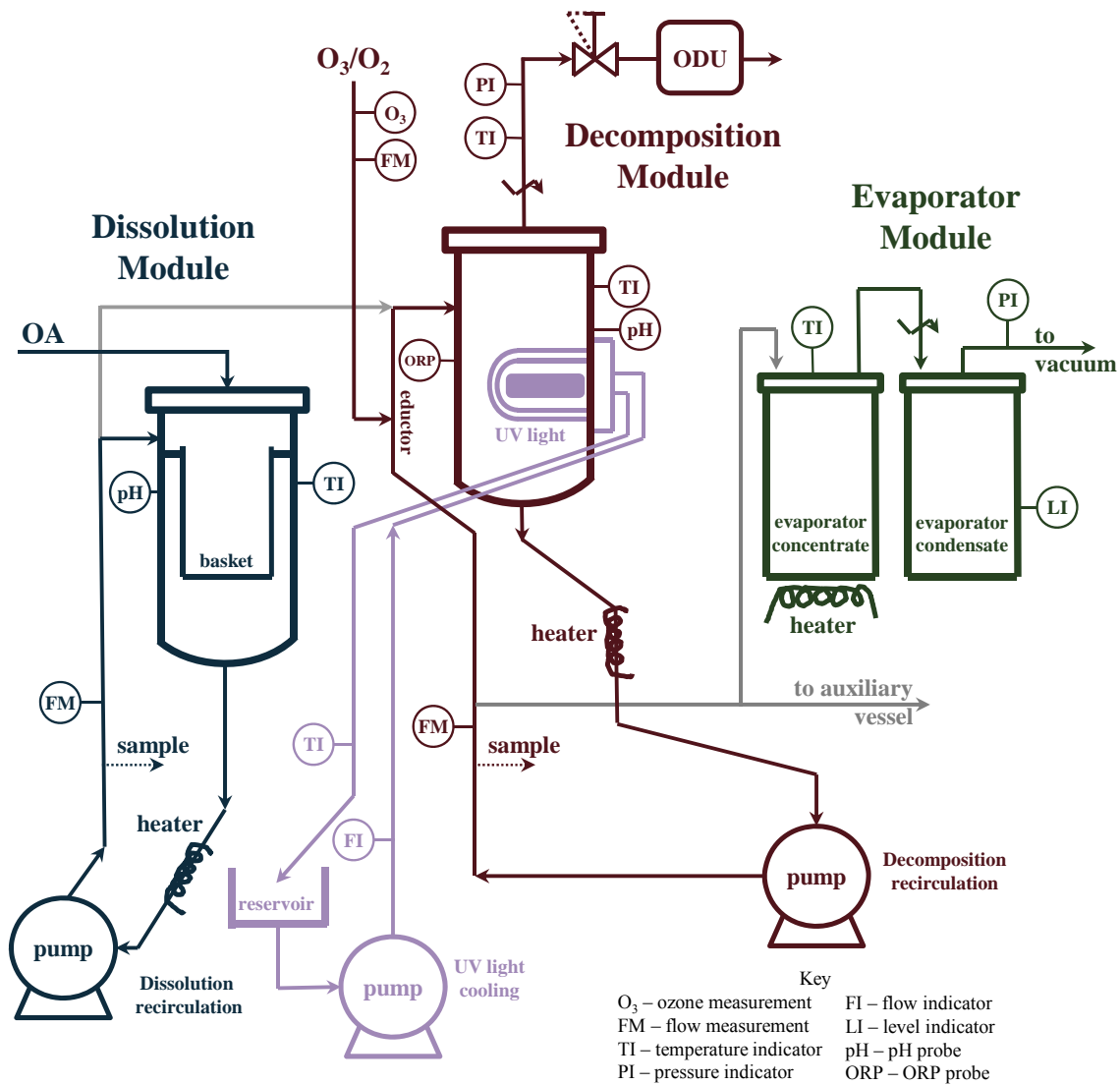


Figure 2-3: Schematic of ECC RWT Equipment Main Cells Skid (roughly related to the equipment elevation photograph of Figure 2-2)

The AREVA ECC RWT equipment (Figure 2-2, Figure 2-3 and Figure 2-4) contains three primary modules, one each for the dissolution of sludge from the treatment-tank (Dissolution Module), the ECC ozone/UV reactor for decomposition of the oxalate (Decomposition Module), and the removal of water for the recovery of dissolution fluid volume (Evaporator Module). The Dissolution Module contains a basket to hold the sludge, a heater to allow for dissolution at 70 °C, and a recirculation loop with a pump to allow for mixing, sampling, and transfer. The Decomposition Module contains a reaction loop where ozone contacts the fluid through an eductor and a vessel that allows for gas/liquid separation and the application of UV light via a medium pressure source. Similar to the Dissolution Module, the Decomposition Module contains a heater to allow for reaction at 70 °C and a recirculation loop with a pump to allow for mixing, sampling, and transfer. The Decomposition Module has the ability to be operated both with and

without the application of UV light to the fluid. The Decomposition Module includes a system for cleaning the UV light sheath with OA. The Evaporator is a vacuum evaporation system heated to 70 °C. The resultant evaporator concentrate is removed from the system manually via the removable evaporation chamber. Each module includes thermocouples to monitor the fluid temperature. The Dissolution Module additionally contains a probe to monitor the fluid pH. The Decomposition Module includes probes to monitor both the fluid pH and the oxidation/reduction potential (ORP). Offgas is sent through an Ozone Destruction Unit (ODU) to decompose ozone to below 0.1 ppmv before being vented to the Shielded Cell atmosphere.

An ozone generator was placed behind Shielded Cells B-Block for use in this study. Control modules for the ECC RWT equipment were accessible in the operations area in front of B-Block Cells 11 and 12. The modules outside of the cell are pictured in Figure 2-5.

Refer to the ECC RWT equipment Piping and Instrumentation Diagram for component labeling on the AREVA-supplied equipment.¹¹

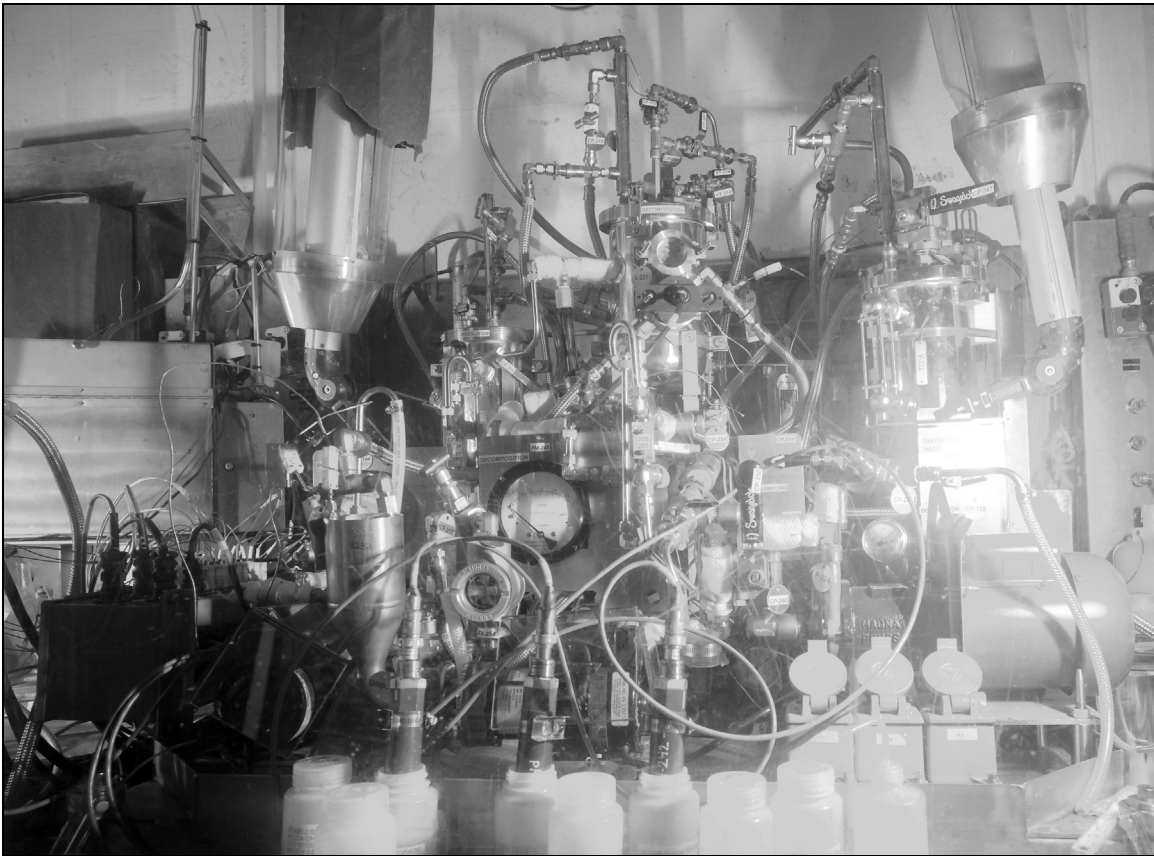


Figure 2-4: ECC RWT Equipment in SRNL High Level Shielded Cells, B-Block Cell 11

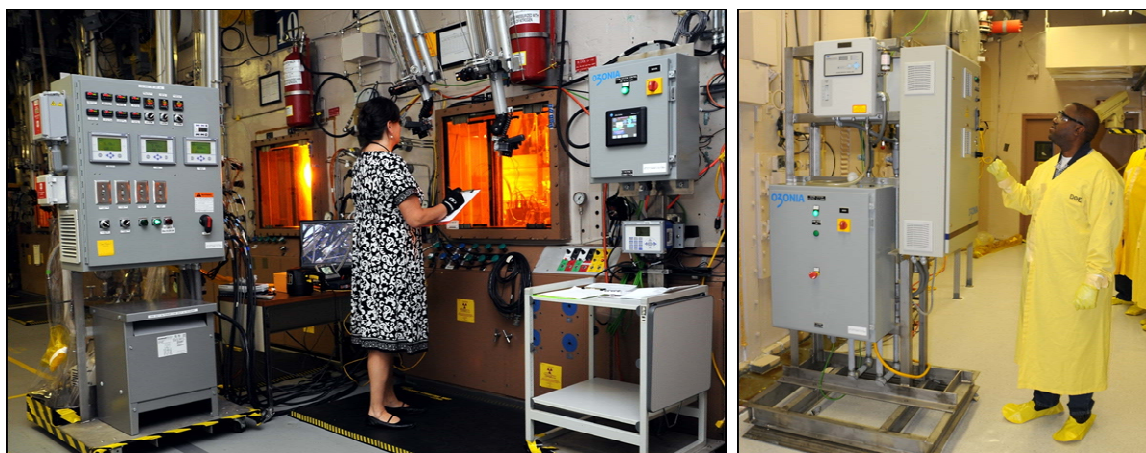


Figure 2-5: ECC RWT Equipment in the Shielded Cells Operating Area (left) and Ozone Generator behind B-Block Cells (right)

Additional ancillary equipment was provided by SRNL. Fabrication of the service plugs for shielded cell installation was performed by SRNL. SRNL used an auxiliary vessel (a 4L HDPE bottle with a magnetic stir bar on a stir plate) for splitting the material leaving the Decomposition Module so that representative portions could be stored in separate deposition bottles and other portions processed in the Evaporation Module. Fluids were able to be removed from the Dissolution and Decomposition Modules of the ECC RWT equipment using ports on the recirculation loops. A peristaltic pump with tygon tubing and stainless steel dip legs was used whenever material needed to be removed from the auxiliary vessels. SRNL also provided the deposition bottles, the equipment required for holding the bottles at deposition tank temperatures, and the equipment for sampling the deposition bottles. Portions of material prior to and after evaporation were stored at Deposition Tank conditions and sampled periodically. Storage bottles were PMP or Teflon for caustic storage conditions and glass for near neutral storage conditions. Bottles held at 50 °C were stored in a 20L water bath. Bottles held at 70 °C were stored in a drying oven. Temperature, pH, and ORP were periodically monitored in these Deposition Tank storage bottles using equipment procured by SRNL.

Based on an assumed 2000 gallon batch size for a field implementation of ECC, the scale of the Decomposition Module in the ECC RWT is between 1:2400 and 1:3000.

2.2.2 Treatment Tank Residual Sludge Testing for Rheology and Particle Size

Duplicate glass vessels were prepared by SRNL glassblowers for the purpose of conducting the OA dissolution batch contacts with both the Tank 5F and 12H sludge samples. The vessels were 140 mm in diameter and approximately 330 mm tall. The vessels were designed to fit into free-standing, metal-jacketed heating mantles. The tops of the vessels were fitted with: 1) a sample loading and pour spout with a plastic cap, 2) a calibrated thermocouple attached through an o-ring seal, and 3) a ground glass joint with an attached air-cooled condenser. The samples were heated in the vessels without agitation using a dual temperature controller with over-temperature protection. The thermocouples extended to within a couple of inches of the vessel bottoms. After initial temperature stabilization, the sample temperatures were recorded once or twice per day and the temperature was confirmed to be within five degrees of the target temperature (70 ± 5 °C) once

per night during off-shift hours. Once the temperature stabilized, all recorded temperatures during the dissolution batch contacts were 70 ± 1 °C. During heater operation, water condensation was never observed at a height greater than one inch above the bottom of the condensers. At the conclusion of each sludge/OA batch contact, the materials were removed from the vessels for rheology measurements and characterization.

2.2.3 RWT Equipment and Measurement Calibrations

Many instruments involved with the ECC RWT were utilized inside of the SRNL shielded cells in radioactive service. For such equipment, it is often not practical to perform post-testing calibration in accordance with the Measurement and Test Equipment (M&TE) program because the components will become too contaminated to be submitted to the SRS Standards Laboratory. For such equipment, pre-test calibration or calibration checking will be performed in accordance with the M&TE program. Prior to removal from the shielded cells for end-of-use disposal, some M&TE will be cross checked for measurement consistency.

Thermocouples that measure the temperatures inside of the three main process vessels (TI-140, TI-141, TI-240, TI-241, TI-340 and TI-341) underwent pre-test calibration checking and were placed in the M&TE program. Thermocouples measuring the temperature of Deposition Bottle storage conditions and the Treatment Tank dissolution underwent pre-test calibration checking and were placed in the M&TE program. Pressure gauges PI-247, PI-342 and PI-440 were calibrated and placed in the M&TE program. Pressure Relief Valves CP-444 and CP-209 were bench tested as required by SRS Pressure Safety. In-cell balances and weight sets were already part of and maintained within the M&TE program.

The pH meters used on the ECC RWT Equipment and used in the Deposition Bottles were checked using pH buffers over an appropriate range and were done in a manner consistent with program requirements for Analytical Measurement Systems. The ORP probes were checked by Light's Solution and Zobell's Solution in a manner consistent with program requirements for Analytical Measurement Systems. Calibration checks of pH and ORP probes contained in ECC RWT Equipment chambers were performed prior to and after each OA batch. The potential failure of probes during testing was known to be a risk. One pH probe failed during a run (Test 2, OA batch 1 Decomposition) and one pH probe failed during a storage period. Additionally, the behavior of the pH measurement during Test 2, OA batch 2 Dissolution was consistent with the probe not being submerged in the Dissolution Module fluid.

The liquid/slurry flow meters FM-144 and FM-245 provide important indications to the equipment operator so that the status of flow within the equipment is known but are not intended to provide useful measurement data during testing. Thus, FM-144 and FM-245 were not calibrated or placed in the M&TE program. The process high concentration ozone monitor O3-441 was not calibrated or placed in the M&TE program and was used in the condition as provided by AREVA. The other ambient ozone monitors (O3-443 and O3-230) were not recalibrated by SRNL because they were not used as the primary Industrial Health monitor for ozone gas.

2.3 Testing Procedure

2.3.1 *ECC Process Real Waste Testing*

Use of the ECC RWT equipment was governed by a Technical Reference procedure.¹² Table 2-3 contains the ECC RWT system inputs. Each test used either 48.1 grams of Tank 12H sludge or 65.1 grams of Tank 5F sludge based on the design volume of the ECC RWT equipment vessels and sample availability. Each OA batch required the fill level of the Dissolution Module to be at least 3.2 L. Dissolution was accomplished with a 2 wt% OA feed heated to 70 °C for an 8 hour period with 1 gallon per minute recirculation. Decomposition of OA in the Decomposition Module was initiated by heating each batch of dissolved sludge to 70 °C with a recirculation of 1.8 gallons per minute (if attainable). Once at temperature, 2 L per minute of a 5 wt% ozone in oxygen stream was fed into the system with a back pressure of 8 psi. Decomposition was continued until, through a combination of pH measurement and permanganate end-of-process testing, it was determined that the soluble oxalate concentration was less than 100 ppm.

Table 2-3: Nominal Operating Parameters and Targets for ECC Real Waste Testing

Feed	
Sludge (Tank 12H)	48.1 g
Sludge (Tank 5F)	65.1 g
OA	3.2 L/batch
Dissolution	
Feed OA Conc.	2 wt%
Temperature	70 °C
Recirculation Rate	1 gpm
Time	8 hr
Decomposition	
Temperature	70 °C
Pressure	8 psig
Recirculation Rate	1.8 gpm
Feed Gas Flow	2 L/min
Feed Gas O ₃ Conc.	5 wt%
Final Oxalate Conc.	< 100 mg/L
Evaporation	
Temperature	70 °C
Water Removal	≥ 85 %

2.3.2 Analytical Measurements

Table 2-4 lists the planned analyses for each type of sample taken during the ECC RWT. The primary analytes obtained by each method are also listed, although not all analytes were detectable in every sample and some additional analytes were attainable from some of the methods without additional effort. The different types of samples were taken on frequencies that have four different bases: per tank sludge sample, per sludge dissolution test, per OA batch, and per deposition bottle. The analysis plan was developed with the intent of providing enough information to provide a mass balance around key liquid and solid components throughout the process. However, complete closure of the mass balances remains unlikely due to holdup of material within the RWT equipment.

The initial feed materials were characterized for metals and radioactive components using two digestion methods and triplicate analysis. After each test, the residual insoluble solids in the dissolution module were removed and a portion of the solids were analyzed for metals and radioactive components.

Samples from the recirculation loops of the Dissolution and Decomposition Modules were either liquids or slurries depending on the stage at which sampling was performed. For some sample analysis the composition of the slurry was desired, but for most sample analysis the composition of the soluble components in the slurry was desired. To obtain analysis of the soluble components in the slurry, the slurry was filtered by passing the material through a 0.45 micron nylon syringe filter. Ideally, the smallest filter size possible should be used to minimize the impact of very small particles influencing the measurement. Initial attempts were made to use smaller pore size (0.1 micron) nylon filters for this testing. However, for the slurries encountered during the ECC RWT with Tank 5F and 12H feed sludges, the 0.1 micron filters proved impractical to use due to the great resistance of the material to flow through the membrane.

At the completion of Dissolution Module operation, samples were taken from the recirculation loop prior to transfer of material to the Decomposition Module. Dissolution Module samples were filtered and the filtrate analyzed in duplicate for anions (including oxalate) by ion chromatography (IC), metals (including iron and aluminum) by inductively coupled plasma-emissions spectroscopy (ICP-ES), other radiochemical (Pu, gamma, Sr-90) and ionic components (CO_3). Single acid dissolutions of the slurry samples were prepared and analyzed for metals and radiochemical components. Residual solids isolated from the Dissolution Module at the conclusion of each test were digested by the aqua regia method and analyzed for metals and radiochemical components.

During Decomposition Module operation, 3 to 5 intermediate samples were taken from the recirculation loop at intervals during OA decomposition. The intermediate Decomposition Module samples were filtered and analyzed by IC, ICP-ES, ICP-MS, and radiochemistry (Pu). Final Decomposition Module samples were filtered and analyzed in duplicate for a large suite of soluble anions, metals, and radiochemical components. Single acid dissolutions of the Decomposition Module slurry samples were prepared and analyzed for a subset of the metal and radiochemical components.

No samples were taken directly from the evaporator pot, though Deposition Tank Storage Condition 6 corresponds to unadjusted sludge after evaporation.

Table 2-4: ECC RWT Sampling and Analysis Plan

Analysis	Primary Analytes	Initial Feeds, Prior to Testing one to two dissolution methods, in triplicate	Residual Solids in Dissolution Module 1 per test, in duplicate	Dissolution Module Final Samples, Filtrate 1 per OA batch, in duplicate	Dissolution Module Final Samples, Slurry 1 per OA batch	Decomposition Module Intermediate Samples, Filtrate 3 to 5 per OA batch during decomposition	Decomposition Module Final Samples, Filtrate 1 per OA batch, in duplicate	Decomposition Module Final Samples, Slurry 1 per OA batch	Deposition Tank Intermediate Samples, Filtrate 2 per storage bottle	Deposition Tank Final Samples, Filtrate 1 per storage bottle in duplicate
ICP-ES	Al, Ca, Cr, Fe, Gd, Mn, Na, Ni, P, S, Si, Sr, Ti	X	X	X	X	X	X	X	X	X
ICP-MS	Th-232, U-235, Np-237, U-238	X	X	X	X	X	X	X	X	X
Pu TTA	Pu-238, Pu-239/240	X	X	X	X	X	X	X	X	X
CVAA	Hg	X	X	X	X		X	X		X
Sr-90	Sr-90	X	X	X	X		X	X		X
Gamma	Cs-137	X	X	X			X			X
Am/Cm	Am-241, Am-242m, Cm 242, Cm-244	X	X	X			X			X
IC Anions	C ₂ O ₄ , NO ₃ , NO ₂ , SO ₄ , Cl, F			X		X	X			X
TIC/TOC	CO ₃ , TOC			X			X			X
wet chem	Free OH or Free H			X			X			X
Pu-241	Pu-241	X								
Cs-rem gamma	Co-60, Sn-126, Eu-154	X								

Each test had nine corresponding Deposition Tank storage bottles. The storage bottles were mixed prior to sampling but were not mixed continuously. Three samples were taken from each of the storage bottles, filtered with a 0.45 micron filter, and the filtrate prepared for analysis. Intermediate samples were filtered, acidified, and analyzed by IC, ICP-ES, ICP-MS, and radiochemistry (Pu). The sample taken after approximately two to three weeks of storage was filtered, acidified, and analyzed for the entire chemical and radiological suite. The bulk slurry was analyzed for a subset of the metal and radiochemical components. A subset of decomposition bottles were sampled after an additional two weeks of equilibration, with the filtrate analyzed by the suite used for the intermediate deposition bottle samples.

All isotopes of uranium and plutonium were not necessarily tracked through all stages of the process because some isotopes of these elements were difficult and costly to determine in the soluble fraction due to the low concentrations. However, since the isotopic ratios have been established for the feed materials through the initial characterizations, estimates of the complete isotopics can be calculated from the results provided for other Pu and U isotopes.

For the majority of the analytical methods of Table 2-4, 1-sigma uncertainties are nominally 10% or less. Sample preparations were performed in a manner as not to add significantly to the overall 10% uncertainty of the analytical methods. Some radiochemical preparations followed by counting methods had 1-sigma uncertainties greater than 10% when activity was low (approaching the detection limit):

- Am/Cm measurements in the Deposition Tank samples had uncertainties $\gg 10\%$.
- Pu-238 and Pu-239/240 measurements in the Test 2 Deposition Tank samples had uncertainties $\gg 10\%$.

Calorimetric spot tests were conducted in the shielded cells near the end of each decomposition test to confirm that the oxalate anion levels in the spent acid solution had decreased to the target level of 100 ppm. Tests were conducted using small glass vials containing 150 mg of $\text{MnSO}_4 \cdot \text{H}_2\text{O}$. A sample of solution was isolated from the decomposition module and a 5-mL portion was filtered through a 0.45 μm syringe filter directly into the vial. Then 1 mL of 50 wt% (9.1 M) sulfuric acid was transferred into the vial and the vial was capped and shaken to promote dissolution of the manganese sulfate solids. A manual pipet was used to transfer 0.2 mL of 0.1 M potassium permanganate solution into the vial and the vial was capped and shaken again. The color of the solution was observed and recorded after two minutes. A purple or pink colored solution was indication that the oxalate concentration was at or below the target concentration. Note that the amount of permanganate added to the solution was in excess of the amount required to react with 100 mg/L of oxalate anion, but previous spot tests had confirmed that this was the excess required to observe the color through the cell window.

2.3.3 Rheology, Settling Rate, and Particle Size Measurements

Rheology, particle size and/or settling behavior were investigated at key stages in the process. This information aids in the understanding of the removal of undissolved solids from the treatment tank, the flow of solids through the ECC process, and the effects of newly precipitated solids on downstream processes. The key process stages and the applicable test series are outlined in the schematic of Figure 2-6.

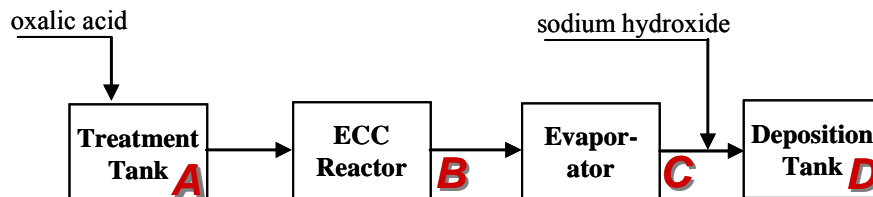


Figure 2-6: Key process stages for rheology, particle size and/or settling behavior testing

- A: In the Treatment Tank
Purpose: Provide input on the impact of sludge dissolution on the ability of the slurry pumps to adequately mix the tank contents. Provide evidence that the undissolved solids during and after ECC are easier to suspend and remain suspended as compared to the original sludge heel.
Measurements: Rheology of residual undissolved solids at concentrated solids levels, particle size distribution of residual undissolved solids.
Methodology: In order to determine the rheology of the residual Treatment Tank heel, separate sludge dissolution testing was performed outside of the AREVA-supplied ECC RWT Equipment dissolution module. The results are contained in this report.
- B: After ECC Reactor, prior to Evaporator or pH adjustment
Purpose: Help determine the flow characteristics in the recirculation loop following oxalate decomposition.
Measurement: Rheology at process-related solids concentration, particle size distribution
Methodology: At the end of solubility testing, slurry from deposition bottles for Storage Condition 3 (see Table 2-2) were measured with the rheometer and submitted for Microtrac particle size analysis.
- C: After Evaporator, prior to pH adjustment
Purpose: In the full scale ECC process, the concentrated material in the evaporator bottoms must be pumped to the deposition tank.
Measurement: rheology at process-related solids concentration, particle size distribution
Methodology: At the end of the solubility testing, slurries from deposition bottles for Storage Condition 6 (see Table 2-2) were measured with the rheometer and submitted for Microtrac particle size analysis.
- D. In the Deposition Tank at nominal conditions
Purpose: Supernate will be decanted from the deposition tank to and processed in a HLW evaporator system. Settling information is needed to help justify the feasibility of decanting supernate from the deposition tank during periods of ECC processing.
Measurement: settling time, particle size distribution
Methodology. Storage Condition 2 is the applicable condition for F-Area deposition bottles and Storage Condition 5 is the applicable condition for H-Area deposition bottles (see Table 2-2). Small samples were collected from the applicable deposition bottles and submitted for Microtrac particle size analysis. At the completion of the solubility testing, the applicable storage bottles were removed from the 50 °C water bath, agitated, and held quiescent to allow the solids to settle. Periodic observations of solids settling (level, behavior, etc.) were made and recorded until the supernatant liquid had clarified.

2.3.3.1 Treatment Tank Rheology Testing

Rheology testing was conducted on samples of the Tank 5F and 12H sludge which had previously been dried at 50 °C. These samples are referred to as “Treatment Tank” samples, since they are believed to be representative of the materials in the front portion of the ECC process which are located in the tank to be treated for sludge heel removal. Sub-samples of each sludge sample were initially pre-wetted with water to produce 50 weight percent slurries and allowed to stand overnight to promote hydration of the solid phase. Sludge/OA slurries were then prepared for baseline rheology measurements at initial sludge solids loadings of 13 and 26 weight percent.

Corrections were made to account for the initial water added for hydration to produce final slurries containing 0.224 M oxalic acid (2 wt% OA).

Baseline rheology measurements were conducted on these slurries at two solids concentrations and three temperatures. It was observed during rheological evaluations that significant portions of the initial slurries settled quickly. This sample characteristic was particularly pronounced for the Tank 5F samples where it appeared that most of the sample settled within five minutes. The rapid settling rate prevented the measurement of the rheological properties of the fully suspended sample by standard methods. Therefore, the rheology data collected for the Tank 5F sample is representative of only the small fraction of the solid materials that remained suspended on the measurement timescale. The suspendable solids for these samples appeared to be so low that the rheological properties measured for the Tank 5F samples were expected to be similar to those of pure OA or spent acid generated in the ECC process. Portions of the Tank 12H slurry used for baseline rheology measurements also settled during initial evaluations as exhibited by the presence of significant amounts of dark solids in the bottom of the rheology cup at the conclusion of each measurement. The Tank 12H rheology sample appeared to contain two types of solids with very different settling rates. The lighter colored solids in this sample remained well suspended over the rheology measurement timescale. The Tank 12H samples were observed to change over the first few days of acid contact. After 4 days, most of the concentrated Tank 12H slurry remained suspended throughout the analysis with few settled solids being observed.

Sub-samples of selected Tank 5F and 12H slurries from the baseline rheology testing were collected to analyze the samples for weight percent solids and determine the amount of “suspendable” solids in the samples. Collection of approximately 3 mL slurry samples was accomplished using a disposable plastic pipet approximately 1 minute after agitating the samples in the same manner that the samples were agitated prior to rheology measurements.

After the completion of the baseline testing, the slurries used for rheology measurements were augmented to give a total of 62.9 g of Tank 5F solids and 45.6 g of Tank 12H solids. The resulting slurries were transferred to the batch contact dissolution vessels. Additional 2 wt% OA was added to the vessels to give a total of 2.9 L for the Tank 5F slurry and 2.3 L for the Tank 12H slurry. The resulting acid:sludge mass ratios for each sample were approximately 50:1. The dissolution vessels were then heated to 70 °C and held at that temperature for seven days. At the conclusion of the first batch contact period, the solution in the Tank 5F slurry was yellow-green in color while the solution in the Tank 12H slurry was slightly yellow. Most of the solution was subsequently removed from the settled solids in each vessel using a peristaltic pump. The solids were then suspended in the remaining solution and the resulting slurries were removed from the vessels for midpoint rheology measurements.

Samples were prepared for rheology measurements by further reducing the volume of spent OA in the slurries. As was observed for the baseline measurements, most of the Tank 5F solids settled quickly, such that meaningful rheology measurements were not practical. As a result, measurements were only conducted at one solids loading at 30 °C for this sample. The Tank 12F slurry was easily suspended with few rapidly settling particles being observed. Rheology measurements were conducted for this sample at two solids loadings and three temperatures. Sub-samples of the Tank 12H slurry were collected as described for the baseline samples with the goal of determining the “suspendable” weight percent solids levels. Samples of the filtered spent OA were also collected in order to determine the weight percent of dissolved solids. In this case, the samples were filtered through 0.45 µm Nylon filter units.

After the completion of the midpoint measurements and sampling, all of the remaining solids used for rheology measurements were returned to the dissolution vessels along with fresh 2 wt% OA totaling 2.9 L for the Tank 5F sample and 2.3 L for the Tank 12H sample (approximately 50:1 acid:sludge based on the original sludge mass). The vessels were then heated to 70 °C and held at that temperature for three days. At the conclusion of the second batch contact period (endpoint), the solutions above the solids were lighter in color than the solutions isolated after the first OA contact (midpoint). Most of the solution was subsequently removed from the settled solids in each vessel using a peristaltic pump and the damp solids were isolated for endpoint rheology analysis.

As was observed for the baseline and midpoint rheology samples, most of the Tank 5F solids settled within minutes. As a result, rheology testing was only conducted at one solids loading at 30 °C. The Tank 12H slurry was easily suspended and suitable for analysis and rheology data was collected for this sample at two solids loadings and three temperatures. Limitations on the minimum volume of material needed for rheology measurements (~50 mL) and the small amount of solids remaining for this sample prohibited the preparation of Tank 12H slurries containing greater than 5 wt% total solids. Slurry sub-samples (filtered and unfiltered) were collected as described for the baseline and midpoint samples. Single sub-samples of the baseline, midpoint, and endpoint rheology materials were also collected for particle size analysis. See Section 2.3.4 for details.

Rheology measurements were also conducted on four deposition tank samples generated during other ECC testing. The samples included Tank 5F and 12H product materials generated during oxalate destruction tests. These samples are referred to as “Deposition” samples because they are believed to be representative of ECC process product materials deposited in the receipt tank. The samples included materials which had been evaporated and materials which were not exposed to evaporation. Rheology measurements were conducted on all four samples at the as-received solids loading levels and at two temperatures.

2.3.3.2 Weight Percent Solids Measurements

Weight percent solids measurements were conducted by drying samples in a drying oven at 112 °C until no additional weight loss was observed. The weight percent of dissolved solids is defined as the weight of dissolved solids divided by the weight of initial filtered supernatant (x100). For these measurements, the supernatant was filtered using a 0.45 µm Nylon filter. The weight percent of total solids is defined as the total weight of solids (dissolved and insoluble) divided by the weight of initial slurry (x100). Weight percent dissolved and total solids measurements were conducted on single or on duplicate samples. The collection of replicate samples for analysis was prevented in some cases by sample size limitations.

2.3.3.3 Rheology Measurement Equipment, Methodology, and Data Analysis

The rheological properties of the samples were determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. Heating/cooling of the cup/sample/bob is accomplished through a jacket that surrounds the sample cup. The shear stress is determined from the torque measurement and is independent of the rheological properties. Conditions that impact the measured torque are: slip (material does not properly adhere to the

rotor or cup), phase separation (buildup of liquid layer on rotor), sedimentation (particles settling out of the shearing zone), homogeneous sample (void of air), lack of sample (gap not filled), excess sample (primarily impacts rheologically thin fluids), completely filling up the void below the bob (air buffer that is now filled with fluid) and Taylor vortices. The first five items yield lower stresses and the last three add additional stresses. The shear rate is geometrically determined using the equations of change (continuity and motion) and is that for a Newtonian fluid. This approach assumes that the flow field is fully developed and the flow is laminar. The shear rate can be calculated for non-Newtonian fluid using the measured data by fitting this data to the rheological model or corrected as recommended by Darby⁸. In either case, for shear thinning, non-Newtonian fluids typical of SRS sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a thinner fluid. Flow curve corrections will not be performed in this task, which results in slightly high measurements of fluid viscosity.

The shape, dimensions, and geometric constants for the MV I rotor used for these measurements are provided in Table 1. Based on the expected particle size of the materials in this investigation, the MV 1 rotor was considered most appropriate. Prior to performing the measurements, the rotors and cups were inspected for physical damage. The torque/speed sensors and temperature bath were verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard, using the MV I rotor. The resulting flow curve was then fitted assuming a Newtonian fluid and the calculated viscosity was required to be within $\pm 10\%$ of the reported NIST viscosity at a given temperature for the system to be considered functionally operable. A N10 oil standard was used to verify system operability prior to the sludge measurements.

The flow curves for the sludge were fitted to the down curves using the Bingham Plastic rheological model, Equation q, where τ is the measured stress (Pa), τ_0 is the Bingham Plastic yield stress (Pa), μ_∞ is the plastic viscosity (Pa-sec), and $\dot{\gamma}$ is the measured shear rate (sec^{-1}).

$$\tau = \tau_0 + \mu_\infty \dot{\gamma} \quad (1)$$

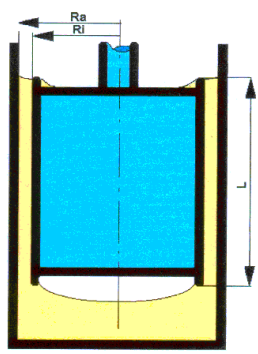
All rheology measurements were conducted in duplicate. Between each rheology measurement, the sample cup was lowered, the sample was transferred to a bottle, and the sample cup was inspected to evaluate whether the solids remained suspended during the measurement. The sample bottle was then capped and shaken to promote agitation. The sample was immediately transferred back to the rheology cup and placed in the instrument for analysis. Loading the sample into the instrument typically took approximately four minutes and eleven minutes was required to complete each rheological measurement. Therefore, slurry samples for which the particles settled in <15 minutes were not considered suitable for rheology measurements.

The temperature of the samples was not measured directly during rheological measurements. Rather, the temperature was monitored and controlled using two thermocouples immersed within the heating fluid flow loop. One thermocouple was located in the flow loop at the heater location before the rotor and cup and the second thermocouple was located in the flow loop after the rotor and cup. This second thermocouple was believed to be most representative of the sample temperature and was used for temperature monitoring and control. During all analysis the temperature readout from the second thermocouple was typically within ± 2 °C of the target value, but occasionally the deviation from the target was ± 3 °C. Due to the settling properties of the samples, it was not possible to allow as much time for temperature equilibration of the system as recommended. Rheology measurements were typically conducted within approximately one

minute after the sample was loaded. Therefore, the actual sample temperatures may be slightly lower than the target values during the high temperature measurements. For each given sample and solids loading, all measurements were conducted in sequence and the time required for lowering the rotor and mixing the sample was kept short such that sample cooling during this time period was minimized. Tank 5F and 12H treatment tank samples were analyzed at 30, 40, and 50 °C, while the deposition tank samples were only analyzed at 30 and 45 °C.

All viscosity results are reported in centipoise (cP) and all yield stress values are reported in pascals (Pa). Negative yield stress values were calculated for numerous samples analyzed due to difficulties in accurately adjusting the instrumental zero set point. This issue is apparent in the data plots for many thin samples in Appendix B, where the shear stress values on the down curve reached zero at a shear rate near 100 s⁻¹. This is believed to be primarily an issue for the thin samples, since the shear stress values for thicker samples typically returned to the initial zero point.

Table 2-5: MV I Rotor Specifications and Flow Curve Program

Rotor Design	Dimensions and Flow Curve Program	
	Rotor Type	MV I
	Rotor radius - R_i (mm)	20.04
	Cup Radius - R_a (mm)	21.0
	Height of rotor - L (mm)	60
	Sample Volume (cm ³) minimum	40
	A factor (Pa/%torque)	3.22
	M factor (s ⁻¹ /RPM)	11.7
	Shear rate range (s ⁻¹)	0 – 600
	Ramp up time (min)	5
	Hold time (min)	1
	Ramp down time (min)	5

2.3.4 Particle Size and Settling Rate Measurements

Particle size distribution determination was accomplished by removing small amounts (approximately 0.03 grams) of solids from the shielded cells for measurement by a Microtrac particle size analyzer. The solid samples were suspended in liquids with similar characteristics to their slurry conditions. The dilution fluids were 0.00625 M nitric acid for Treatment Tank samples; 0.02 M sodium nitrate for post ECC Reactor fluids prior to pH adjustment; and 0.5 M sodium hydroxide with 0.02 M sodium nitrate for caustic Deposition Tank fluids.

Simple settling tests were also performed in the shielded cells. Applicable deposition bottles were agitated and then held quiescent. The appearance of the liquid and the sludge level were observed periodically until the supernatant liquid had clarified.

3.0 Results and Discussion

3.1 Initial Sludge Preparation and Characterization

The ECC RWT used two actual-waste sludge materials as process feeds, Tank 5F sludge and Tank 12H sludge. Additional actual-waste sludge from Tank 51H was used as deposition tank heel material.

The sample used to represent F-Area PUREX sludge was a portion of Tank 5F sample FTF-05-06-55. This sample was of the Tank 5F sludge material prior to Tank 5F chemical cleaning and had been characterized and used in previous testing.¹³ Prior to use, the sample material was washed with 0.01 M NaOH to remove salts and dried at 50 °C. Subsamples of the washed and dried sludge were dissolved by aqua regia and peroxide fusion digestion methods and analyzed for chemical and radioactive components. Table 3-1 and Table 3-2 contain the analytical results for the Tank 5F sample used in the testing. The Tank 5F material was stored in its mostly dry state until used in ECC testing.

The sample used to represent H-Area HM sludge was a combination of several samples from Tank 12H taken from 2008 through 2010. Through waste removal activities, it was noted that the Tank 12H sludge was layered, with uranium enrichment and thorium content varying as layers were encountered. Because this Tank 12H material is a composite of several samples taken at different stages of waste removal, it is a blend of layers and does not represent any individual layer. Similar to the Tank 5F material, the Tank 12H samples were washed with 0.01 M NaOH to reduce the hydroxide concentration and remove other salts and dried at 50 °C. Table 3-3 and Table 3-4 contain the analytical results for Tank 12H sludge material prepared for ECC testing. The Tank 12H material was stored in its mostly dry state until used in ECC testing.

Additionally, a small amount of dilute sludge slurry was needed as a heel for the deposition bottles. For this heel material, we used sludge solids and sludge supernate from Tank 51H combined with additional simulated sludge supernate. The actual-waste sludge sample used was HTF-51-07-121. A previous characterization report exists for this sample and this material was not characterized again prior to use.¹⁴ The purpose of the solids in this material is to provide metal compounds to potentially be affected by the addition of ECC product material. The purpose of the liquid portion of this material is to maintain a pH appropriate to the Deposition Tank. As the sludge sample material was taken at the end of an aluminum dissolution campaign, the sludge supernate contained relatively high hydroxide content, more akin to a salt supernate. The deposition tank heel mixture contained ~4% concentrated Tank 51H slurry, ~25% clarified Tank 51H supernate, and the balance (~71%) 4 M NaOH.

From the characterization, the primary component of the Tank 5F sludge is iron while the primary component of the Tank 12H sludge is aluminum. Both sludge samples contain significant iron, aluminum, manganese, sodium, and uranium. Additionally, the Tank 5F sludge contains significant nickel while the Tank 12H sludge contains significant mercury and thorium. Both samples contain high activities of Sr-90, but there are differences in the radioactive isotope profiles between the two materials with respect to Pu-238, Pu-239, uranium enrichment, and other isotopes.

Table 3-1: Characterization of Tank 5F Sludge Sample Available for ECC Actual-Waste Testing

analyte	method	units	aqua regia average	per. fusion average	overall average	st. dev.	g / g Fe	g / g Al
Ag	ICP-ES	mg/kg	<1.55E+02	<3.89E+02	<2.72E+02	--	--	--
Al	ICP-ES	mg/kg	1.49E+04	1.78E+04	1.64E+04	2.0E+03	4.5E-02	1.0E+00
B	ICP-ES	mg/kg	<8.02E+01	<3.40E+02	<2.10E+02	--	--	--
Ba	ICP-ES	mg/kg	1.57E+03	1.68E+03	1.63E+03	1.2E+02	4.5E-03	9.9E-02
Be	ICP-ES	mg/kg	<5.07E+00	<1.05E+01	<7.80E+00	--	--	--
Ca	ICP-ES	mg/kg	3.12E+03	6.01E+03	4.57E+03	1.60E+03	1.3E-02	2.8E-01
Cd	ICP-ES	mg/kg	8.00E+01	<1.92E+02	8.00E+01	7.9E+00	2.2E-04	4.9E-03
Ce	ICP-ES	mg/kg	2.03E+03	2.55E+03	2.29E+03	3.2E+02	6.3E-03	1.4E-01
Co	ICP-ES	mg/kg	2.06E+02	2.52E+02	2.29E+02	3.0E+01	6.3E-04	1.4E-02
Cr	ICP-ES	mg/kg	4.98E+02	4.56E+02	4.77E+02	5.6E+01	1.3E-03	2.9E-02
Cu	ICP-ES	mg/kg	4.45E+02	4.78E+02	4.62E+02	3.4E+01	1.3E-03	2.8E-02
Fe	ICP-ES	mg/kg	3.65E+05	3.59E+05	3.62E+05	2.1E+04	1.0E+00	2.2E+01
Gd	ICP-ES	mg/kg	<2.00E+02	<1.93E+02	<1.96E+02	--	--	--
K	ICP-ES	mg/kg	6.62E+02	<3.71E+03	6.62E+02	9.7E+01	1.8E-03	4.1E-02
La	ICP-ES	mg/kg	9.49E+02	1.05E+03	9.98E+02	8.4E+01	2.8E-03	6.1E-02
Li	ICP-ES	mg/kg	4.00E+02	4.75E+02	4.38E+02	5.0E+01	1.2E-03	2.7E-02
Mg	ICP-ES	mg/kg	4.78E+02	6.97E+02	5.88E+02	1.30E+02	1.6E-03	3.6E-02
Mn	ICP-ES	mg/kg	6.39E+04	6.96E+04	6.68E+04	5.2E+03	1.8E-01	4.1E+00
Mo	ICP-ES	mg/kg	7.22E+01	<2.60E+02	1.66E+02	1.04E+02	4.6E-04	1.0E-02
Na	ICP-ES	mg/kg	3.37E+04	--	3.37E+04	3.6E+03	9.3E-02	2.1E+00
Ni	ICP-ES	mg/kg	3.38E+04	3.82E+04	3.60E+04	3.4E+03	9.9E-02	2.2E+00
P	ICP-ES	mg/kg	<3.08E+02	<7.71E+02	<5.39E+02	--	--	--
Pb	ICP-ES	mg/kg	3.89E+02	<6.79E+02	3.89E+02	1.98E+02	1.1E-03	2.4E-02
S	ICP-ES	mg/kg	<2.17E+03	<6.81E+03	<4.49E+03	--	--	--
Sb	ICP-ES	mg/kg	<4.99E+02	<6.25E+02	<5.62E+02	--	--	--
Si	ICP-ES	mg/kg	5.30E+03	1.08E+04	8.07E+03	3.05E+03	2.2E-02	4.9E-01
Sn	ICP-ES	mg/kg	<4.98E+02	<7.79E+02	<6.39E+02	--	--	--
Sr	ICP-ES	mg/kg	7.48E+02	8.12E+02	7.80E+02	6.0E+01	2.2E-03	4.8E-02
Th	ICP-ES	mg/kg	<9.22E+01	<8.09E+02	<4.51E+02	--	1.2E-03	2.8E-02
Ti	ICP-ES	mg/kg	1.86E+02	2.11E+02	1.99E+02	1.9E+01	5.5E-04	1.2E-02
U	ICP-ES	mg/kg	7.26E+04	8.50E+04	7.88E+04	8.4E+03	2.2E-01	4.8E+00
V	ICP-ES	mg/kg	<8.70E+00	<4.72E+01	<2.80E+01	--	--	--
Zn	ICP-ES	mg/kg	2.26E+02	2.64E+02	2.45E+02	2.6E+01	6.8E-04	1.5E-02
Zr	ICP-ES	mg/kg	3.49E+03	--	3.49E+03	3.5E+02	9.6E-03	2.1E-01
Co	ICP-MS	mg/kg	2.08E+02	2.34E+02	2.21E+02	1.4E+01	6.1E-04	1.4E-02
Hg	CVAA	mg/kg	6.95E+02	--	6.95E+02	8.7E+01	1.9E-03	4.3E-02
Pb	ICP-MS	mg/kg	3.58E+02	3.90E+02	3.74E+02	2.6E+01	1.0E-03	2.3E-02
Th-232	ICP-MS	mg/kg	<3.19E+01	1.04E+01	1.04E+01	7E-01	2.9E-05	6.4E-04
U-234	ICP-MS	mg/kg	<4.53E+01	6.06E+00	6.06E+00	4.2E-01	1.7E-05	3.7E-04
U-235	ICP-MS	mg/kg	4.42E+02	5.39E+02	4.90E+02	6.1E+01	1.4E-03	3.0E-02
U-236	ICP-MS	mg/kg	2.53E+01	2.54E+01	2.54E+01	3.9E+00	7.0E-05	1.6E-03
Np-237	ICP-MS	mg/kg	4.90E+01	5.32E+01	5.11E+01	3.0E+00	1.4E-04	3.1E-03
U-238	ICP-MS	mg/kg	7.22E+04	8.58E+04	7.90E+04	8.7E+03	2.2E-01	4.8E+00
Pu-239	ICP-MS	mg/kg	9.81E+01	1.07E+02	1.03E+02	1.2E+01	2.8E-04	6.3E-03
Pu-240	ICP-MS	mg/kg	<2.72E+01	8.69E+00	8.69E+00	1.33E+00	2.4E-05	5.3E-04
mass 241	ICP-MS	mg/kg	<2.72E+01	1.41E+01	1.41E+01	1.9E+00	3.9E-05	8.6E-04

Table 3-2: Characterization of Tank 5F Sludge Sample Available for ECC Actual-Waste Testing, continued

analyte	method	units	per. fusion average	st. dev.	mCi / g Fe	mCi / g Al
Pu-238	PuTTA	dpm/g	4.04E+06	2.3E+05	5.0E-03	1.1E-01
Pu-239/240	PuTTA	dpm/g	2.04E+07	1.5E+06	2.5E-02	5.6E-01
Pu-241	Pu-241	dpm/g	<2.09E+07	--	--	--
Co-60	gamma	dpm/g	1.96E+07	1.9E+06	2.4E-02	5.4E-01
Cs-137	gamma	dpm/g	2.39E+09	1.3E+08	3.0E+00	6.6E+01
Eu-154	gamma	dpm/g	4.52E+07	2.4E+06	5.6E-02	1.2E+00
Eu-155	gamma	dpm/g	<6.26E+06	--	--	--
Np-237	gamma	dpm/g	<1.04E+07	--	--	--
Am-241	gamma	dpm/g	9.69E+07	5.5E+06	1.2E-01	2.7E+00
Co-60	Cs-rem γ	dpm/g	1.74E+07	1.0E+06	2.2E-02	4.8E-01
Eu-154	Cs-rem γ	dpm/g	3.97E+07	9E+05	4.9E-02	1.1E+00
Eu-155	Cs-rem γ	dpm/g	<6.42E+06	--	--	--
Am-241	Cs-rem γ	dpm/g	8.47E+07	2.9E+06	1.1E-01	2.3E+00
Sr-90	Sr-90	dpm/g	7.81E+10	1.55E+10	9.7E+01	2.2E+03
Am-241	Am/Cm	dpm/g	8.47E+07	2.9E+06	1.1E-01	2.3E+00
Am-243	Am/Cm	dpm/g	5.79E+05	4.7E+04	7.2E-04	1.6E-02
Am-242m	Am/Cm	dpm/g	1.89E+05	4.4E+04	2.4E-04	5.2E-03
Cm-243	Am/Cm	dpm/g	<1.50E+06	--	--	--
Cm-245	Am/Cm	dpm/g	<6.30E+05	--	--	--
Cm-247	Am/Cm	dpm/g	<3.11E+05	--	--	--
Cf-249	Am/Cm	dpm/g	<3.18E+05	--	--	--
Cf-251	Am/Cm	dpm/g	<2.29E+05	--	--	--
Cm-242	Am/Cm	dpm/g	1.57E+05	3.7E+04	1.9E-04	4.3E-03
Cm-244	Am/Cm	dpm/g	5.29E+06	4.1E+05	6.6E-03	1.5E-01

Table 3-3: Characterization of Tank 12H Sludge Sample Available for ECC Actual-Waste Testing

analyte	method	units	aqua regia average	per. fusion average	overall average	st. dev.	g / g Fe	g / g Al
Ag	ICP-ES	mg/kg	8.96E+01	<2.05E+02	8.96E+01	5.4E+00	2.0E-03	3.4E-04
Al	ICP-ES	mg/kg	2.68E+05	2.56E+05	2.62E+05	1.2E+04	5.8E+00	1.0E+00
B	ICP-ES	mg/kg	<1.31E+02	<1.68E+02	<1.50E+02	--	--	--
Ba	ICP-ES	mg/kg	5.24E+02	4.94E+02	5.09E+02	2.6E+01	1.1E-02	1.9E-03
Be	ICP-ES	mg/kg	<8.93E+00	<2.10E+01	<1.50E+01	--	--	--
Ca	ICP-ES	mg/kg	2.46E+03	4.12E+03	3.29E+03	9.44E+02	7.3E-02	1.3E-02
Cd	ICP-ES	mg/kg	<6.25E+01	<1.22E+02	<9.24E+01	--	--	--
Ce	ICP-ES	mg/kg	8.39E+02	<1.25E+03	8.39E+02	2.0E+01	1.9E-02	3.2E-03
Co	ICP-ES	mg/kg	<6.32E+01	<8.83E+01	<7.58E+01	--	--	--
Cr	ICP-ES	mg/kg	2.29E+02	2.32E+02	2.31E+02	9E+00	5.1E-03	8.8E-04
Cu	ICP-ES	mg/kg	5.50E+02	5.52E+02	5.51E+02	2.5E+01	1.2E-02	2.1E-03
Fe	ICP-ES	mg/kg	4.55E+04	4.49E+04	4.52E+04	1.8E+03	1.0E+00	1.7E-01
Gd	ICP-ES	mg/kg	1.65E+02	<2.02E+02	1.65E+02	2E+00	3.6E-03	6.3E-04
K	ICP-ES	mg/kg	<1.49E+03	3.27E+03	3.27E+03	1.3E+02	7.2E-02	1.2E-02
La	ICP-ES	mg/kg	4.60E+02	3.40E+02	4.00E+02	7.6E+01	8.9E-03	1.5E-03
Li	ICP-ES	mg/kg	1.12E+02	<1.65E+02	1.12E+02	7E+00	2.5E-03	4.3E-04
Mg	ICP-ES	mg/kg	1.18E+03	1.09E+03	1.14E+03	8E+01	2.5E-02	4.3E-03
Mn	ICP-ES	mg/kg	2.94E+04	2.69E+04	2.81E+04	1.6E+03	6.2E-01	1.1E-01
Mo	ICP-ES	mg/kg	<9.31E+01	<2.73E+02	<1.83E+02	--	--	--
Na	ICP-ES	mg/kg	4.23E+04	--	4.23E+04	3.1E+03	9.4E-01	1.6E-01
Ni	ICP-ES	mg/kg	4.62E+03	4.62E+03	4.62E+03	1.6E+02	1.0E-01	1.8E-02
P	ICP-ES	mg/kg	7.58E+02	<8.11E+02	7.58E+02	5.2E+01	1.7E-02	2.9E-03
Pb	ICP-ES	mg/kg	<2.72E+02	<6.98E+02	<4.85E+02	--	--	--
S	ICP-ES	mg/kg	<1.39E+03	<1.43E+04	<7.86E+03	--	--	--
Sb	ICP-ES	mg/kg	<1.54E+02	<6.57E+02	<4.06E+02	--	--	--
Si	ICP-ES	mg/kg	2.59E+03	2.72E+03	2.66E+03	1.08E+02	5.9E-02	1.0E-02
Sn	ICP-ES	mg/kg	<3.19E+02	<8.19E+02	<5.69E+02	--	--	--
Sr	ICP-ES	mg/kg	1.97E+02	2.35E+02	2.16E+02	2.5E+01	4.8E-03	8.2E-04
Th	ICP-ES	mg/kg	1.73E+04	1.36E+04	1.55E+04	2.3E+03	3.4E-01	5.9E-02
Ti	ICP-ES	mg/kg	1.03E+02	1.13E+02	1.08E+02	7E+00	2.4E-03	4.1E-04
U	ICP-ES	mg/kg	6.00E+03	<7.17E+03	6.00E+03	2.0E+02	1.3E-01	2.3E-02
V	ICP-ES	mg/kg	<4.46E+01	<9.87E+01	<7.17E+01	--	--	--
Zn	ICP-ES	mg/kg	3.56E+02	3.30E+02	3.43E+02	2.0E+01	7.6E-03	1.3E-03
Zr	ICP-ES	mg/kg	1.46E+03	--	1.46E+03	5E+01	3.2E-02	5.6E-03
Co	ICP-MS	mg/kg	2.61E+01	2.85E+01	2.73E+01	3.3E+00	6.0E-04	1.0E-04
Pb	ICP-MS	mg/kg	2.98E+01	3.15E+01	3.07E+01	8.2E+00	6.8E-04	1.2E-04
Hg	CVAA	mg/kg	1.85E+04	--	1.85E+04	1.2E+03	4.1E-01	7.0E-02
Th-232	ICP-MS	mg/kg	1.67E+04	1.38E+04	1.52E+04	1.8E+03	3.4E-01	5.8E-02
U-233	ICP-MS	mg/kg	1.30E+01	9.29E+00	1.11E+01	2.8E+00	2.5E-04	4.2E-05
U-234	ICP-MS	mg/kg	1.00E+01	1.06E+01	1.03E+01	1.2E+00	2.3E-04	3.9E-05
U-235	ICP-MS	mg/kg	1.01E+02	9.16E+01	9.63E+01	7.4E+00	2.1E-03	3.7E-04
U-236	ICP-MS	mg/kg	1.29E+01	1.37E+01	1.33E+01	1.9E+00	2.9E-04	5.1E-05
Np-237	ICP-MS	mg/kg	1.35E+01	1.35E+01	1.35E+01	1.9E+00	3.0E-04	5.2E-05
U-238	ICP-MS	mg/kg	5.66E+03	4.83E+03	5.25E+03	5.5E+02	1.2E-01	2.0E-02
Pu-239	ICP-MS	mg/kg	2.15E+02	1.88E+02	2.02E+02	2.3E+01	4.5E-03	7.7E-04
Pu-240	ICP-MS	mg/kg	2.26E+01	2.05E+01	2.16E+01	2.2E+00	4.8E-04	8.2E-05

Table 3-4: Characterization of Tank 12H Sludge Sample Available for ECC Actual-Waste Testing, continued

analyte	method	units	overall average	st. dev.	mCi / g Fe	mCi / g Al
Pu-238	PuTTA	dpm/g	7.18E+08	5.2E+07	7.2E+00	1.2E+00
Pu-239/240	PuTTA	dpm/g	3.06E+07	2.4E+06	3.0E-01	5.3E-02
Pu-241	Pu-241	dpm/g	1.74E+08	1.3E+07	1.7E+00	3.0E-01
Cs-137	gamma	dpm/g	2.48E+08	2.1E+07	2.5E+00	4.3E-01
Co-60	Cs-rem g	dpm/g	4.42E+05	3.5E+04	4.4E-03	7.6E-04
Eu-152	Cs-rem g	dpm/g	2.43E+06	1.3E+05	2.4E-02	4.2E-03
Eu-154	Cs-rem g	dpm/g	3.15E+07	2.6E+06	3.1E-01	5.4E-02
Eu-155	Cs-rem g	dpm/g	2.10E+06	4E+04	2.1E-02	3.6E-03
Am-241	Cs-rem g	dpm/g	3.90E+07	3.5E+06	3.9E-01	6.7E-02
Sr-90	Sr-90	dpm/g	2.04E+10	2.0E+09	2.0E+02	3.5E+01
Am-241	Am/Cm	dpm/g	3.90E+07	3.5E+06	3.9E-01	6.7E-02
Am-243	Am/Cm	dpm/g	3.53E+04	3.0E+03	3.5E-04	6.1E-05
Am-242m	Am/Cm	dpm/g	2.83E+04	4.63E+04	2.8E-04	4.9E-05
Cm-243	Am/Cm	dpm/g	<1.52E+05	--	--	--
Cm-245	Am/Cm	dpm/g	<1.25E+05	--	--	--
Cm-247	Am/Cm	dpm/g	<1.18E+05	--	--	--
Cf-249	Am/Cm	dpm/g	<1.18E+05	--	--	--
Cf-251	Am/Cm	dpm/g	<9.78E+04	--	--	--
Cm-242	Am/Cm	dpm/g	2.34E+04	3.83E+04	2.3E-04	4.0E-05
Cm-244	Am/Cm	dpm/g	1.03E+06	1.3E+05	1.0E-02	1.8E-03

3.2 ECC Processing

The primary ECC process testing involved the dissolution of actual waste sludge using 2 wt% OA (Dissolution), the decomposition of OA and the precipitation of sludge by oxidation initiated with dissolved ozone in the ECC reactor (Decomposition), and liquid removal by a vacuum evaporator (Evaporation). This section contains the conditions and real-time observations made during the primary ECC process testing. The observations related to solubilities and sample analytical results for the dissolution and decomposition processes are discussed in the next section (Section 3.3). Outside of the primary ECC process, the storage of materials at different conditions for investigation of solubility effects is discussed in a separate section (Section 3.4).

Previous Table 2-1 contains a description of the ECC tests that were initially scheduled to be performed. This report contains the results of the testing performed without use of the UV light, Test 1 using Tank 12H sludge feed material and Test 2 using Tank 5F sludge feed material. Prior to initiating testing with actual-waste sludge, a test was performed that only involved the decomposition of 2 wt% OA (without running the other modules, without sludge feed, and without chemical and radiological analysis).

Table 3-5 contains information on the material balance for the two ECC real waste tests. We determined that the dissolution module must contain at least 3.2 L of material in order for the pH probe to be in contact with the solution. However, when the RWT equipment transfer system is used between the Dissolution and Decomposition Module, there is significant holdup in the lines (600 mL to 1 L). The total volume in the Dissolution Module was 3.2L or greater for each test even though the volume of fresh 2 wt% OA was often less than 3.2 L. The Decomposition Module product volume estimate is a more appropriate indication of the amount of material that was reacted in the ECC Decomposition Module than is the Dissolution Module OA volume feed.

Table 3-5: Throughput Data ECC Real Waste Tests 1 and 2

Test 1		Test 2	
Initial Tank 12H Sludge Feed (dry)	48.1 g	Initial Tank 5F Sludge Feed (dry)	65.1 g
Residual After OA Batch 3 (dry)	13.1 g	Residual After OA Batch 3 (dry)	12.7 g
Sludge Removal (dry basis)	73%	Sludge Removal (dry basis)	80%
OA Batch 1		OA Batch 1	
Oxalic Acid Feed	3.2 L	Oxalic Acid Feed	3.2 L
Dissolution Time	10.5 hr	Dissolution Time	8 hr
Decomposition Module Product	2.2 L	Decomposition Module Product	2.8 L
Evaporator Module Feed	1.75 L	Evaporator Module Feed	2.0 L
Evaporator Module Output (as slurry)	189 g	Evaporator Module Output (as slurry)	310 g
OA Batch 2		OA Batch 2	
Oxalic Acid Feed	3.0 L	Oxalic Acid Feed	2.8 L
Dissolution Time	8 hr	Dissolution Time	8 hr
Decomposition Module Product	2.8 L	Decomposition Module Product	2.3 L
Evaporator Module Feed	1.95 L	Evaporator Module Feed	1.5 L
Evaporator Module Output (as slurry)	351 g	Evaporator Module Output (as slurry)	123 g
OA Batch 3		OA Batch 3	
Oxalic Acid Feed	3.0 L	Oxalic Acid Feed	2.8 L
Dissolution Time	8 hr	Dissolution Time	8 hr
Decomposition Module Product	3.0 L	Decomposition Module Product	2.85 L
Evaporator Module Feed	1.95 L	Evaporator Module Feed	2.2 L
Evaporator Module Output (as slurry)	276 g	Evaporator Module Output (as slurry)	187 g

During the two tests, the sludge removal did not meet the target of 95% removal by a combination of dissolution with OA and transfer of insoluble solids. Test 1 showed an approximately 73% removal of Tank 12H sludge and Test 2 showed an approximately 80% removal of Tank 5F sludge. Although the dissolution in this testing did not meet the stated goal, the dissolution time (or cycle) in this testing was not representative of the ECC flowsheet as the dissolution time was limited to only 8 hours. The 1 gpm circulation rate, with the discharge of the fluid onto the surface of the dissolution module fluid, results in a level of mixing at the bottom of the dissolution basket that is conservatively low. Visual observation of the sludge in the dissolution basket (made between OA batches) suggested that sludge suspension was not fully achieved during testing. Figure 3-1 contains a photograph of the dissolution basket made after the first OA batch for Test 1. The tank 12H sludge was wetter and noticeably lighter in shade after the first OA batch. Even though this sludge had a particle size that would allow for it to be swept through the mesh, a significant portion of the undissolved solids remained in the dissolution basket.

Appendix A contains a series of tables (Table A-1 through Table A-25) that list the process conditions as a function of time for each of the dissolution, decomposition, and evaporation runs performed. Dissolution and evaporation data were recorded hourly. Decomposition data was recorded on a 15 minute interval except for periods where pH readings were changing rapidly enough to warrant more frequent observation.

Figure 3-2 through Figure 3-8 contain plots of the in-process pH and ORP measurement data during the ECC decomposition testing. During decomposition testing, pH was used as the indicator of OA decomposition. The pH generally started low, near the final pH of the OA and dissolved sludge mixture measured at the end of the dissolution process. As the ECC decomposition proceeded, the pH gradually increased, accelerating as pH approached 7. The pH then leveled off, usually above pH 8. Overall, the ORP measurements were less regular than the pH measurements. ORP remained constant for the most part during the initial periods where pH was changing slowly. Then a change in the ORP measurement was usually noted during periods when pH was changing more rapidly. This period was usually followed by another period of nearly constant ORP.

Figure 3-2 contains the pH and ORP trends for the case where no sludge feed was added and 2 wt% OA was decomposed. Because magnetite and hematite dissolution testing had occurred previously using the ECC RWT equipment and cleaning of the equipment was not perfect, we consider this testing to also contain small amounts of iron. The appearance of the decomposition module effluent supported our assumption that the OA decomposition test contained iron. The OA decomposition test without sludge feed was the only decomposition test in this set that was split into two parts due to the long run time. The first 10.5 hours of run time was performed continuously, followed by a system shutdown overnight, with the additional run time resuming when reaction conditions were reestablished 13.5 hours later. The shutdown period is evident in Figure 3-2 by a slight discontinuity in the pH measurement, which indicates that some decomposition reaction may have continued to occur during the shutdown and restart period. The OA decomposition test without added sludge is distinguished by the relatively long reaction time required (greater than 13 hours) and by the relatively fast pH change from 2 to 7, which took less than 1.5 hours. The ORP was steady throughout the first portion of processing, at 650 to 700 mV. When the pH started changing rapidly, the ORP measurement increased to above 1000 mV and then settled to around 900 mV at the completion of the decomposition reaction.

Figure 3-3, Figure 3-4, and Figure 3-5 contain the pH and ORP trends for Test 1 (Tank 12H sludge) OA decomposition for the first, second, and third batches, respectively. The three batches show similar trends in pH during OA decomposition, with the major differences being the initial pH and the amount of time required for OA decomposition. In all cases, once the pH had risen to 3, it took an additional three hours for the pH to raise to 7 and above. However, since the starting pH was higher for the earlier batches than for the later batches, the amount of time to get to pH 3 increased with subsequent batches. The threshold of pH 3 was crossed at 30 minutes, five hours, and eight hours for the first, second and third OA batches, respectively. The change in pH temporarily slowed around pH 4, possibly indicating that solids were precipitating or some other factor was contributing to pH buffering. Once again, the ORP measurement fluctuated during periods of more rapid pH change.

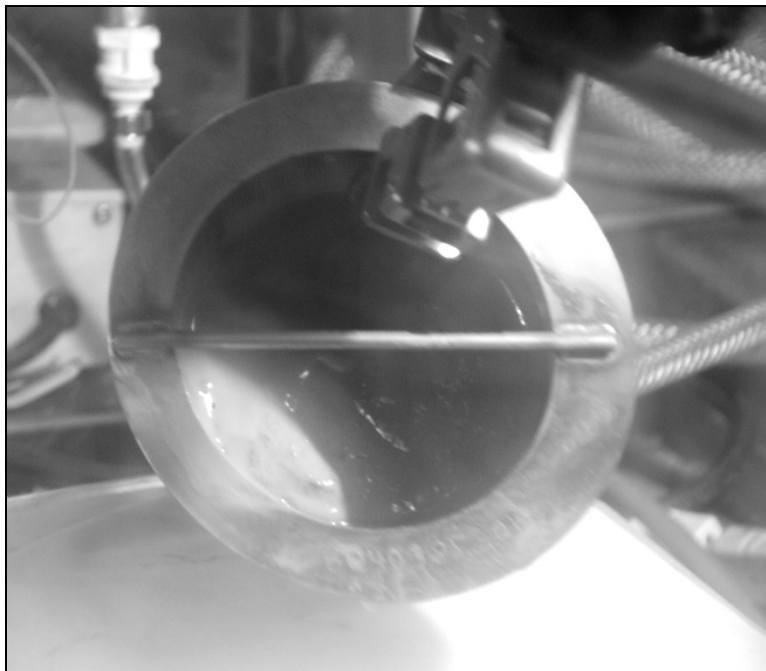


Figure 3-1: Sludge remaining in Dissolution Module basket after Test 1, OA Batch 1

Figure 3-6, Figure 3-7, and Figure 3-8 contain the pH and ORP trends for Test 2 (Tank 5F sludge) OA decomposition for the first, second and third batches, respectively. In Figure 3-6, the pH measurements during the first OA decomposition batch are not correct but are shown to illustrate the real-time information available at the time of the experiment. Inspection of the pH probe after the first OA decomposition batch revealed that the probe was damaged either prior to or during the decomposition batch. Because the pH measurement is the primary indication of the progress of the decomposition reaction, decomposition of the first OA batch was performed for much longer than necessary. Based on the steadiness of the ORP measurement after about five hours of the decomposition reaction, it is likely that the target oxalate decomposition was attained in about six hours. Accounting for the pH probe problem with the first OA batch, similar trends in pH were noted for Test 2 as were seen in Test 1. Each subsequent OA decomposition batch started at a lower pH, had a higher soluble oxalate concentration, and took a longer time to show large increases in the pH measurement, resulting in overall longer decomposition times required for the later batches. The pH and ORP trends for Test 2 are internally consistent but did not show the same exact shape as the trends for Test 1, possibly indicating slightly different chemistry. Overall, Test 2 OA decomposition was accomplished in less time than for Test 1, possibly due to the larger amount of iron in the Tank 5F sludge than in the Tank 12H sludge. The differences in the time required for decomposition may be due to the differences in concentrations of species like iron, which may catalyze the OA decomposition reactions.

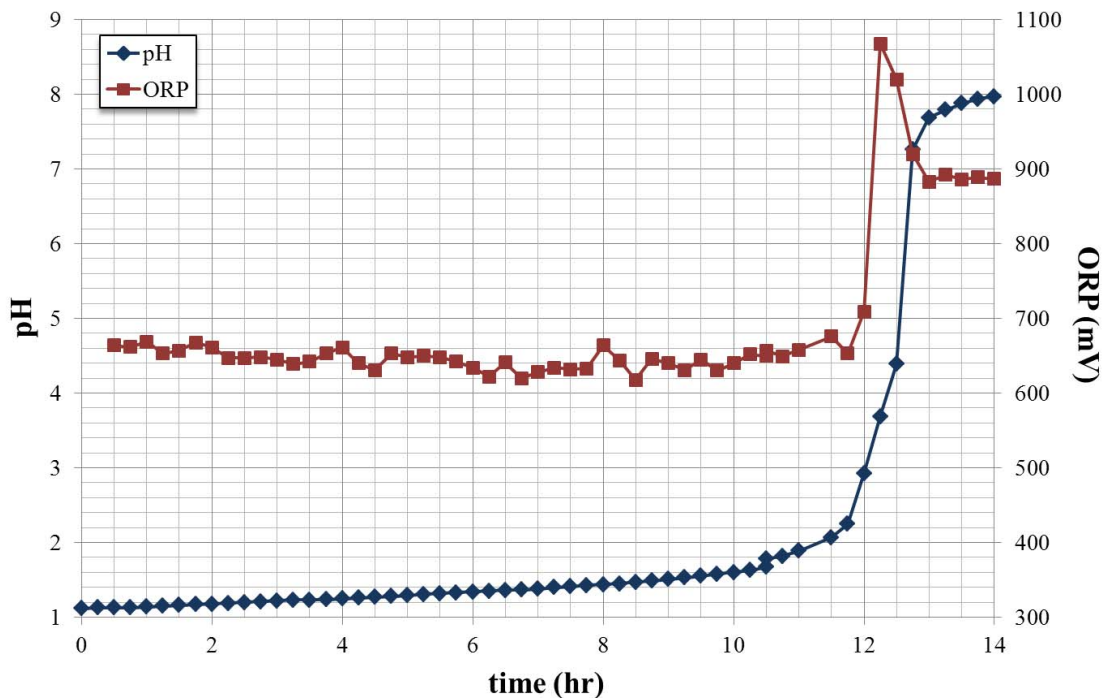


Figure 3-2: Trends for pH and ORP during Decomposition of Oxalic Acid by Ozone

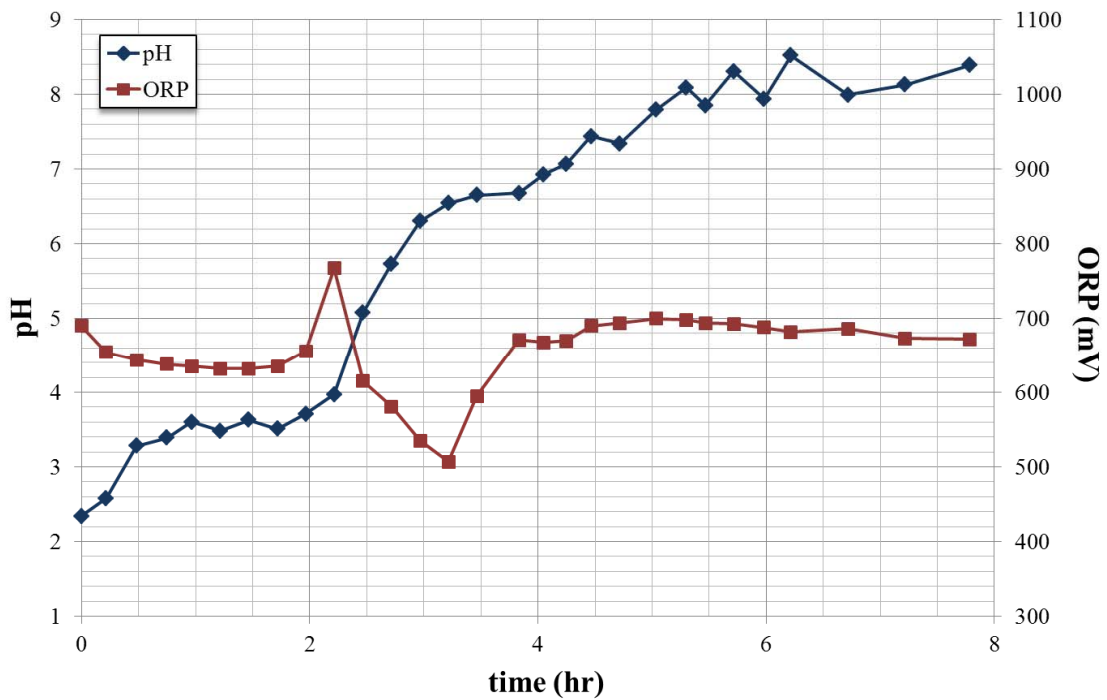


Figure 3-3: Trends for pH and ORP during Decomposition Test 1 (Tank 12H) OA Batch 1

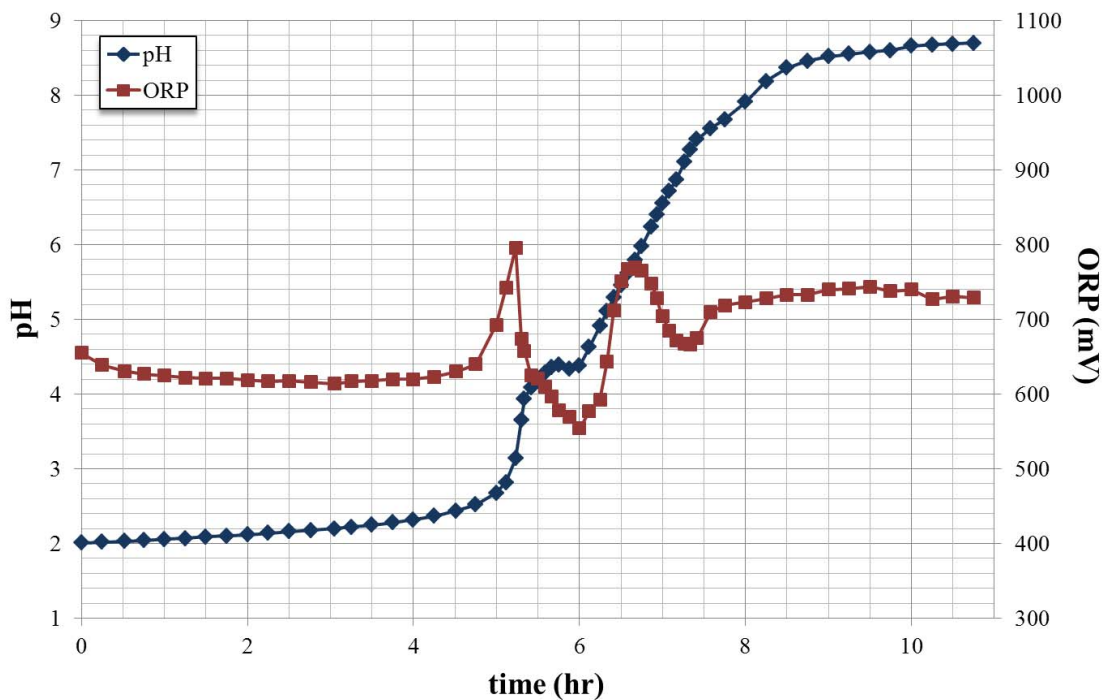


Figure 3-4: Trends for pH and ORP during Decomposition Test 1 (Tank 12H) OA Batch 2

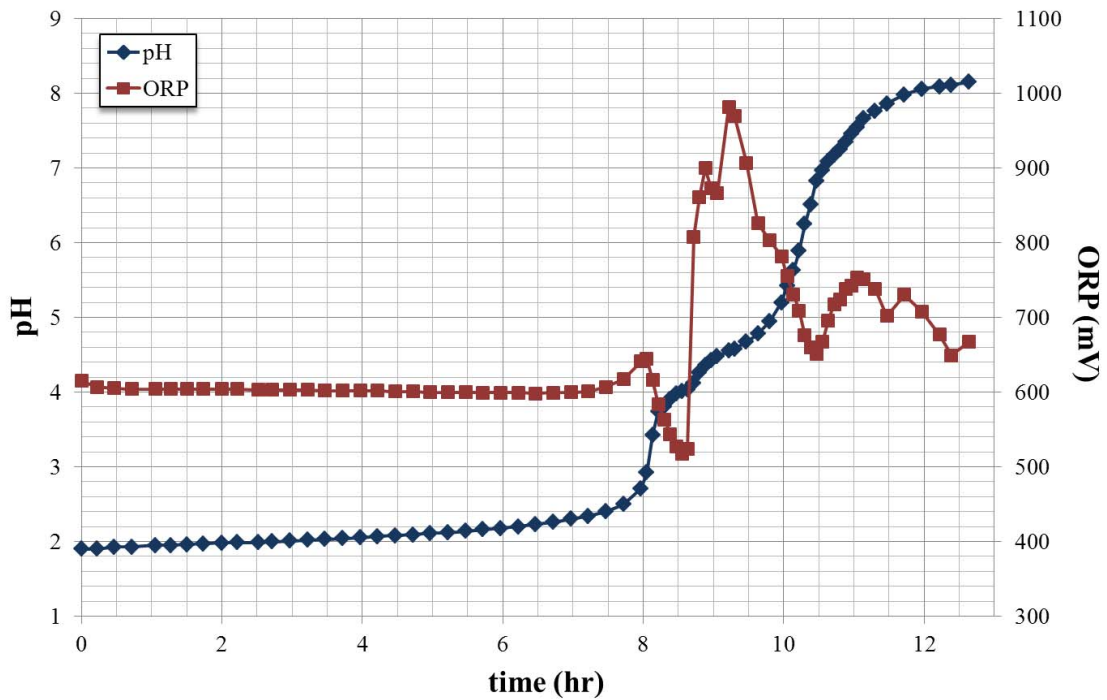


Figure 3-5: Trends for pH and ORP during Decomposition Test 1 (Tank 12H) OA Batch 3

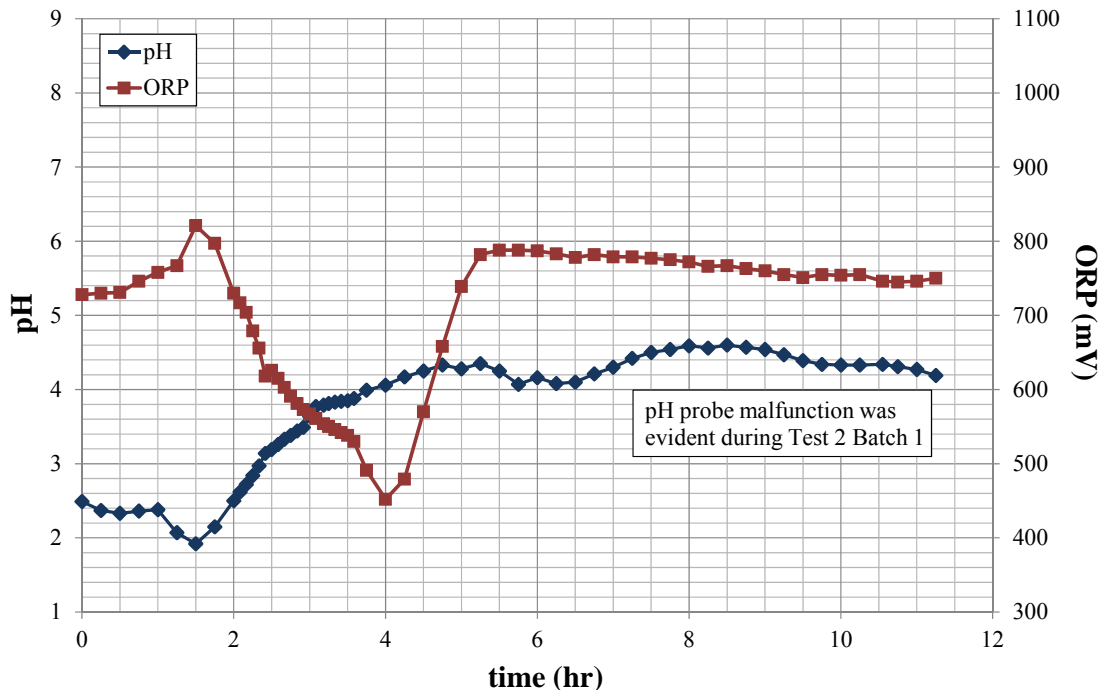


Figure 3-6: Trends for pH and ORP during Decomposition Test 2 (Tank 5F) OA Batch 1

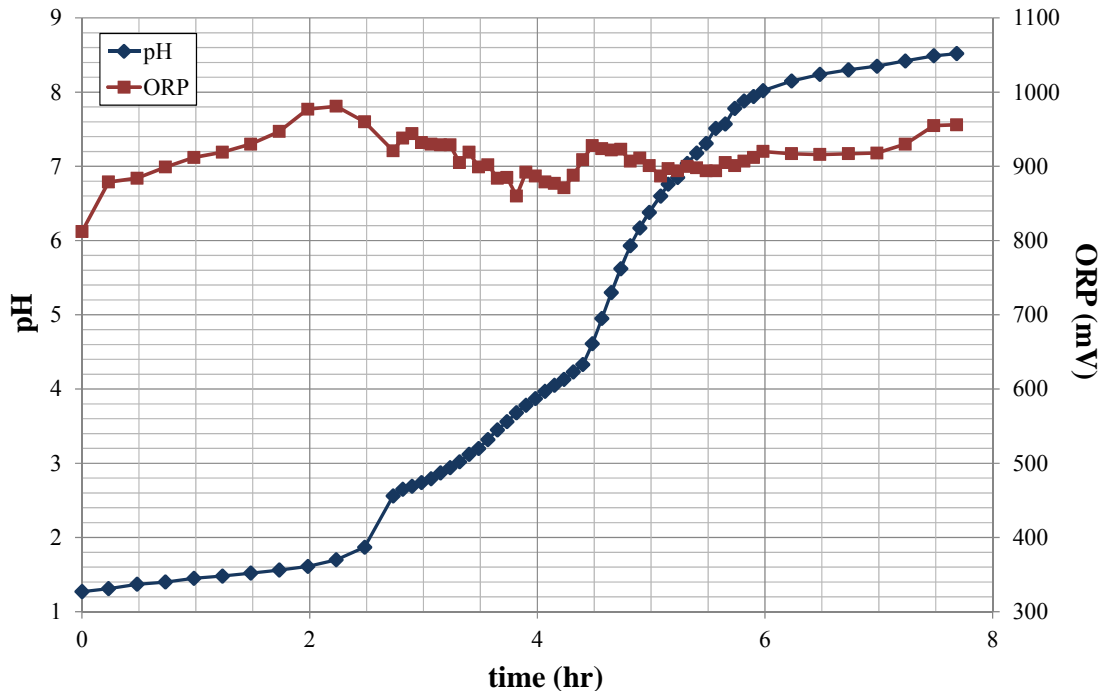


Figure 3-7: Trends for pH and ORP during Decomposition Test 2 (Tank 5F) OA Batch 2

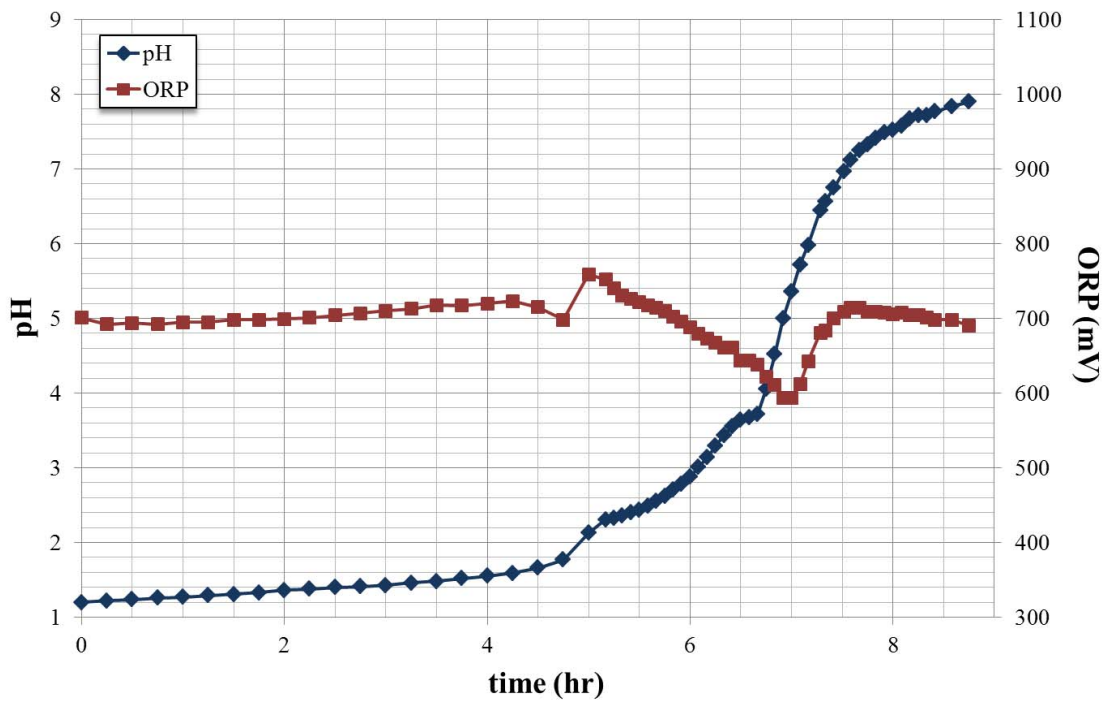


Figure 3-8: Trends for pH and ORP during Decomposition Test 2 (Tank 5F) OA Batch 3

3.3 Dissolution and Decomposition Chemistry

In this section, data is presented for the soluble and insoluble portions analyzed after the sludge dissolution process and during and after the OA decomposition process.

An indicator of the effectiveness of the sludge dissolution with 2 wt% OA is the analysis of the residual undissolved sludge and comparison of the components in the residual sludge to the components in the initial sludge feed. Table 3-6 and Table 3-7 contain the analytical results for the residual undissolved sludge from Test 1 and Table 3-8 and Table 3-9 contain the analogous results for Test 2. The results are given on a dry basis. The average and standard deviation of duplicate analyses are given.

By comparison of the species concentrations in the feed and the residue and considering the mass of residue recovered, a mass balance is used to calculate the "% removal" for each species. Note that these percent removal values are sensitive to the ability to recover sludge from the Dissolution Module manually and are thus biased high. This bias is insignificant for high % removal results (i.e. 99%) but is very significant for moderate to low % removal results (i.e., 30%). Thus, this bias may cause low removal results to be overestimated.

The ratios of various species to iron and aluminum in the residual sludge are provided in Table 3-6 through Table 3-9. The "% change" values for the relative amount of a component in the residual sludge are calculated as indicators of relative dissolution effectiveness of each component as compared with iron and aluminum. A "% change" value of zero indicates that the ratio of the species to iron or aluminum did not change when comparing the residual sludge to the feed sludge. Negative "% change" values (approaching -100%) indicate reduced concentrations of the species in the residual sludge in comparison with iron or aluminum and signify relatively favorable dissolution and removal of those species. Positive "% change" values indicate larger concentrations of the species in the residual sludge in comparison with iron or aluminum and signify relatively unfavorable dissolution and removal of those species.

For each test, the major component in the sludge (aluminum for Test 1 and iron for Test 2) remained the major component in the residual sludge. Comparison of the two tests reveals that the major component of the sludge tends to be amongst the most resistant to dissolution when based on the quantity in the residual material. For example, the high-aluminum Tank 12H sludge had a larger percentage removal of iron than aluminum while the high-iron Tank 5F sludge had a larger percentage removal of aluminum than iron. Residuals remaining in the dissolution vessel at the conclusion of Test 1 were primarily aluminum (accounts for over 90% of the mass assuming boehmite is the major form) with small amounts of iron and manganese. Residuals remaining in the dissolution vessel at the conclusion of Test 2 were primarily iron (accounts for 66% of the mass assuming hematite is the major form) with small amounts of manganese and nickel.

Removal of uranium from the sludge by dissolution with 2 wt% OA was very effective for both the Tank 12H and Tank 5F sludge. The dissolution and removal of Pu was not as effective, but the percent removal was on the same order and typically intermediate to that of iron and aluminum. The strontium removal for Tank 5F was 92 to 96% and for Tank 12H was 77 to 79%. Some minor metals were not removed effectively (<40% removal) from the dissolution vessel of the RWT equipment with 2 wt% OA. For Test 1, these metals included the lanthanides and actinides Ce, Gd, La, Th, Eu, Am, and possibly Np. For Test 2, OA was more effective at removing the minor metals and only thorium was removed at a level below 40%.

Table 3-6: Residual Sludge in Dissolution Module from Test 1 (Tank 12H)

analyte	units	residue average	st. dev.	feed average	% removal	g / g Fe			g / g Al		
						residue	feed	% change	residue	feed	% change
Ag	mg/kg	<2.69E+01	--	8.96E+01	>92%	--	2.0E-03	--	--	3.4E-04	--
Al	mg/kg	5.17E+05	4.4E+04	2.62E+05	46%	1.4E+01	5.8E+00	140%	1.0E+00	1.0E+00	0%
Ba	mg/kg	3.43E+02	4.1E+01	5.09E+02	82%	9.3E-03	1.1E-02	-18%	6.6E-04	1.9E-03	-66%
Be	mg/kg	1.82E+00	3.65E-01	<1.50E+01	--	4.9E-05	--	--	3.5E-06	--	--
Ca	mg/kg	6.93E+02	4.8E+01	3.29E+03	94%	1.9E-02	7.3E-02	-74%	1.3E-03	1.3E-02	-89%
Ce	mg/kg	1.75E+03	3.03E+02	8.39E+02	43%	4.7E-02	1.9E-02	154%	3.4E-03	3.2E-03	6%
Co	mg/kg	2.87E+01	2.4E+00	<7.58E+01	--	7.7E-04	--	--	5.6E-05	--	--
Cr	mg/kg	8.76E+01	2.3E+01	2.31E+02	90%	2.4E-03	5.1E-03	-54%	1.7E-04	8.8E-04	-81%
Cu	mg/kg	4.86E+02	7.3E+01	5.51E+02	76%	1.3E-02	1.2E-02	8%	9.4E-04	2.1E-03	-55%
Fe	mg/kg	3.71E+04	1E+03	4.52E+04	78%	1.0E+00	1.0E+00	0%	7.2E-02	1.7E-01	-58%
Gd	mg/kg	3.75E+02	6.3E+01	1.65E+02	38%	1.0E-02	3.6E-03	177%	7.3E-04	6.3E-04	15%
K	mg/kg	<5.54E+02	--	3.27E+03	>95%	--	7.2E-02	--	--	1.2E-02	--
La	mg/kg	9.02E+02	1.5E+02	4.00E+02	39%	2.4E-02	8.9E-03	175%	1.7E-03	1.5E-03	14%
Li	mg/kg	1.41E+01	1.6E+00	1.12E+02	97%	3.8E-04	2.5E-03	-85%	2.7E-05	4.3E-04	-94%
Mg	mg/kg	4.03E+02	2.8E+01	1.14E+03	90%	1.1E-02	2.5E-02	-57%	7.8E-04	4.3E-03	-82%
Mn	mg/kg	5.10E+03	6.8E+02	2.81E+04	95%	1.4E-01	6.2E-01	-78%	9.9E-03	1.1E-01	-91%
Na	mg/kg	6.28E+02	2.6E+01	4.23E+04	99.6%	1.7E-02	9.4E-01	-98%	1.2E-03	1.6E-01	-99%
Ni	mg/kg	5.42E+03	6.3E+02	4.62E+03	68%	1.5E-01	1.0E-01	43%	1.0E-02	1.8E-02	-40%
P	mg/kg	<3.02E+02	--	7.58E+02	>89%	--	1.7E-02	--	--	2.9E-03	--
Si	mg/kg	1.97E+02	3.80E+01	2.66E+03	98%	5.3E-03	5.9E-02	-91%	3.8E-04	1.0E-02	-96%
Sr	mg/kg	1.82E+02	1.1E+01	2.16E+02	77%	4.9E-03	4.8E-03	3%	3.5E-04	8.2E-04	-57%
Th	mg/kg	3.76E+04	3.0E+03	1.55E+04	34%	1.0E+00	3.4E-01	196%	7.3E-02	5.9E-02	23%
Ti	mg/kg	1.10E+02	1.0E+01	1.08E+02	72%	3.0E-03	2.4E-03	24%	2.1E-04	4.1E-04	-48%
U	mg/kg	<5.52E+02	--	6.00E+03	>97%	--	1.3E-01	--	--	2.3E-02	--
V	mg/kg	1.10E+02	2.2E+01	<7.17E+01	--	3.0E-03	--	--	2.1E-04	--	--
Zn	mg/kg	3.75E+02	4.8E+01	3.43E+02	70%	1.0E-02	7.6E-03	33%	7.3E-04	1.3E-03	-45%
Zr	mg/kg	3.53E+02	6.4E+01	1.46E+03	93%	9.5E-03	3.2E-02	-70%	6.8E-04	5.6E-03	-88%
Co	mg/kg	2.08E+01	1E+00	2.73E+01	79%	5.6E-04	6.0E-04	-7%	4.0E-05	1.0E-04	-61%
Hg	mg/kg	3.93E+02	2.85E+00	1.85E+04	99%	1.1E-02	4.1E-01	-97%	7.6E-04	7.0E-02	-99%
Pb	mg/kg	<6.28E+01	--	3.07E+01	>44%	--	6.8E-04	--	--	1.2E-04	--
Th-232	mg/kg	3.49E+04	3.28E+03	1.52E+04	37%	9.4E-01	3.4E-01	180%	6.8E-02	5.8E-02	17%
Np-237	mg/kg	<3.39E+01	--	1.35E+01	>32%	--	3.0E-04	--	--	5.2E-05	--
U-238	mg/kg	1.05E+02	2.21E+01	5.25E+03	99%	2.8E-03	1.2E-01	-98%	2.0E-04	2.0E-02	-99%
Pu-239	mg/kg	2.03E+02	3.47E+01	2.02E+02	73%	5.5E-03	4.5E-03	23%	3.9E-04	7.7E-04	-49%
Pu-240	mg/kg	2.44E+01	4.56E+00	2.16E+01	69%	6.6E-04	4.8E-04	38%	4.7E-05	8.2E-05	-43%

Table 3-7: Residual Sludge in Dissolution Module from Test 1 (Tank 12H), continued

analyte	units	residue average	st. dev.	feed average	% removal	mCi / g Fe			mCi / g Al		
						residue	feed	% change	residue	feed	% change
Pu-238	dpm/g	1.01E+09	2.25E+08	7.18E+08	62%	1.2E+01	7.2E+00	71%	8.8E-01	1.2E+00	-29%
Pu-239/240	dpm/g	4.52E+07	1.1E+07	3.06E+07	60%	5.5E-01	3.0E-01	80%	3.9E-02	5.3E-02	-25%
Co-60	dpm/g	<7.48E+05	--	4.42E+05	>54%	--	4.4E-03	--	--	7.6E-04	--
Cs-137	dpm/g	7.05E+06	1.0E+06	2.48E+08	99%	8.6E-02	2.5E+00	-97%	6.1E-03	4.3E-01	-99%
Eu-154	dpm/g	6.62E+07	6.1E+06	3.15E+07	43%	8.0E-01	3.1E-01	156%	5.8E-02	5.4E-02	7%
Eu-155	dpm/g	<4.41E+06	--	2.10E+06	>43%	--	2.1E-02	--	--	3.6E-03	--
Sr-90	dpm/g	1.54E+10	7.1E+08	2.04E+10	79%	1.87E+02	2.0E+02	-8%	1.34E+01	3.5E+01	-62%
Am-241	dpm/g	1.06E+08	1.3E+07	3.90E+07	26%	1.29E+00	3.9E-01	232%	9.27E-02	6.7E-02	38%
Am-243	dpm/g	1.10E+05	5.1E+03	3.53E+04	--	1.34E-03	3.5E-04	--	9.61E-05	6.1E-05	--
Am-242m	dpm/g	2.19E+04	2.4E+03	2.83E+04	--	2.66E-04	2.8E-04	--	1.91E-05	4.9E-05	--
Cm-242	dpm/g	1.80E+04	2.2E+05	2.34E+04	--	2.19E-04	2.3E-04	--	1.57E-05	4.0E-05	--
Cm-244	dpm/g	3.12E+06	8.8E+04	1.03E+06	17%	3.79E-02	1.0E-02	271%	2.72E-03	1.8E-03	54%

The final pH values and the molar concentrations of selected species for each dissolution batch are provided in Table 3-10. For each test, the highest pH was observed for Batch 1 and the lowest for Batch 3, indicating that the sludge was less effective at acid neutralization with repeated contacts. Lower pH values were observed for the Tank 5F material, where the final pH for batches 2 and 3 were near that of 2 wt% OA. The Tank 12H material contains more base equivalents due to the presence of more residual basic salts or basic metal oxides and hydroxides such as boehmite (AlOOH), which is the primary aluminum phase in this sludge.¹⁵

The liquid phase concentrations of the primary sludge components (Al and Fe) indicate the effectiveness of OA for dissolution over the 8 hour test period. Boehmite is not highly soluble in oxalic acid. An aluminum concentration of 0.09 M was observed in previous testing with pure boehmite in 1.1 wt% OA at 70 °C (pH 1.3, 3 week contact) when nitric acid was added for pH control.¹⁵ The aluminum concentration for Test 1 was near 0.008 M for all three batches and significant residual aluminum remained (Table 3-6) after Batch 3. The low aluminum concentrations may be the result of slow boehmite dissolution kinetics in oxalic acid. For Test 2, nearly all of the aluminum had dissolved by the completion of Batch 3. Opposite trends were observed in the iron concentrations for Tests 1 and 2, where nearly all of the iron was dissolved from the Test 1 material by the completion of Batch 3, while significant iron remained in the Test 2 residue (Table 3-8). In previous testing with pure hematite (Fe₂O₃), the iron solubility in 1 wt% OA was approximately 0.06 M at 50 °C (pH 1.5, 6 week contact).¹⁶ In 2 wt% OA, the iron concentration would be expected to approach 0.12 M under these same conditions, however, other metals in sludge compete for the added oxalate anion, resulting in a lower iron solubility. In addition, dissolution times for Tests 1 and 2 ranged from 8-11 hours (Table 3-5) and previous testing indicated that this timescale is insufficient to reach equilibrium for hematite dissolution in OA.¹⁶ Successively lower iron concentrations were observed for each Test 2 batch, despite the fact that 25% of the iron remained at the conclusion of Batch 3. The soluble iron concentration for Batch 3 was nearly 70% lower than that observed for Batch 1. This is an indication that the iron particles remaining after the initial OA batches dissolve more slowly and longer OA contact times might be needed to increase the dissolution efficiency for F Area tanks.

Trends in the oxalate concentrations in Table 3-10 are not fully understood. For Tests 1 and 2, the Batch 1 oxalate concentrations were only 19 and 41%, respectively, of the initial OA concentration (2 wt% OA corresponds to 0.224 M oxalate). The measured oxalate concentrations increased during successive batches for each test. The Batch 3 oxalate concentrations were 93% for Test 1 and 82% for Test 2, respectively, of the initial value. The low measured oxalate concentrations for early process batches could be due to: 1) analytical errors for highly complexed oxalate anion, 2) precipitation of oxalate compounds (including formation of films), or 3) decomposition of oxalate anion during dissolution.

Table 3-8: Residual Sludge in Dissolution Module from Test 2 (Tank 5F)

analyte	units	residue average	st. dev.	feed average	% removal	g / g Fe			g / g Al		
						residue	feed	% change	residue	feed	% change
Al	mg/kg	3.37E+03	1E+02	1.64E+04	96%	7.3E-03	4.5E-02	-84%	1.0E+00	1.0E+00	0%
Ba	mg/kg	6.04E+02	4.5E+01	1.63E+03	93%	1.3E-03	4.5E-03	-71%	1.8E-01	9.9E-02	80%
Be	mg/kg	1.03E+01	6.7E-01	<7.80E+00	--	2.2E-05	--	--	3.1E-03	--	--
Ca	mg/kg	8.51E+02	5.4E+01	4.57E+03	96%	1.8E-03	1.3E-02	-85%	2.5E-01	2.8E-01	-10%
Cd	mg/kg	6.31E+01	1.9E+00	8.00E+01	85%	1.4E-04	2.2E-04	-38%	1.9E-02	4.9E-03	282%
Ce	mg/kg	2.64E+03	4.1E+02	2.29E+03	78%	5.7E-03	6.3E-03	-9%	7.8E-01	1.4E-01	459%
Co	mg/kg	3.17E+02	5.4E+01	2.29E+02	73%	6.9E-04	6.3E-04	9%	9.4E-02	1.4E-02	572%
Cr	mg/kg	5.18E+01	5.7E+00	4.77E+02	98%	1.1E-04	1.3E-03	-91%	1.5E-02	2.9E-02	-47%
Cu	mg/kg	9.56E+02	8.2E+01	4.62E+02	60%	2.1E-03	1.3E-03	63%	2.8E-01	2.8E-02	903%
Fe	mg/kg	4.61E+05	3.9E+04	3.62E+05	75%	1.0E+00	1.0E+00	0%	1.4E+02	2.2E+01	516%
Gd	mg/kg	1.80E+02	2E+00	<1.96E+02	--	3.9E-04	--	--	5.3E-02	--	--
K	mg/kg	<3.43E+02	--	6.62E+02	>90%	--	1.8E-03	--	--	4.1E-02	--
La	mg/kg	1.45E+03	1.7E+02	9.98E+02	72%	3.1E-03	2.8E-03	14%	4.3E-01	6.1E-02	603%
Li	mg/kg	2.90E+01	4.5E+00	4.38E+02	99%	6.3E-05	1.2E-03	-95%	8.6E-03	2.7E-02	-68%
Mg	mg/kg	2.90E+02	1E+01	5.88E+02	90%	6.3E-04	1.6E-03	-61%	8.6E-02	3.6E-02	139%
Mn	mg/kg	3.45E+04	4.9E+03	6.68E+04	90%	7.5E-02	1.8E-01	-59%	1.0E+01	4.1E+00	150%
Mo	mg/kg	<3.06E+01	--	1.66E+02	>96%	--	4.6E-04	--	--	1.0E-02	--
Na	mg/kg	2.26E+03	1.6E+02	3.37E+04	99%	4.9E-03	9.3E-02	-95%	6.7E-01	2.1E+00	-68%
Ni	mg/kg	5.73E+04	9.6E+03	3.60E+04	69%	1.2E-01	9.9E-02	25%	1.7E+01	2.2E+00	672%
P	mg/kg	2.29E+02	3.0E+01	<5.39E+02	--	5.0E-04	--	--	6.8E-02	--	--
Pb	mg/kg	3.17E+02	2.4E+01	3.89E+02	84%	6.9E-04	1.1E-03	-36%	9.4E-02	2.4E-02	295%
Si	mg/kg	1.23E+03	2.6E+02	8.07E+03	97%	2.7E-03	2.2E-02	-88%	3.6E-01	4.9E-01	-26%
Sr	mg/kg	3.39E+02	2.4E+01	7.80E+02	92%	7.4E-04	2.2E-03	-66%	1.0E-01	4.8E-02	111%
Th	mg/kg	<3.06E+02	--	<4.51E+02	--	--	1.2E-03	--	--	2.8E-02	--
Ti	mg/kg	6.67E+01	6.2E+00	1.99E+02	93%	1.4E-04	5.5E-04	-74%	2.0E-02	1.2E-02	63%
Zn	mg/kg	5.28E+02	6.6E+01	2.45E+02	58%	1.1E-03	6.8E-04	69%	1.6E-01	1.5E-02	943%
Zr	mg/kg	9.00E+02	1.8E+01	3.49E+03	95%	2.0E-03	9.6E-03	-80%	2.7E-01	2.1E-01	25%
Co	mg/kg	2.88E+02	5.4E+01	2.21E+02	75%	6.2E-04	6.1E-04	2%	8.5E-02	1.4E-02	531%
Hg	mg/kg	9.10E+02	2.30E+02	6.95E+02	74%	2.0E-03	1.9E-03	3%	2.7E-01	4.3E-02	535%
Pb	mg/kg	2.03E+02	1.7E-01	3.74E+02	89%	4.4E-04	1.0E-03	-57%	6.0E-02	2.3E-02	163%
Th-232	mg/kg	4.50E+01	7.0E+00	1.04E+01	16%	9.77E-05	2.9E-05	240%	1.3E-02	6.4E-04	1997%
Np-237	mg/kg	2.55E+01	3.5E+00	5.11E+01	90%	5.53E-05	1.4E-04	-61%	7.54E-03	3.1E-03	141%
U-238	mg/kg	5.35E+02	3.1E+01	7.90E+04	99.9%	1.16E-03	2.2E-01	-99%	1.59E-01	4.8E+00	-97%
Pu-239	mg/kg	6.76E+01	1.17E+01	1.03E+02	87%	1.47E-04	2.8E-04	-48%	2.00E-02	6.3E-03	219%
Pu-240	mg/kg	6.28E+00	2.10E+00	8.69E+00	86%	1.36E-05	2.4E-05	-43%	1.86E-03	5.3E-04	250%
m-241(Am,Pu)	mg/kg	1.98E+01	6.7E+00	1.41E+01	73%	4.30E-05	3.9E-05	10%	5.87E-03	8.6E-04	580%

Table 3-9: Residual Sludge in Dissolution Module from Test 2 (Tank 5F), continued

analyte	units	residue average	st. dev.	feed average	% removal	mCi / g Fe			mCi / g Al		
						residue	feed	% change	residue	feed	% change
Pu-238	dpm/g	3.71E+06	2.6E+05	4.04E+06	82%	3.63E-03	5.0E-03	-28%	4.96E-01	1.1E-01	345%
Pu-239/240	dpm/g	1.39E+07	8E+05	2.04E+07	87%	1.36E-02	2.5E-02	-46%	1.85E+00	5.6E-01	231%
Cs-137	dpm/g	1.04E+07	6.1E+05	2.39E+09	99.9%	1.02E-02	3.0E+00	-100%	1.39E+00	6.6E+01	-98%
Co-60	dpm/g	2.00E+07	2.3E+06	1.96E+07	80%	1.95E-02	2.4E-02	-20%	2.67E+00	5.4E-01	394%
Eu-154	dpm/g	5.54E+07	5.0E+06	4.52E+07	76%	5.42E-02	5.6E-02	-4%	7.40E+00	1.2E+00	494%
Sr-90	dpm/g	1.74E+10	1.5E+09	7.81E+10	96%	1.70E+01	9.7E+01	-82%	2.32E+03	2.2E+03	8%
Am-241	dpm/g	1.17E+08	3.9E+07	8.47E+07	73%	1.15E-01	1.1E-01	9%	1.57E+01	2.3E+00	571%
Am-243	dpm/g	7.91E+05	2.49E+05	5.79E+05	73%	7.74E-04	7.2E-04	7%	1.06E-01	1.6E-02	562%
Am-242m	dpm/g	2.55E+05	1.23E+05	1.89E+05	74%	2.49E-04	2.4E-04	6%	3.41E-02	5.2E-03	553%
Cm-242	dpm/g	2.11E+05	1.02E+05	1.57E+05	74%	2.06E-04	1.9E-04	6%	2.81E-02	4.3E-03	552%
Cm-244	dpm/g	5.39E+06	1.90E+06	5.29E+06	80%	5.28E-03	6.6E-03	-20%	7.20E-01	1.5E-01	394%

Table 3-10: Summary of Soluble Components at Completion of Dissolution Step

Test	OA Batch	pH	Oxalate (M)	Al (M)	Fe (M)	Na (M)
1 (Tank 12H)	1	2.34	0.091	0.009	0.008	0.030
	2	2.01	0.170	0.008	0.004	0.008
	3	1.90	0.209	0.007	0.001	0.002
2 (Tank 5F)	1	2.20	0.043	0.011	0.049	0.009
	2	1.27	0.118	0.003	0.025	0.005
	3	1.20	0.183	0.001	0.015	0.001

Table 3-11 through Table 3-16 contain the results for the soluble components during OA decomposition by application of ozone at 70 °C. Each table includes several columns of data that represent the composition of a series of samples taken as a function of time as the OA decomposition progressed during each batch. The column labeled "initial" shows an average of the duplicate analysis of the filtered samples taken from the Dissolution Module at the conclusion of the dissolution period. The columns labeled "1st sample," "2nd sample," etc. show the single analyses of the intermediate Decomposition Module samples. The "final sample" is an average of the duplicate analysis of the filtered samples taken from the Decomposition Module after the pH and permanganate end-of-process test indicated that OA decomposition was complete. Included for reference in the Decomposition chemistry tables are the run time and the pH and ORP readings as the time of sampling.

Two anomalies are evident in these Decomposition Module sample results. These anomalies occurred for the third sample of Test 1 OA batch 1 and the second sample of Test 1 OA batch 2. Inconsistent high levels of many insoluble components indicate that solids likely bypassed the syringe filters for these samples and are included in the sample results.

In general, most sludge components show the highest soluble concentrations in the initial dissolved sludge and early decomposition samples. As the sludge components drop out of solution, subsequent decomposition samples show lower concentrations of soluble sludge components until the end of the decomposition process that typically has the lowest concentration of soluble sludge components. This is true for Fe, Al, Mn, Sr-90 and is typically true for U and Pu. This behavior does not hold for typical supernate components such as sodium and Cs-137, which remain largely soluble throughout the OA decomposition. Figure 3-9 contains a photograph of the intermediate samples and final sample from the decomposition portion of Test 1 OA Batch 2. This figure, which is representative of the observations of Decomposition Module samples from all other OA Batches during ECC RWT, illustrates the increase in precipitated solids as the OA decomposition progresses. This observation is also consistent with Decomposition Module samples taken during ECC simulant testing.¹⁷

During the ECC testing, approximately 5 moles of ozone was fed to the Decomposition Module for every mole of oxalate decomposed. This molar ratio was lower than the baseline 3:1 ozone to oxalate ratio that was easily attained during simulant testing.¹⁷ The relatively low utilization of ozone in the ECC RWT may be due to several factors.

- Production of ozone was at a lower concentration (5 wt% versus the baseline 10 wt%).
- The ozone generator was located away from the RWT equipment, with the feed gas having to travel through over 20 feet of tubing between the ozone generator and the Decomposition Module. This extended distance may allow a portion of the ozone to decompose prior to introduction to the decomposition module.
- The ECC RWT Decomposition Module pressure was nominally 8 psig, which is lower than the baseline pressure. This lower pressure would result in a lower soluble ozone concentration and result in a higher fraction of the ozone exiting the Decomposition Module in the offgas stream.
- The Decomposition Module recirculation rate was not optimized and was maintained at the maximum capability of pump P-224, which was often less than 1.5 gpm.

Table 3-17 and Table 3-18 contain the results for the bulk analysis of the slurries at the end of the dissolution and decomposition processes for Tests 1 and 2, respectively. These results indicate the composition of the slurries within the process and are provided for reference.

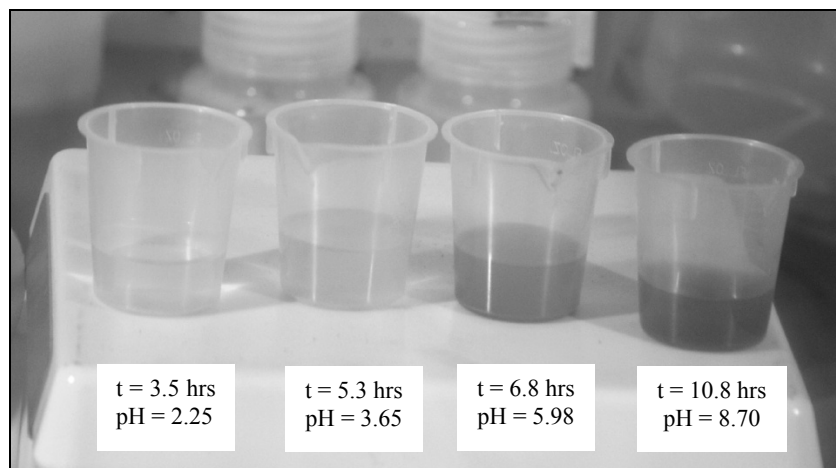


Figure 3-9: Samples of Decomposition Module during Test 1, OA Batch 2.

**Table 3-11: Soluble Components throughout ECC Oxalate Decomposition:
Test 1 (Tank 12H) OA Batch 1**

analyte	units	initial	1st sample	2nd sample	3rd sample*	final sample
time	hr	0.0	1.1	4.6	5.1	7.6
pH	--	2.34	3.48	7.34	7.79	8.39
ORP	mV	690	632	694	700	672
Oxalate	mg/L	8.05E+03	1.49E+03	4.00E+02	7.84E+03*	<4.79E+01
TOC	mg C/L	3.88E+03	--	--	--	<2.97E+02
Nitrate	mg/L	3.21E+02	--	--	--	2.71E+02
Ag	mg/L	6.64E-01	<3.72E-01	<2.32E-01	<2.25E-01	<2.77E-01
Al	mg/L	2.35E+02	1.32E+02	3.51E+00	3.68E+02*	2.93E+01
Ba	mg/L	4.42E+00	1.28E-01	1.28E-02	5.16E+00*	<9.56E-03
Ca	mg/L	2.90E+01	9.08E+00	2.62E+00	3.32E+01*	1.65E+00
Cd	mg/L	2.72E-01	<7.70E-02	<4.80E-02	3.43E-01*	<5.73E-02
Cr	mg/L	4.17E+00	6.72E+00	5.66E+00	5.27E+00*	6.61E+00
Cu	mg/L	3.86E+00	2.13E+00	3.87E-01	1.65E+00*	<1.24E-01
Fe	mg/L	4.43E+02	3.62E+00	3.39E-01	4.53E+02*	4.97E-01
K	mg/L	1.47E+01	1.10E+01	8.32E+00	8.39E+00*	<5.83E+00
La	mg/L	2.29E-01	<1.67E-01	<1.04E-01	<1.01E-01	<1.24E-01
Li	mg/L	<2.84E-01	<3.72E-01	<2.32E-01	3.25E-01*	<2.77E-01
Mg	mg/L	1.42E+01	1.44E+01	6.73E+00	1.56E+01*	1.97E-01
Mn	mg/L	3.21E+02	3.90E-01	9.92E-01	3.00E+02*	3.20E+00
Na	mg/L	6.79E+02	6.75E+02	5.33E+02	7.45E+02	5.27E+02
Ni	mg/L	1.39E+01	9.41E+00	2.42E+00	3.13E+00*	<3.06E-01
P	mg/L	8.92E+00	<4.21E+00	<2.62E+00	1.12E+01*	<3.13E+00
S	mg/L	1.96E+01	<1.93E+01	1.56E+01	1.82E+01*	<1.75E+01
Si	mg/L	3.82E+01	1.86E+01	2.55E+00	4.45E+01*	<1.86E+00
Sr	mg/L	2.19E+00	5.55E-01	1.38E-01	2.53E+00*	1.72E-02
Th	mg/L	1.66E+01	<1.41E+00	<8.80E-01	1.16E+01*	<1.05E+00
Ti	mg/L	9.79E-01	<1.03E-01	<6.40E-02	1.31E+00*	<7.65E-02
U	mg/L	8.73E+01	6.59E+01	2.56E+01	9.86E+01*	3.52E+01
Zn	mg/L	1.26E+00	2.95E-01	<1.44E-01	2.53E-01*	<1.72E-01
Zr	mg/L	1.64E+01	<6.42E-02	<4.00E-02	2.00E+01*	<4.78E-02
Hg	mg/L	1.18E+01	--	--	--	<3.08E+00
Co	mg/L	9.89E-02	<2.57E-02	<1.60E-02	3.22E-02*	<1.91E-02
Pb	mg/L	3.96E-01	<7.70E-02	<4.80E-02	3.77E-01*	<5.73E-02
m-232 (Th)	mg/L	1.55E+01	3.34E-01	1.63E-02	1.02E+01*	<1.91E-02
m-233 (U)	mg/L	1.03E-01	9.75E-02	3.33E-02	1.11E-01*	5.38E-02
m-234 (U)	mg/L	1.18E-01	1.09E-01	4.99E-02	1.25E-01*	5.76E-02
m-235 (U)	mg/L	1.43E+00	1.20E+00	4.81E-01	1.76E+00*	6.30E-01
m-236 (U)	mg/L	1.79E-01	1.43E-01	4.88E-02	1.91E-01*	6.39E-02
m-237 (Np)	mg/L	1.21E-01	1.03E-01	4.53E-02	1.20E-01*	4.05E-02
m-238 (U,Pu)	mg/L	8.08E+01	6.37E+01	2.54E+01	9.48E+01*	3.38E+01
m-239 (Pu)	mg/L	1.79E+00	1.10E+00	5.30E-01	1.12E+00*	6.14E-01
m-240 (Pu)	mg/L	1.69E-01	1.04E-01	6.25E-02	1.13E-01*	6.15E-02
Pu-238	dpm/mL	6.46E+06	4.21E+06	2.06E+06	4.19E+06*	2.20E+06
Pu-239/240	dpm/mL	3.46E+05	2.20E+05	1.04E+05	2.20E+05*	1.13E+05
Sr-90	dpm/mL	1.28E+08	--	--	--	8.17E+05
Cs-137	dpm/mL	3.55E+06	--	--	--	2.64E+06
Am-241	dpm/mL	5.84E+03	--	--	--	6.74E+03
Cm-244	dpm/mL	6.42E+02	--	--	--	5.14E+01

* high result is possibly due to filter breakthrough or misidentified sample

**Table 3-12: Soluble Components throughout ECC Oxalate Decomposition:
Test 1 (Tank 12H) OA Batch 2**

analyte	units	initial	1st sample	2nd sample*	3rd sample	final sample
time	hr	0.0	3.5	5.3	6.8	10.8
pH	--	2.01	2.25	3.65	5.98	8.70
ORP	mV	655	618	674	765	7.29
Oxalate	mg/L	1.50E+04	2.88E+03	1.27E+03	7.12E+02	<4.98E+01
TOC	mg C/L	4.65E+03	--	--	--	<3.09E+02
Nitrate	mg/L	7.64E+01	--	--	--	8.71E+01
Ag	mg/L	<2.69E-01	3.33E-01	<2.75E-01	<2.95E-01	<2.63E-01
Al	mg/L	2.19E+02	2.71E+02	3.19E+02*	9.09E+01	1.51E+01
Ba	mg/L	1.42E+00	2.03E+00	5.79E+00*	4.80E-01	<9.08E-03
Ca	mg/L	9.84E+00	1.48E+01	2.70E+01*	1.24E+01	7.59E-01
Cd	mg/L	8.82E-02	1.34E-01	6.39E-01*	<6.10E-02	<5.45E-02
Cr	mg/L	8.75E+00	9.44E+00	8.98E+01*	8.62E+00	1.01E+01
Cu	mg/L	1.59E+00	2.27E+00	3.59E+00*	1.14E+00	<1.18E-01
Fe	mg/L	1.97E+02	2.64E+02	7.33E+02*	1.12E+01	5.51E-01
K	mg/L	1.90E+01	1.17E+01	1.09E+01*	1.06E+01	6.71E+00
La	mg/L	3.31E-01	4.04E-01	4.11E+00*	<1.32E-01	<1.18E-01
Li	mg/L	<2.69E-01	<2.86E-01	5.49E+00*	<2.95E-01	<2.63E-01
Mg	mg/L	4.42E+00	6.31E+00	9.76E+00*	6.48E+00	1.11E-01
Mn	mg/L	1.00E+02	1.48E+02	1.53E+02*	3.27E+00	2.09E+00
Na	mg/L	1.94E+02	2.08E+02	2.73E+02	2.01E+02	1.88E+02
Ni	mg/L	8.35E+00	1.35E+01	1.95E+01*	1.19E+01	<2.90E-01
P	mg/L	3.54E+00	4.01E+00	2.15E+01*	<3.34E+00	<2.98E+00
S	mg/L	<1.39E+01	<1.48E+01	2.37E+01*	<1.53E+01	<1.36E+01
Si	mg/L	1.09E+01	1.75E+01	8.31E+01*	1.12E+01	<1.77E+00
Sr	mg/L	7.22E-01	1.03E+00	2.42E+00*	6.97E-01	1.09E-02
Th	mg/L	1.05E+01	1.28E+01	8.80E+00*	<1.12E+00	<9.98E-01
Ti	mg/L	5.17E-01	7.39E-01	1.67E+00*	<8.14E-02	<7.26E-02
U	mg/L	2.50E+01	3.26E+01	3.49E+01*	2.32E+01	<1.47E+01
Zn	mg/L	6.71E-01	1.10E+00	4.17E+00*	2.43E-01	<1.63E-01
Zr	mg/L	5.77E+00	8.04E+00	8.83E+00*	<5.09E-02	<4.54E-02
Hg	mg/L	2.70E+00	--	--	--	2.54E+00
Co	mg/L	8.98E-02	1.44E-01	2.09E-01*	2.15E-02	<9.08E-03
Pb	mg/L	1.33E-01	2.11E-01	1.48E+00*	<6.10E-02	3.70E-02
m-232 (Th)	mg/L	1.09E+01	1.24E+01	8.05E+00*	1.11E-01	<9.08E-03
m-233 (U)	mg/L	4.03E-02	<3.94E-02	4.58E-02*	<4.07E-02	<1.82E-02
m-234 (U)	mg/L	3.75E-02	4.22E-02	5.27E-02*	4.09E-02	<9.08E-03
m-235 (U)	mg/L	4.25E-01	5.67E-01	5.94E-01*	3.89E-01	<5.34E-02
m-236 (U)	mg/L	4.73E-02	7.30E-02	8.12E-02*	4.78E-02	<1.82E-02
m-237 (Np)	mg/L	3.05E-02	4.57E-02	6.75E-02*	3.87E-02	<9.08E-03
m-238 (U,Pu)	mg/L	2.33E+01	2.85E+01	3.18E+01*	2.09E+01	2.64E+00
m-239 (Pu)	mg/L	4.80E-01	6.98E-01	5.38E+00*	4.62E-01	7.00E-02
m-240 (Pu)	mg/L	4.73E-02	7.14E-02	4.11E-01*	4.92E-02	<9.58E-03
Pu-238	dpm/mL	1.73E+06	2.33E+06	2.51E+06*	1.72E+06	2.48E+05
Pu-239/240	dpm/mL	8.53E+04	1.20E+05	1.27E+05*	8.94E+04	1.25E+04
Sr-90	dpm/mL	5.21E+07	--	--	--	1.68E+05
Cs-137	dpm/mL	1.03E+06	--	--	--	9.85E+05
Am-241	dpm/mL	1.96E+04	--	--	--	2.21E+03
Cm-244	dpm/mL	9.15E+02	--	--	--	7.08E+00

* high result is possibly due to filter breakthrough

**Table 3-13: Soluble Components throughout ECC Oxalate Decomposition:
Test 1 (Tank 12H) OA Batch 3**

analyte	units	initial	1st sample	2nd sample	3rd sample	4th sample	5th sample	final sample
time	hr	0.0	4.2	8.5	9.9	10.3	10.8	12.5
pH	--	1.90	2.07	3.98	5.20	6.25	7.25	8.11
ORP	mV	615	602	527	782	676	724	649
Oxalate	mg/L	1.84E+04	5.67E+03	6.59E+02	4.82E+02	1.85E+02	<9.26E+01	<3.96E+01
TOC	mg C/L	4.95E+03	--	--	--	--	--	<2.45E+02
Nitrate	mg/L	<3.84E+01	--	--	--	--	--	<3.96E+01
Ag	mg/L	<2.81E-01	<2.76E-01	<2.58E-01	<2.54E-01	<2.68E-01	<2.69E-01	<2.72E-01
Al	mg/L	1.94E+02	2.73E+02	2.85E+02	9.03E+01	2.47E+01	8.52E-01	2.10E+00
Ba	mg/L	4.44E-01	1.12E+00	9.80E-01	7.73E-01	3.22E-01	8.15E-02	<9.37E-03
Ca	mg/L	3.65E+00	8.37E+00	7.66E+00	7.42E+00	6.36E+00	4.56E+00	7.71E-01
Cd	mg/L	<5.82E-02	6.28E-02	8.20E-02	<5.26E-02	<5.55E-02	<5.56E-02	<5.62E-02
Cr	mg/L	3.02E+00	5.93E+00	5.77E+00	3.56E+00	4.03E+00	3.60E+00	5.32E+00
Cu	mg/L	7.72E-01	1.51E+00	1.43E+00	8.52E-01	6.81E-01	<1.20E-01	<1.22E-01
Fe	mg/L	7.36E+01	1.65E+02	9.36E+01	1.96E+00	7.09E-01	4.58E-01	6.99E-01
K	mg/L	1.58E+01	1.17E+01	6.84E+00	9.91E+00	<5.57E+00	9.24E+00	<5.64E+00
La	mg/L	4.71E-01	5.27E-01	2.58E-01	<1.14E-01	<1.20E-01	<1.20E-01	<1.22E-01
Li	mg/L	<2.81E-01	<2.76E-01	<2.58E-01	<2.54E-01	<2.68E-01	<2.69E-01	<2.72E-01
Mg	mg/L	1.25E+00	2.95E+00	2.68E+00	2.67E+00	2.62E+00	1.92E+00	1.18E-01
Mn	mg/L	2.45E+01	7.71E+01	6.43E+01	2.00E+00	5.85E-01	4.61E-01	2.27E+00
Na	mg/L	3.58E+01	6.98E+01	5.45E+01	5.23E+01	5.14E+01	5.07E+01	5.09E+01
Ni	mg/L	7.82E+00	1.16E+01	1.17E+01	1.17E+01	1.09E+01	6.70E-01	<3.00E-01
P	mg/L	<3.18E+00	<3.12E+00	<2.92E+00	<2.88E+00	<3.04E+00	<3.04E+00	<3.07E+00
S	mg/L	<1.46E+01	<1.43E+01	<1.34E+01	<1.31E+01	<1.39E+01	<1.39E+01	<1.41E+01
Si	mg/L	2.14E+00	8.82E+00	8.07E+00	4.72E+00	2.95E+00	<1.81E+00	<1.83E+00
Sr	mg/L	2.13E-01	5.65E-01	5.14E-01	4.87E-01	3.54E-01	2.55E-01	1.36E-02
Th	mg/L	1.33E+01	1.71E+01	6.64E+00	<9.64E-01	<1.02E+00	<1.02E+00	<1.03E+00
Ti	mg/L	2.31E-01	5.61E-01	1.50E-01	<7.01E-02	<7.41E-02	<7.41E-02	<7.50E-02
U	mg/L	<1.57E+01	1.54E+01	<1.44E+01	<1.42E+01	<1.50E+01	<1.50E+01	<1.52E+01
Zn	mg/L	7.56E-01	8.96E-01	8.74E-01	5.92E-01	2.57E-01	<1.67E-01	<1.69E-01
Zr	mg/L	2.23E+00	5.00E+00	2.10E+00	<4.38E-02	<4.63E-02	<4.63E-02	<4.69E-02
Hg	mg/L	1.67E+00	--	--	--	--	--	2.97E+00
Co	mg/L	6.29E-02	1.47E-01	1.42E-01	4.00E-02	2.29E-02	<9.26E-03	<9.37E-03
Pb	mg/L	<8.10E-02	1.11E-01	7.57E-02	<2.63E-02	<3.84E-02	1.23E-01	<2.81E-02
m-232 (Th)	mg/L	1.38E+01	1.69E+01	6.87E+00	6.71E-02	2.12E-02	1.30E-02	<9.37E-03
m-233 (U)	mg/L	1.13E-02	2.38E-02	2.35E-02	1.79E-02	<1.85E-02	<1.85E-02	<1.87E-02
m-234 (U)	mg/L	1.06E-02	2.36E-02	2.62E-02	1.17E-02	1.30E-02	<9.26E-03	<9.37E-03
m-235 (U)	mg/L	7.92E-02	2.39E-01	2.09E-01	1.49E-01	1.34E-01	<9.26E-03	<9.37E-03
m-236 (U)	mg/L	<1.94E-02	2.62E-02	3.67E-02	2.10E-02	<1.85E-02	<1.85E-02	<1.87E-02
m-237 (Np)	mg/L	<1.46E-02	2.91E-02	2.06E-02	1.52E-02	1.32E-02	<9.26E-03	<9.37E-03
m-238 (U,Pu)	mg/L	4.09E+00	1.27E+01	1.14E+01	7.77E+00	6.84E+00	4.89E-02	2.53E-02
m-239 (Pu)	mg/L	1.27E-01	3.66E-01	3.10E-01	2.77E-01	2.50E-01	1.49E-02	<9.37E-03
m-240 (Pu)	mg/L	<1.94E-02	4.07E-02	3.31E-02	2.70E-02	2.49E-02	<9.26E-03	<9.37E-03
Pu-238	dpm/mL	5.12E+05	1.36E+06	1.14E+06	1.05E+06	9.72E+05	2.37E+04	7.30E+03
Pu-239/240	dpm/mL	2.49E+04	6.99E+04	6.02E+04	5.40E+04	5.08E+04	1.25E+03	<6.45E+02
Sr-90	dpm/mL	1.54E+07	--	--	--	--	--	7.82E+05
Cs-137	dpm/mL	2.31E+05	--	--	--	--	--	3.14E+05
Am-241	dpm/mL	3.46E+04	--	--	--	--	--	1.24E+03
Cm-244	dpm/mL	1.91E+03	--	--	--	--	--	1.11E+01

**Table 3-14: Soluble Components throughout ECC Oxalate Decomposition:
Test 2 (Tank 5F) OA Batch 1**

analyte	units	initial	1st sample	2nd sample	3rd sample	final sample
time	hrs	0	2.5	5.1	9.7	10.9
pH	--	2.20	--	--	--	--
ORP	mV	728	626	739	755	746
Oxalate	mg/L	3.75E+03	3.13E+02	6.51E+02	<8.93E+01	<7.82E+01
TOC	mg C/L	1.30E+03	--	--	--	<7.82E+01
Nitrate	mg/L	1.15E+02	--	--	--	1.03E+02
Carbonate	mg/L	4.15E+02	--	--	--	1.07E+03
Ag	mg/L	9.59E-01	<6.62E-01	<7.21E-01	<6.43E-01	<6.80E-01
Al	mg/L	2.95E+02	2.70E+02	3.87E+01	2.82E+01	2.81E+01
Ba	mg/L	2.58E+01	9.75E+00	3.76E-01	5.80E-02	6.61E-02
Ca	mg/L	5.94E+01	4.44E+01	1.50E+01	4.53E+00	3.75E+00
Cd	mg/L	9.04E-01	6.35E-01	<1.60E-01	<1.43E-01	<1.51E-01
Cr	mg/L	1.40E+01	1.21E+01	1.05E+01	1.04E+01	9.54E+00
Cu	mg/L	7.13E+00	6.30E+00	4.07E+00	3.79E+00	3.44E+00
Fe	mg/L	2.72E+03	8.47E+02	5.46E+01	5.48E+00	5.08E+00
Gd	mg/L	5.47E+00	4.83E+00	3.17E+00	2.90E+00	2.78E+00
K	mg/L	2.00E+01	1.88E+01	1.51E+01	1.35E+01	<1.42E+01
La	mg/L	4.16E+00	8.49E-01	<4.31E-01	<3.84E-01	<4.06E-01
Li	mg/L	6.64E+00	6.23E+00	5.51E+00	4.53E+00	3.94E+00
Mg	mg/L	6.92E+00	6.41E+00	3.03E+00	1.96E-01	1.75E-01
Mn	mg/L	8.77E+02	7.24E+02	1.80E+01	4.61E+00	4.11E+00
Na	mg/L	7.97E+02	7.10E+02	7.07E+02	7.03E+02	6.52E+02
Ni	mg/L	4.37E+01	3.31E+01	4.74E+00	<7.14E-01	<7.56E-01
Si	mg/L	1.76E+02	1.25E+02	1.81E+01	7.53E+00	6.62E+00
Sr	mg/L	1.29E+01	8.35E+00	1.54E+00	2.90E-01	2.18E-01
Th	mg/L	5.30E+00	4.16E+00	2.78E+00	<2.45E+00	<2.59E+00
Ti	mg/L	2.37E+00	6.07E-01	3.76E-01	3.57E-01	3.28E-01
U	mg/L	1.66E+03	1.45E+03	8.62E+02	7.95E+02	7.28E+02
Zn	mg/L	6.63E-01	5.43E-01	3.56E-01	3.17E-01	3.57E-01
Zr	mg/L	6.49E+01	7.17E-01	<2.81E-01	<2.50E-01	<2.64E-01
Hg	mg/L	7.29E+00	--	--	--	<2.83E-01
Pb	mg/L	4.68E+00	<2.53E+00	<1.38E+00	<1.23E+00	<1.65E+00
m-232 (Th)	mg/L	<9.64E-01	<9.20E-01	<5.01E-01	<4.46E-01	<4.72E-01
m-235 (U)	mg/L	1.17E+01	9.93E+00	6.05E+00	5.36E+00	4.58E+00
m-236 (U)	mg/L	<9.64E-01	<6.90E-01	4.23E-01	3.53E-01	<4.72E-01
m-237 (Np)	mg/L	<9.64E-01	6.12E-01	<2.51E-01	<2.23E-01	1.02E+00
m-238 (U,Pu)	mg/L	1.72E+03	1.45E+03	8.84E+02	7.38E+02	6.73E+02
m-239 (Pu)	mg/L	1.28E+00	<6.90E-01	<3.76E-01	<3.35E-01	3.16E+01
m-240 (Pu)	mg/L	<1.45E+00	<6.90E-01	<3.76E-01	<3.35E-01	1.82E+00
Pu-238	dpm/mL	4.13E+04	3.58E+04	1.94E+04	7.40E+03	7.37E+03
Pu-239/240	dpm/mL	1.27E+05	3.68E+04	8.36E+03	5.46E+03	4.88E+03
Sr-90	dpm/mL	1.15E+09	--	--	--	1.56E+07
Cs-137	dpm/mL	4.91E+07	--	--	--	2.46E+07
Am-241	dpm/mL	2.25E+04	--	--	--	6.50E+02
Am-243	dpm/mL	1.56E+02	--	--	--	<4.92E+02
Am-242m	dpm/mL	4.67E+01	--	--	--	<7.65E+01
Cm-242	dpm/mL	3.86E+01	--	--	--	<6.32E+01
Cm-244	dpm/mL	1.17E+03	--	--	--	2.36E+02

**Table 3-15: Soluble Components throughout ECC Oxalate Decomposition:
Test 2 (Tank 5F) OA Batch 2**

analyte	units	initial	1st sample	2nd sample	final sample
time	hrs	0	3.5	5.0	7.6
pH	--	1.27	3.20	6.38	8.52
ORP	mV	812	899	901	956
Oxalate	mg/L	1.04E+04	<8.94E+01	4.04E+02	<8.20E+01
TOC	mg C/L	3.73E+03	--	--	<1.67E+02
Nitrate	mg/L	<6.22E+01	--	--	<8.20E+01
Carbonate	mg/L	<6.53E+02	--	--	<4.10E+02
Ag	mg/L	<6.79E-01	<6.43E-01	<5.81E-01	<7.06E-01
Al	mg/L	7.04E+01	1.56E+02	2.59E+01	5.69E+00
Ba	mg/L	7.71E+00	7.74E+00	7.35E-01	8.97E-01
Ca	mg/L	1.32E+01	2.79E+01	9.29E+00	1.48E+00
Cd	mg/L	4.93E-01	7.64E-01	3.51E-01	4.51E-01
Cr	mg/L	4.80E+00	6.80E+00	4.09E+00	5.23E+00
Cu	mg/L	1.95E+00	3.19E+00	1.59E+00	<7.45E-01
Fe	mg/L	1.42E+03	8.29E+02	3.60E+00	4.59E+00
Gd	mg/L	<1.02E+00	2.25E+00	1.56E+00	<1.06E+00
K	mg/L	<1.42E+01	1.42E+01	1.26E+01	<1.47E+01
La	mg/L	1.72E+00	6.19E-01	<3.47E-01	<4.22E-01
Li	mg/L	2.39E+00	3.28E+00	2.81E+00	2.60E+00
Mg	mg/L	2.22E+00	4.23E+00	3.49E+00	1.32E-01
Mn	mg/L	1.75E+02	2.88E+02	4.81E-01	1.74E+00
Na	mg/L	1.07E+02	2.03E+02	1.74E+02	1.70E+02
Ni	mg/L	1.57E+01	3.70E+01	1.66E+01	<7.85E-01
Si	mg/L	3.43E+01	8.94E+01	2.51E+01	<2.16E+00
Sr	mg/L	2.59E+00	4.92E+00	8.80E-01	3.07E-02
Th	mg/L	<2.65E+00	<2.45E+00	<2.21E+00	<2.69E+00
Ti	mg/L	1.13E+00	2.68E-01	1.74E-01	<1.86E-01
U	mg/L	2.05E+02	5.88E+02	3.63E+02	5.19E+01
Zn	mg/L	<3.46E-01	4.51E-01	<2.67E-01	<3.24E-01
Zr	mg/L	1.45E+01	4.38E-01	<2.26E-01	<2.75E-01
Hg	mg/L	3.74E+00	--	--	<2.94E-01
Pb	mg/L	2.08E+00	<1.23E+00	<8.88E-01	<6.89E-02
m-232 (Th)	mg/L	1.32E+00	<4.47E-01	<3.23E-01	<1.96E-02
m-235 (U)	mg/L	1.33E+00	3.78E+00	2.33E+00	3.30E-01
m-236 (U)	mg/L	<4.72E-01	<3.35E-01	<2.42E-01	2.42E-02
m-237 (Np)	mg/L	1.07E+00	2.80E-01	1.67E-01	<1.96E-02
m-238 (U,Pu)	mg/L	1.85E+02	5.79E+02	3.64E+02	4.99E+01
m-239 (Pu)	mg/L	3.21E+01	<3.35E-01	<2.42E-01	2.33E-02
m-240 (Pu)	mg/L	1.94E+00	<3.35E-01	<2.42E-01	<2.94E-02
Pu-238	dpm/mL	4.10E+04	7.16E+04	3.15E+04	4.42E+03
Pu-239/240	dpm/mL	3.89E+04	4.80E+04	1.83E+04	2.79E+03
Sr-90	dpm/mL	1.83E+08	--	--	2.58E+06
Cs-137	dpm/mL	7.52E+06	--	--	7.33E+06
Am-241	dpm/mL	1.55E+04	--	--	1.97E+03
Am-243	dpm/mL	<8.21E+01	--	--	<4.46E+01
Am-242m	dpm/mL	3.18E+01	--	--	<2.58E+00
Cm-242	dpm/mL	2.64E+01	--	--	<2.14E+00
Cm-244	dpm/mL	1.19E+03	--	--	4.44E+02

**Table 3-16: Soluble Components throughout ECC Oxalate Decomposition:
Test 2 (Tank 5F) OA Batch 3**

analyte	units	initial	1st sample	2nd sample	3rd sample	final sample
time	hrs	0	3.3	5.8	7.3	8.6
pH	--	1.20	1.46	2.71	6.45	7.84
ORP	mV	701	713	702	681	698
Oxalate	mg/L	1.61E+04	<9.57E+01	<9.29E+01	<9.50E+01	<7.98E+01
TOC	mg C/L	3.95E+03	--	--	--	<7.98E+01
Nitrate	mg/L	<6.70E+01	--	--	--	<7.98E+01
Carbonate	mg/L	<7.37E+02	--	--	--	<3.99E+02
Ag	mg/L	<6.61E-01	<6.89E-01	<6.69E-01	<6.84E-01	<6.67E-01
Al	mg/L	2.56E+01	6.91E+01	7.18E+01	1.88E+00	<1.54E+00
Ba	mg/L	3.29E+00	6.11E+00	5.35E+00	8.12E-01	7.46E-01
Ca	mg/L	4.89E+00	1.38E+01	1.29E+01	4.49E+00	1.07E+00
Cd	mg/L	5.09E-01	6.99E-01	6.32E-01	3.90E-01	3.62E-01
Cr	mg/L	2.85E+00	5.26E+00	4.70E+00	2.14E+00	2.97E+00
Cu	mg/L	1.04E+00	1.99E+00	2.02E+00	<7.22E-01	<7.04E-01
Fe	mg/L	8.17E+02	1.45E+03	7.42E+02	4.49E+00	1.72E+00
Gd	mg/L	<9.91E-01	<1.03E+00	<1.00E+00	<1.03E+00	<1.00E+00
K	mg/L	<1.38E+01	<1.44E+01	<1.39E+01	<1.43E+01	<1.39E+01
La	mg/L	1.23E+00	2.16E+00	7.18E-01	<4.09E-01	<3.98E-01
Li	mg/L	<7.32E-01	1.28E+00	9.94E-01	8.98E-01	<6.76E-01
Mg	mg/L	9.18E-01	1.93E+00	1.92E+00	1.33E+00	1.09E-01
Mn	mg/L	8.14E+01	1.56E+02	1.57E+02	2.33E-01	3.61E-01
Na	mg/L	2.68E+01	4.92E+01	4.07E+01	3.89E+01	3.74E+01
Ni	mg/L	1.01E+01	2.02E+01	3.42E+01	6.88E+00	<7.41E-01
Si	mg/L	1.06E+01	4.04E+01	3.91E+01	7.86E+00	<2.40E+00
Sr	mg/L	9.96E-01	2.61E+00	2.41E+00	3.59E-01	<2.78E-02
Th	mg/L	<2.51E+00	4.35E+00	<2.54E+00	<2.60E+00	<2.54E+00
Ti	mg/L	5.35E-01	1.36E+00	<1.76E-01	<1.81E-01	<1.76E-01
U	mg/L	4.05E+01	2.19E+02	2.14E+02	2.25E+01	<1.51E+01
Zn	mg/L	<3.13E-01	3.49E-01	4.50E-01	<3.14E-01	<3.06E-01
Zr	mg/L	4.41E+00	1.81E+01	2.15E+00	<2.66E-01	<2.59E-01
Hg	mg/L	1.11E+00	--	--	--	<2.78E-01
Pb	mg/L	9.38E-01	1.90E+00	<5.54E-01	<6.62E-02	<6.49E-02
m-232 (Th)	mg/L	9.17E-01	3.00E+00	8.06E-01	<1.90E-02	<1.85E-02
m-235 (U)	mg/L	2.82E-01	1.27E+00	1.50E+00	1.53E-01	<1.85E-02
m-236 (U)	mg/L	<1.87E-02	<1.44E-01	<1.39E-01	<1.43E-02	<1.85E-02
m-237 (Np)	mg/L	5.72E-02	1.51E-01	1.70E-01	3.52E-02	<1.85E-02
m-238 (U,Pu)	mg/L	4.38E+01	2.01E+02	2.06E+02	2.44E+01	1.24E+00
m-239 (Pu)	mg/L	1.15E-01	4.50E-01	2.93E-01	5.79E-02	<1.85E-02
m-240 (Pu)	mg/L	<2.75E-02	<1.44E-01	<1.39E-01	<1.43E-02	<2.78E-02
Pu-238	dpm/mL	3.11E+04	8.61E+04	6.23E+04	2.01E+04	6.43E+02
Pu-239/240	dpm/mL	2.24E+04	4.79E+04	3.00E+04	1.01E+04	<3.46E+02
Sr-90	dpm/mL	7.78E+07	--	--	--	7.65E+05
Cs-137	dpm/mL	1.78E+06	--	--	--	3.05E+06
Am-241	dpm/mL	2.74E+04	--	--	--	9.16E+02
Am-243	dpm/mL	1.96E+02	--	--	--	<3.00E+01
Am-242m	dpm/mL	6.37E+01	--	--	--	<3.38E+00
Cm-242	dpm/mL	5.27E+01	--	--	--	<2.80E+00
Cm-244	dpm/mL	1.58E+03	--	--	--	1.04E+02

Table 3-17: Test 1 Dissolution and Decomposition Module Product Slurry Analysis

analyte	units	dissolution slurry			decomposition slurry		
		OA batch 1	OA batch 2	OA batch 3	OA batch 1	OA batch 2	OA batch 3
Ag	mg/kg	<2.46E+00	<2.23E+00	<2.49E+00	<2.37E+00	<2.31E+00	<2.39E+00
Al	mg/kg	9.93E+02	3.23E+02	3.46E+02	3.64E+02	1.99E+02	1.47E+02
Ba	mg/kg	5.47E+00	1.33E+00	5.46E-01	2.24E+00	9.83E-01	4.09E-01
Be	mg/kg	<1.35E-01	<1.22E-01	<1.36E-01	<1.30E-01	<1.27E-01	<1.31E-01
Ca	mg/kg	3.55E+01	1.08E+01	4.98E+00	1.66E+01	8.37E+00	5.18E+00
Cd	mg/kg	5.89E-01	<4.73E-01	<5.29E-01	<5.03E-01	<4.92E-01	<5.07E-01
Cr	mg/kg	5.82E+00	9.15E+00	3.98E+00	6.52E+00	1.09E+01	5.85E+00
Cu	mg/kg	4.93E+00	1.72E+00	<1.07E+00	2.48E+00	1.01E+00	<1.03E+00
Fe	mg/kg	5.80E+02	1.87E+02	1.05E+02	2.69E+02	1.32E+02	5.96E+01
Gd	mg/kg	<1.31E+00	<1.19E+00	<1.33E+00	<1.27E+00	<1.24E+00	<1.28E+00
K	mg/kg	<5.06E+01	<4.59E+01	<5.13E+01	<4.88E+01	<4.77E+01	<4.92E+01
La	mg/kg	1.82E+00	<1.31E+00	<1.47E+00	<1.39E+00	<1.36E+00	<1.41E+00
Li	mg/kg	<2.46E+00	<2.23E+00	<2.49E+00	<2.37E+00	<2.31E+00	<2.39E+00
Mg	mg/kg	1.64E+01	4.24E+00	1.85E+00	7.61E+00	3.51E+00	1.76E+00
Mn	mg/kg	3.76E+02	9.32E+01	3.10E+01	1.61E+02	6.98E+01	2.43E+01
Na	mg/kg	7.13E+02	1.69E+02	6.16E+01	5.15E+02	2.09E+02	6.28E+01
Ni	mg/kg	3.34E+01	1.16E+01	1.05E+01	1.64E+01	8.69E+00	7.52E+00
Si	mg/kg	4.59E+01	1.45E+01	<7.49E+00	2.65E+01	1.05E+01	<7.18E+00
Sr	mg/kg	2.64E+00	6.64E-01	2.65E-01	1.08E+00	5.00E-01	2.37E-01
Th	mg/kg	7.33E+01	1.65E+01	1.92E+01	3.29E+01	1.35E+01	<8.96E+00
Ti	mg/kg	1.56E+00	5.18E-01	4.44E-01	9.08E-01	3.49E-01	2.78E-01
U	mg/kg	7.88E+01	<4.57E+01	<5.11E+01	<4.86E+01	<4.75E+01	<4.90E+01
Zn	mg/kg	2.73E+00	1.15E+00	<1.13E+00	1.39E+00	<1.05E+00	<1.08E+00
Zr	mg/kg	1.80E+01	4.74E+00	2.35E+00	7.61E+00	3.12E+00	1.36E+00
Co	mg/kg	3.44E-01	1.32E-01	8.80E-02	9.90E-02	7.20E-02	6.30E-02
Th-232	mg/kg	6.75E+01	1.55E+01	1.83E+01	3.10E+01	1.22E+01	7.32E+00
U-233	mg/kg	<2.52E-01	5.60E-02	<5.12E-02	1.15E-01	<4.76E-02	<4.91E-02
U-234	mg/kg	<1.68E-01	3.50E-02	<3.41E-02	9.00E-02	3.80E-02	<3.27E-02
U-235	mg/kg	1.28E+00	3.46E-01	1.06E-01	8.14E-01	2.10E-01	7.50E-02
U-236	mg/kg	1.94E-01	3.80E-02	<3.41E-02	1.62E-01	4.00E-02	<3.27E-02
Np-237	mg/kg	<2.52E-01	<4.57E-02	<5.12E-02	<9.73E-02	<4.76E-02	<4.91E-02
U-238	mg/kg	7.84E+01	1.81E+01	4.57E+00	4.46E+01	1.28E+01	4.39E+00
Pu-239	mg/kg	1.84E+00	3.93E-01	1.45E-01	9.47E-01	2.73E-01	1.58E-01
Pu-240	mg/kg	<1.68E-01	4.20E-02	<3.41E-02	1.35E-01	<3.17E-02	<3.27E-02
Hg	mg/kg	6.50E+01	9.12E+00	6.72E+00	2.04E+01	1.76E+01	9.46E+00
Pb	mg/kg	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Pu-238	dpm/g	7.01E+06	1.58E+06	5.10E+05	3.79E+06	1.31E+06	4.99E+05
Pu-239/240	dpm/g	3.45E+05	8.27E+04	2.08E+04	1.99E+05	6.29E+04	2.62E+04
Sr-90	dpm/g	1.75E+08	4.53E+07	2.89E+07	8.51E+07	3.84E+07	1.78E+07

Table 3-18: Test 2 Dissolution and Decomposition Module Product Slurry Analysis

analyte	units	dissolution slurry			decomposition slurry		
		OA batch 1	OA batch 2	OA batch 3	OA batch 1	OA batch 2	OA batch 3
Al	mg/kg	3.02E+02	7.22E+01	3.15E+01	1.00E+02	6.19E+01	2.49E+01
Ba	mg/kg	1.92E+01	7.35E+00	2.73E+00	6.32E+00	4.42E+00	1.56E+00
Be	mg/kg	<1.31E-01	<1.27E-01	<1.33E-01	<1.31E-01	<1.32E-01	<1.29E-01
Ca	mg/kg	6.05E+01	1.46E+01	6.54E+00	1.77E+01	1.28E+01	6.20E+00
Cd	mg/kg	<1.15E+00	<1.11E+00	<1.16E+00	<1.15E+00	<1.16E+00	<1.13E+00
Ce	mg/kg	<1.16E+01	<1.12E+01	<1.18E+01	<1.16E+01	<1.17E+01	<1.14E+01
Co	mg/kg	<1.59E+00	<1.54E+00	<1.61E+00	<1.59E+00	<1.60E+00	<1.56E+00
Cr	mg/kg	1.13E+01	3.91E+00	2.15E+00	9.78E+00	5.74E+00	3.25E+00
Cu	mg/kg	<1.04E+00	<9.98E-01	<1.05E+00	<1.03E+00	<1.04E+00	<1.01E+00
Fe	mg/kg	5.20E+02	1.36E+03	8.17E+02	2.53E+02	5.16E+02	3.17E+02
Gd	mg/kg	5.77E+00	<3.41E+00	<3.58E+00	3.72E+00	<3.56E+00	<3.46E+00
K	mg/kg	<4.94E+01	<4.76E+01	<5.01E+01	<4.93E+01	<4.98E+01	<4.84E+01
La	mg/kg	2.27E+00	2.11E+00	1.28E+00	1.13E+00	<8.93E-01	<8.69E-01
Li	mg/kg	8.23E+00	3.10E+00	1.93E+00	4.86E+00	2.95E+00	1.64E+00
Mg	mg/kg	4.97E+00	2.33E+00	9.98E-01	2.68E+00	1.78E+00	9.18E-01
Mn	mg/kg	2.07E+02	1.73E+02	9.31E+01	2.58E+02	1.38E+02	5.35E+01
Mo	mg/kg	<4.40E+00	<4.25E+00	<4.46E+00	<4.40E+00	<4.43E+00	<4.31E+00
Na	mg/kg	7.88E+02	1.01E+02	2.64E+01	6.30E+02	1.57E+02	4.36E+01
Ni	mg/kg	<7.48E+00	1.84E+01	3.04E+01	1.57E+01	1.53E+01	1.46E+01
P	mg/kg	<2.70E+01	<2.60E+01	<2.73E+01	<2.69E+01	<2.71E+01	<2.64E+01
Si	mg/kg	1.49E+02	2.94E+01	<1.63E+01	2.84E+01	2.82E+01	<1.57E+01
Sr	mg/kg	1.19E+01	2.45E+00	1.02E+00	3.18E+00	1.98E+00	7.41E-01
Th	mg/kg	<9.01E+00	<8.68E+00	<9.12E+00	<8.99E+00	<9.07E+00	<8.82E+00
Ti	mg/kg	1.96E+00	1.04E+00	<6.32E-01	<6.23E-01	<6.29E-01	<6.12E-01
U	mg/kg	1.65E+03	2.00E+02	<4.99E+01	8.76E+02	2.28E+02	6.42E+01
Zn	mg/kg	<1.08E+00	<1.05E+00	<1.10E+00	<1.08E+00	<1.09E+00	<1.06E+00
Zr	mg/kg	6.31E+01	1.43E+01	4.21E+00	9.02E-01	5.56E+00	2.72E+00
Co	mg/kg	<6.57E-01	<1.58E-01	2.06E-01	<3.28E-01	<1.65E-01	1.34E-01
Hg	mg/kg	<3.62E+00	<3.48E+00	<3.66E+00	<3.61E+00	3.75E+00	<3.54E+00
Pb	mg/kg	2.92E+00	3.12E+00	9.59E-01	<1.41E+00	1.08E+00	5.04E-01
Th-232	mg/kg	<6.57E-01	1.63E+00	9.71E-01	4.29E-01	1.38E+00	6.46E-01
U-235	mg/kg	1.13E+01	1.27E+00	2.83E-01	5.92E+00	1.63E+00	4.12E-01
U-238	mg/kg	1.64E+03	1.97E+02	4.13E+01	9.11E+02	2.32E+02	6.00E+01
Pu-239	mg/kg	<1.31E+00	4.39E-01	1.18E-01	<6.56E-01	<3.31E-01	<6.44E-02
Pu-238	dpm/g	2.11E+04	5.15E+04	2.70E+04	1.86E+04	3.74E+04	1.67E+04
Pu-239/240	dpm/g	5.48E+04	4.95E+04	2.19E+04	9.56E+03	1.80E+04	1.21E+04

3.4 Deposition Tank Chemistry

For each test, the product slurry from each OA batch was combined in a series of nine storage bottles (eight conditions plus one control) to simulate different potential adjustment scenarios after the dissolution and decomposition processes. Different parameters varied include Deposition Tank storage temperature, whether or not the slurry was evaporated prior to storage, whether or not sodium hydroxide was added to the slurry for pH adjustment, and whether or not dilute Tank 51H slurry was stored with the product slurry to simulate a Deposition Tank heel. An additional control was performed for each test by using dilute slurry of the original feed sludge rather than the ECC process effluent. Table 2-2 contains a description of the eight storage conditions plus the control. Table 3-19 and Table 3-20 contain details of the makeup of each storage condition, plus storage bottle supernatant liquid analysis for Test 1 and 2, respectively. Note that the ECC Batch additions listed in the table are slurry additions. The ECC Batch additions for Deposition Tank conditions with evaporation are more concentrated slurries than the additions for the conditions without evaporation.

Of note in Table 3-19 and Table 3-20 are the analytical results showing supernate concentrations of nitrate, oxalate, and carbonate. For Test 1, the oxalate concentrations for the storage conditions without evaporation were less than the detection limit (approximately <115 mg/L) while the oxalate concentrations for the storage conditions with evaporation were higher (500 to 800 mg/L) due to the concentration factor. For Test 2, the oxalate concentrations for the storage conditions receiving ECC effluent without evaporation were greater than 100 mg/L (265 to 300 mg/L). The higher oxalate concentration in Test 2 Deposition Tank storage bottles may be due to any of several factors: 1) there may have been incomplete oxalate decomposition in one or more batches, 2) there may have been holdup in an area of the transfer system with material that did not receive treatment in the Decomposition Module, and 3) there may have been cross contamination with a heel of 2 wt% OA in the Auxiliary Vessel used for transfers to the Evaporator Module. Also note for Test 2 Storage Condition 4, the oxalate and TOC measurements are inconsistent, with the likely true oxalate concentration in Storage Condition 4 being similar to that for Storage Conditions 5 and 6.

Table 3-19: Deposition Tank (Storage Bottle) Conditions for Test 1

Storage Condition	1	2	3	4	5	6	7	8	9
Temperature (°C)	50	50	50	50	50	50	25**	70	50
Evaporation?	No	No	No	Yes	Yes	Yes	Yes	Yes	No
Container Material	PTFE	PTFE	Glass	PMP	PMP	Glass	PMP	PMP	PMP
Additions:									
Tank 51H Heel (mL)	0	10	0	0	10	0	10	10	10
50 wt% NaOH (mL)	25	25	0	12.5	12.5	0	12.5	12.5	12.5
ECC Batch 1 (g)	144	156	133	24	27	72	28	30	71*
ECC Batch 2 (g)	137	134	139	63	64	81	61	72	73*
ECC Batch 3 (g)	134	132	135	34	63	36	66	64	0
Analysis:									
pH	>12	>12	8.69	>12	>12	9.73	>12	>12	>12
Free Hydroxide (M)	0.77	0.85	<0.12	1.15	1.26	<0.11	1.27	1.29	1.03
Fluoride (mg/L)	<113	<116	<116	<106	<109	<112	<110	<102	<112
Formate (mg/L)	<113	<116	<116	<106	<109	<112	<110	<102	<112
Chloride (mg/L)	<113	<116	<116	<106	<109	<112	<110	<102	<112
Nitrite (mg/L)	<113	<116	<116	<106	164	<112	154	234	<212
Bromide (mg/L)	<113	<116	<116	<106	<109	<112	<110	<102	<112
Nitrate (mg/L)	181	186	128	745	810	1230	837	1140	212
Phosphate (mg/L)	<113	<116	<116	<106	<109	<112	<110	122	<112
Sulfate (mg/L)	<113	<116	<116	<106	<109	<112	<110	<102	<112
Oxalate (mg/L)	<113	<116	<116	585	525	781	463	764	<112
Carbonate (mg/L)	136	139	<116	362	307	502	308	438	<112
TOC (mg C/L)	<113	<116	<116	192	197	223	165	275	816

* deionized water added to control sample

** ambient temperature, approximately 25 °C

Table 3-20: Deposition Tank (Storage Bottle) Conditions for Test 2

Storage Condition	1	2	3	4	5	6	7	8	9
Temperature (°C)	50	50	50	50	50	50	25**	70	50
Evaporation?	No	No	No	Yes	Yes	Yes	No	No	No
Container Material	PMP	PMP	Glass	PMP	PMP	Glass	PMP	PMP	PMP
Additions:									
Tank 51H Heel (mL)	0	10	0	0	10	0	10	10	10
50 wt% NaOH (mL)	25	25	0	12.5	12.5	0	12.5	12.5	12.5
ECC Batch 1 (g)	155	167	151	70	75	163	160	164	66*
ECC Batch 2 (g)	155	159	152	36	42	38	152	156	0
ECC Batch 3 (g)	134	121	147	63	61	55	121	134	55*
Analysis:									
pH	>12	>12	7.52	>12	>12	9.15	>12	>12	>12
Free Hydroxide (M)	0.59	0.63	<0.03	0.98	1.14	<0.04	0.86	0.87	1.46
Fluoride (mg/L)	<115	<110	<67	<78	163	283	<114	<108	<109
Formate (mg/L)	<115	<110	<67	<78	<109	<71	<114	<108	<109
Chloride (mg/L)	<115	<110	<67	<78	<109	<71	<114	<108	<109
Nitrite (mg/L)	<115	<110	<67	<78	283	205	<114	108	305
Bromide (mg/L)	<115	<110	<67	<78	<109	<71	<114	<108	<109
Nitrate (mg/L)	<115	<110	<67	85	479	354	126	108	305
Phosphate (mg/L)	<115	<110	<67	<78	<109	<71	<114	<108	<109
Sulfate (mg/L)	<115	<110	<67	<78	<109	<71	<114	<108	<109
Oxalate (mg/L)	264	265	300	171	1882	1840	252	271	<109
Carbonate (mg/L)	149	155	73	411	413	510	149	152	131
TOC (mg C/L)	<115	<110	87	466	446	453	<114	<108	<109

* deionized water added to control sample

** ambient temperature, approximately 25 °C

Table 3-21 through Table 3-38 contain data and measurements for the soluble components in the storage bottles. Table 3-21 through Table 3-29 are the eight storage bottles plus the control for Test 1 and Table 3-30 through Table 3-38 are for the eight storage bottles plus control for Test 2. Table 3-39 through Table 3-42 contain summaries of the Deposition Tank storage bottle solubility measurements for four of the key species, uranium-238, plutonium-238, iron and aluminum. The summary tables may prove more useful in comparing results for different storage conditions.

The time scale at which the sampling of the Deposition Tank conditions occurred differed for the two tests. The storage bottles for Test 1 were sampled on a longer time basis in order to span storage times of over one month (7, 12, and 35 days). The storage bottles for Test 2 were sampled on a shorter time basis with some overlap of the Test 1 time scale (1, 8, and 15 days).

Note that for Table 3-21 through Table 3-38, the Deposition Tank holding time is calculated from the time of the last process effluent addition (OA Batch 3) to the storage bottles. For Test 1, the time between the batch 1 addition and the batch 3 addition was 2 to 3 days. For Test 2, the time between the batch 1 addition and the batch 3 addition was 7 to 9 days.

The information provided by storage bottle analysis can be used to determine how the following factors influence the solubilities of the species of interest in the Deposition Tank:

- storage time
- processing in ECC reactor
- processing in ECC evaporator
- storage temperature
- pH adjustment
- sludge heel contact

The effect of storage time is indicated by changes in soluble components in each storage bottle sampled at three different time periods (7, 21 and 35 days for Test 1 and 1, 8 and 15 days for Test 2). In general, solubilities of the components of interest did not significantly change as a function of time when compared with the changes in the control storage condition. Uranium in the Test 2 conditions with pH adjustment is an exception to this, where the concentration tends to decrease with time. The soluble plutonium in Test 2 was overall at low levels, but did show an increase with time. This increase, however, only brought the soluble plutonium in line with the levels noted in the control storage sample. The plutonium solubility remained relatively constant during test 1, although it was at higher levels and storage testing was performed over a longer time period.

The effect of processing in the ECC reactor is indicated by differences in the soluble components between Storage Condition 2 and Storage Condition 9. The concentrations of soluble uranium in the baseline adjusted ECC effluent (Storage Condition 2) was higher than that in the control (Storage Condition 9) for Tests 1 and 2. Similarly, the concentration of soluble Pu in the baseline adjusted ECC effluent is higher than the control for Test 1, but is not distinguishably different for Test 2. A similar trend is noted for uranium in Tests 1 and 2 and plutonium in Test 1 when comparing the solubilities at other storage conditions with the control, with the control condition having lower soluble concentrations of these components. Clear trends cannot be noted for iron and aluminum differences between the baseline ECC effluent and the control.

The effect of processing ECC reactor effluent through the ECC evaporator is indicated by differences in the soluble components between Storage Conditions 1 and 4, between Storage Conditions 2 and 5, and between Storage Conditions 3 and 6. The soluble concentrations of many components are higher for the storage conditions that received evaporation than for the storage conditions that did not receive evaporation. This phenomenon is not likely unique to ECC, as soluble concentrations of uranium in Tank Farm evaporator systems have been seen to be high compared to unconcentrated feeds. This can be due to chemistry changes of the solution as the evaporator concentrates ionic components, possible subsaturation of components in the evaporator feed, or possible supersaturation of components in the evaporator product. For example, soluble aluminum concentrations are 4 to 6 times higher in the evaporated and pH adjusted cases than for the only pH adjusted cases. For the similar storage conditions with added heel, the aluminum concentration is 2 to 3 times higher for the evaporated than for the non-evaporated. One extreme case is that of soluble uranium concentration in the unadjusted cases, where the evaporated concentrations were on average 28-times higher than the non-evaporated concentration. For the pH adjusted cases with and without sludge heel, the soluble uranium

concentrations for the evaporated cases averaged less than 2-times higher than for the non-evaporated cases.

The effect of storage temperature on ECC product is indicated by comparing the soluble components for Storage Conditions 7 and 8 with Storage Condition 5 (for Test 1) and Storage Condition 2 (for Test 2). Overall the effect of storage temperature was minor. For iron and aluminum, higher temperatures led to slightly higher soluble concentrations in most cases. Uranium showed a slight trend in the opposite direction, with higher storage temperatures leading to slightly lower soluble concentrations. The trend for soluble plutonium concentration with storage temperature was not clear. There is no evidence that a phenomenon specific to the ECC process is influencing the behavior of these components with respect to storage temperature.

The effect of pH adjustment on ECC product is indicated by differences in the soluble components between Storage Conditions 1 and 3 and between Storage Conditions 4 and 6. Adjustment of the pH tended to increase the soluble concentrations of uranium and plutonium for cases where evaporation was not performed and tended to decrease the concentrations of uranium and plutonium for cases where evaporation was performed. The pH adjustment increased the soluble concentrations of iron and aluminum regardless of whether evaporation was used. In general, the cases with pH adjustment tended to bring the soluble uranium and plutonium concentrations to consistent levels when compared to the unadjusted cases.

The effect of the contact of pH adjusted ECC product with a sludge heel is indicated by differences in the soluble components between Storage Conditions 1 and 2 and between Storage Conditions 4 and 5. Although differences were seen between soluble concentrations of some individual components depending on contact with the sludge heel, no universal trends were noted as a function of contact with the sludge heel.

Table 3-43 and Table 3-44 contain the analyses of the wet solids in the Deposition Tanks for Tests 1 and 2, respectively. These tables show the components of the sludge transferred forward from the ECC process with and without mixing with a sludge heel. Some variation is caused by the incomplete separation of Deposition Tank supernate from the settled sludge.

Table 3-21: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 1: Without Evaporation, Without Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	255	87
Al	mg/L	2.16E+02	2.99E+02	3.33E+02
Ba	mg/L	<5.44E-02	8.77E-01	8.13E-01
Ca	mg/L	7.16E-01	6.75E-01	1.13E+00
Cd	mg/L	<2.81E-01	4.45E-01	3.80E-01
Cr	mg/L	6.91E+00	7.01E+00	6.98E+00
Cu	mg/L	<5.71E-01	<3.00E-01	<3.04E-01
Fe	mg/L	1.08E+00	1.42E+00	1.50E+00
K	mg/L	8.37E+01	<1.20E+01	<1.43E+01
Mg	mg/L	<1.36E-01	<7.50E-02	8.08E-02
Mo	mg/L	<2.80E+00	<1.26E+00	<1.27E+00
Na	mg/L	2.55E+04	--	2.69E+04
Ni	mg/L	<1.45E+00	<7.50E-01	<7.61E-01
P	mg/L	<7.43E+00	<7.69E+00	<7.80E+00
S	mg/L	<3.40E+01	<3.52E+01	4.11E+01
Si	mg/L	1.28E+01	1.42E+01	1.27E+01
U	mg/L	<3.66E+01	3.90E+01	2.67E+01
Zn	mg/L	7.30E-01	5.72E-01	5.28E-01
Zr	mg/L	2.27E-01	<1.22E-01	<1.50E+01
Hg	mg/L	--	1.25E+01	--
m-232 (Th)	mg/L	<4.53E-02	<3.75E-02	<7.61E-02
m-233 (U)	mg/L	<4.53E-02	4.40E-02	6.21E-02
m-234 (U)	mg/L	5.60E-02	<3.75E-02	<5.71E-02
m-235 (U)	mg/L	4.49E-01	5.08E-01	5.34E-01
m-236 (U)	mg/L	6.15E-02	7.14E-02	<7.61E-02
m-237 (Np)	mg/L	<6.80E-02	3.98E-02	<1.33E-01
m-238 (U,Pu)	mg/L	2.59E+01	2.53E+01	2.56E+01
m-239 (Pu)	mg/L	<9.06E-02	8.09E-02	1.06E-01
Pu-238	dpm/mL	2.24E+05	2.33E+05	2.78E+05
Pu-239/240	dpm/mL	1.23E+04	1.40E+04	1.45E+04
Sr-90	dpm/mL	--	6.13E+04	--
Cs-137	dpm/mL	--	1.24E+06	--
Am-241	dpm/mL	--	<1.05E+02	--
Cm-244	dpm/mL	--	<2.87E+00	--

Table 3-22: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 2: Without Evaporation, With Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	47.9	98
Al	mg/L	4.67E+02	5.62E+02	7.05E+02
Ba	mg/L	<5.65E-02	7.56E-01	7.87E-01
Ca	mg/L	1.17E+00	5.38E-01	6.35E-01
Cd	mg/L	<2.92E-01	3.65E-01	4.46E-01
Cr	mg/L	6.94E+00	6.38E+00	7.11E+00
Cu	mg/L	<5.93E-01	<3.00E-01	3.73E-01
Fe	mg/L	3.82E+00	1.56E+00	4.62E+00
K	mg/L	8.05E+01	<1.20E+01	<1.38E+01
Mg	mg/L	<1.41E-01	<7.49E-02	<5.52E-02
Mo	mg/L	<2.91E+00	<1.25E+00	1.33E+00
Na	mg/L	2.64E+04	--	2.55E+04
Ni	mg/L	<1.51E+00	<7.49E-01	<7.36E-01
P	mg/L	9.05E+00	8.86E+00	9.01E+00
S	mg/L	<3.53E+01	<3.51E+01	<3.45E+01
Si	mg/L	2.51E+01	2.45E+01	2.57E+01
U	mg/L	3.81E+01	4.79E+01	4.09E+01
Zn	mg/L	9.98E-01	7.35E-01	8.88E-01
Zr	mg/L	1.46E-01	2.29E-01	<1.45E+01
Hg	mg/L	--	2.79E+01	--
m-232 (Th)	mg/L	<4.71E-02	<3.74E-02	<7.36E-02
m-233 (U)	mg/L	5.81E-02	5.13E-02	5.69E-02
m-234 (U)	mg/L	7.38E-02	7.72E-02	7.21E-02
m-235 (U)	mg/L	6.68E-01	8.25E-01	7.49E-01
m-236 (U)	mg/L	1.18E-01	1.17E-01	9.11E-02
m-237 (Np)	mg/L	<7.06E-02	5.27E-02	<1.29E-01
m-238 (U,Pu)	mg/L	3.95E+01	3.88E+01	3.85E+01
m-239 (Pu)	mg/L	1.64E-01	1.61E-01	1.44E-01
Pu-238	dpm/mL	4.62E+05	4.97E+05	5.34E+05
Pu-239/240	dpm/mL	2.27E+04	2.36E+04	2.78E+04
Sr-90	dpm/mL	--	4.60E+04	--
Cs-137	dpm/mL	--	1.69E+06	--
Am-241	dpm/mL	--	<2.60E+02	--
Cm-244	dpm/mL	--	1.08E+02	--

Table 3-23: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 3: Without Evaporation, Without Sludge Heel, Without pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	8.67	8.71
ORP	mV	--	184	247
Al	mg/L	<3.11E+00	<1.58E+00	1.08E+00
Ba	mg/L	<5.63E-02	7.98E-01	5.85E-02
Ca	mg/L	2.61E+00	1.82E+00	2.03E+00
Cd	mg/L	<2.91E-01	3.88E-01	<6.05E-02
Cr	mg/L	7.39E+00	7.21E+00	7.86E+00
Cu	mg/L	<5.91E-01	<3.04E-01	<1.31E-01
Fe	mg/L	<9.00E-01	8.65E-01	2.72E-01
K	mg/L	7.34E+01	<1.22E+01	8.23E+00
Mg	mg/L	4.41E-01	3.75E-01	3.85E-01
Mo	mg/L	<2.90E+00	<1.27E+00	<5.45E-01
Na	mg/L	2.60E+02	--	<7.45E+02
Ni	mg/L	<1.50E+00	<7.60E-01	<3.23E-01
P	mg/L	<7.69E+00	<7.79E+00	<3.31E+00
S	mg/L	<3.52E+01	<3.56E+01	1.72E+01
Si	mg/L	<4.59E+00	<2.09E+00	1.41E+00
U	mg/L	<3.79E+01	<1.55E+01	6.64E+00
Zn	mg/L	<4.31E-01	<3.14E-01	<1.82E-01
Zr	mg/L	<1.22E-01	<1.24E-01	<6.40E+00
Hg	mg/L	--	2.47E+00	--
m-232 (Th)	mg/L	<4.69E-02	<3.80E-02	<8.07E-02
m-233 (U)	mg/L	<4.69E-02	<3.80E-02	<4.04E-02
m-234 (U)	mg/L	<4.69E-02	<3.80E-02	<6.05E-02
m-235 (U)	mg/L	1.33E-01	<1.33E-01	1.31E-01
m-236 (U)	mg/L	<4.69E-02	<5.70E-02	<8.07E-02
m-237 (Np)	mg/L	<7.03E-02	<3.80E-02	<1.41E-01
m-238 (U,Pu)	mg/L	5.19E+00	5.32E+00	5.31E+00
m-239 (Pu)	mg/L	<9.38E-02	<3.80E-02	<6.05E-02
Pu-238	dpm/mL	1.87E+05	4.05E+04	1.93E+04
Pu-239/240	dpm/mL	8.41E+03	<2.23E+03	9.38E+02
Sr-90	dpm/mL	--	1.05E+06	--
Cs-137	dpm/mL	--	1.33E+06	--
Am-241	dpm/mL	--	<6.78E+02	--
Cm-244	dpm/mL	--	2.42E+01	--

Table 3-24: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 4: With Evaporation, Without Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	55	66.1
Al	mg/L	1.25E+03	1.46E+03	1.58E+03
Ba	mg/L	<5.55E-02	8.56E-01	5.77E-02
Ca	mg/L	1.43E+00	6.56E-01	7.45E-01
Cd	mg/L	<2.87E-01	4.70E-01	<1.42E-01
Cr	mg/L	4.94E+01	4.95E+01	5.00E+01
Cu	mg/L	<5.83E-01	8.51E-01	8.16E-01
Fe	mg/L	4.06E+00	2.99E+00	2.16E+00
K	mg/L	9.04E+01	<1.19E+01	4.35E+01
Mg	mg/L	1.57E-01	<7.44E-02	7.98E-02
Mo	mg/L	5.53E+00	5.45E+00	5.23E+00
Na	mg/L	4.12E+04	--	4.22E+04
Ni	mg/L	<1.48E+00	<7.44E-01	<7.10E-01
P	mg/L	1.44E+01	1.53E+01	1.22E+01
S	mg/L	4.08E+01	<3.49E+01	<3.33E+01
Si	mg/L	4.58E+01	4.83E+01	4.74E+01
U	mg/L	7.68E+01	8.57E+01	6.54E+01
Zn	mg/L	2.69E+00	2.56E+00	2.19E+00
Zr	mg/L	2.64E-01	2.05E-01	<1.40E+01
Hg	mg/L	--	2.75E+01	--
m-232 (Th)	mg/L	<4.62E-02	<3.72E-02	<7.10E-02
m-233 (U)	mg/L	9.53E-02	8.95E-02	9.49E-02
m-234 (U)	mg/L	1.27E-01	1.14E-01	1.09E-01
m-235 (U)	mg/L	1.38E+00	1.38E+00	1.09E+00
m-236 (U)	mg/L	1.79E-01	1.25E-01	1.22E-01
m-237 (Np)	mg/L	<6.94E-02	<3.72E-02	<1.24E-01
m-238 (U,Pu)	mg/L	7.77E+01	6.99E+01	6.41E+01
m-239 (Pu)	mg/L	1.01E-01	6.91E-02	1.13E-01
Pu-238	dpm/mL	3.55E+05	3.11E+05	3.25E+05
Pu-239/240	dpm/mL	1.92E+04	1.62E+04	1.61E+04
Sr-90	dpm/mL	--	8.57E+04	--
Cs-137	dpm/mL	--	7.18E+06	--
Am-241	dpm/mL	--	<1.74E+02	--
Cm-244	dpm/mL	--	9.05E+01	--

Table 3-25: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 5: With Evaporation, With Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	28.8	67.9
Al	mg/L	1.19E+03	1.25E+03	1.48E+03
Ba	mg/L	<5.39E-02	7.69E-01	5.93E-02
Ca	mg/L	1.27E+00	6.21E-01	7.34E-01
Cd	mg/L	<2.78E-01	4.50E-01	<1.46E-01
Cr	mg/L	4.52E+01	4.06E+01	4.52E+01
Cu	mg/L	<5.65E-01	4.47E-01	4.83E-01
Fe	mg/L	2.04E+00	2.43E+00	1.70E+00
K	mg/L	8.91E+01	1.32E+01	4.27E+01
Mg	mg/L	<1.35E-01	<7.52E-02	7.30E-02
Mo	mg/L	4.64E+00	4.19E+00	4.72E+00
Na	mg/L	3.76E+04	--	3.59E+04
Ni	mg/L	<1.44E+00	<7.52E-01	<7.30E-01
P	mg/L	1.37E+01	1.35E+01	1.24E+01
S	mg/L	3.84E+01	<3.53E+01	5.22E+01
Si	mg/L	3.99E+01	3.85E+01	4.08E+01
U	mg/L	7.43E+01	7.28E+01	6.40E+01
Zn	mg/L	2.21E+00	2.06E+00	1.69E+00
Zr	mg/L	1.88E-01	1.65E-01	<1.44E+01
Hg	mg/L	--	2.68E+01	--
m-232 (Th)	mg/L	<4.49E-02	<3.76E-02	<7.30E-02
m-233 (U)	mg/L	1.25E-01	7.89E-02	1.05E-01
m-234 (U)	mg/L	1.51E-01	1.52E-01	1.08E-01
m-235 (U)	mg/L	1.35E+00	1.28E+00	1.23E+00
m-236 (U)	mg/L	1.50E-01	1.48E-01	2.02E-01
m-237 (Np)	mg/L	7.23E-02	4.85E-02	<1.28E-01
m-238 (U,Pu)	mg/L	7.78E+01	6.79E+01	6.27E+01
m-239 (Pu)	mg/L	1.31E-01	1.36E-01	8.74E-02
Pu-238	dpm/mL	3.44E+05	2.91E+05	2.52E+05
Pu-239/240	dpm/mL	1.95E+04	1.51E+04	9.76E+03
Sr-90	dpm/mL	--	8.10E+04	--
Cs-137	dpm/mL	--	6.96E+06	--
Am-241	dpm/mL	--	<8.02E+01	--
Cm-244	dpm/mL	--	3.94E+02	--

Table 3-26: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 6: With Evaporation, Without Sludge Heel, Without pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	9.77	9.68
ORP	mV	--	143	223
Al	mg/L	9.56E+00	7.83E+00	7.63E+00
Ba	mg/L	<5.80E-02	8.94E-01	5.95E-02
Ca	mg/L	2.53E+00	2.10E+00	2.51E+00
Cd	mg/L	<3.00E-01	5.10E-01	<7.14E-02
Cr	mg/L	5.90E+01	5.81E+01	6.17E+01
Cu	mg/L	<6.09E-01	<3.41E-01	<1.55E-01
Fe	mg/L	<9.28E-01	8.70E-01	2.67E-01
K	mg/L	8.75E+01	2.77E+01	4.84E+01
Mg	mg/L	1.84E-01	1.55E-01	2.38E-01
Mo	mg/L	3.98E+00	4.24E+00	4.59E+00
Na	mg/L	2.38E+03	--	2.39E+03
Ni	mg/L	<1.55E+00	<8.54E-01	<3.81E-01
P	mg/L	<7.93E+00	<8.75E+00	<3.90E+00
S	mg/L	5.26E+01	4.08E+01	4.15E+01
Si	mg/L	<4.73E+00	<2.35E+00	1.37E+00
U	mg/L	1.84E+02	2.01E+02	2.11E+02
Zn	mg/L	<4.45E-01	<3.52E-01	<2.14E-01
Zr	mg/L	<1.26E-01	<1.39E-01	<7.55E+00
Hg	mg/L	--	1.59E+01	--
m-232 (Th)	mg/L	<4.83E-02	<1.07E-01	<2.38E-01
m-233 (U)	mg/L	1.52E-01	2.52E-01	2.95E-01
m-234 (U)	mg/L	1.93E-01	2.73E-01	3.15E-01
m-235 (U)	mg/L	1.45E+00	3.52E+00	3.36E+00
m-236 (U)	mg/L	1.85E-01	4.09E-01	4.04E-01
m-237 (Np)	mg/L	<7.25E-02	<1.07E-01	<4.17E-01
m-238 (U,Pu)	mg/L	8.25E+01	1.94E+02	1.88E+02
m-239 (Pu)	mg/L	1.13E-01	4.62E-01	4.73E-01
Pu-238	dpm/mL	1.35E+06	1.33E+06	1.86E+06
Pu-239/240	dpm/mL	6.73E+04	6.76E+04	9.45E+04
Sr-90	dpm/mL	--	9.78E+05	--
Cs-137	dpm/mL	--	1.06E+07	--
Am-241	dpm/mL	--	<4.34E+02	--
Cm-244	dpm/mL	--	<2.51E+01	--

Table 3-27: Soluble Components in the Deposition Tank for the Tank 12H ECC No-Light Test, Storage Condition 7: With Evaporation, With Sludge Heel, With pH Adjustment, T = ~25 °C (ambient).

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	74	96
Al	mg/L	6.83E+02	7.30E+02	7.45E+02
Ba	mg/L	<5.17E-02	8.27E-01	7.15E-02
Ca	mg/L	6.89E-01	7.33E-01	7.68E-01
Cd	mg/L	<2.67E-01	4.60E-01	<1.53E-01
Cr	mg/L	4.43E+01	4.50E+01	4.68E+01
Cu	mg/L	<5.43E-01	<2.91E-01	<3.05E-01
Fe	mg/L	1.67E+00	2.11E+00	1.51E+00
K	mg/L	8.79E+01	<1.17E+01	4.12E+01
Mg	mg/L	<1.29E-01	7.28E-02	1.10E-01
Mo	mg/L	3.37E+00	3.42E+00	3.36E+00
Na	mg/L	3.64E+04	--	3.87E+04
Ni	mg/L	<1.38E+00	<7.28E-01	<7.63E-01
P	mg/L	8.62E+00	9.93E+00	8.85E+00
S	mg/L	4.73E+01	<3.41E+01	<3.58E+01
Si	mg/L	2.34E+01	2.58E+01	2.63E+01
U	mg/L	6.20E+01	6.85E+01	6.23E+01
Zn	mg/L	1.22E+00	1.23E+00	9.06E-01
Zr	mg/L	2.67E-01	<1.18E-01	<1.51E+01
Hg	mg/L	--	2.24E+01	--
m-232 (Th)	mg/L	<4.31E-02	<3.64E-02	<7.63E-02
m-233 (U)	mg/L	9.22E-02	8.66E-02	1.04E-01
m-234 (U)	mg/L	1.03E-01	1.09E-01	8.17E-02
m-235 (U)	mg/L	1.17E+00	1.13E+00	1.01E+00
m-236 (U)	mg/L	1.49E-01	1.52E-01	1.13E-01
m-237 (Np)	mg/L	<6.46E-02	3.82E-02	<1.34E-01
m-238 (U,Pu)	mg/L	6.36E+01	6.03E+01	5.75E+01
m-239 (Pu)	mg/L	8.96E-02	6.53E-02	8.69E-02
Pu-238	dpm/mL	2.74E+05	2.06E+05	2.41E+05
Pu-239/240	dpm/mL	1.61E+04	1.28E+04	1.24E+04
Sr-90	dpm/mL	--	8.12E+04	--
Cs-137	dpm/mL	--	6.83E+06	--
Am-241	dpm/mL	--	<1.45E+02	--
Cm-244	dpm/mL	--	9.93E+01	--

Table 3-28: Soluble Components in the Deposition Tank for Test 1 (Tank 12H No-Light), Storage Condition 8: With Evaporation, With Sludge Heel, With pH Adjustment, T = 70 °C.

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	25	70.6
Al	mg/L	1.46E+03	1.58E+03	1.95E+03
Ba	mg/L	<5.68E-02	6.56E-01	6.04E-02
Ca	mg/L	1.09E+00	5.65E-01	6.45E-01
Cd	mg/L	<2.94E-01	3.29E-01	<1.49E-01
Cr	mg/L	4.65E+01	4.57E+01	5.76E+01
Cu	mg/L	<5.97E-01	6.76E-01	7.38E-01
Fe	mg/L	2.44E+00	1.92E+00	1.62E+00
K	mg/L	8.87E+01	1.57E+01	5.24E+01
Mg	mg/L	<1.42E-01	<6.36E-02	<5.57E-02
Mo	mg/L	4.31E+00	4.40E+00	5.32E+00
Na	mg/L	3.40E+04	--	4.14E+04
Ni	mg/L	<1.52E+00	<6.36E-01	<7.43E-01
P	mg/L	1.25E+01	9.62E+00	1.24E+01
S	mg/L	5.11E+01	<2.98E+01	5.65E+01
Si	mg/L	3.24E+01	3.26E+01	3.91E+01
U	mg/L	5.62E+01	5.39E+01	4.35E+01
Zn	mg/L	1.66E+00	1.28E+00	1.38E+00
Zr	mg/L	1.23E-01	1.19E-01	<1.47E+01
Hg	mg/L	--	2.72E+01	--
m-232 (Th)	mg/L	<4.74E-02	<3.18E-02	<7.43E-02
m-233 (U)	mg/L	9.57E-02	7.27E-02	7.62E-02
m-234 (U)	mg/L	9.23E-02	8.91E-02	6.97E-02
m-235 (U)	mg/L	1.07E+00	8.83E-01	8.21E-01
m-236 (U)	mg/L	1.50E-01	1.46E-01	9.84E-02
m-237 (Np)	mg/L	<7.10E-02	<3.18E-02	<1.30E-01
m-238 (U,Pu)	mg/L	6.01E+01	4.55E+01	4.45E+01
m-239 (Pu)	mg/L	<9.47E-02	4.83E-02	5.78E-02
Pu-238	dpm/mL	3.06E+05	1.83E+05	2.09E+05
Pu-239/240	dpm/mL	1.07E+04	<4.34E+03	1.00E+04
Sr-90	dpm/mL	--	8.19E+04	--
Cs-137	dpm/mL	--	7.62E+06	--
Am-241	dpm/mL	--	<2.60E+02	--
Cm-244	dpm/mL	--	<2.04E+01	--

**Table 3-29: Soluble Components in the Deposition Tank for the Tank 12H Control
(without ECC Processing): Without Evaporation, With Sludge Heel, With pH Adjustment,
T = 50 °C.**

analyte	units	deposition tank holding time		
		7 days	21 days	35 days
pH	--	--	>12	>12
ORP	mV	--	68	68.9
Al	mg/L	4.57E+02	6.71E+02	8.42E+02
Ba	mg/L	<5.29E-02	7.45E-01	5.27E-02
Ca	mg/L	1.30E+00	1.04E+00	1.04E+00
Cd	mg/L	<2.73E-01	4.65E-01	<1.41E-01
Cr	mg/L	<8.99E-01	6.10E-01	7.19E-01
Cu	mg/L	<5.55E-01	4.53E-01	3.69E-01
Fe	mg/L	1.03E+00	1.78E+00	1.17E+00
K	mg/L	6.64E+01	<1.20E+01	<1.32E+01
Mg	mg/L	<1.32E-01	<7.47E-02	6.59E-02
Mo	mg/L	<2.72E+00	<1.25E+00	<1.18E+00
Na	mg/L	3.41E+04	--	3.68E+04
Ni	mg/L	<1.41E+00	<7.47E-01	<7.03E-01
P	mg/L	<7.23E+00	<7.66E+00	<7.21E+00
S	mg/L	<3.30E+01	<3.50E+01	3.59E+01
Si	mg/L	1.50E+01	1.40E+01	1.42E+01
U	mg/L	<3.56E+01	<1.52E+01	<1.43E+01
Zn	mg/L	3.50E-01	3.17E-01	<4.04E-01
Zr	mg/L	<1.15E-01	<1.21E-01	<1.39E+01
Hg	mg/L	--	4.50E+01	--
m-232 (Th)	mg/L	<4.41E-02	<3.73E-02	<7.03E-02
m-233 (U)	mg/L	<4.41E-02	<3.73E-02	<3.52E-02
m-234 (U)	mg/L	<4.41E-02	<3.73E-02	<5.27E-02
m-235 (U)	mg/L	1.46E-01	<1.31E-01	1.20E-01
m-236 (U)	mg/L	<4.41E-02	<5.60E-02	<7.03E-02
m-237 (Np)	mg/L	<6.61E-02	3.75E-02	<1.23E-01
m-238 (U,Pu)	mg/L	1.40E+00	1.67E+00	1.81E+00
m-239 (Pu)	mg/L	<8.81E-02	<3.73E-02	<5.27E-02
Pu-238	dpm/mL	2.32E+04	6.91E+03	5.26E+03
Pu-239/240	dpm/mL	3.11E+03	<9.99E+02	5.92E+02
Sr-90	dpm/mL	--	4.71E+05	--
Cs-137	dpm/mL	--	1.59E+06	--
Am-241	dpm/mL	--	<1.33E+03	--
Cm-244	dpm/mL	--	<1.23E+02	--

Table 3-30: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 1: Without Evaporation, Without Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	69	--	48.2
Al	mg/L	1.03E+02	1.16E+02	8.69E+01
Ba	mg/L	7.78E-01	8.25E-01	<3.04E-01
Ca	mg/L	5.08E-01	4.36E-01	1.11E+00
Cd	mg/L	4.32E-01	4.44E-01	<1.52E-01
Cr	mg/L	5.65E+00	5.82E+00	4.21E+00
Fe	mg/L	1.59E+00	1.57E+00	2.11E+00
K	mg/L	<1.42E+01	1.35E+01	1.49E+01
Li	mg/L	1.45E+00	1.50E+00	1.21E+00
Mg	mg/L	<5.70E-02	<5.75E-02	1.28E-01
Mn	mg/L	<3.80E-02	<1.05E-01	<1.04E-01
Mo	mg/L	<1.27E+00	<1.28E+00	<1.27E+00
Na	mg/L	2.28E+04	2.29E+04	1.80E+04
Ni	mg/L	<7.59E-01	<7.67E-01	<7.59E-01
P	mg/L	<7.78E+00	<7.86E+00	<7.78E+00
S	mg/L	<3.56E+01	<3.60E+01	<3.56E+01
Si	mg/L	2.77E+01	2.85E+01	2.19E+01
Sr	mg/L	<2.85E-02	<2.88E-02	<2.85E-02
U	mg/L	1.22E+02	7.27E+01	4.42E+01
Zr	mg/L	3.51E-01	1.63E-01	<1.23E-01
Hg	mg/L	--	--	7.40E-01
Oxalate	mg/L	--	--	1.42E+02
m-234 (U)	mg/L	<1.78E-01	<4.79E-02	<1.52E-01
m-235 (U)	mg/L	9.31E-01	4.51E-01	3.29E-01
m-236 (U)	mg/L	<1.19E-01	<4.79E-02	<9.49E-02
m-237 (Np)	mg/L	<1.19E-01	<2.64E-01	<5.69E-02
m-238 (U,Pu)	mg/L	1.22E+02	7.46E+01	4.15E+01
m-239 (Pu)	mg/L	<1.78E-01	<7.19E-02	<1.14E-01
Pu-238	dpm/mL	8.35E+02	8.12E+02	1.20E+02
Pu-239/240	dpm/mL	<2.33E+03	7.02E+02	9.96E+01
Sr-90	dpm/mL	--	--	<1.57E+05
Cs-137	dpm/mL	--	--	4.02E+06
Am-241	dpm/mL	--	--	<9.35E+02
Cm-244	dpm/mL	--	--	<8.93E+01

Table 3-31: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 2: Without Evaporation, With Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	44	--	44
Al	mg/L	1.89E+02	2.01E+02	2.08E+02
Ba	mg/L	1.04E+00	7.51E-01	<3.27E-01
Ca	mg/L	7.75E-01	3.73E-01	1.25E+00
Cd	mg/L	5.82E-01	3.38E-01	<1.63E-01
Cr	mg/L	5.92E+00	5.79E+00	6.01E+00
Fe	mg/L	1.89E+00	1.59E+00	1.12E+00
K	mg/L	<1.76E+01	1.43E+01	1.63E+01
Li	mg/L	1.47E+00	1.48E+00	1.65E+00
Mg	mg/L	<7.05E-02	<5.33E-02	1.28E-01
Mn	mg/L	<4.70E-02	<9.77E-02	<1.12E-01
Mo	mg/L	<1.57E+00	<1.19E+00	<1.37E+00
Na	mg/L	2.31E+04	2.44E+04	2.68E+04
Ni	mg/L	<9.40E-01	<7.11E-01	<8.17E-01
P	mg/L	<9.63E+00	<7.29E+00	<8.38E+00
S	mg/L	<4.41E+01	<3.33E+01	<3.83E+01
Si	mg/L	3.13E+01	3.20E+01	3.37E+01
Sr	mg/L	<3.52E-02	<2.67E-02	<3.06E-02
U	mg/L	1.13E+02	7.25E+01	5.83E+01
Zr	mg/L	3.41E-01	1.73E-01	1.28E-01
Hg	mg/L	--	--	1.61E+00
Oxalate	mg/L	--	--	2.04E+02
m-234 (U)	mg/L	<2.21E-01	<4.44E-02	<1.63E-01
m-235 (U)	mg/L	7.91E-01	5.11E-01	3.95E-01
m-236 (U)	mg/L	<1.47E-01	<4.44E-02	<1.02E-01
m-237 (Np)	mg/L	<1.47E-01	<2.44E-01	<6.13E-02
m-238 (U,Pu)	mg/L	1.06E+02	7.48E+01	5.62E+01
m-239 (Pu)	mg/L	<2.21E-01	<6.66E-02	<1.23E-01
Pu-238	dpm/mL	6.99E+02	3.54E+02	7.68E+02
Pu-239/240	dpm/mL	<1.34E+03	3.81E+02	2.99E+02
Sr-90	dpm/mL	--	--	<3.04E+05
Cs-137	dpm/mL	--	--	1.79E+07
Am-241	dpm/mL	--	--	<4.06E+02
Cm-244	dpm/mL	--	--	3.35E+02

Table 3-32: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 3: Without Evaporation, Without Sludge Heel, Without pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	7.52	--	7.51
ORP	mV	232	--	260
Al	mg/L	6.92E-01	1.65E+00	7.47E-01
Ba	mg/L	9.27E-01	8.22E-01	<1.30E-01
Ca	mg/L	2.37E+00	1.82E+00	2.20E+00
Cd	mg/L	4.58E-01	3.95E-01	<6.02E-02
Cr	mg/L	5.05E+00	4.79E+00	4.42E+00
Fe	mg/L	2.07E+00	2.15E+00	8.67E-01
K	mg/L	1.25E+01	<1.20E+01	1.28E+01
Li	mg/L	2.68E+00	2.37E+00	2.40E+00
Mg	mg/L	1.58E+00	1.48E+00	1.40E+00
Mn	mg/L	8.08E-01	7.59E-01	4.70E-01
Mo	mg/L	<5.28E-01	<1.25E+00	<5.42E-01
Na	mg/L	3.20E+02	<6.89E+02	2.76E+02
Ni	mg/L	2.99E+00	2.02E+00	1.30E+00
P	mg/L	<3.21E+00	<7.66E+00	<3.29E+00
S	mg/L	1.88E+01	<3.50E+01	<1.51E+01
Si	mg/L	4.61E+00	4.81E+00	4.30E+00
Sr	mg/L	1.53E-01	1.21E-01	1.13E-01
U	mg/L	1.07E+02	9.31E+01	8.14E+01
Zr	mg/L	<4.89E-02	<1.21E-01	<5.02E-02
Hg	mg/L	--	--	<5.02E-01
Oxalate	mg/L	--	--	2.21E+02
m-234 (U)	mg/L	<1.84E-01	<4.67E-02	<1.61E-01
m-235 (U)	mg/L	6.79E-01	6.25E-01	5.83E-01
m-236 (U)	mg/L	<1.22E-01	<4.67E-02	<1.00E-01
m-237 (Np)	mg/L	<1.22E-01	<2.57E-01	6.82E-02
m-238 (U,Pu)	mg/L	9.87E+01	9.62E+01	8.35E+01
m-239 (Pu)	mg/L	<1.84E-01	8.87E-02	<1.20E-01
Pu-238	dpm/mL	4.20E+02	5.24E+02	1.48E+02
Pu-239/240	dpm/mL	<6.46E+02	1.49E+02	<7.29E+01
Sr-90	dpm/mL	--	--	1.06E+07
Cs-137	dpm/mL	--	--	1.30E+07
Am-241	dpm/mL	--	--	<1.39E+03
Cm-244	dpm/mL	--	--	7.38E+02

Table 3-33: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 4: With Evaporation, Without Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	54	--	63
Al	mg/L	4.26E+02	4.77E+02	5.03E+02
Ba	mg/L	8.38E-01	8.02E-01	<3.23E-01
Ca	mg/L	5.99E-01	3.94E-01	1.35E+00
Cd	mg/L	4.06E-01	3.92E-01	<1.62E-01
Cr	mg/L	3.95E+01	4.05E+01	4.33E+01
Fe	mg/L	1.84E+00	1.99E+00	1.63E+00
K	mg/L	8.73E+01	9.44E+01	9.64E+01
Li	mg/L	6.84E+00	7.23E+00	7.47E+00
Mg	mg/L	<6.10E-02	<5.70E-02	1.31E-01
Mn	mg/L	<4.06E-02	<1.04E-01	<1.11E-01
Mo	mg/L	2.91E+00	2.29E+00	2.74E+00
Na	mg/L	2.99E+04	2.85E+04	3.01E+04
Ni	mg/L	<8.13E-01	<7.59E-01	<8.09E-01
P	mg/L	1.31E+01	1.46E+01	1.49E+01
S	mg/L	4.34E+01	<3.56E+01	<3.79E+01
Si	mg/L	6.58E+01	6.78E+01	7.33E+01
Sr	mg/L	<3.05E-02	<2.85E-02	<3.03E-02
U	mg/L	1.50E+02	7.08E+01	6.01E+01
Zr	mg/L	2.44E-01	1.61E-01	<1.31E-01
Hg	mg/L	--	--	2.89E+00
Oxalate	mg/L	--	--	1.14E+03
m-234 (U)	mg/L	<1.91E-01	<4.75E-02	<1.62E-01
m-235 (U)	mg/L	9.70E-01	4.32E-01	3.39E-01
m-236 (U)	mg/L	<1.27E-01	<4.75E-02	<1.01E-01
m-237 (Np)	mg/L	<1.27E-01	<2.61E-01	<6.06E-02
m-238 (U,Pu)	mg/L	1.50E+02	7.50E+01	5.53E+01
m-239 (Pu)	mg/L	<1.91E-01	8.42E-02	<1.21E-01
Pu-238	dpm/mL	2.60E+02	2.61E+02	7.83E+02
Pu-239/240	dpm/mL	<3.90E+02	6.01E+02	5.26E+02
Sr-90	dpm/mL	--	--	<2.77E+05
Cs-137	dpm/mL	--	--	1.91E+08
Am-241	dpm/mL	--	--	<3.12E+02
Cm-244	dpm/mL	--	--	8.79E+01

Table 3-34: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 5: With Evaporation, With Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	54	--	64
Al	mg/L	4.72E+02	5.03E+02	5.32E+02
Ba	mg/L	7.64E-01	7.30E-01	<3.14E-01
Ca	mg/L	4.75E-01	3.54E-01	1.33E+00
Cd	mg/L	4.13E-01	4.22E-01	<1.57E-01
Cr	mg/L	4.01E+01	3.91E+01	4.17E+01
Fe	mg/L	2.26E+00	2.21E+00	1.78E+00
K	mg/L	8.63E+01	8.72E+01	9.08E+01
Li	mg/L	7.90E+00	7.69E+00	8.10E+00
Mg	mg/L	<5.33E-02	<5.18E-02	2.11E-01
Mn	mg/L	<3.55E-02	<9.50E-02	1.42E-01
Mo	mg/L	2.47E+00	2.13E+00	2.31E+00
Na	mg/L	3.28E+04	3.27E+04	3.24E+04
Ni	mg/L	<7.10E-01	<6.91E-01	<7.84E-01
P	mg/L	1.31E+01	1.19E+01	1.27E+01
S	mg/L	3.47E+01	<3.24E+01	3.85E+01
Si	mg/L	6.83E+01	6.76E+01	7.29E+01
Sr	mg/L	<2.66E-02	<2.59E-02	<2.94E-02
U	mg/L	1.60E+02	7.33E+01	6.14E+01
Zr	mg/L	3.86E-01	2.76E-01	2.50E-01
Hg	mg/L	--	--	5.11E+00
Oxalate	mg/L	--	--	1.21E+03
m-234 (U)	mg/L	<1.67E-01	<4.32E-02	<1.57E-01
m-235 (U)	mg/L	1.04E+00	4.79E-01	4.08E-01
m-236 (U)	mg/L	<1.11E-01	<4.32E-02	<9.80E-02
m-237 (Np)	mg/L	<1.11E-01	<2.38E-01	<5.88E-02
m-238 (U,Pu)	mg/L	1.52E+02	7.46E+01	5.80E+01
m-239 (Pu)	mg/L	<1.67E-01	8.98E-02	<1.18E-01
Pu-238	dpm/mL	1.07E+03	1.12E+03	1.76E+03
Pu-239/240	dpm/mL	<6.13E+02	4.91E+02	1.08E+03
Sr-90	dpm/mL	--	--	<2.95E+05
Cs-137	dpm/mL	--	--	9.72E+07
Am-241	dpm/mL	--	--	<4.79E+01
Cm-244	dpm/mL	--	--	3.54E+01

Table 3-35: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 6: With Evaporation, Without Sludge Heel, Without pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	9.27	--	9.02
ORP	mV	144	--	51.1
Al	mg/L	<1.57E+01	2.45E+01	2.49E+01
Ba	mg/L	6.90E-01	7.61E-01	<3.77E-01
Ca	mg/L	5.75E+00	5.98E+00	6.80E+00
Cd	mg/L	4.66E-01	4.11E-01	<1.89E-01
Cr	mg/L	5.31E+01	5.16E+01	5.27E+01
Fe	mg/L	1.04E+00	1.23E+00	8.90E-01
K	mg/L	8.48E+01	8.10E+01	7.51E+01
Li	mg/L	1.29E+01	1.10E+01	1.04E+01
Mg	mg/L	6.47E-01	4.26E-01	5.24E-01
Mn	mg/L	4.45E-01	6.81E-01	7.78E-01
Mo	mg/L	4.14E+00	2.45E+00	4.22E+00
Na	mg/L	3.06E+03	2.75E+03	2.78E+03
Ni	mg/L	1.15E+00	<8.01E-01	<9.43E-01
P	mg/L	<2.93E+00	<8.21E+00	<9.66E+00
S	mg/L	2.38E+01	<3.76E+01	<4.42E+01
Si	mg/L	4.45E+00	<2.20E+00	<2.59E+00
Sr	mg/L	3.41E-01	2.75E-01	3.10E-01
U	mg/L	2.50E+03	2.34E+03	2.46E+03
Zr	mg/L	<2.23E+00	<1.30E-01	<1.53E-01
Hg	mg/L	--	--	9.49E-01
Oxalate	mg/L	--	--	1.27E+03
m-234 (U)	mg/L	2.23E-01	<1.00E+00	<4.71E+00
m-235 (U)	mg/L	1.64E+01	1.49E+01	1.50E+01
m-236 (U)	mg/L	1.34E+00	<1.00E+00	<2.95E+00
m-237 (Np)	mg/L	<1.12E-01	<5.51E+00	<1.77E+00
m-238 (U,Pu)	mg/L	2.33E+03	2.44E+03	2.36E+03
m-239 (Pu)	mg/L	1.74E-01	1.96E+00	<3.53E+00
Pu-238	dpm/mL	1.59E+02	1.60E+02	1.89E+02
Pu-239/240	dpm/mL	<3.21E+02	<3.99E+02	8.70E+01
Sr-90	dpm/mL	--	--	1.87E+07
Cs-137	dpm/mL	--	--	9.28E+07
Am-241	dpm/mL	--	--	--
Cm-244	dpm/mL	--	--	--

Table 3-36: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 7: Without Evaporation, With Sludge Heel, With pH Adjustment, T = ~25 °C (ambient).

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	56	--	63.5
Al	mg/L	1.38E+02	1.55E+02	1.54E+02
Ba	mg/L	7.78E-01	7.30E-01	<3.19E-01
Ca	mg/L	4.77E-01	3.61E-01	1.26E+00
Cd	mg/L	3.68E-01	3.59E-01	<1.60E-01
Cr	mg/L	5.31E+00	5.49E+00	5.67E+00
Fe	mg/L	1.68E+00	1.61E+00	2.08E+00
K	mg/L	<1.43E+01	1.43E+01	1.62E+01
Li	mg/L	1.54E+00	1.68E+00	1.71E+00
Mg	mg/L	<5.73E-02	<5.15E-02	1.75E-01
Mn	mg/L	<3.82E-02	<9.44E-02	1.20E-01
Mo	mg/L	<1.28E+00	<1.15E+00	<1.34E+00
Na	mg/L	2.62E+04	2.51E+04	2.81E+04
Ni	mg/L	<7.64E-01	<6.87E-01	<7.98E-01
P	mg/L	<7.83E+00	<7.04E+00	<8.18E+00
S	mg/L	4.35E+01	<3.22E+01	<3.74E+01
Si	mg/L	2.39E+01	2.59E+01	2.73E+01
Sr	mg/L	<2.86E-02	<2.58E-02	<2.99E-02
U	mg/L	1.30E+02	1.03E+02	8.87E+01
Zr	mg/L	4.87E-01	3.05E-01	2.64E-01
Hg	mg/L	--	--	6.58E-01
Oxalate	mg/L	--	--	1.10E+02
m-234 (U)	mg/L	<1.79E-01	4.43E-02	<1.60E-01
m-235 (U)	mg/L	1.04E+00	6.71E-01	6.08E-01
m-236 (U)	mg/L	<1.19E-01	<4.29E-02	<9.97E-02
m-237 (Np)	mg/L	<1.19E-01	<2.36E-01	<5.98E-02
m-238 (U,Pu)	mg/L	1.33E+02	1.05E+02	8.55E+01
m-239 (Pu)	mg/L	<1.79E-01	8.84E-02	<1.20E-01
Pu-238	dpm/mL	8.55E+02	5.66E+02	1.07E+03
Pu-239/240	dpm/mL	<1.08E+03	3.71E+02	3.40E+02
Sr-90	dpm/mL	--	--	<3.13E+05
Cs-137	dpm/mL	--	--	1.56E+07
Am-241	dpm/mL	--	--	<2.66E+02
Cm-244	dpm/mL	--	--	6.60E+01

Table 3-37: Soluble Components in the Deposition Tank for Test 2 (Tank 5F No-Light), Storage Condition 8: Without Evaporation, With Sludge Heel, With pH Adjustment, T = 70 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	58	--	65.5
Al	mg/L	1.67E+02	1.77E+02	1.89E+02
Ba	mg/L	7.43E-01	7.59E-01	<3.08E-01
Ca	mg/L	5.68E-01	5.21E-01	1.50E+00
Cd	mg/L	4.07E-01	3.58E-01	<1.54E-01
Cr	mg/L	5.60E+00	5.55E+00	6.08E+00
Fe	mg/L	2.54E+00	1.86E+00	1.90E+00
K	mg/L	1.29E+01	1.36E+01	1.75E+01
Li	mg/L	1.79E+00	1.77E+00	1.99E+00
Mg	mg/L	<5.25E-02	8.54E-02	1.73E-01
Mn	mg/L	<3.50E-02	<9.89E-02	1.25E-01
Mo	mg/L	<1.17E+00	<1.20E+00	<1.29E+00
Na	mg/L	2.53E+04	2.50E+04	2.55E+04
Ni	mg/L	<7.00E-01	<7.19E-01	<7.70E-01
P	mg/L	<7.17E+00	<7.37E+00	<7.89E+00
S	mg/L	<3.28E+01	<3.37E+01	<3.61E+01
Si	mg/L	2.69E+01	2.73E+01	3.09E+01
Sr	mg/L	<2.62E-02	<2.70E-02	<2.89E-02
U	mg/L	7.27E+01	4.24E+01	3.93E+01
Zr	mg/L	3.28E-01	1.39E-01	<1.25E-01
Hg	mg/L	--	--	<4.81E-01
Oxalate	mg/L	--	--	1.73E+02
m-234 (U)	mg/L	<1.64E-01	<1.80E-02	<1.54E-01
m-235 (U)	mg/L	4.50E-01	2.88E-01	2.34E-01
m-236 (U)	mg/L	<1.09E-01	<1.80E-02	<9.63E-02
m-237 (Np)	mg/L	<1.09E-01	<9.89E-02	<5.78E-02
m-238 (U,Pu)	mg/L	6.85E+01	4.45E+01	3.36E+01
m-239 (Pu)	mg/L	<1.64E-01	3.87E-02	<1.16E-01
Pu-238	dpm/mL	1.98E+03	<6.10E+02	9.11E+02
Pu-239/240	dpm/mL	<2.81E+03	1.14E+03	5.15E+02
Sr-90	dpm/mL	--	--	<2.99E+05
Cs-137	dpm/mL	--	--	1.52E+07
Am-241	dpm/mL	--	--	<2.36E+02
Cm-244	dpm/mL	--	--	3.09E+01

Table 3-38: Soluble Components in the Deposition Tank for the Tank 5F Control (without ECC Processing): Without Evaporation, With Sludge Heel, With pH Adjustment, T = 50 °C.

analyte	units	deposition tank holding time		
		1 day	8 days	15 days
pH	--	>12	--	>12
ORP	mV	59	--	54.2
Al	mg/L	3.52E+02	3.80E+02	3.79E+02
Ba	mg/L	8.65E-01	7.57E-01	<2.93E-01
Ca	mg/L	6.86E-01	4.38E-01	1.16E+00
Cd	mg/L	4.65E-01	4.16E-01	<1.46E-01
Cr	mg/L	1.08E+00	1.06E+00	1.35E+00
Fe	mg/L	1.43E+00	1.34E+00	2.28E+00
K	mg/L	2.15E+01	1.73E+01	1.89E+01
Li	mg/L	<6.86E-01	<6.46E-01	<6.68E-01
Mg	mg/L	<5.64E-02	<5.31E-02	1.83E-01
Mn	mg/L	<3.76E-02	<9.74E-02	2.15E-01
Mo	mg/L	<1.26E+00	<1.19E+00	<1.23E+00
Na	mg/L	4.68E+04	4.20E+04	4.35E+04
Ni	mg/L	<7.52E-01	<7.08E-01	<7.32E-01
P	mg/L	<7.71E+00	6.73E+00	<7.50E+00
S	mg/L	3.84E+01	<3.32E+01	3.87E+01
Si	mg/L	4.41E+01	6.02E+01	6.36E+01
Sr	mg/L	<2.82E-02	<2.66E-02	<2.74E-02
U	mg/L	<1.53E+01	<1.44E+01	<1.49E+01
Zr	mg/L	<1.22E-01	<1.15E-01	<1.19E-01
Hg	mg/L	--	--	2.33E+00
Oxalate	mg/L	--	--	<9.14E+01
m-234 (U)	mg/L	<1.41E-02	<4.43E-03	<1.46E-01
m-235 (U)	mg/L	4.12E-02	4.74E-02	6.76E-02
m-236 (U)	mg/L	<9.40E-03	<4.43E-03	<9.14E-02
m-237 (Np)	mg/L	<9.40E-03	<2.43E-02	<5.49E-02
m-238 (U,Pu)	mg/L	5.91E+00	6.87E+00	6.70E+00
m-239 (Pu)	mg/L	<1.41E-02	8.71E-03	<1.10E-01
Pu-238	dpm/mL	<8.96E+02	<7.71E+02	7.38E+02
Pu-239/240	dpm/mL	<1.17E+03	9.56E+02	1.28E+02
Sr-90	dpm/mL	--	--	<2.69E+05
Cs-137	dpm/mL	--	--	2.49E+07
Am-241	dpm/mL	--	--	<9.69E+01
Cm-244	dpm/mL	--	--	2.62E+02

Table 3-39: Deposition Tank Storage Condition Summary for Soluble U-238 (mg/L)

Storage Condition	Temperature (°C)	Evaporation?	pH Adjust?	Sludge Heel?	U-238 (mg/L)					
					Test 1 (Tank 12H)			Test 2 (Tank 5F)		
					7 days	21 days	35 days	1 day	8 days	15 days
1	50	No	Yes	No	2.59E+01	2.53E+01	2.56E+01	1.22E+02	7.46E+01	4.15E+01
2	50	No	Yes	Yes	3.95E+01	3.88E+01	3.85E+01	1.06E+02	7.48E+01	5.62E+01
3	50	No	No	No	5.19E+00	5.32E+00	5.31E+00	9.87E+01	9.62E+01	8.35E+01
4	50	Yes	Yes	No	7.77E+01	6.99E+01	6.41E+01	1.50E+02	7.50E+01	5.53E+01
5	50	Yes	Yes	Yes	7.78E+01	6.79E+01	6.27E+01	1.52E+02	7.46E+01	5.80E+01
6	50	Yes	No	No	8.25E+01	1.94E+02	1.88E+02	2.33E+03	2.44E+03	2.36E+03
7	~25	*	Yes	Yes	6.36E+01	6.03E+01	5.75E+01	1.33E+02	1.05E+02	8.55E+01
8	70	*	Yes	Yes	6.01E+01	4.55E+01	4.45E+01	6.85E+01	4.45E+01	3.36E+01
9	50	No	Yes	Yes	1.40E+00	1.67E+00	1.81E+00	5.91E+00	6.87E+00	6.70E+00

* With evaporation for Test 1, Without evaporation for Test 2

Table 3-40: Deposition Tank Storage Condition Summary for Soluble Pu-238 (dpm/mL)

Storage Condition	Temperature (°C)	Evaporation?	pH Adjust?	Sludge Heel?	Pu-238 (dpm/mL)					
					Test 1 (Tank 12H)			Test 2 (Tank 5F)		
					7 days	21 days	35 days	1 day	8 days	15 days
1	50	No	Yes	No	2.24E+05	2.33E+05	2.78E+05	8.35E+02	8.12E+02	1.20E+02
2	50	No	Yes	Yes	4.62E+05	4.97E+05	5.34E+05	6.99E+02	3.54E+02	7.68E+02
3	50	No	No	No	1.87E+05	4.05E+04	1.93E+04	4.20E+02	5.24E+02	1.48E+02
4	50	Yes	Yes	No	3.55E+05	3.11E+05	3.25E+05	2.60E+02	2.61E+02	7.83E+02
5	50	Yes	Yes	Yes	3.44E+05	2.91E+05	2.52E+05	1.07E+03	1.12E+03	1.76E+03
6	50	Yes	No	No	1.35E+06	1.33E+06	1.86E+06	1.59E+02	1.60E+02	1.89E+02
7	~25	*	Yes	Yes	2.74E+05	2.06E+05	2.41E+05	8.55E+02	5.66E+02	1.07E+03
8	70	*	Yes	Yes	3.06E+05	1.83E+05	2.09E+05	1.98E+03	<6.10E+02	9.11E+02
9	50	No	Yes	Yes	2.32E+04	6.91E+03	5.26E+03	<8.96E+02	<7.71E+02	7.38E+02

* With evaporation for Test 1, Without evaporation for Test 2

Table 3-41: Deposition Tank Storage Condition Summary for Soluble Iron (mg/L)

Storage Condition	Temperature (°C)	Evaporation?	pH Adjust?	Sludge Heel?	Fe (mg/L)					
					Test 1 (Tank 12H)			Test 2 (Tank 5F)		
					7 days	21 days	35 days	1 day	8 days	15 days
1	50	No	Yes	No	1.08E+00	1.42E+00	1.50E+00	1.59E+00	1.57E+00	2.11E+00
2	50	No	Yes	Yes	3.82E+00	1.56E+00	4.62E+00	1.89E+00	1.59E+00	1.12E+00
3	50	No	No	No	<9.00E-01	8.65E-01	2.72E-01	2.07E+00	2.15E+00	8.67E-01
4	50	Yes	Yes	No	4.06E+00	2.99E+00	2.16E+00	1.84E+00	1.99E+00	1.63E+00
5	50	Yes	Yes	Yes	2.04E+00	2.43E+00	1.70E+00	2.26E+00	2.21E+00	1.78E+00
6	50	Yes	No	No	<9.28E-01	8.70E-01	2.67E-01	1.04E+00	1.23E+00	8.90E-01
7	~25	*	Yes	Yes	1.67E+00	2.11E+00	1.51E+00	1.68E+00	1.61E+00	2.08E+00
8	70	*	Yes	Yes	2.44E+00	1.92E+00	1.62E+00	2.54E+00	1.86E+00	1.90E+00
9	50	No	Yes	Yes	1.03E+00	1.78E+00	1.17E+00	1.43E+00	1.34E+00	2.28E+00

* With evaporation for Test 1, Without evaporation for Test 2

Table 3-42: Deposition Tank Storage Condition Summary for Soluble Aluminum (mg/L)

Storage Condition	Temperature (°C)	Evaporation?	pH Adjust?	Sludge Heel?	Al (mg/L)					
					Test 1 (Tank 12H)			Test 2 (Tank 5F)		
					7 days	21 days	35 days	1 day	8 days	15 days
1	50	No	Yes	No	2.16E+02	2.99E+02	3.33E+02	1.03E+02	1.16E+02	8.69E+01
2	50	No	Yes	Yes	4.67E+02	5.62E+02	7.05E+02	1.89E+02	2.01E+02	2.08E+02
3	50	No	No	No	<3.11E+00	<1.58E+00	1.08E+00	6.92E-01	1.65E+00	7.47E-01
4	50	Yes	Yes	No	1.25E+03	1.46E+03	1.58E+03	4.26E+02	4.77E+02	5.03E+02
5	50	Yes	Yes	Yes	1.19E+03	1.25E+03	1.48E+03	4.72E+02	5.03E+02	5.32E+02
6	50	Yes	No	No	9.56E+00	7.83E+00	7.63E+00	<1.57E+01	2.45E+01	2.49E+01
7	~25	*	Yes	Yes	6.83E+02	7.30E+02	7.45E+02	1.38E+02	1.55E+02	1.54E+02
8	70	*	Yes	Yes	1.46E+03	1.58E+03	1.95E+03	1.67E+02	1.77E+02	1.89E+02
9	50	No	Yes	Yes	4.57E+02	6.71E+02	8.42E+02	3.52E+02	3.80E+02	3.79E+02

* With evaporation for Test 1, Without evaporation for Test 2

Table 3-43: Analysis of Test 1 Deposition Tank Solids, Wet Basis

Storage Condition:		1	2	3	4	5	6	7	8	9
Ag	mg/kg	<1.43E+01	<1.40E+01	<1.39E+01	<1.40E+01	<1.43E+01	<1.38E+01	<1.43E+01	<1.40E+01	<1.43E+01
Al	mg/kg	1.40E+03	3.66E+03	1.19E+04	6.12E+03	4.89E+03	2.01E+04	6.62E+03	2.14E+03	5.30E+03
B	mg/kg	<1.98E+01	<1.93E+01	<1.93E+01	<1.93E+01	<1.98E+01	<1.91E+01	<1.98E+01	<1.93E+01	<1.98E+01
Ba	mg/kg	3.80E+01	6.30E+01	6.33E+01	7.32E+01	5.71E+01	8.85E+01	5.30E+01	2.52E+01	4.95E+01
Be	mg/kg	<7.86E-01	<7.65E-01	<7.63E-01	<7.65E-01	<7.84E-01	<7.56E-01	<7.84E-01	<7.65E-01	<7.84E-01
Ca	mg/kg	2.75E+02	4.62E+02	4.06E+02	5.17E+02	5.67E+02	6.01E+02	4.62E+02	2.18E+02	4.38E+02
Cd	mg/kg	3.54E+00	7.74E+00	6.01E+00	6.88E+00	7.65E+00	7.65E+00	8.82E+00	4.59E+00	1.14E+01
Ce	mg/kg	<6.95E+01	<6.76E+01	<6.74E+01	<6.76E+01	1.14E+03	<6.68E+01	1.19E+02	<6.76E+01	<6.93E+01
Cr	mg/kg	1.03E+01	1.14E+01	1.15E+01	5.98E+01	4.73E+01	6.34E+01	5.39E+01	6.50E+01	1.56E+01
Cu	mg/kg	5.16E+01	7.93E+01	8.03E+01	1.06E+02	7.40E+01	1.12E+02	7.33E+01	3.71E+01	6.64E+01
Fe	mg/kg	4.41E+03	7.23E+03	7.25E+03	9.08E+03	7.11E+03	1.04E+04	6.87E+03	3.26E+03	5.38E+03
Gd	mg/kg	<2.11E+01	<2.06E+01	<2.05E+01	<2.06E+01	<2.11E+01	<2.03E+01	<2.11E+01	<2.06E+01	<2.11E+01
K	mg/kg	<2.96E+02	<2.88E+02	<2.87E+02	<2.88E+02	<2.95E+02	<2.84E+02	<2.95E+02	<2.88E+02	<2.95E+02
La	mg/kg	1.88E+01	3.10E+01	3.13E+01	3.59E+01	3.09E+01	4.20E+01	2.73E+01	1.34E+01	5.01E+01
Li	mg/kg	<6.58E+00	7.17E+00	<6.39E+00	7.36E+00	<6.57E+00	8.60E+00	<6.57E+00	<6.41E+00	<6.57E+00
Mg	mg/kg	1.15E+02	2.01E+02	1.83E+02	2.15E+02	1.89E+02	2.58E+02	1.89E+02	8.67E+01	1.78E+02
Mn	mg/kg	2.48E+03	4.09E+03	4.26E+03	4.70E+03	3.54E+03	5.85E+03	3.21E+03	1.55E+03	2.48E+03
Mo	mg/kg	<2.21E+01	<2.15E+01	<2.15E+01	<2.15E+01	<2.21E+01	<2.13E+01	<2.21E+01	<2.15E+01	<2.21E+01
Na	mg/kg	2.52E+04	2.43E+04	<7.04E+02	4.09E+04	3.35E+04	2.72E+03	3.49E+04	4.06E+04	3.45E+04
Ni	mg/kg	3.05E+02	4.65E+02	4.63E+02	7.30E+02	5.46E+02	7.15E+02	5.73E+02	2.73E+02	5.16E+02
P	mg/kg	<1.61E+02	<1.57E+02	<1.56E+02	<1.57E+02	<1.61E+02	<1.55E+02	<1.61E+02	<1.57E+02	<1.61E+02
S	mg/kg	<7.37E+02	<7.17E+02	<7.16E+02	<7.17E+02	<7.35E+02	<7.09E+02	<7.35E+02	<7.17E+02	<7.35E+02
Sb	mg/kg	<1.05E+02	<1.02E+02	<1.02E+02	<1.02E+02	<1.05E+02	<1.01E+02	<1.05E+02	<1.02E+02	<1.05E+02
Si	mg/kg	1.03E+02	2.12E+02	4.70E+02	2.28E+02	2.39E+02	7.09E+02	2.16E+02	1.47E+02	9.48E+01
Sn	mg/kg	<5.51E+01	<5.36E+01	<5.35E+01	<5.36E+01	<5.50E+01	<5.30E+01	<5.50E+01	<5.36E+01	<5.50E+01
Sr	mg/kg	1.88E+01	3.04E+01	3.00E+01	3.62E+01	2.79E+01	4.22E+01	2.63E+01	1.26E+01	2.42E+01
Th	mg/kg	5.69E+02	8.93E+02	9.48E+02	1.14E+03	8.11E+02	1.30E+03	7.33E+02	3.57E+02	1.28E+03
Ti	mg/kg	1.97E+01	3.28E+01	2.30E+01	3.98E+01	2.85E+01	4.75E+01	2.30E+01	1.01E+01	1.24E+01
U	mg/kg	<2.94E+02	<2.87E+02	4.25E+02	8.22E+02	<2.94E+02	4.42E+02	6.68E+02	4.56E+02	6.61E+02
V	mg/kg	<4.62E+00	<4.49E+00	<4.48E+00	<4.49E+00	<4.61E+00	<4.44E+00	<4.61E+00	<4.49E+00	<4.61E+00
Zn	mg/kg	1.47E+01	2.71E+01	3.50E+01	3.40E+01	2.81E+01	5.18E+01	2.62E+01	1.56E+01	3.30E+01
Zr	mg/kg	1.35E+02	2.28E+02	2.34E+02	2.68E+02	2.01E+02	2.32E+02	1.86E+02	9.01E+01	1.72E+02
Co	mg/kg	5.06E+00	<4.78E+00	<4.77E+00	5.69E+00	5.17E+00	6.58E+00	<4.90E+00	<4.78E+00	<4.90E+00
Hg	mg/kg	2.33E+02	6.66E+02	1.11E+03	1.02E+03	9.66E+02	1.42E+03	9.72E+02	4.10E+02	2.22E+03
Pb	mg/kg	<9.82E+00	1.37E+01	<1.02E+01	1.48E+01	1.12E+01	<9.94E+00	<1.11E+01	2.03E+01	1.48E+01
m-230 (Th)	mg/kg	<2.95E+00	<2.87E+00	<2.86E+00	<2.87E+00	<2.94E+00	<2.84E+00	<2.94E+00	<2.87E+00	<2.94E+00
m-232 (Th)	mg/kg	4.73E+02	6.59E+02	9.08E+02	1.02E+03	7.53E+02	1.26E+03	6.38E+02	3.48E+02	1.24E+03
m-233 (U)	mg/kg	<1.96E+00	<1.91E+00	<1.91E+00	1.96E+00	<1.96E+00	<1.89E+00	<1.96E+00	<1.91E+00	<1.96E+00
m-234 (U)	mg/kg	<4.91E+00	<4.78E+00	<4.77E+00	<4.78E+00	<4.90E+00	<4.73E+00	<4.90E+00	<4.78E+00	<4.90E+00
m-235 (U)	mg/kg	<4.91E+00	<4.78E+00	9.82E+00	1.60E+01	1.08E+01	9.01E+00	1.44E+01	1.09E+01	8.62E+00
m-236 (U)	mg/kg	<1.96E+00	<1.91E+00	2.06E+00	<1.91E+00	<1.96E+00	<1.89E+00	<1.96E+00	<1.91E+00	<1.96E+00
m-237 (Np)	mg/kg	<1.96E+00	<1.91E+00	<1.91E+00	<1.91E+00	<1.96E+00	2.61E+00	<1.96E+00	<1.91E+00	<1.96E+00
m-238 (U,Pu)	mg/kg	3.63E+01	1.64E+02	5.51E+02	8.12E+02	6.53E+02	5.53E+02	7.87E+02	4.86E+02	8.05E+02
m-239 (Pu)	mg/kg	1.13E+01	1.28E+01	1.70E+01	2.27E+01	1.92E+01	2.66E+01	1.88E+01	1.22E+01	1.79E+01
m-240 (Pu)	mg/kg	<1.96E+00	<1.91E+00	2.04E+00	3.69E+00	2.38E+00	2.59E+00	2.86E+00	<1.91E+00	<1.96E+00
m-241 (Am,Pu)	mg/kg	<1.96E+00	<1.91E+00	<1.91E+00	<1.91E+00	<1.96E+00	<1.89E+00	<1.96E+00	<1.91E+00	<1.96E+00
Pu-238	dpm/mL	4.54E+07	5.91E+07	7.49E+07	9.56E+07	8.40E+07	1.04E+08	8.65E+07	4.21E+07	8.25E+07
Pu-239/240	dpm/mL	2.32E+06	3.28E+06	3.68E+06	4.37E+06	4.11E+06	5.05E+06	3.93E+06	2.03E+06	3.58E+06
Sr-90	dpm/mL	1.47E+09	2.85E+09	2.65E+09	2.54E+09	2.28E+09	3.15E+09	1.90E+09	1.07E+09	1.80E+09
Cs-137	dpm/mL	1.03E+06	2.68E+06	4.44E+06	7.48E+06	9.39E+06	2.01E+07	9.73E+06	1.07E+07	2.32E+06
Eu-154	dpm/mL	<2.02E+06	2.04E+06	2.03E+06	1.52E+06	2.37E+06	2.51E+06	2.29E+06	<9.20E+05	3.60E+06

Table 3-44: Analysis of Test 2 Deposition Tank Solids, Wet Basis

Storage Condition:		1	2	3	4	5	6	7	8	9
Ag	mg/kg	<1.44E+01	2.38E+01	2.80E+01	3.93E+01	3.80E+01	8.07E+01	<1.41E+01	<1.41E+01	4.74E+01
Al	mg/kg	3.65E+02	8.42E+02	2.89E+03	1.43E+03	1.76E+03	8.88E+03	7.89E+02	3.70E+02	1.86E+03
B	mg/kg	<1.99E+01	<2.00E+01	<1.97E+01	<1.96E+01	<1.95E+01	<2.00E+01	<1.95E+01	<1.95E+01	<1.96E+01
Ba	mg/kg	1.46E+02	2.68E+02	2.78E+02	3.14E+02	2.76E+02	7.16E+02	1.21E+02	1.33E+02	1.58E+02
Be	mg/kg	<7.88E-01	<7.90E-01	<7.78E-01	<7.77E-01	<7.74E-01	7.92E-01	<7.72E-01	<7.71E-01	<7.77E-01
Ca	mg/kg	3.13E+02	5.93E+02	2.40E+02	7.68E+02	7.96E+02	1.59E+03	3.35E+02	4.00E+02	5.38E+02
Cd	mg/kg	6.99E+00	1.43E+01	9.83E+00	1.48E+01	1.60E+01	2.81E+01	7.43E+00	9.44E+00	1.63E+01
Ce	mg/kg	<6.96E+01	<6.99E+01	<6.88E+01	<6.86E+01	<6.84E+01	<7.00E+01	<6.83E+01	<6.81E+01	8.80E+01
Cr	mg/kg	1.49E+01	2.08E+01	4.18E+01	5.99E+01	6.11E+01	1.08E+02	1.55E+01	1.58E+01	5.11E+01
Cu	mg/kg	4.34E+01	7.26E+01	4.99E+01	1.28E+02	1.35E+02	1.56E+02	3.98E+01	4.85E+01	7.71E+01
Fe	mg/kg	1.74E+04	2.65E+04	1.87E+04	4.61E+04	4.47E+04	8.04E+04	1.40E+04	1.64E+04	1.87E+04
Gd	mg/kg	<2.12E+01	3.45E+01	2.41E+01	6.73E+01	7.00E+01	7.93E+01	<2.08E+01	2.22E+01	2.45E+01
K	mg/kg	<2.96E+02	<2.97E+02	<2.93E+02	<2.92E+02	<2.91E+02	4.40E+02	<2.90E+02	<2.90E+02	<2.92E+02
La	mg/kg	2.96E+01	4.39E+01	3.64E+01	6.76E+01	6.72E+01	1.18E+02	2.42E+01	2.84E+01	1.23E+02
Li	mg/kg	2.21E+01	4.01E+01	2.49E+01	8.87E+01	8.51E+01	1.53E+02	2.20E+01	2.11E+01	3.75E+01
Mg	mg/kg	4.24E+01	1.04E+02	4.87E+01	1.20E+02	1.49E+02	2.05E+02	5.99E+01	7.20E+01	1.65E+02
Mn	mg/kg	4.93E+03	9.06E+03	9.54E+03	1.01E+04	8.64E+03	2.45E+04	4.01E+03	4.35E+03	6.61E+03
Mo	mg/kg	<2.22E+01	<2.22E+01	<2.19E+01	<2.18E+01	<2.18E+01	<2.23E+01	<2.17E+01	<2.17E+01	<2.18E+01
Na	mg/kg	2.34E+04	2.48E+04	<7.18E+02	2.91E+04	3.28E+04	6.30E+03	2.52E+04	2.45E+04	4.42E+04
Ni	mg/kg	6.24E+02	9.30E+02	8.24E+02	1.38E+03	1.39E+03	2.40E+03	4.73E+02	5.47E+02	3.55E+03
P	mg/kg	<1.61E+02	<1.62E+02	<1.60E+02	<1.59E+02	<1.59E+02	<1.62E+02	<1.58E+02	<1.58E+02	<1.59E+02
S	mg/kg	<7.38E+02	<7.41E+02	<7.30E+02	<7.28E+02	<7.25E+02	<7.43E+02	<7.24E+02	<7.23E+02	<7.28E+02
Sb	mg/kg	<1.05E+02	<1.05E+02	<1.04E+02	1.19E+02	1.19E+02	2.34E+02	<1.03E+02	<1.03E+02	<1.04E+02
Si	mg/kg	4.85E+02	7.12E+02	7.57E+02	1.59E+03	1.52E+03	1.07E+03	3.55E+02	3.96E+02	4.57E+02
Sn	mg/kg	<5.52E+01	<5.54E+01	<5.46E+01	<5.45E+01	<5.42E+01	<5.55E+01	<5.42E+01	<5.41E+01	<5.45E+01
Sr	mg/kg	6.33E+01	1.14E+02	1.02E+02	1.54E+02	1.37E+02	3.42E+02	5.33E+01	6.05E+01	6.80E+01
Th	mg/kg	5.50E+01	8.48E+01	<5.21E+01	1.40E+02	1.36E+02	2.75E+02	<5.17E+01	<5.15E+01	<5.19E+01
Ti	mg/kg	1.09E+01	1.57E+01	<3.70E+00	2.92E+01	3.10E+01	4.67E+01	1.00E+01	1.09E+01	1.17E+01
U	mg/kg	6.54E+03	1.11E+04	8.09E+03	2.19E+04	2.26E+04	2.78E+04	5.40E+03	7.07E+03	7.44E+03
V	mg/kg	<4.63E+00	<4.64E+00	<4.57E+00	<4.56E+00	<4.54E+00	<4.65E+00	<4.54E+00	<4.53E+00	<4.56E+00
Zn	mg/kg	7.58E+00	1.38E+01	6.71E+00	2.04E+01	2.43E+01	2.70E+01	8.88E+00	9.83E+00	4.17E+01
Zr	mg/kg	2.45E+02	3.77E+02	1.91E+02	8.10E+02	7.63E+02	1.61E+03	2.06E+02	2.44E+02	3.31E+02
Co	mg/kg	<3.94E+00	6.81E+00	9.24E+00	8.38E+00	9.48E+00	1.13E+01	<3.86E+00	<3.85E+00	1.88E+01
Hg	mg/kg	7.95E+01	4.48E+02	1.65E+02	3.04E+02	6.15E+02	5.88E+02	2.99E+02	2.47E+02	1.03E+03
Pb	mg/kg	<2.42E+01	4.22E+01	<3.14E+01	6.40E+01	6.90E+01	1.04E+02	<2.32E+01	<2.56E+01	4.46E+01
m-230 (Th)	mg/kg	<7.87E+00	<7.91E+00	<7.78E+00	<7.77E+00	<7.74E+00	<7.92E+00	<7.72E+00	<7.71E+00	<7.77E+00
m-232 (Th)	mg/kg	3.93E+01	6.74E+01	3.59E+01	1.21E+02	1.24E+02	2.06E+02	3.68E+01	4.95E+01	2.35E+01
m-233 (U)	mg/kg	<7.87E+00	<7.91E+00	<7.78E+00	<7.77E+00	<7.74E+00	<7.92E+00	<7.72E+00	<7.71E+00	<7.77E+00
m-234 (U)	mg/kg	<5.91E+00	<5.93E+00	<5.84E+00	<5.83E+00	<5.80E+00	<5.94E+00	<5.79E+00	<5.78E+00	<5.83E+00
m-235 (U)	mg/kg	4.04E+01	7.96E+01	5.25E+01	1.48E+02	1.55E+02	1.74E+02	3.46E+01	4.85E+01	4.75E+01
m-236 (U)	mg/kg	<3.94E+00	4.67E+00	<3.89E+00	7.34E+00	7.58E+00	7.27E+00	<3.86E+00	<3.85E+00	<3.88E+00
m-237 (Np)	mg/kg	<7.87E+00	<7.91E+00	<7.78E+00	<7.77E+00	<7.74E+00	1.17E+01	<7.72E+00	<7.71E+00	<7.77E+00
m-238 (U,Pu)	mg/kg	6.33E+03	1.18E+04	7.58E+03	2.31E+04	2.38E+04	2.73E+04	5.12E+03	7.15E+03	7.07E+03
m-239 (Pu)	mg/kg	7.17E+00	1.01E+01	7.32E+00	1.34E+01	1.48E+01	2.69E+01	6.99E+00	5.79E+00	1.27E+01
m-240 (Pu)	mg/kg	<3.94E+00	<3.95E+00	<3.89E+00	<3.88E+00	<3.87E+00	<3.96E+00	<3.86E+00	<3.85E+00	<3.88E+00
m-241 (Am,Pu)	mg/kg	<3.94E+00	<3.95E+00	<3.89E+00	<3.88E+00	<3.87E+00	<3.96E+00	<3.86E+00	<3.85E+00	<3.88E+00
Pu-238	dpm/mL	1.33E+08	8.54E+08	1.36E+08	3.47E+08	1.02E+09	5.81E+08	5.66E+08	7.32E+08	2.87E+09
Pu-239/240	dpm/mL	7.07E+07	1.45E+08	8.58E+07	2.17E+08	2.34E+08	4.11E+08	7.37E+07	9.77E+07	2.25E+08
Sr-90	dpm/mL	<4.72E+09	<8.10E+09	<1.01E+10	<9.71E+09	<1.04E+10	<2.43E+10	<4.51E+09	<4.77E+09	<4.74E+09
Co-60	dpm/mL	<6.06E+05	3.93E+05	3.50E+05	4.08E+05	4.79E+05	9.83E+05	<4.88E+05	<5.99E+05	1.10E+06
Cs-137	dpm/mL	1.82E+07	2.18E+07	1.40E+08	7.02E+07	9.80E+07	8.14E+08	1.66E+07	1.72E+07	6.28E+07
Eu-154	dpm/mL	--	--	--	--	--	--	--	--	5.05E+06
Am-241	dpm/mL	--	--	--	--	--	--	--	--	7.83E+06

3.5 Treatment Tank Sludge Physical and Flow Properties

Measurement of the flow properties and behavior of the solids during and after the dissolution process in the Treatment Tank contributes to the understanding of how the solids may be transferred out of the tank. As a background for the ECC process, SRR has theorized that over the course of sludge heel chemical cleaning by multiple batches of heated OA, the particle size distribution shifts toward smaller particle sizes and the rheology becomes more favorable for the suspension and transfer of undissolved solids out of the tank.

3.5.1 *Rheology*

Baseline, midpoint, and endpoint Tank 5F and 12H slurry samples were prepared and characterized as described in detail in the Experimental Section. Baseline samples correspond to the original sludge materials. Rheology on the baseline samples was conducted after exposure of the sludge to 2 wt% OA at acid:sludge mass phase ratios of approximately 3 and 7 (correspond to sludge solids of 13 and 26 wt%, respectively). Midpoint and endpoint sludge samples were intended to represent sludge in the treatment tank near the middle and end (respectively) of the ECC process. These samples were generated by exposing the sludge samples to two successive contacts with 2 wt. % OA using an acid:sludge mass ratio of approximately 50:1 for each acid contact. Acid contacts were conducted without agitation at 70 °C. Based on previous testing, it was expected that these conditions would result in partial dissolution of the sludge samples while leaving sufficient residual materials for characterization. Seven days was used for the first OA contact (midpoint) and 3 days was used for the second OA contact (endpoint). For many samples, rheology testing was conducted at two solids loading levels referred to as dilute and concentrated. Rheology testing for the midpoint and endpoint samples was conducted in the spent OA solutions used for the dissolution batch contacts. Weight percent solids and particle size measurements were conducted on sub-samples of each sample type in order to aid in the interpretation of the rheology data.

For comparison, rheology measurements were conducted using the same equipment on 2 wt% OA (0.224 M) solution containing no dissolved sludge materials across the temperature range of interest. The results are provided in Table 3-45. As expected, 2 wt% OA solution is thin and exhibits ideal Newtonian behavior with no yield stress. Yield stress values within ± 0.5 Pa of zero should be treated as zero values for this instrument. As the solution temperature was increased from 30 to 50 °C, the viscosity decreased by approximately 19% from 1.79 to 1.45 cP. A viscosity approaching 2 cP at 30 °C is significantly higher than expected for 2 wt% OA. At 20 °C the viscosity of 2 wt% OA is reported to be 1.04 cP.¹⁸ Based on this result and the previously discussed issues with adjusting the instrumental zero point (Section 2.3), this instrument and experimental methodology are not believed to be highly accurate for the measurement of low viscosity fluids. For comparison, the viscosity of water decreases from 0.80 to 0.65 cP as the temperature is increased from 30 to 50 °C.¹⁸

Rheology data for the baseline, midpoint, and endpoint Tank 5F/OA slurries is provided in Table 3-46. For all Tank 5F treatment tank samples, most of the solids settled quickly (within 5 minutes) and the slurries were not well suited for rheological measurement. Nonetheless, rheology data was collected on the baseline material at two solids loadings and three temperatures. Although the dilute and concentrated Tank 5F baseline slurry samples were known

to contain 15 and 28 wt% total solids, respectively, the suspendable solids were determined to be <5 wt% for both samples (see Table 3-43). Furthermore, the measured viscosities for both the dilute and concentrated samples of 1.6 to 2.1 cP were only slightly higher than was observed for OA acid at the same temperatures and the yield stress values were all near zero. The results are consistent with visual observations regarding particle settling and indicate that the rheology of the baseline material was representative of the liquid phase alone rather than a solid suspension. Presumably, the Tank 5F baseline slurry characteristics were dominated by iron oxide and oxyhydroxide phases, which are known to be primary chemical constituents of this sludge material. It appears based on these results that the removal of these dense and rapidly settling phases from the tanks will be best accomplished by dissolution rather than suspension. Fortunately, the iron phases tend to dissolve well in OA. The settling characteristics of the midpoint and endpoint samples were similar to the baseline material. As a result, rheology was only conducted on these materials at a single solids loading and temperature. The viscosities of both samples were in the range observed for the baseline material.

Rheology data for the Tank 12H/OA slurries is provided in Table 3-47. The initial baseline Tank 12H slurry samples were observed to contain particles which settled quickly as well as particles which remained suspended. Sub-samples of the dilute and concentrated slurries collected within one minute after sample agitation were determined to contain 17 and 25 wt. % total solids, respectively. All of the dilute baseline Tank 12H slurry samples exhibited Newtonian behavior with essentially no yield stress. However, later samples analyzed after aging for an additional day (see second set of results for Baseline Dilute material at 30 and 40 °C) showed signs of thickening and better particle suspension. Similarly, the concentrated baseline slurry was observed to exhibit non-Newtonian behavior characteristic of a Bingham Plastic material in some later measurements (T = 40 and 50 °C). Since this behavior appeared to be increasing with time, the baseline concentrated slurry sample was left over the weekend to allow time for sample stabilization. Subsequent rheology data collected at 30 °C on the concentrated sample after 4 days of aging revealed that the viscosity (6.3 cP) and yield stress (4.9 Pa) had increased considerably relative to initial measurements at this temperature.

Rheology data was also collected for the Tank 12H midpoint and endpoint slurries at three temperatures and two solids loadings. Due to sample size limitations, it was not possible to prepare slurries at total solids loading levels greater than 10 weight percent. The midpoint concentrate sample contained the highest solids loading of any sample (9.6 wt. %) and exhibited the highest viscosity and yield stress. The stress versus strain curve for one rheology measurement at 40 °C on the midpoint concentrate sample is provided in Figure 3-10. The rheological characteristics of this sample were typical of a Bingham Plastic material and the average viscosity and yield stress values for duplicate measurements were 6.6 cP and 5.5 Pa, respectively. Temperature variations had minimal effects on the rheological properties across the range of interest (30-50 °C). The rheological properties were primarily affected by the solids loading. At total solids loadings <5 wt. % the Tank 12H slurries also behaved as Bingham Plastic materials. The stress versus strain curve for the Endpoint Concentrate sample is provided in Figure 3-11. The midpoint dilute and endpoint concentrate samples serendipitously contained similar weight percent total solids (5-6 wt. %) and had similar viscosities (2-3 cP) and yield stress (0.6-0.8 Pa) values. These results indicate that the rheological properties of the Tank 12H slurry changed little between the midpoint and endpoint samples. In contrast, very high weight percent solids levels in the baseline materials only resulted in moderate viscosity and yield stress values. This characteristic of the baseline slurry may have been associated with the fact that the sludge sample had been dried prior to testing. The rheological properties of the Tank 12H slurries are presumably dominated by aluminum phases which are known to be the primary chemical constituents. In contrast to the solids in the Tank 5F slurry, the Tank 12H solids form

suspensions which are somewhat stable over the timescale of rheology measurements. These solids are therefore expected to be more suitable for suspension within and transfer from the waste tanks.

Table 3-45: Rheology Data for 2 wt% OA Solution at Various Temperatures

Temperature (°C)	Viscosity (cP)	Yield Stress (Pa)
30	1.79	-0.25
40	1.61	-0.25
50	1.45	-0.25

Table 3-46: Rheology Data for Tank 5F Sludge/OA (2 wt%) Slurry

Sample ^a	Total Solids (Wt. %) ^b	Temperature (°C)	Viscosity (cP)	Yield Stress (Pa)
Baseline Dilute ^c	2.8	30	2.10	-0.31
Baseline Dilute ^c	2.8	40	1.89	-0.28
Baseline Dilute ^c	2.8	50	1.62	-0.27
Baseline Concentrate ^c	4.2	30	2.16	-0.30
Baseline Concentrate ^c	4.2	40	2.04	-0.26
Baseline Concentrate ^c	4.2	50	1.81	-0.26
Midpoint	NA	30	1.76	-0.30
Endpoint	NA	30	2.13	-0.30

^a significant settling observed on analysis timescale for all samples

^b represents total suspendable and dissolved solids; as-prepared Baseline dilute and concentrate samples contained 15 and 28 wt. % total solids, respectively, based on added sludge and accounting for dissolved oxalic acid

^c solution contained 3.6 wt. % soluble solids

Table 3-47: Rheology Data for Tank 12H Sludge/OA (2 wt%) Slurry

Sample	Total Solids ^a (Wt. %)	Temperature (°C)	Viscosity (cP)	Yield Stress (Pa)
Baseline Dilute ^b	16.8	30	2.26	-0.31
Baseline Dilute ^b	16.8	40	2.26	-0.31
Baseline Dilute ^b	16.8	50	2.38	-0.20
Baseline Dilute ^c	16.8	30	2.45	0.07
Baseline Dilute ^c	16.8	40	2.16	0.19
Baseline Concentrate ^b	25.2	30	2.56	0.42
Baseline Concentrate ^b	25.2	40	2.77	0.85
Baseline Concentrate ^b	25.2	50	3.16	1.37
Baseline Concentrate ^d	25.2	30	6.27	4.93
Midpoint Dilute ^e	5.7	30	2.68	0.67
Midpoint Dilute ^e	5.7	40	2.34	0.78
Midpoint Dilute ^e	5.7	50	2.33	0.77
Midpoint Concentrate	9.6	30	6.52	5.37
Midpoint Concentrate	9.6	40	6.58	5.52
Midpoint Concentrate	9.6	50	6.90	6.20
Endpoint Dilute	3.3	30	2.16	0.01
Endpoint Dilute	3.3	40	1.95	0.07
Endpoint Dilute	3.3	50	1.97	0.10
Endpoint Concentrate	5.0	30	2.65	0.61
Endpoint Concentrate	5.0	40	2.43	0.67
Endpoint Concentrate	5.0	50	2.26	0.77

^a represents total suspendable and dissolved solids; as-prepared Baseline dilute and concentrate samples contained 15 and 28 wt. % total solids, respectively, based on added sludge and accounting for dissolved oxalic acid

^b significant settling observed on analysis timescale

^c after 1 day of storage at ambient temperature

^d after 4 days storage at ambient temperature

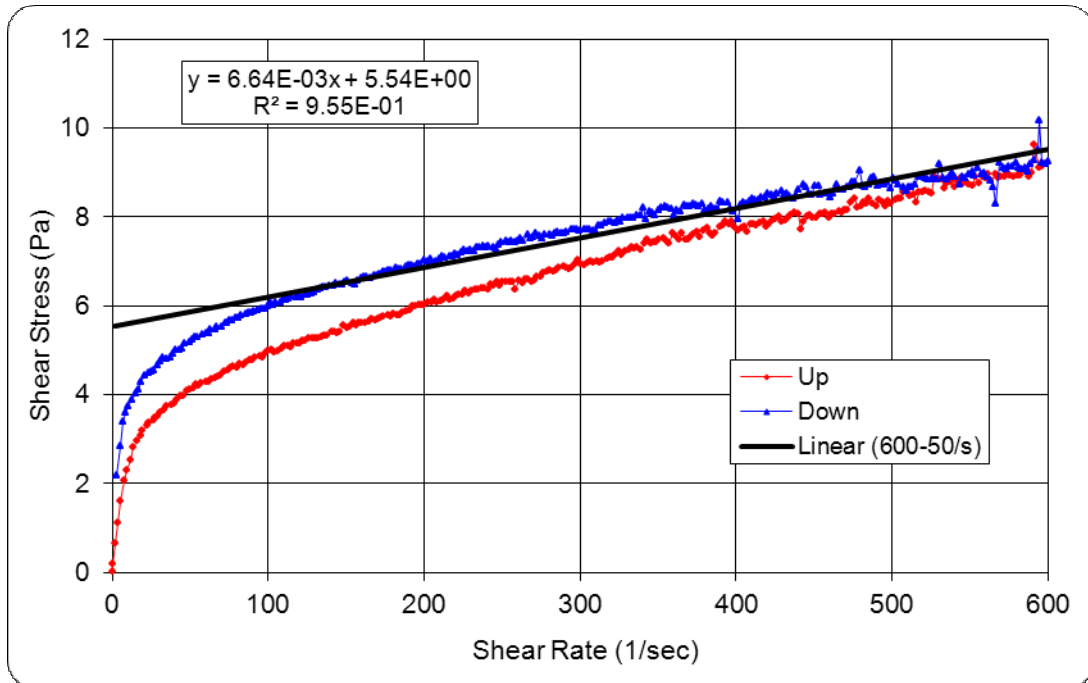


Figure 3-10: Rheology Results for the Concentrated Tank 12H Slurry Midpoint Sample at 40 °C.

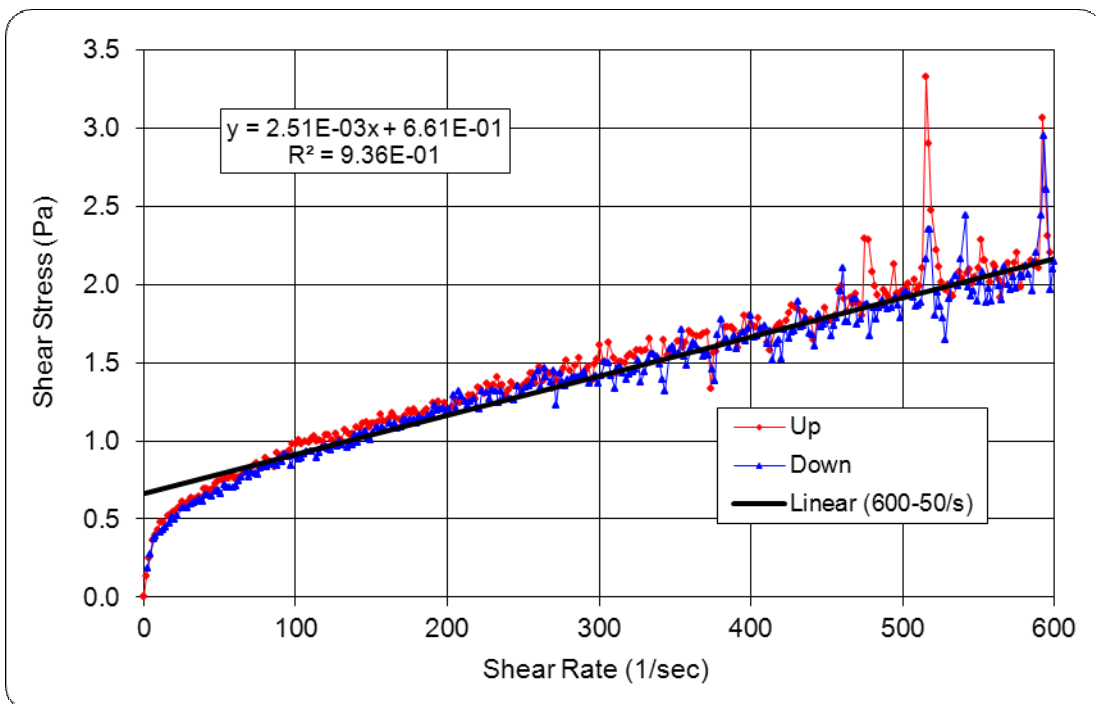


Figure 3-11: Rheology Results for the Concentrated Tank 12H Slurry Endpoint Sample at 40 °C.

3.5.2 Particle size

Individual sub-samples of the baseline, midpoint, and endpoint Tank 5F and 12H materials were submitted for particle size analysis. Complete analysis results for each sample are provided in Appendix B (Figure B-75 through Figure B-80). The Tank 5F particles were larger (mean volume range: 37-118 μm) than the Tank 12H particles (mean volume range: 3.7-11.7 μm). The baseline Tank 5F material was bimodal with a significant population of particles in the 1-10 μm range. The midpoint and endpoint Tank 5F samples contained a smaller fraction of particles below 10 μm . Trends in the distributions also indicate that the larger particles were dissolving in the Tank 5F slurry as a result of OA contact. The mean volume observed for the endpoint sample was below 40 μm with few particles (<10 percentile) greater than 100 μm . Evidence for the dissolution of smaller particles was also observed for the Tank 12H sample. The baseline material contained a significant number of particles below 1 μm (25 percentile), while the midpoint and endpoint samples contained smaller fractions of particles below 1 μm . Little difference was observed between the midpoint and endpoint Tank 12H samples, which is consistent with the fact that little sludge dissolution occurred during the second and third ECC process batches for Test 1 (Section 3-3).

3.5.3 Settling

As stated previously, Tank 5F sludge showed quick settling both prior to and after partial dissolution with OA. A portion of the solids settled quickly enough that difficulty was encountered removing the solids from the dissolution apparatus. For example, when a bottle with the residual solids is agitated and poured, the solids are large enough that they do not stay suspended long enough to be poured out of the bottle. Figure 3-12 contains a picture of the material after the first OA dissolution for the Treatment Tank rheology test. On the left is a cylinder with the portion that was able to be poured from the bottle. Within 8 minutes, the major sludge level was at around 50% of the height of the liquid in the cylinder. The residual materials in the bottle on the right are a significant portion of the quickly settling solids that could not be poured from the bottle.



Figure 3-12: Tank 5F Sludge Intermediate Dissolution Sample, a Graduated Cylinder with the Fluid Portion After 8 Minutes of Settling (left) and the Fast-Settling Portion Not Removed From the Sample Bottle (right)

3.6 ECC Product Sludge Physical and Flow Properties

The following are the expectations held prior to investigating the physical properties of the products of the ECC process. The particle size of the recently precipitated sludge at nearly neutral pH might be expected to be very small in comparison with typical Tank Farm sludge, and thus may not settle as quickly as typical high-pH sludge. The solids concentration of the ECC reactor product slurry was low, and might be expected to have a relatively low viscosity and nearly Newtonian rheology. Slurries leaving the evaporator would have higher solids concentrations than those prior to evaporation, so changes in flow behavior might be expected. However, it was expected that the rheology would still be favorable to pumping. After hydroxide adjustment, the particle size was expected to be large enough for the sludge solids to settle quickly so that outages would not be required to provide deposition tank settling time. The testing was designed to either confirm or contradict these expectations.

3.6.1 *Rheology*

Rheology data for the Tank 5F and 12H Deposition Tank samples are provided in Table 3-4. The rheology measurements were performed for Storage Conditions 3 and 6, which correspond to the unadjusted material from the ECC reactor without and with evaporation, respectively. Note that the rheology was measured only after the completion of the Deposition Tank testing, so some changes may have occurred during the several weeks of storage at 50°C. The rheology was measured on the suspended slurry from the storage bottle with no additional solid/liquid separation. Weight percent solids measurements were not conducted on these samples, but it was known based on the sample preparation details that these samples contained low solids levels. Partial settling of the solids was observed during all rheology measurements for the Tank 5F deposition samples and during one measurement with the Tank 12H samples. As expected based

on the low solids levels in the samples, all measured viscosities were low (2-3 cP) and no samples exhibited a significant yield stress. The measured viscosities for these samples was only slightly higher than was observed for pure OA (Table 3-1). Deposition Tank storage conditions that included evaporation for removal of 85% of the water added during the ECC dissolution process did not result in slurries that exhibited significant yield stresses.

Table 3-4. Rheology Data for Tank 5F and 12H Deposition Tank Samples

Sample	Temperature (°C)	Viscosity (cP)	Yield Stress (Pa)
Tank 12 No Evaporation (T1SC3)	30	1.88	-0.29
Tank 12 No Evaporation (T1SC3) ^a	45	1.65	-0.24
Tank 12 Evaporated (T1SC6)	30	1.88	-0.29
Tank 12 Evaporated (T1SC6)	45	2.26	-0.30
Tank 5 No Evaporation (T2SC3) ^a	30	2.03	-0.31
Tank 5 No Evaporation (T2SC3) ^a	45	1.66	-0.28
Tank 5 Evaporated (T2SC6) ^a	30	1.80	-0.28
Tank 5 Evaporated (T2SC6) ^a	45	2.15	-0.32

^a partial settling observed on analysis timescale

3.6.2 Particle size

Particle size measurements for Deposition Tank storage bottle samples are included in Appendix B. Note that the particle size analysis was performed only after the completion of the Deposition Tank testing, so some changes may have occurred during the several weeks of storage at 50°C.

Figure B-75 and Figure B-76 show the particle size analyses for the Tank 5F and 12H sample material, respectively, at Treatment Tank conditions. From these results it is clear that the initial Tank 12H material has a narrower size range with the volume percentage centering around 2 µm, while the initial Tank 5F material has a very large particle size range centering around 80 µm. Figure B-84 and Figure B-88 contain the Deposition Tank control samples for Tank 12H and 5F materials, respectively, and are consistent with the Treatment Tank analysis with the addition of another solids source with a relatively large particle size (from the sludge heel addition). Figure B-81, Figure B-83, and Figure B-82 show the Tank 12H ECC Reactor product at positions B, C, and D of Figure 2-6, respectively. The Tank 12H ECC product is consistently narrow in particle size range centering around 2 µm, with the added sludge heel evident in Figure B-82 as a completely separate narrow distribution centering around ~200 µm. Figure B-86 and Figure B-87 show the Tank 5F ECC Reactor product at positions B and C of, respectively. However, Figure B-85 does not correspond directly to Figure 2-6 because the nominal F-Area Deposition Tank condition does not include evaporation. Unadjusted Tank 5F ECC product that was not evaporated had a particle size distribution that centered below 2 µm, while the evaporated and the not evaporated pH adjusted products had particle size distributions that centered around 3 µm. Despite the relatively large particle size of the initial Tank 5F sludge heel material, the Tank 5F ECC Reactor product was comparable to that of Tank 12H.

3.6.3 Settling

A visual settling test was performed for the unadjusted ECC reactor products (Tests 1 and 2, Storage Condition 3), the nominally adjusted products (Test 1 Storage Condition 5 and Test 2 Storage Condition 2) and the adjusted controls (Tests 1 and 2, Storage Condition 9). Figure 3-13 shows an image of the storage bottles for these conditions after 8 hours of settling. A larger series of such photographs from 0 hours of settling through 150 hours of settling are included in Appendix B, Figure B-89 through Figure B-92.

By the one and two hour settling observations, layers of solids were evident for all bottles except for the unadjusted Test 2 ECC product (Test 2 Storage Condition 3). By the six and eight hour observations of the settling, all supernatant liquids showed significant transparency except for the unadjusted Test 2 ECC product (Test 2 Storage Condition 3). By comparing the adjusted ECC products with the adjusted control samples, settling behavior is comparable. Thus, settling of adjusted ECC products is not expected to differ from that of Tank 5F or Tank 12H sludge. However, unadjusted Tank 5F ECC product may require adjustment if normal sludge settling behavior is desired.

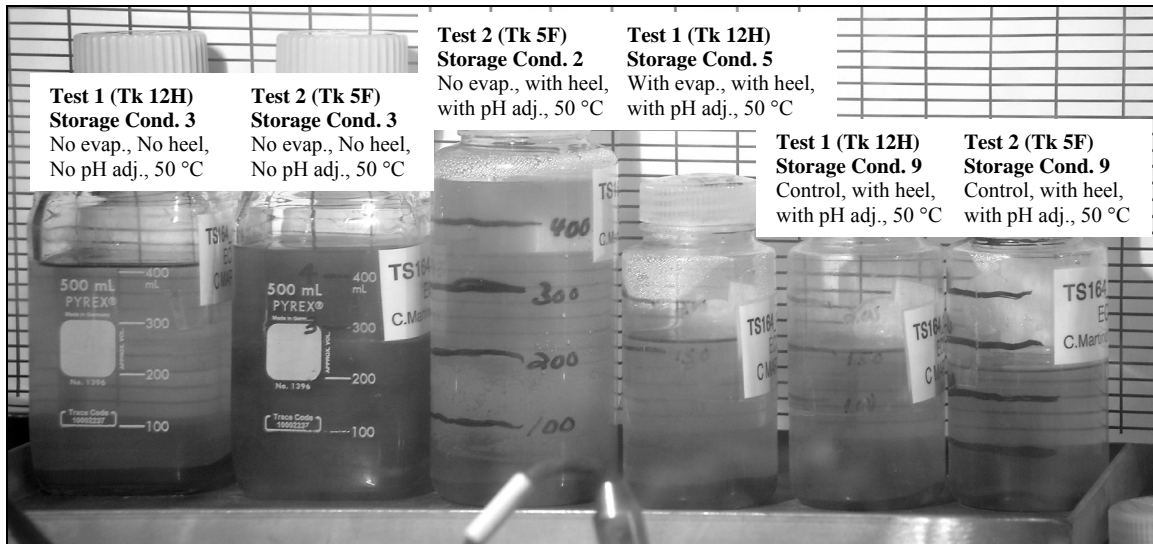


Figure 3-13: ECC Products after 8 Hours of Settling

4.0 Conclusions

SRNL conducted two tests using actual SRS waste material from Tanks 5F and 12H. Testing involved sludge dissolution with an initial 2 wt% OA, the decomposition of the oxalates by ozonolysis, the evaporation of a portion of the material for water removal, and tracking the concentrations of key components in the Deposition Tank. The UV light was not utilized in the decomposition testing. The following are results of the ECC RWT:

- Using three OA batches per test, Test 1 showed an approximately 73% removal of Tank 12H sludge and Test 2 showed an approximately 80% removal of Tank 5F sludge. Removal includes both dissolution of soluble components and partial transfer of insoluble solids. The dissolution process was constrained to eight hours per OA batch and thus did not precisely represent the in tank dissolution process.
- Residuals remaining in the dissolution vessel at the conclusion of Test 1 were primarily aluminum (accounts for over 90% of the mass assuming boehmite is the major form) with small amounts of iron and manganese. Residuals remaining in the dissolution vessel at the conclusion of Test 2 were primarily iron (accounts for 66% of the mass assuming hematite is the major form) with small amounts of manganese and nickel.
- Some minor metals were not removed effectively (<40% removal) from the dissolution vessel of the RWTE with 2 wt% OA. For Test 1, these metals included the lanthanides and actinides Ce, Gd, La, Th, Eu, Am, and possibly Np. For Test 2, OA was more effective at removing the minor metals and only thorium was removed at a level below 40%.
- Decomposition of 2 wt% OA to levels of <100 ppm using 2 L/min of 5 wt% ozone at 70 °C, a pressure of 8 psi, and a liquid recirculation rate of 1.8 gal/min required 8 to 14 hours for nominal 3.2L batches. The pH and oxidation/reduction potential (ORP) were tracked during decomposition testing.
- Sludge components were tracked during OA decomposition, showing that most components have the highest soluble levels in the initial dissolved sludge and early decomposition samples and exhibit lower soluble levels as OA decomposition progresses. Samples from the end of the decomposition process typically have the lowest soluble level of sludge components.
- Over the time period studied, the changes to the solubilities of important components in the Deposition Tank storage tests were insignificant. .
- Deposition Tank storage testing for Tank 12H ECC processing indicated higher solubilities for uranium and plutonium when compared with the control condition. Similar testing for Tank 5F indicated higher solubilities for uranium when compared with the control condition. The solubilities of uranium and plutonium remained within the levels expected for Tank Farm high pH supernatant liquids, which are relatively low compared to the levels in the sludge solids.

- Use of the ECC Evaporator generally led to higher soluble component concentrations in the Deposition Tank storage tests.
- Over the range of 25 to 70 °C, the storage temperature was not a major factor affecting soluble concentrations during the Deposition Tank storage tests.
- Tank 5F sludge/OA slurries, believed to representative of ECC Treatment Tank materials, were not suitable for rheology measurements, due to the fact that most of the sludge particles settled rapidly. Chemical dissolution of these materials is likely necessary for the removal these materials from the treatment tank. Since iron oxide and hydroxide materials are primary chemical constituents for Tank 5F sludge, chemical dissolution in OA should be effective.
- Tank 12H sludge/OA slurries, believed to representative of ECC Treatment Tank materials, formed suspensions which were suitable for rheology measurements. Stress versus strain curves observed for these materials revealed that the rheological properties were characteristic of Bingham Plastic materials. For sludge/acid slurries containing 10 weight percent total solids, the measured viscosity and yield stress values were 7 cP and 6 Pa, respectively. Only minor effects on the rheological properties were observed across the temperature range of 30 to 50 °C. Little difference was observed between the process midpoint and endpoint samples. Based on these results, the transfer of significant amounts of Tank 12H sludge materials from the treatment tank by suspension (rather than dissolution) appears possible.
- Rheology testing of the Tank 5F and 12H Deposition Tank samples revealed that these slurries were thin with little yield stress. Even the ECC product slurries that were evaporated to remove $\geq 85\%$ of the water had low enough insoluble solids content as to not exhibit a significant yield stress.
- Settling of the adjusted Deposition Tank solids was comparable to the control and is fast enough as to not likely delay processing. Only the Tank 5F product without pH adjustment exhibited settling significantly slower than the control sample of sludge that did not go through the ECC process.

5.0 Recommendations, Path Forward or Future Work

Testing of the Decomposition portion of the ECC process utilizing the UV light is in progress. The scope of the testing involves processing two dissolved sludge batches for each of the two sludge feeds through the Decomposition Module. Decomposition will be tracked in a manner consistent with Figure 3-2 through Figure 3-8. Sampling will be performed to provide information analogous to that of Table 3-11 through Table 3-16. Future testing using the UV light does not include additional Dissolution and Evaporation testing. Rheology, Settling, and Particle Size measurements are also not planned to be performed.

6.0 References

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Appendix A. Dissolution, Decomposition, and Evaporation Process Data

Table A-1: Oxalic Acid Decomposition (without actual tank waste sludge)

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/19/11 07:45	0	70.2	8.5	1.55	1.12	--
04/19/11 08:00	0.25	70.0	8.5	1.55	1.13	--
04/19/11 08:15	0.50	70.0	8.5	1.50	1.13	664
04/19/11 08:30	0.75	70.0	8.5	1.50	1.13	662
04/19/11 08:45	1.00	70.0	8.0	1.50	1.14	669
04/19/11 09:00	1.25	70.0	8.0	1.50	1.15	653
04/19/11 09:15	1.50	70.0	8.0	1.50	1.16	657
04/19/11 09:30	1.75	70.0	8.0	1.50	1.17	668
04/19/11 09:45	2.00	70.0	8.5	1.50	1.18	661
04/19/11 10:00	2.25	70.0	8.5	1.50	1.19	647
04/19/11 10:15	2.50	70.0	8.5	1.50	1.20	647
04/19/11 10:30	2.75	70.0	8.0	1.50	1.21	648
04/19/11 10:45	3.00	70.0	8.0	1.50	1.22	645
04/19/11 11:00	3.25	70.0	8.0	1.50	1.23	639
04/19/11 11:15	3.50	69.9	7.5	1.50	1.23	642
04/19/11 11:30	3.75	70.0	7.5	1.50	1.24	653
04/19/11 11:45	4.00	70.0	7.5	1.50	1.25	661
04/19/11 12:00	4.25	70.0	7.3	1.50	1.26	640
04/19/11 12:15	4.50	70.0	8.3	1.50	1.27	631
04/19/11 12:30	4.75	70.0	8.3	1.49	1.28	653
04/19/11 12:45	5.00	70.0	8.3	1.50	1.29	648
04/19/11 13:00	5.25	70.0	8.3	1.50	1.31	650
04/19/11 13:15	5.50	70.0	8.3	1.50	1.32	648
04/19/11 13:30	5.75	70.0	8.3	1.50	1.33	642
04/19/11 13:45	6.00	70.0	8.3	1.50	1.34	634
04/19/11 14:00	6.25	70.0	8.3	1.50	1.35	622
04/19/11 14:15	6.50	70.0	8.3	1.50	1.36	641
04/19/11 14:30	6.75	70.0	8.0	1.50	1.37	620
04/19/11 14:45	7.00	70.0	8.0	1.50	1.38	628
04/19/11 15:00	7.25	70.0	7.5	1.50	1.40	634
04/19/11 15:15	7.50	70.0	8.5	1.50	1.41	632
04/19/11 15:30	7.75	70.0	8.5	1.50	1.43	633
04/19/11 15:45	8.00	70.0	8.5	1.50	1.44	664
04/19/11 16:00	8.25	70.0	8.5	1.50	1.45	644

Table A-2: Oxalic Acid Decomposition (without actual tank waste sludge), continued

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/19/11 16:15	8.50	70.0	8.5	1.50	1.47	618
04/19/11 16:30	8.75	70.0	8.5	1.50	1.49	646
04/19/11 16:45	9.00	70.0	8.0	1.50	1.51	640
04/19/11 17:00	9.25	70.0	8.0	1.50	1.53	631
04/19/11 17:15	9.50	70.0	8.0	1.50	1.55	645
04/19/11 17:30	9.75	70.0	8.0	1.50	1.58	630
04/19/11 17:45	10.00	70.0	8.0	1.50	1.60	640
04/19/11 18:00	10.25	70.0	8.0	1.50	1.63	652
04/19/11 18:15	10.50	70.0	8.0	1.50	1.67	650
04/20/11 07:45	10.50	70.0	8.5	1.50	1.78	657
04/20/11 08:00	10.75	70.0	8.5	1.50	1.82	649
04/20/11 08:15	11.00	70.0	8.5	1.50	1.89	658
04/20/11 08:45	11.50	69.9	8.5	1.50	2.07	676
04/20/11 09:00	11.75	70.1	8.5	1.50	2.25	653
04/20/11 09:15	12.00	70.0	9.0	1.50	2.93	709
04/20/11 09:30	12.25	70.0	9.0	1.58	3.69	1068
04/20/11 09:45	12.50	69.9	9.0	1.55	4.39	1020
04/20/11 10:00	12.75	70.0	8.5	1.50	7.26	920
04/20/11 10:15	13.00	70.0	8.5	1.50	7.69	883
04/20/11 10:30	13.25	70.0	8.5	1.50	7.79	892
04/20/11 10:45	13.50	70.0	8.5	1.50	7.88	886
04/20/11 11:00	13.75	70.0	8.5	1.50	7.94	889
04/20/11 11:15	14.00	70.0	8.5	1.50	7.97	887

Table A-3: Test 1 (Tank 12H) OA Batch 1 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH
04/20/11 10:48	0.0	65.2	1.0	1.52
04/20/11 11:02	0.2	70.1	1.0	1.86
04/20/11 11:49	1.0	70.1	1.0	2.50
04/20/11 12:49	2.0	70.0	1.0	3.10
04/20/11 13:49	3.0	70.0	1.0	3.40
04/20/11 14:49	4.0	70.0	1.0	3.50
04/20/11 15:49	5.0	70.0	1.0	3.55
04/20/11 16:49	6.0	70.0	1.0	3.56
04/20/11 17:49	7.0	70.0	1.0	3.81
04/20/11 18:55	8.1	70.5	1.0	4.06
04/25/11 08:30	8.1	69.2	1.0	1.95
04/25/11 08:46	8.4	70.4	1.0	2.33
04/25/11 09:01	8.6	69.9	1.0	2.45
04/25/11 09:15	8.9	70.1	1.0	2.66
04/25/11 09:33	9.2	70.0	1.0	2.82
04/25/11 09:47	9.4	70.1	1.0	3.03
04/25/11 10:02	9.7	70.0	1.0	3.17
04/25/11 10:15	9.9	70.0	1.0	3.34
04/25/11 10:35	10.2	70.1	1.0	3.57
04/25/11 10:46	10.4	70.0	1.0	3.68
04/25/11 11:00	10.6	70.6	1.0	3.41

Table A-4: Test 1 (Tank 12H Sludge) OA Batch 1 Decomposition Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/25/11 11:03	--	40.3	0.0	1.50	1.42	438
04/25/11 11:17	--	54.2	0.0	1.50	1.69	454
04/25/11 11:32	--	63.3	0.0	1.50	1.77	467
04/25/11 12:00	--	70.7	0.0	1.50	1.82	464
04/25/11 12:17	0.00	70.2	9.0	1.60	2.34	690
04/25/11 12:30	0.22	70.0	8.5	1.50	2.58	654
04/25/11 12:46	0.48	69.9	8.5	1.50	3.28	644
04/25/11 13:02	0.75	70.0	8.3	1.54	3.39	638
04/25/11 13:15	0.97	70.0	8.3	1.53	3.60	635
04/25/11 13:30	1.22	70.0	8.3	1.53	3.48	632
04/25/11 13:45	1.47	70.0	8.2	1.35	3.63	632
04/25/11 14:00	1.72	70.0	8.2	1.50	3.51	635
04/25/11 14:15	1.97	70.0	8.2	1.50	3.71	656
04/25/11 14:30	2.22	70.0	8.2	1.50	3.97	767
04/25/11 14:45	2.47	70.0	8.1	1.50	5.08	615
04/25/11 15:00	2.72	70.0	8.1	1.48	5.73	581
04/25/11 15:15	2.97	70.0	8.1	1.50	6.30	535
04/25/11 15:30	3.22	70.0	8.0	1.45	6.54	507
04/25/11 15:45	3.47	70.0	8.0	1.45	6.65	595
04/25/11 16:07	3.83	70.1	8.2	1.45	6.68	671
04/25/11 16:20	4.05	70.0	8.0	1.45	6.93	668
04/25/11 16:32	4.25	70.0	8.0	1.45	7.07	670
04/25/11 16:45	4.47	70.0	8.0	1.45	7.44	690
04/25/11 17:00	4.72	70.0	8.0	1.45	7.34	694
04/25/11 17:19	5.03	70.0	7.8	1.45	7.79	700
04/25/11 17:35	5.30	70.0	7.8	1.42	8.09	698
04/25/11 17:45	5.47	70.0	7.8	1.40	7.85	694
04/25/11 18:00	5.72	70.0	7.8	1.40	8.31	693
04/25/11 18:16	5.98	70.0	7.8	1.40	7.94	688
04/25/11 18:30	6.22	70.0	7.5	1.40	8.52	682
04/25/11 19:00	6.72	70.0	7.5	1.40	7.99	686
04/25/11 19:30	7.22	70.0	7.5	1.40	8.13	673
04/25/11 20:04	7.78	70.0	7.3	1.40	8.39	672

Table A-5: Test 1 (Tank 12H) OA Batch 1 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
04/26/11 09:22	0.0	24.3	23.0	< 1000
04/26/11 10:19	0.9	76.3	0.0	< 1000
04/26/11 10:30	1.1	72.3	18.0	< 1000
04/26/11 11:30	2.1	67.2	18.0	< 1000
04/26/11 12:30	3.1	67.3	22.0	< 1000
04/26/11 13:33	4.2	70.6	18.5	< 1000
04/26/11 14:30	5.1	71.6	18.5	1100
04/26/11 15:30	6.1	70.9	19.0	1100
04/26/11 17:02	7.7	70.0	18.0	1100
04/26/11 17:30	8.1	70.3	19.0	1100
04/26/11 18:28	9.1	70.3	18.0	1180
04/26/11 18:47	9.4	70.3	18.0	1180
04/26/11 20:20	9.4	30.7	24.5	1180
04/26/11 20:46	9.8	52.5	25.0	1220
04/26/11 20:57	10.0	57.6	23.5	1320
04/26/11 21:21	10.4	57.7	23.5	1460

Table A-6: Test 1 (Tank 12H) OA Batch 2 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH
04/26/11 05:00	0.0	69.7	1.04	1.61
04/26/11 06:00	1.0	70.0	1.04	1.66
04/26/11 07:00	2.0	70.0	1.04	1.65
04/26/11 08:00	3.0	70.0	1.04	1.64
04/26/11 09:06	4.1	70.0	1.04	1.64
04/26/11 10:00	5.0	70.0	1.04	1.64
04/26/11 11:00	6.0	70.0	1.04	1.64
04/26/11 12:00	7.0	70.0	1.02	1.64
04/26/11 13:00	8.0	70.0	1.02	1.64
04/26/11 14:00	9.0	59.3	0.00	1.62

Table A-7: Test 1 (Tank 12H Sludge) OA Batch 2 Decomposition Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/26/11 14:45	--	70.2	0.0	1.50	1.99	461
04/26/11 15:00	--	70.2	0.0	1.52	1.99	458
04/26/11 15:15	0.00	69.9	8.0	1.55	2.01	655
04/26/11 15:30	0.25	70.1	8.0	1.55	2.02	639
04/26/11 15:46	0.52	70.0	8.0	1.55	2.03	631
04/26/11 16:00	0.75	70.0	8.0	1.55	2.04	627
04/26/11 16:15	1.00	70.0	8.0	1.55	2.06	625
04/26/11 16:30	1.25	70.0	8.0	1.55	2.07	622
04/26/11 16:45	1.50	69.9	8.2	1.55	2.09	621
04/26/11 17:00	1.75	70.0	8.2	1.55	2.10	621
04/26/11 17:15	2.00	70.0	8.2	1.55	2.12	619
04/26/11 17:30	2.25	70.0	8.2	1.55	2.14	617
04/26/11 17:45	2.50	70.0	8.2	1.55	2.16	618
04/26/11 18:01	2.77	70.0	8.0	1.55	2.18	616
04/26/11 18:18	3.05	70.0	8.0	1.55	2.20	614
04/26/11 18:30	3.25	70.0	8.0	1.45	2.22	617
04/26/11 18:45	3.50	70.0	8.0	1.45	2.25	618
04/26/11 19:00	3.75	70.0	8.0	1.45	2.28	620
04/26/11 19:15	4.00	70.0	8.0	1.45	2.32	620
04/26/11 19:30	4.25	70.0	7.5	1.45	2.37	623
04/26/11 19:46	4.52	70.0	7.5	1.45	2.44	630
04/26/11 20:00	4.75	70.0	7.5	1.45	2.52	640
04/26/11 20:15	5.00	70.0	7.5	1.45	2.68	693
04/26/11 20:22	5.12	69.9	7.5	1.45	2.82	742
04/26/11 20:29	5.23	70.0	7.5	1.45	3.14	796
04/26/11 20:33	5.30	70.0	--	--	3.65	674
04/26/11 20:35	5.33	70.0	--	--	3.94	658
04/26/11 20:40	5.42	70.0	--	--	4.09	625
04/26/11 20:45	5.50	70.0	7.0	1.47	4.17	621
04/26/11 20:50	5.58	70.0	--	--	4.28	610
04/26/11 20:55	5.67	70.0	7.5	1.45	4.36	597
04/26/11 21:00	5.75	69.9	7.0	1.45	4.39	578
04/26/11 21:08	5.88	70.0	8.0	1.45	4.34	570
04/26/11 21:15	6.00	70.0	8.0	1.45	4.38	554

Table A-8: Test 1 (Tank 12H Sludge) OA Batch 2 Decomposition Module Data, continued

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/26/11 21:22	6.12	70.0	--	--	4.63	577
04/26/11 21:30	6.25	70.0	8.0	1.45	4.91	593
04/26/11 21:35	6.33	70.0	--	--	5.11	644
04/26/11 21:40	6.42	70.0	--	--	5.29	712
04/26/11 21:45	6.50	70.0	8.0	1.40	5.46	751
04/26/11 21:50	6.58	70.0	--	--	5.62	768
04/26/11 21:55	6.67	70.0	--	--	5.79	770
04/26/11 22:00	6.75	70.0	8.0	1.40	5.98	765
04/26/11 22:07	6.87	70.0	--	--	6.24	748
04/26/11 22:11	6.93	70.0	--	--	6.40	728
04/26/11 22:15	7.00	70.1	8.0	1.40	6.55	704
04/26/11 22:20	7.08	70.0	--	--	6.72	685
04/26/11 22:25	7.17	70.0	--	--	6.87	672
04/26/11 22:31	7.27	70.0	8.0	1.40	7.11	668
04/26/11 22:35	7.33	70.0	--	--	7.27	666
04/26/11 22:40	7.42	70.0	--	--	7.41	675
04/26/11 22:50	7.58	70.0	8.0	1.40	7.56	710
04/26/11 23:00	7.75	70.0	7.5	1.40	7.68	719
04/26/11 23:15	8.00	70.0	7.5	1.40	7.91	723
04/26/11 23:30	8.25	70.0	7.5	1.40	8.19	728
04/26/11 23:45	8.50	70.0	7.5	1.40	8.37	733
04/27/11 00:00	8.75	70.0	7.5	1.38	8.46	733
04/27/11 00:15	9.00	69.9	7.5	1.40	8.52	740
04/27/11 00:30	9.25	70.0	7.5	1.40	8.55	741
04/27/11 00:45	9.50	69.9	7.5	1.40	8.58	744
04/27/11 01:00	9.75	70.0	7.5	1.40	8.60	738
04/27/11 01:15	10.00	69.9	7.5	1.38	8.66	740
04/27/11 01:30	10.25	70.1	7.5	1.38	8.68	727
04/27/11 01:45	10.50	69.9	7.0	1.38	8.69	731
04/27/11 02:00	10.75	70.0	7.0	1.38	8.70	729
04/27/11 02:07	10.87	--	--	--	--	--

Table A-9: Test 1 (Tank 12H) OA Batch 2 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
04/27/11 13:33	0.0	26.2	23.5	<1000
04/27/11 14:05	0.5	58.8	24.0	<1000
04/27/11 14:40	1.1	70.0	21.0	<1000
04/27/11 15:01	1.5	70.0	19.5	<1000
04/27/11 16:00	2.4	70.0	19.0	<1000
04/27/11 17:00	3.5	69.5	19.5	1200
04/27/11 18:00	4.5	69.8	19.5	1600

Table A-10: Test 1 (Tank 12H) OA Batch 3 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH
04/27/11 17:32	0.0	65.8	1.0	2.16
04/27/11 18:00	0.5	70.1	1.0	2.16
04/27/11 19:00	1.5	70.0	1.0	2.10
04/27/11 20:02	2.5	70.0	1.05	2.04
04/27/11 21:00	3.5	70.0	1.05	2.02
04/27/11 22:00	4.5	70.0	1.0	2.01
04/27/11 23:09	5.6	70.0	1.0	2.00
04/28/11 00:00	6.5	70.0	1.0	2.00
04/28/11 01:00	7.5	70.0	1.0	2.01
04/28/11 02:01	8.5	70.0	1.0	2.00
04/28/11 02:15	8.7	--	--	--

Table A-11 Test 1 (Tank 12H Sludge) OA Batch 3 Decomposition Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/28/11 02:23	--	46.4	0.0	1.55	2.08	662
04/28/11 03:02	0.00	67.1	8.0	1.50	1.90	615
04/28/11 03:15	0.22	70.6	8.0	1.50	1.90	607
04/28/11 03:30	0.47	70.0	8.0	1.50	1.93	605
04/28/11 03:45	0.72	69.9	8.0	1.50	1.93	604
04/28/11 04:05	1.05	70.0	8.0	1.50	1.95	604
04/28/11 04:18	1.27	70.0	7.5	1.50	1.95	604
04/28/11 04:32	1.50	70.0	7.0	1.50	1.96	604
04/28/11 04:46	1.73	70.0	8.0	1.50	1.97	604
04/28/11 05:02	2.00	70.0	8.0	1.50	1.98	604
04/28/11 05:15	2.22	70.0	8.0	1.50	1.99	604
04/28/11 05:33	2.52	69.9	8.0	1.50	1.99	603
04/28/11 05:45	2.72	69.9	8.2	1.50	2.00	603
04/28/11 06:00	2.97	70.0	8.2	1.50	2.01	603
04/28/11 06:15	3.22	70.0	8.2	1.50	2.02	603
04/28/11 06:30	3.47	70.0	7.5	1.50	2.03	602
04/28/11 06:45	3.72	70.0	7.5	1.50	2.04	602
04/28/11 07:00	3.97	70.0	7.5	1.50	2.05	602
04/28/11 07:15	4.22	70.0	8.0	1.50	2.07	602
04/28/11 07:30	4.47	70.0	8.0	1.35	2.08	601
04/28/11 07:45	4.72	70.0	8.0	1.50	2.09	601
04/28/11 08:00	4.97	70.0	8.0	1.50	2.11	600
04/28/11 08:15	5.22	70.0	8.0	1.50	2.12	600
04/28/11 08:30	5.47	70.0	8.0	1.50	2.14	600
04/28/11 08:45	5.72	70.0	8.3	1.50	2.16	599
04/28/11 09:00	5.97	70.0	8.3	1.50	2.18	599
04/28/11 09:15	6.22	69.9	8.3	1.50	2.20	599
04/28/11 09:30	6.47	70.0	8.3	1.50	2.23	598
04/28/11 09:45	6.72	70.0	8.3	1.50	2.26	599
04/28/11 10:00	6.97	70.0	8.3	1.50	2.30	600
04/28/11 10:15	7.22	70.0	7.5	1.50	2.34	601
04/28/11 10:30	7.47	69.9	8.0	1.50	2.40	607
04/28/11 10:45	7.72	70.1	8.0	1.50	2.50	617
04/28/11 11:00	7.97	70.0	8.0	1.50	2.71	641
04/28/11 11:05	8.05	69.9	8.0	1.50	2.92	645
04/28/11 11:10	8.13	70.0	7.9	1.50	3.42	616
04/28/11 11:15	8.22	70.0	7.9	1.50	3.74	584

Table A-12: Test 1 (Tank12H Sludge) OA Batch 3 Decomposition Module Data, continued

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
04/28/11 11:20	8.30	69.9	7.8	1.50	3.80	563
04/28/11 11:25	8.38	69.9	7.8	1.50	3.90	544
04/28/11 11:30	8.47	70.0	7.8	1.50	3.98	527
04/28/11 11:35	8.55	70.0	7.7	1.50	4.01	517
04/28/11 11:40	8.63	70.0	7.8	1.50	4.03	524
04/28/11 11:45	8.72	70.0	7.9	1.50	4.12	808
04/28/11 11:50	8.80	70.0	7.8	1.50	4.26	861
04/28/11 11:55	8.88	70.0	7.8	1.50	4.36	900
04/28/11 12:00	8.97	70.0	7.7	1.50	4.43	873
04/28/11 12:05	9.05	70.0	7.6	1.50	4.48	866
04/28/11 12:15	9.22	70.0	8.5	1.50	4.55	982
04/28/11 12:20	9.30	70.0	8.3	1.50	4.58	970
04/28/11 12:30	9.47	70.0	8.3	1.50	4.67	907
04/28/11 12:40	9.63	70.0	8.2	1.50	4.78	826
04/28/11 12:50	9.80	70.0	8.2	1.50	4.95	803
04/28/11 13:00	9.97	70.0	8.1	1.50	5.20	782
04/28/11 13:05	10.05	70.0	8.1	1.50	5.42	755
04/28/11 13:10	10.13	70.0	8.1	1.50	5.63	731
04/28/11 13:15	10.22	70.0	8.1	1.50	5.89	709
04/28/11 13:20	10.30	70.0	8.1	1.50	6.25	676
04/28/11 13:25	10.38	70.1	8.1	1.50	6.51	660
04/28/11 13:30	10.47	70.0	8.1	1.50	6.83	651
04/28/11 13:35	10.55	70.0	8.0	1.50	6.97	668
04/28/11 13:40	10.63	70.0	8.0	1.50	7.09	696
04/28/11 13:45	10.72	70.0	8.0	1.50	7.18	718
04/28/11 13:50	10.80	70.0	8.0	1.50	7.25	724
04/28/11 13:55	10.88	70.0	8.0	1.50	7.35	738
04/28/11 14:00	10.97	70.0	8.0	1.48	7.46	743
04/28/11 14:05	11.05	70.1	8.0	1.48	7.54	753
04/28/11 14:10	11.13	70.0	8.0	1.47	7.66	751
04/28/11 14:20	11.30	70.0	8.0	1.45	7.76	738
04/28/11 14:30	11.47	69.9	8.0	1.45	7.86	702
04/28/11 14:45	11.72	70.1	8.0	1.45	7.98	730
04/28/11 15:00	11.97	70.0	8.0	1.45	8.06	708
04/28/11 15:15	12.22	69.9	8.0	1.45	8.09	677
04/28/11 15:25	12.38	70.0	8.0	1.45	8.11	649
04/28/11 15:40	12.63	69.0	8.0	1.45	8.15	667

Table A-13: Test 1 (Tank12H) OA Batch 3 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
04/28/11 18:15	0.0	--	21.0	<1000
04/28/11 18:50	0.6	68.8	20.0	<1000
04/28/11 19:07	0.9	68.7	20.0	<1000
04/28/11 19:40	1.4	69.9	19.5	<1000
04/28/11 19:55	1.7	70.2	19.5	<1000
04/28/11 20:00	1.8	70.0	20.0	<1000
04/28/11 21:00	2.8	69.8	20.0	<1000
04/28/11 22:00	3.8	68.8	20.0	<1000
04/28/11 23:00	4.8	69.0	20.0	1300
04/28/11 23:40	5.4	68.9	20.0	1450
04/29/11 00:02	5.8	68.9	20.0	1570
04/29/11 00:07	5.9	68.9	20.0	1600
04/29/11 00:17	6.0	68.7	20.0	>1600

Table A-14: Test 2 (Tank 5F) OA Batch 1 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH
05/05/11 08:28	0.0	65.1	1.25	2.77
05/05/11 08:43	0.2	70.0	1.25	2.87
05/05/11 09:43	1.3	70.2	1.25	3.65
05/05/11 10:43	2.3	70.0	1.25	4.00
05/05/11 11:43	3.2	69.9	1.25	2.89
05/05/11 12:48	4.3	70.0	1.25	2.18
05/05/11 13:48	5.3	70.0	1.25	2.15
05/05/11 14:48	6.3	70.0	1.25	2.18
05/05/11 16:51	8.4	70.0	1.25	2.20

Table A-15: Test 2 (Tank 5F Sludge) OA Batch 1 Decomposition Module Data

Note: the pH probe was malfunctioning during this data set

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/09/11 06:45	--	32.3	0.0	1.60	2.09	499
05/09/11 07:45	0.00	66.8	8.0	1.50	2.49	728
05/09/11 08:00	0.25	69.4	8.0	1.50	2.37	730
05/09/11 08:15	0.50	70.4	8.0	1.50	2.33	731
05/09/11 08:30	0.75	70.0	8.0	1.50	2.36	746
05/09/11 08:45	1.00	70.1	8.0	1.50	2.38	758
05/09/11 09:00	1.25	70.1	8.0	1.50	2.07	767
05/09/11 09:15	1.50	69.9	7.0	1.50	1.92	821
05/09/11 09:30	1.75	70.1	8.0	1.50	2.15	797
05/09/11 09:45	2.00	69.9	8.0	1.50	2.50	730
05/09/11 09:50	2.08	70.0	8.0	1.50	2.62	717
05/09/11 09:55	2.17	70.0	8.0	1.50	2.72	704
05/09/11 10:00	2.25	70.0	8.0	1.40	2.84	679
05/09/11 10:05	2.33	70.0	8.0	1.40	2.97	656
05/09/11 10:10	2.42	70.0	8.0	1.50	3.14	618
05/09/11 10:15	2.50	70.0	8.0	1.50	3.19	626
05/09/11 10:20	2.58	70.0	8.0	1.50	3.26	615
05/09/11 10:25	2.67	70.0	8.0	1.50	3.33	603
05/09/11 10:30	2.75	69.9	8.0	1.50	3.38	591
05/09/11 10:35	2.83	70.0	7.5	1.50	3.44	581
05/09/11 10:40	2.92	70.0	7.5	1.47	3.49	573
05/09/11 10:45	3.00	70.0	7.5	1.48	3.64	567
05/09/11 10:50	3.08	70.0	7.5	1.48	3.77	561
05/09/11 10:56	3.18	70.0	7.5	1.50	3.79	554
05/09/11 11:00	3.25	70.0	7.5	1.50	3.81	550
05/09/11 11:05	3.33	70.0	7.5	1.50	3.83	546
05/09/11 11:10	3.42	70.0	7.5	1.50	3.84	542
05/09/11 11:15	3.50	70.0	7.5	1.50	3.85	538
05/09/11 11:20	3.58	70.0	7.5	1.50	3.88	530
05/09/11 11:30	3.75	70.0	7.5	1.50	3.99	491
05/09/11 11:45	4.00	69.9	7.5	1.50	4.06	452
05/09/11 12:00	4.25	70.0	7.5	1.50	4.17	479
05/09/11 12:15	4.50	70.0	7.5	1.50	4.25	570
05/09/11 12:30	4.75	70.0	7.5	1.42	4.33	658

Table A-16: Test 2 (Tank 5F Sludge) OA Batch 1 Decomposition Module Data, continued

Note: the pH probe was malfunctioning during this data set

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/09/11 12:45	5.00	70.0	7.5	1.42	4.28	739
05/09/11 13:00	5.25	70.0	7.5	1.42	4.35	782
05/09/11 13:15	5.50	70.0	7.5	1.42	4.25	788
05/09/11 13:30	5.75	70.0	7.5	1.42	4.07	788
05/09/11 13:45	6.00	70.0	7.5	1.40	4.16	787
05/09/11 14:00	6.25	70.0	7.5	1.40	4.08	783
05/09/11 14:15	6.50	70.0	7.2	1.41	4.10	778
05/09/11 14:30	6.75	70.0	8.3	1.45	4.21	782
05/09/11 14:45	7.00	70.1	8.3	1.45	4.30	779
05/09/11 15:00	7.25	70.0	8.0	1.45	4.42	779
05/09/11 15:15	7.50	70.1	8.3	1.45	4.50	777
05/09/11 15:30	7.75	70.0	8.3	1.46	4.54	775
05/09/11 15:45	8.00	70.0	8.3	1.46	4.59	772
05/09/11 16:00	8.25	70.0	8.3	1.46	4.56	766
05/09/11 16:15	8.50	70.0	8.3	1.46	4.60	767
05/09/11 16:30	8.75	70.0	8.3	1.46	4.57	763
05/09/11 16:45	9.00	70.0	8.5	1.48	4.54	760
05/09/11 17:00	9.25	70.1	8.5	1.48	4.47	755
05/09/11 17:15	9.50	70.0	8.5	1.48	4.39	751
05/09/11 17:30	9.75	70.0	8.5	1.48	4.34	755
05/09/11 17:45	10.00	70.0	8.5	1.48	4.33	754
05/09/11 18:00	10.25	70.0	8.5	1.48	4.33	755
05/09/11 18:18	10.55	70.0	8.5	1.48	4.34	746
05/09/11 18:30	10.75	70.0	8.5	1.48	4.31	745
05/09/11 18:45	11.00	69.9	8.5	1.48	4.27	746
05/09/11 19:00	11.25	70.0	8.5	1.48	4.19	750

Table A-17: Test 2 (Tank 5F) OA Batch 1 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
05/11/11 06:29	0.0	--	15.5	<1000
05/11/11 07:00	0.5	69.1	20.0	<1000
05/11/11 08:00	1.5	70.0	20.0	<1000
05/11/11 09:00	2.5	69.3	20.0	<1000
05/11/11 10:00	3.5	68.9	20.0	1200
05/11/11 11:00	4.5	68.7	20.0	1580
05/11/11 11:20	4.8	68.7	20.0	>1600

Table A-18: Test 2 (Tank 5F) OA Batch 2 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH*
05/11/11 07:17	0.0	67.1	1.03	1.89
05/11/11 08:00	0.7	70.0	1.03	3.05*
05/11/11 08:11	0.9	70.0	1.03	3.30*
05/11/11 09:00	1.7	70.0	1.03	3.73*
05/11/11 10:00	2.7	70.0	1.03	4.07*
05/11/11 11:00	3.7	70.0	1.03	4.35*
05/11/11 12:00	4.7	70.0	1.03	4.48*
05/11/11 13:00	5.7	70.0	1.03	4.52*
05/11/11 15:15	8.0	70.0	1.03	4.55*

* pH behavior indicates probe not in contact with Dissolution Module fluid.

Table A-19: Test 2 (Tank 5F Sludge) OA Batch 2 Decomposition Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/12/11 06:10	--	22.0	0.0	1.50	--	--
05/12/11 07:01	0.00	65.5	8.0	1.50	1.27	812
05/12/11 07:15	0.23	69.6	8.3	1.50	1.31	879
05/12/11 07:30	0.48	70.4	8.0	1.50	1.37	884
05/12/11 07:45	0.73	70.0	8.0	1.50	1.40	899
05/12/11 08:00	0.98	70.0	8.0	1.50	1.45	912
05/12/11 08:15	1.23	70.0	8.0	1.50	1.48	919
05/12/11 08:30	1.48	70.0	8.0	1.50	1.52	930
05/12/11 08:45	1.73	70.0	8.0	1.50	1.56	947
05/12/11 09:00	1.98	70.0	8.5	1.50	1.61	977
05/12/11 09:15	2.23	70.0	8.5	1.50	1.70	981
05/12/11 09:30	2.48	70.0	8.5	1.50	1.87	960
05/12/11 09:45	2.73	70.0	8.5	1.50	2.56	921
05/12/11 09:50	2.82	69.9	8.5	1.48	2.65	938
05/12/11 09:55	2.90	70.0	8.0	1.35	2.69	944
05/12/11 10:00	2.98	70.0	8.5	1.35	2.74	932
05/12/11 10:05	3.07	70.0	8.0	1.35	2.79	930
05/12/11 10:10	3.15	70.0	8.0	1.35	2.87	929
05/12/11 10:15	3.23	70.0	8.0	1.35	2.94	929
05/12/11 10:20	3.32	70.0	8.0	1.35	3.02	905
05/12/11 10:25	3.40	70.0	8.0	1.35	3.12	919
05/12/11 10:30	3.48	70.0	8.0	1.35	3.20	899
05/12/11 10:35	3.57	70.0	8.0	1.35	3.32	902
05/12/11 10:40	3.65	70.0	8.0	1.23	3.45	884
05/12/11 10:45	3.73	70.0	8.0	1.23	3.56	885
05/12/11 10:50	3.82	69.9	7.5	1.23	3.68	860
05/12/11 10:55	3.90	69.9	8.0	1.23	3.78	892
05/12/11 11:00	3.98	70.0	8.0	1.23	3.87	887
05/12/11 11:05	4.07	70.0	8.0	1.22	3.97	879
05/12/11 11:10	4.15	70.0	8.0	1.22	4.05	877
05/12/11 11:15	4.23	70.0	8.0	1.22	4.13	871
05/12/11 11:20	4.32	70.0	8.0	1.20	4.23	888
05/12/11 11:25	4.40	70.1	7.5	1.20	4.33	909
05/12/11 11:30	4.48	70.0	8.0	1.20	4.61	928

Table A-20: Test 2 (Tank 5F Sludge) OA Batch 2 Decomposition Module Data, continued

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/12/11 11:35	4.57	70.0	7.5	1.20	4.95	924
05/12/11 11:40	4.65	70.0	8.0	1.20	5.30	922
05/12/11 11:45	4.73	70.0	8.0	1.20	5.62	923
05/12/11 11:50	4.82	70.0	7.5	1.20	5.93	907
05/12/11 11:55	4.90	70.0	7.5	1.20	6.17	911
05/12/11 12:00	4.98	70.0	7.5	1.20	6.38	901
05/12/11 12:06	5.08	70.0	7.5	1.20	6.60	887
05/12/11 12:10	5.15	70.0	7.5	1.20	6.76	897
05/12/11 12:15	5.23	70.0	8.0	1.20	6.85	894
05/12/11 12:20	5.32	70.0	8.0	1.20	7.04	900
05/12/11 12:25	5.40	70.0	8.0	1.20	7.18	898
05/12/11 12:30	5.48	70.0	8.0	1.20	7.31	896
05/12/11 12:35	5.57	70.0	8.0	1.20	7.51	894
05/12/11 12:40	5.65	70.0	8.0	1.20	7.57	905
05/12/11 12:45	5.73	70.0	8.0	1.20	7.78	901
05/12/11 12:50	5.82	70.0	8.0	1.20	7.88	907
05/12/11 12:55	5.90	70.0	8.0	1.20	7.94	912
05/12/11 13:00	5.98	70.0	8.0	1.20	8.02	920
05/12/11 13:15	6.23	70.0	8.0	1.20	8.15	917
05/12/11 13:30	6.48	70.1	8.0	1.20	8.24	916
05/12/11 13:45	6.73	70.0	8.0	1.20	8.30	917
05/12/11 14:00	6.98	70.0	8.0	1.20	8.35	918
05/12/11 14:15	7.23	70.0	7.5	1.08	8.42	930
05/12/11 14:30	7.48	70.0	7.5	1.08	8.49	955
05/12/11 14:42	7.68	70.0	7.5	1.20	8.52	956

Table A-21: Test 2 (Tank 5F) OA Batch 2 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
05/16/11 10:58	0.0	21.0	19.5	<1000
05/16/11 11:33	0.6	70.1	19.5	<1000
05/16/11 12:30	1.5	69.3	19.5	<1000
05/16/11 13:00	2.0	69.0	19.5	<1000
05/16/11 14:00	3.0	69.6	19.5	<1000
05/16/11 14:40	3.7	69.1	19.5	1200
05/16/11 15:00	4.0	69.2	19.5	1300

Table A-22: Test 2 (Tank 5F) OA Batch 3 Dissolution Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Flow (gal/min)	pH
05/16/11 08:19	0.0	65.3	1.05	1.25
05/16/11 08:30	0.2	70.0	1.05	1.24
05/16/11 09:30	1.2	70.0	1.05	1.21
05/16/11 10:30	2.2	70.1	1.05	1.11
05/16/11 11:30	3.2	70.0	1.05	1.09
05/16/11 12:30	4.2	70.0	1.05	1.08
05/16/11 13:30	5.2	70.0	1.05	1.08
05/16/11 14:30	6.2	70.0	1.05	1.07
05/16/11 15:30	7.2	70.0	1.05	1.07
05/16/11 16:29	8.2	70.0	1.10	1.06

Table A-23: Test 2 (Tank 5F Sludge) OA Batch 3 Decomposition Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/17/11 06:20		18.6	0.0	1.50	1.22	624
05/17/11 07:32		64.1	8.0	1.50	1.20	679
05/17/11 07:45	0.00	66.8	8.0	1.58	1.20	701
05/17/11 08:00	0.25	69.8	8.0	1.58	1.22	692
05/17/11 08:15	0.50	70.3	7.7	1.58	1.24	694
05/17/11 08:30	0.75	69.9	8.0	1.58	1.26	692
05/17/11 08:45	1.00	70.0	8.0	1.57	1.27	695
05/17/11 09:00	1.25	70.0	8.0	1.57	1.29	695
05/17/11 09:15	1.50	70.0	8.0	1.57	1.31	698
05/17/11 09:30	1.75	70.1	8.0	1.57	1.33	698
05/17/11 09:45	2.00	70.1	8.5	1.57	1.36	699
05/17/11 10:00	2.25	70.0	8.5	1.57	1.38	701
05/17/11 10:15	2.50	69.9	8.5	1.57	1.40	704
05/17/11 10:30	2.75	69.9	8.8	1.57	1.41	707
05/17/11 10:45	3.00	70.0	8.8	1.57	1.43	710
05/17/11 11:00	3.25	70.0	9.0	1.58	1.46	713
05/17/11 11:15	3.50	70.0	9.0	1.57	1.48	718
05/17/11 11:30	3.75	70.0	8.5	1.47	1.52	717
05/17/11 11:45	4.00	70.0	8.5	1.45	1.55	720
05/17/11 12:00	4.25	70.0	8.5	1.45	1.59	723
05/17/11 12:15	4.50	70.0	8.5	1.45	1.66	715
05/17/11 12:30	4.75	70.0	8.5	1.42	1.77	698
05/17/11 12:45	5.00	70.0	8.5	1.40	2.13	759
05/17/11 12:55	5.17	70.0	8.5	1.40	2.31	752
05/17/11 13:00	5.25	70.0	8.5	1.40	2.33	740
05/17/11 13:05	5.33	70.0	8.5	1.38	2.36	731
05/17/11 13:10	5.42	70.0	8.5	1.38	2.40	726
05/17/11 13:15	5.50	70.0	8.5	1.38	2.44	722
05/17/11 13:20	5.58	70.0	8.5	1.38	2.49	718
05/17/11 13:25	5.67	70.0	8.5	1.38	2.55	714
05/17/11 13:30	5.75	70.0	8.5	1.38	2.62	710
05/17/11 13:35	5.83	70.1	8.5	1.38	2.71	702
05/17/11 13:40	5.92	70.0	8.2	1.38	2.78	696
05/17/11 13:45	6.00	70.0	8.5	1.38	2.88	688

Table A-24: Test 2 (Tank 5F Sludge) OA Batch 3 Decomposition Module Data, continued

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (psi)	Flow (gal/min)	pH	ORP (mV)
05/17/11 13:50	6.08	70.0	8.5	1.38	3.01	679
05/17/11 13:55	6.17	70.0	8.5	1.38	3.14	673
05/17/11 14:00	6.25	70.0	8.5	1.38	3.29	667
05/17/11 14:05	6.33	70.0	8.5	1.38	3.44	661
05/17/11 14:10	6.42	70.0	8.5	1.38	3.55	661
05/17/11 14:15	6.50	70.0	8.5	1.38	3.64	644
05/17/11 14:20	6.58	70.0	8.5	1.38	3.68	644
05/17/11 14:25	6.67	70.0	8.5	1.38	3.72	638
05/17/11 14:30	6.75	70.1	8.5	1.38	4.05	622
05/17/11 14:35	6.83	70.0	8.5	1.38	4.52	611
05/17/11 14:40	6.92	70.0	8.5	1.38	5.00	594
05/17/11 14:45	7.00	70.0	8.5	1.38	5.36	594
05/17/11 14:50	7.08	70.0	8.5	1.38	5.72	612
05/17/11 14:55	7.17	69.9	8.5	1.38	5.98	643
05/17/11 15:02	7.28	70.0	8.5	1.35	6.45	681
05/17/11 15:05	7.33	70.0	8.5	1.35	6.57	684
05/17/11 15:10	7.42	70.0	8.5	1.35	6.75	700
05/17/11 15:16	7.52	70.0	8.5	1.32	6.97	709
05/17/11 15:20	7.58	69.9	8.5	1.32	7.12	714
05/17/11 15:25	7.67	70.0	8.5	1.32	7.25	714
05/17/11 15:30	7.75	70.0	8.5	1.32	7.33	709
05/17/11 15:35	7.83	70.0	8.5	1.32	7.41	709
05/17/11 15:40	7.92	70.0	8.4	1.32	7.49	708
05/17/11 15:45	8.00	70.1	8.4	1.32	7.52	706
05/17/11 15:50	8.08	70.0	8.5	1.32	7.58	708
05/17/11 15:55	8.17	70.0	8.5	1.30	7.67	704
05/17/11 16:00	8.25	70.0	8.5	1.35	7.72	704
05/17/11 16:05	8.33	70.0	8.3	1.32	7.72	701
05/17/11 16:10	8.42	70.0	8.3	1.31	7.77	698
05/17/11 16:20	8.58	69.9	8.2	1.31	7.84	698
05/17/11 16:30	8.75	70.0	8.2	1.31	7.90	690

Table A-25: Test 2 (Tank 5F) OA Batch 3 Evaporator Module Data

Date/Time (mm/dd/yy hh:mm)	Run Time (hr)	Temperature (°C)	Pressure (in. Hg)	Condensate (mL)
05/19/11 08:26	0.0	18.2	18.0	<1000
05/19/11 09:02	0.6	70.5	18.0	<1000
05/19/11 10:00	1.6	70.4	20.0	<1000
05/19/11 11:00	2.6	70.3	20.0	<1000
05/19/11 12:00	3.6	69.5	20.0	<1000
05/19/11 13:00	4.6	69.5	20.0	1200
05/19/11 14:00	5.6	69.6	20.0	1520
05/19/11 14:20	5.9	69.6	20.0	>1600

Appendix B. Particle Size, Rheology, and Settling Data

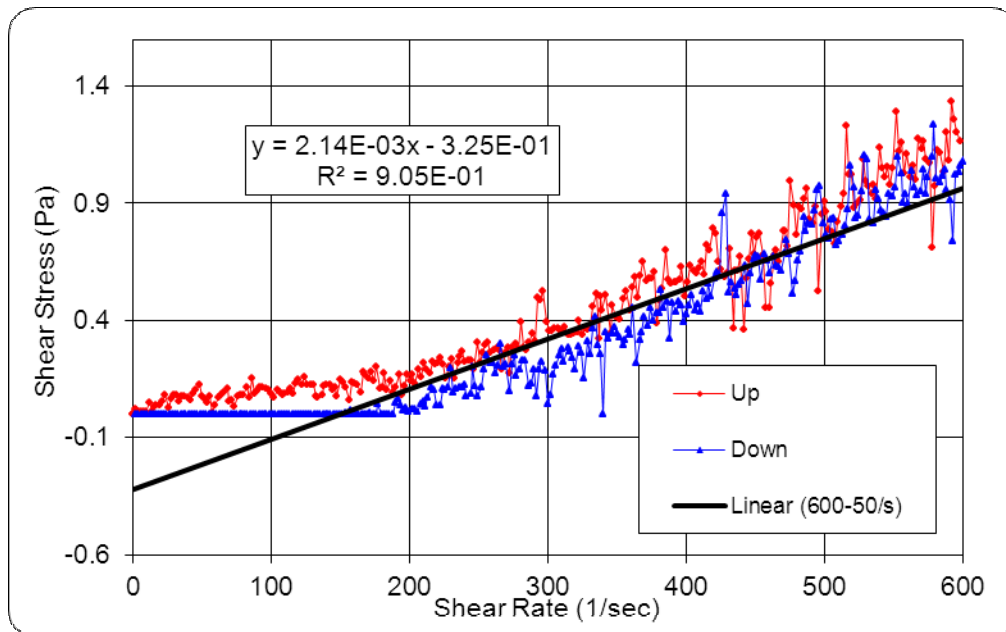


Figure B-1: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 30 °C (First Replicate).

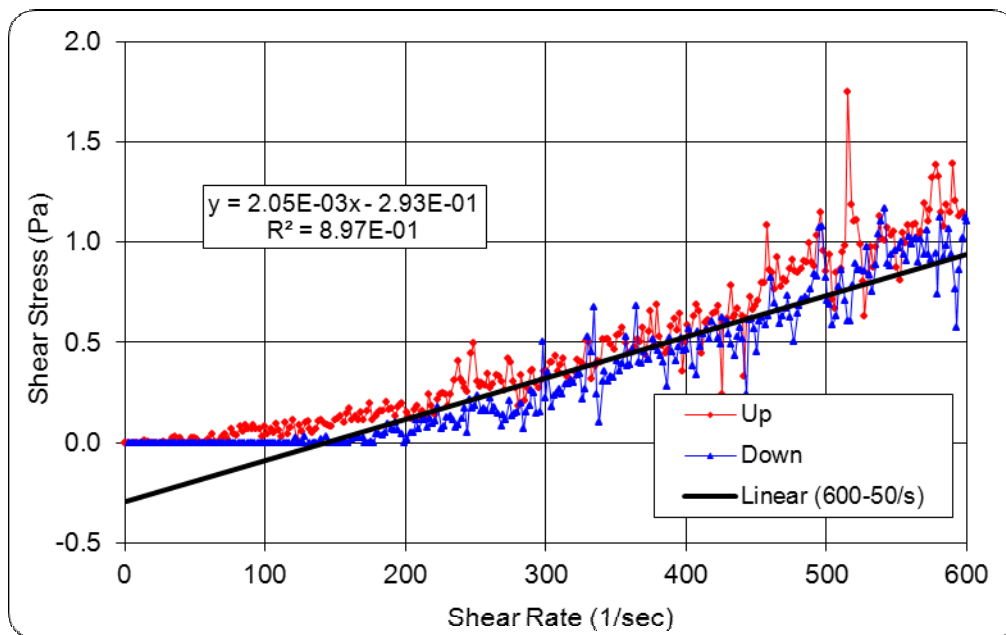


Figure B-2: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 30 °C (Second Replicate).

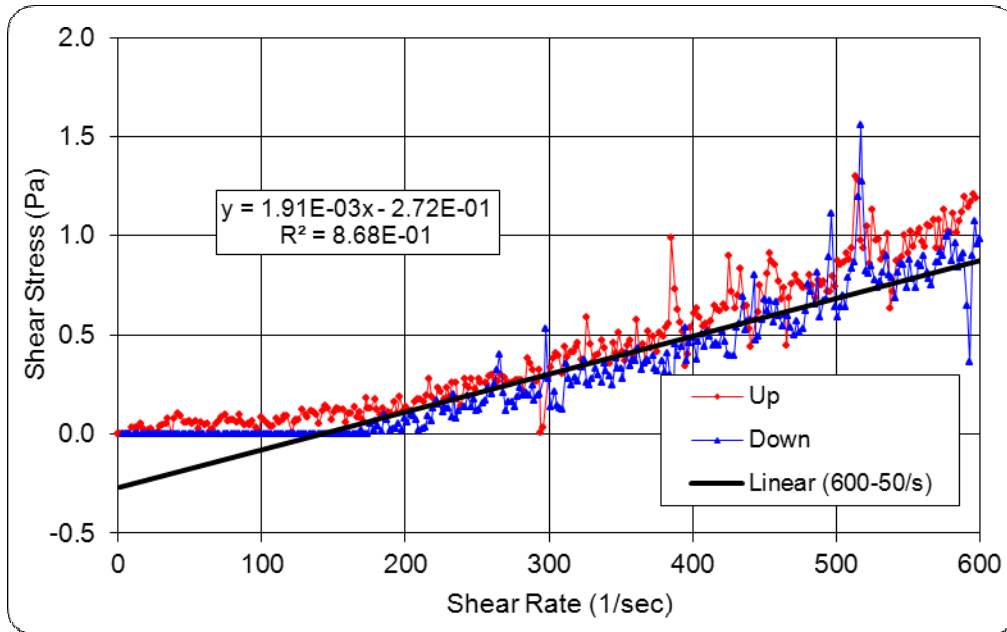


Figure B-3: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 40 °C (First Replicate).

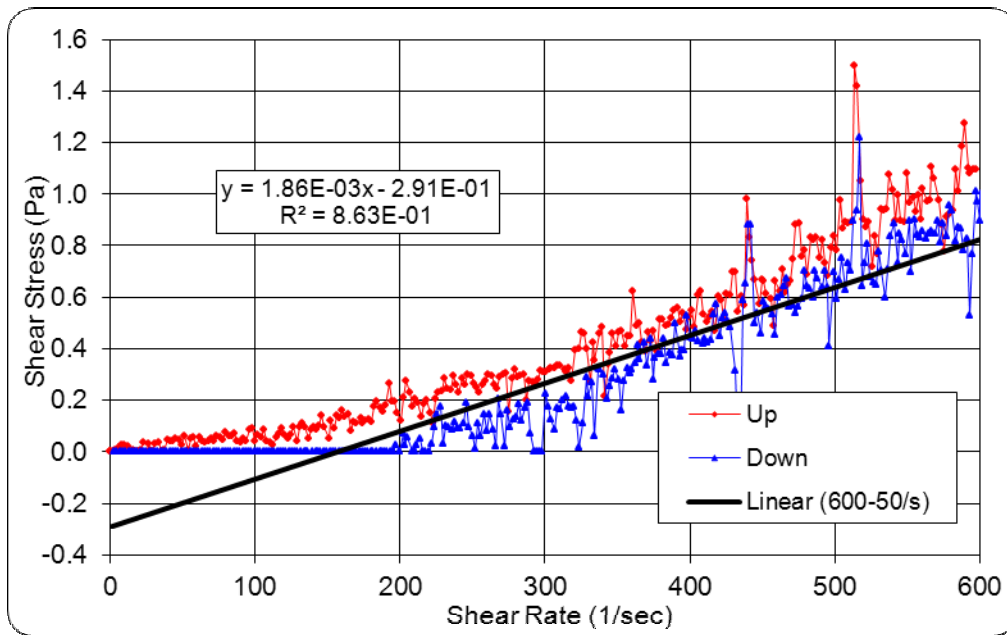


Figure B-4: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 40 °C (Second Replicate).

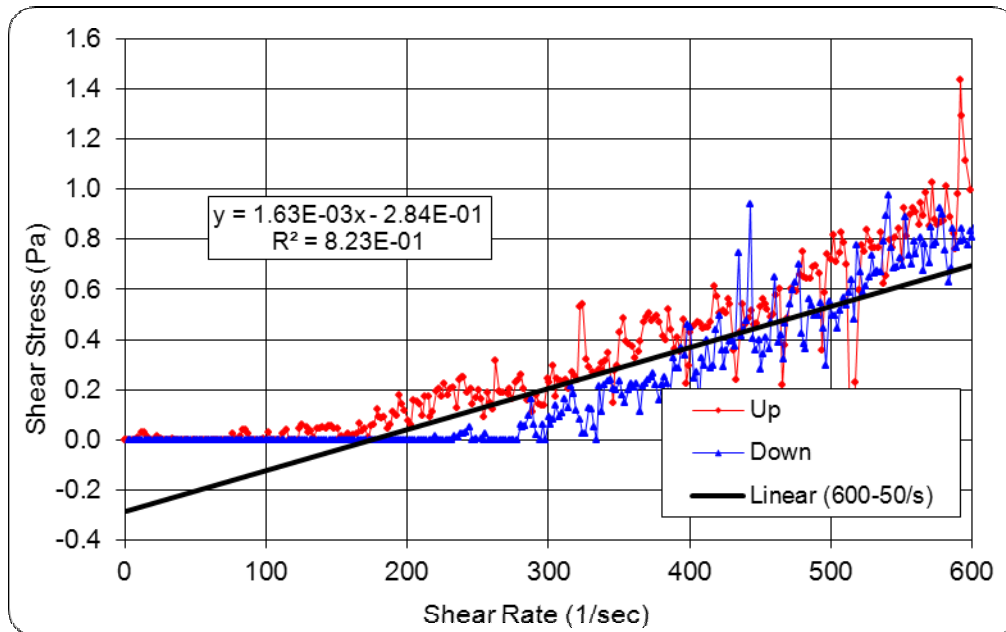


Figure B-5: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 50 °C (First Replicate).

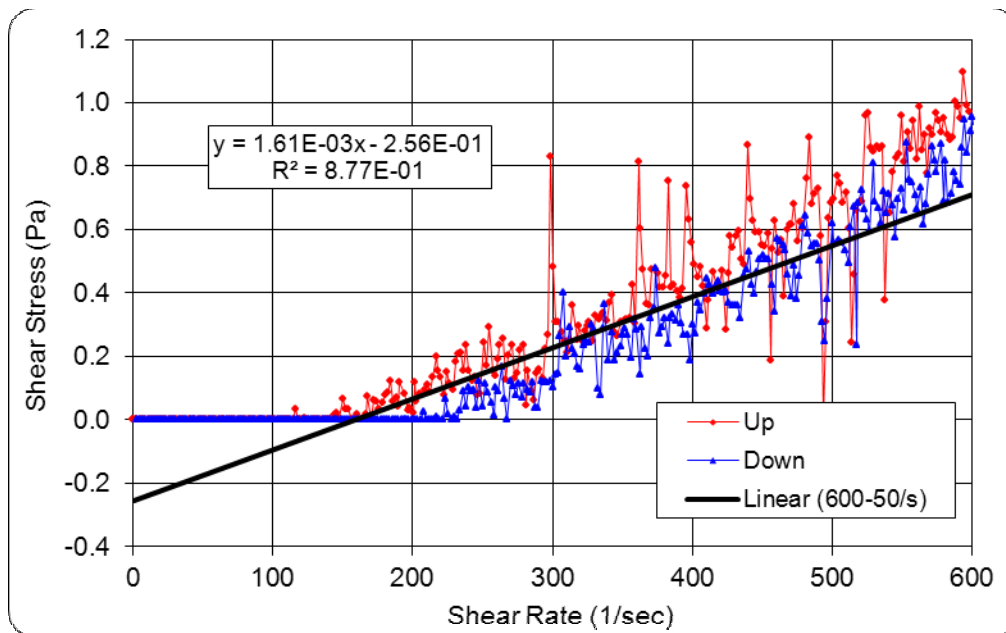


Figure B-6: Stress Versus Strain Curve for Tank 5F Baseline Dilute at 50 °C (Second Replicate).

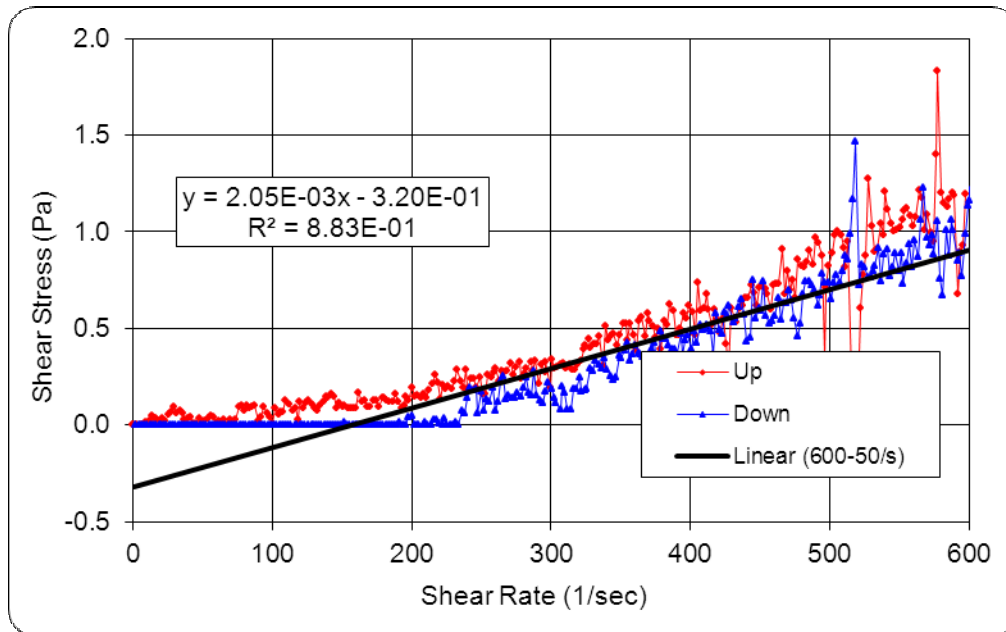


Figure B-7: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 30 °C (First Replicate).

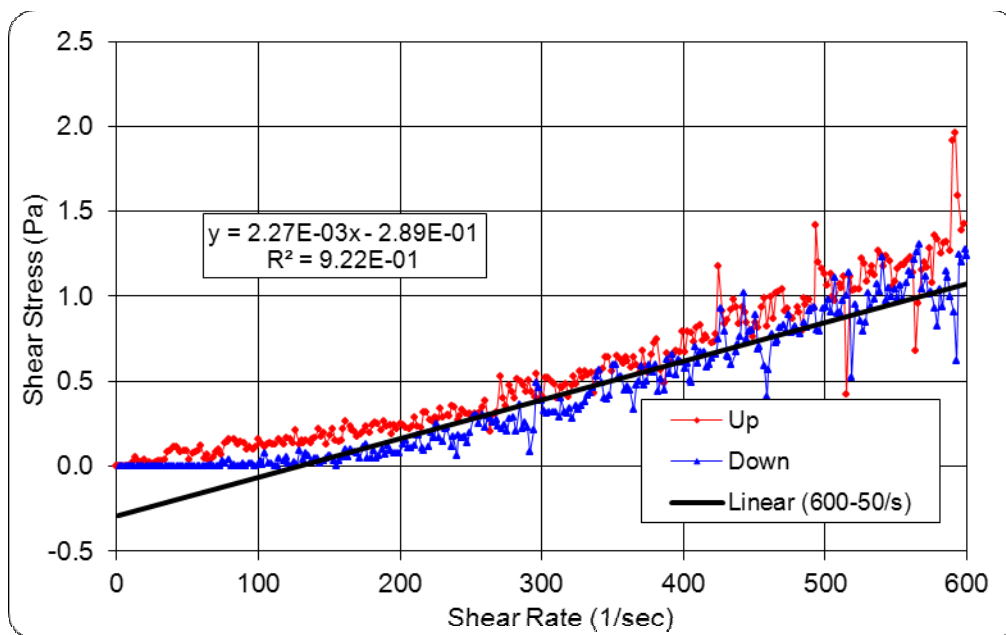


Figure B-8: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 30 °C (Second Replicate).

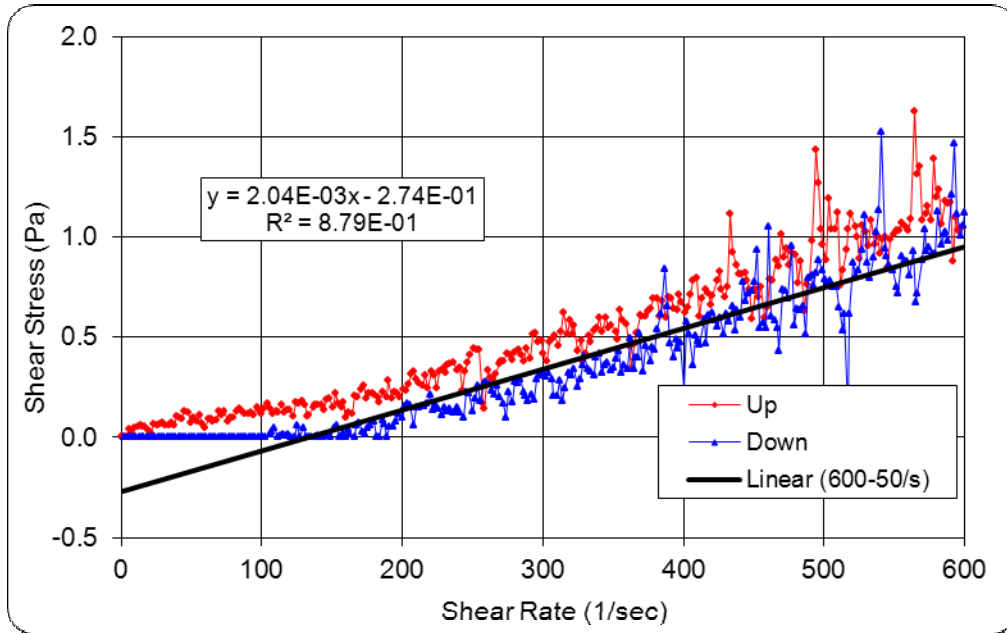


Figure B-9: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 40 °C (First Replicate).

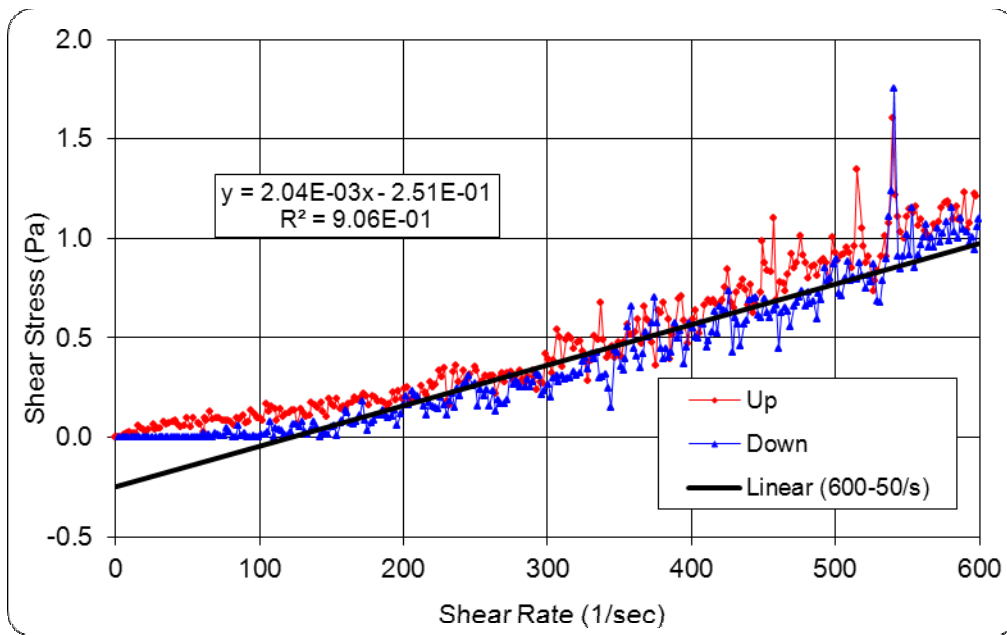


Figure B-10: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 40 °C (Second Replicate).

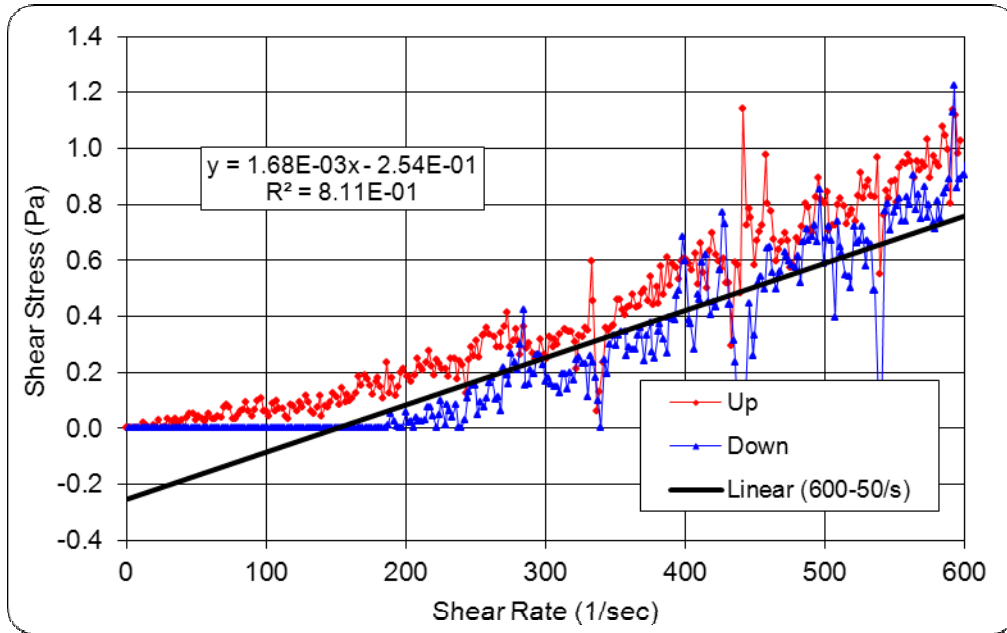


Figure B-11: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 50 °C (First Replicate).

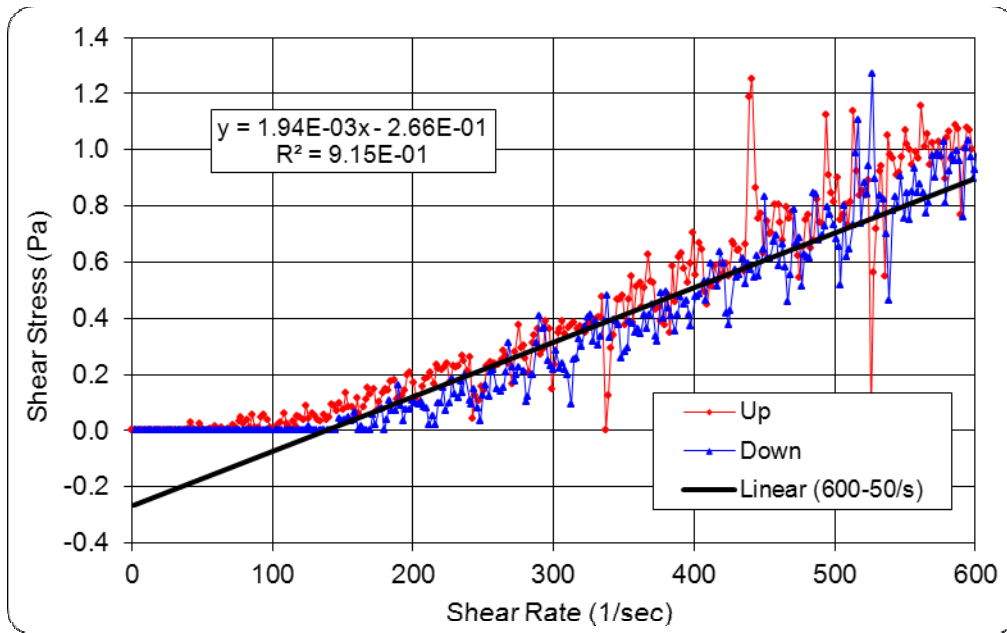


Figure B-12: Stress Versus Strain Curve for Tank 5F Baseline Concentrate at 50 °C (Second Replicate).

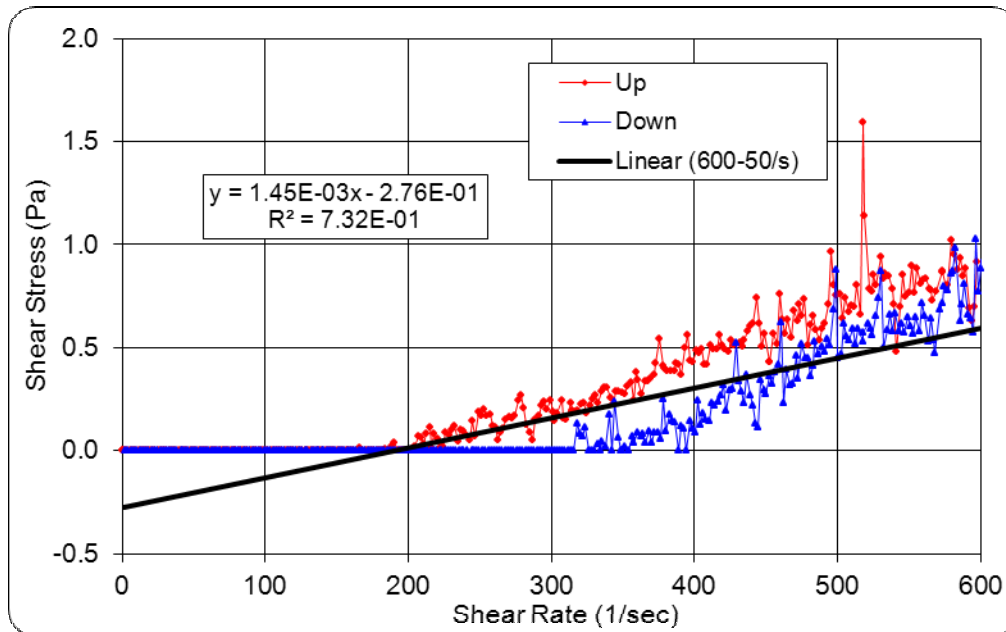


Figure B-13: Stress Versus Strain Curve for Tank 5F Midpoint Concentrate at 30 °C (First Replicate).

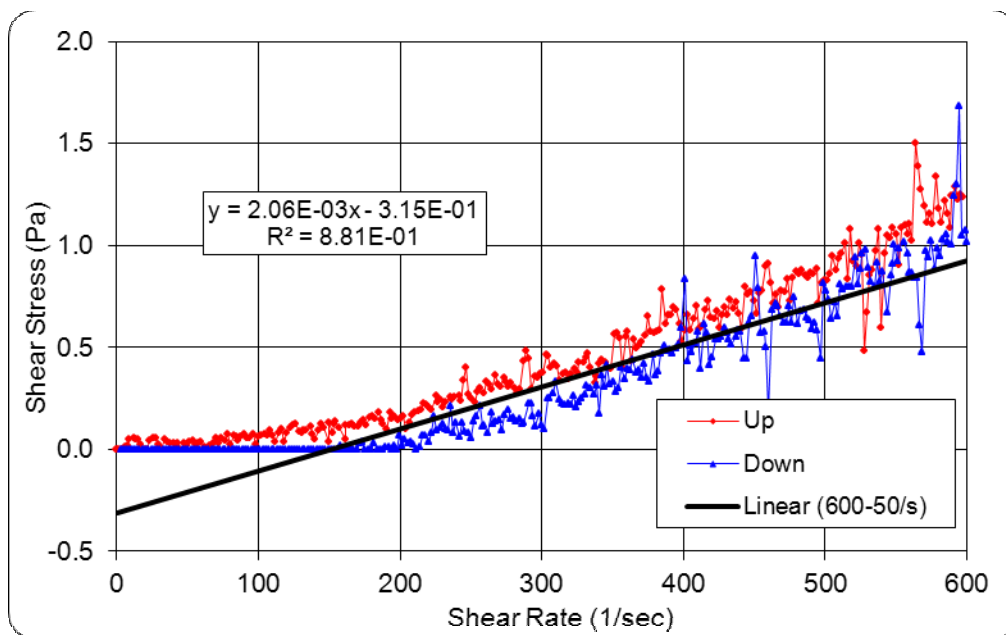


Figure B-14: Stress Versus Strain Curve for Tank 5F Midpoint Concentrate at 30 °C (Second Replicate).

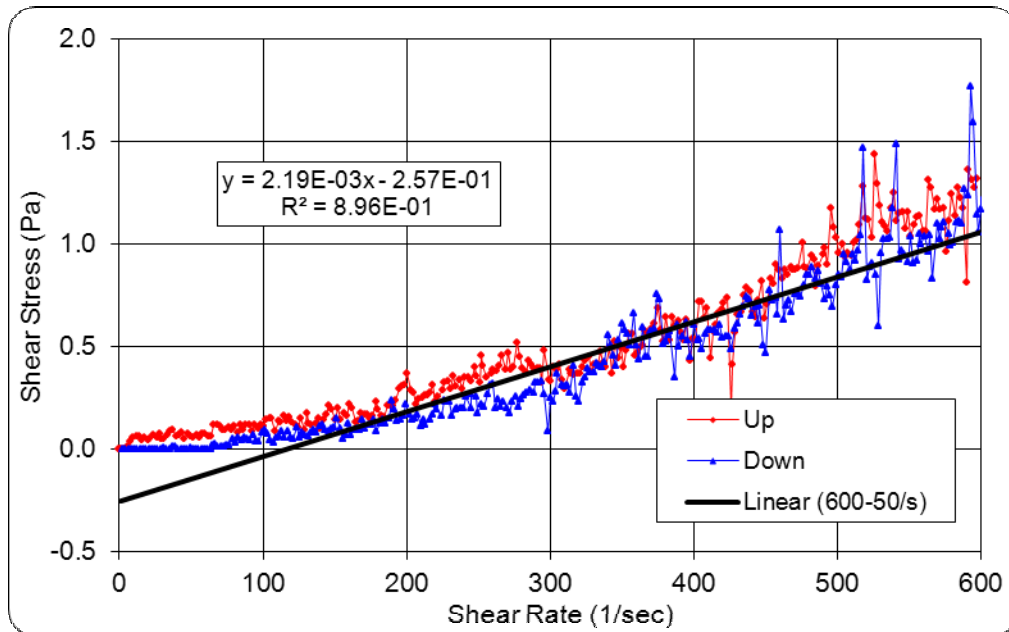


Figure B-15: Stress Versus Strain Curve for Tank 5F Endpoint Concentrate at 30 °C (First Replicate).

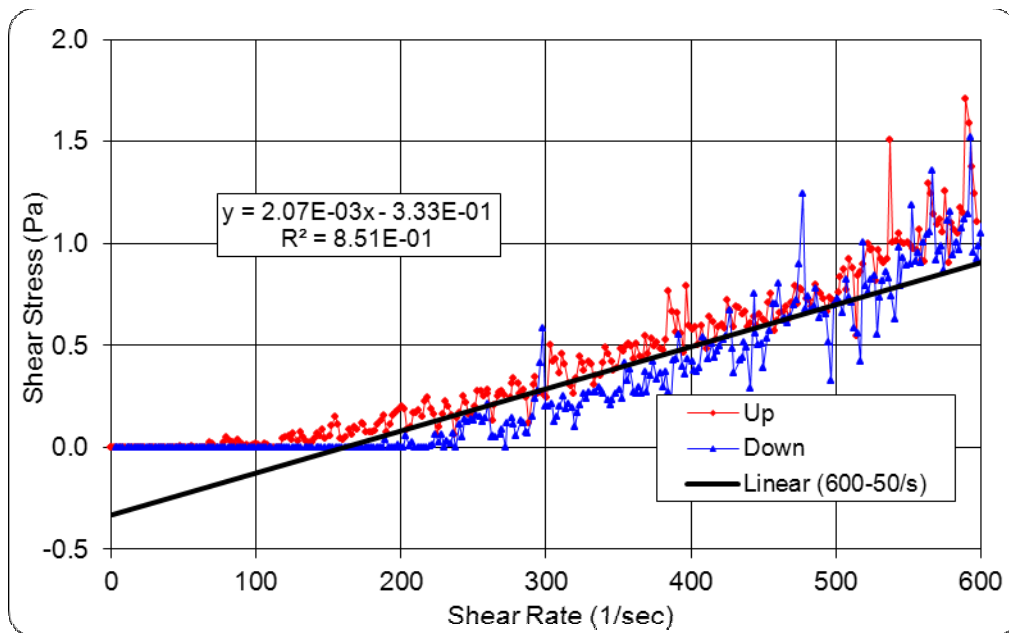


Figure B-16: Stress Versus Strain Curve for Tank 5F Endpoint Concentrate at 30 °C (Second Replicate).

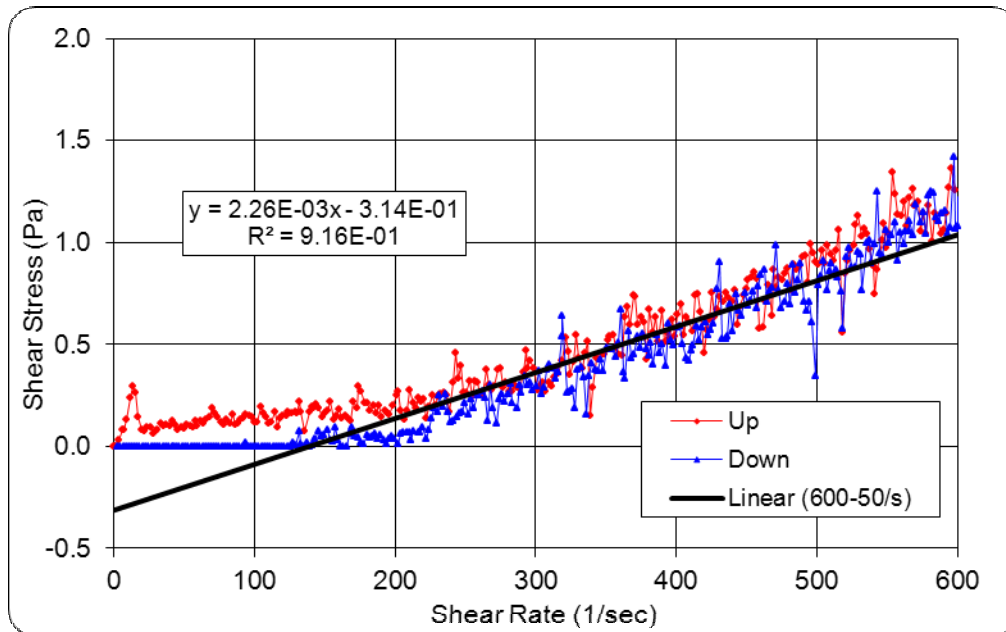


Figure B-17: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 30 °C (First Replicate)

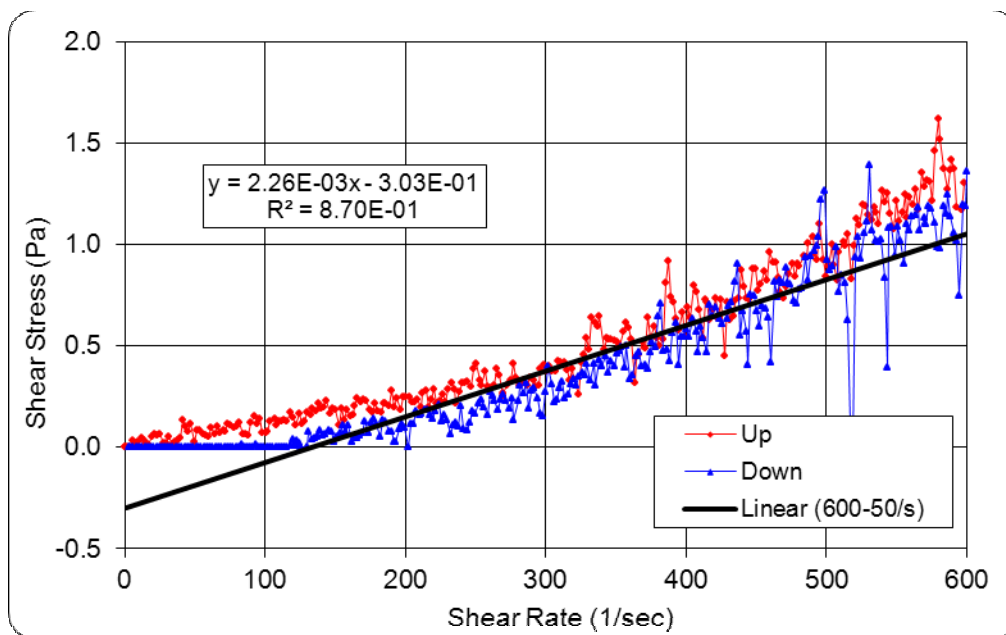


Figure B-18: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 30 °C (Second Replicate)

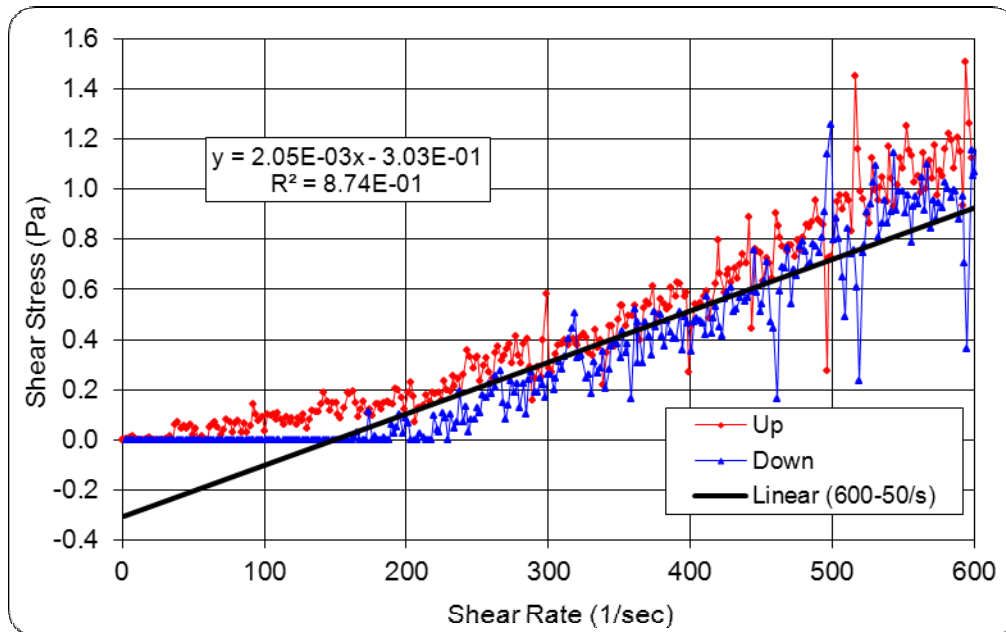


Figure B-19: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 40 °C (First Replicate)

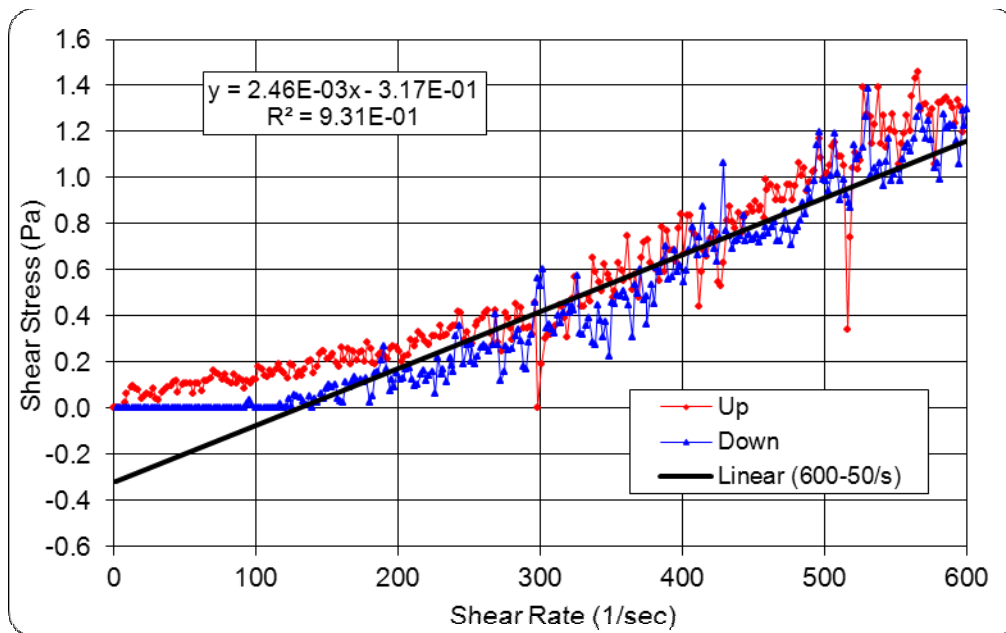


Figure B-20: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 40 °C (Second Replicate)

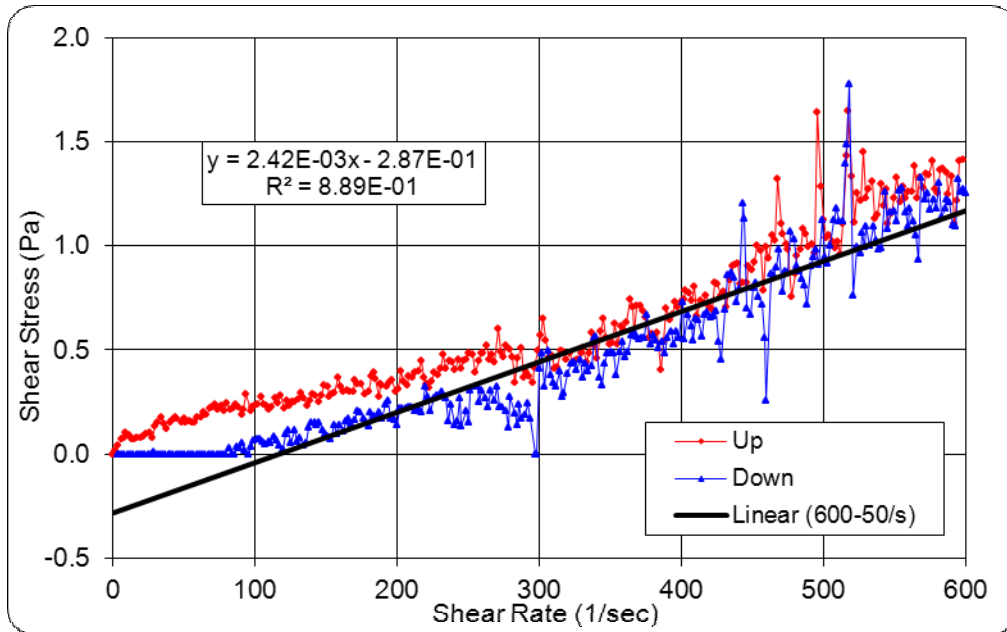


Figure B-21: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 50 °C (First Replicate)

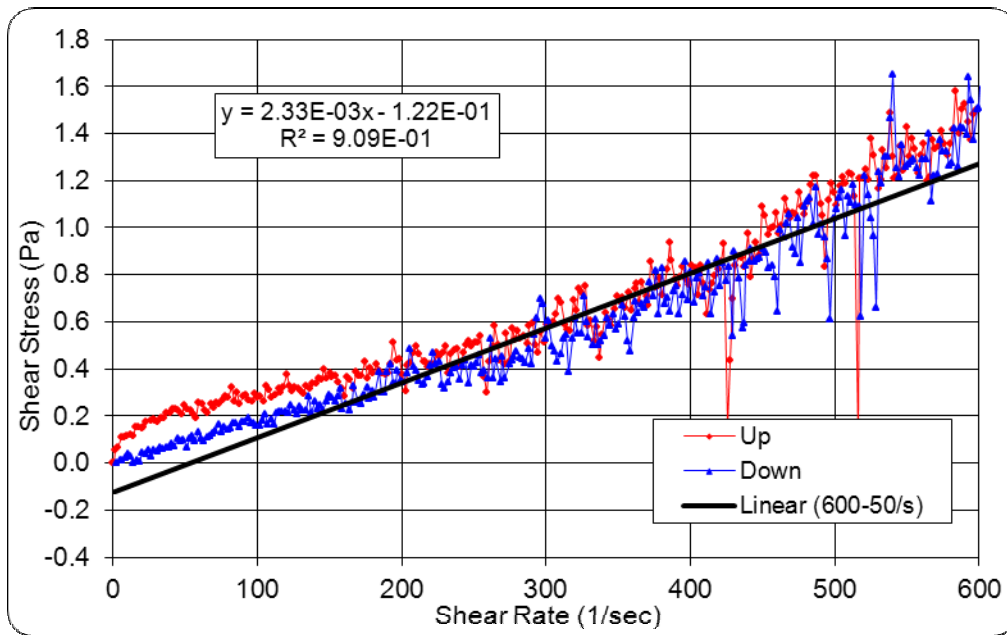


Figure B-22: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 50 °C (Second Replicate)

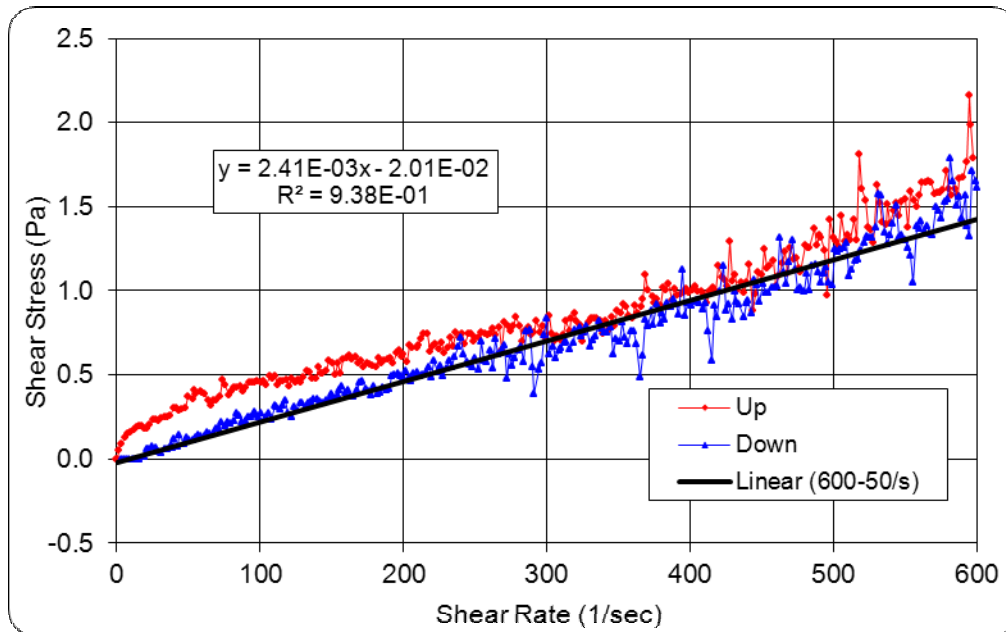


Figure B-23: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 30 °C after 1 Day (First Replicate)

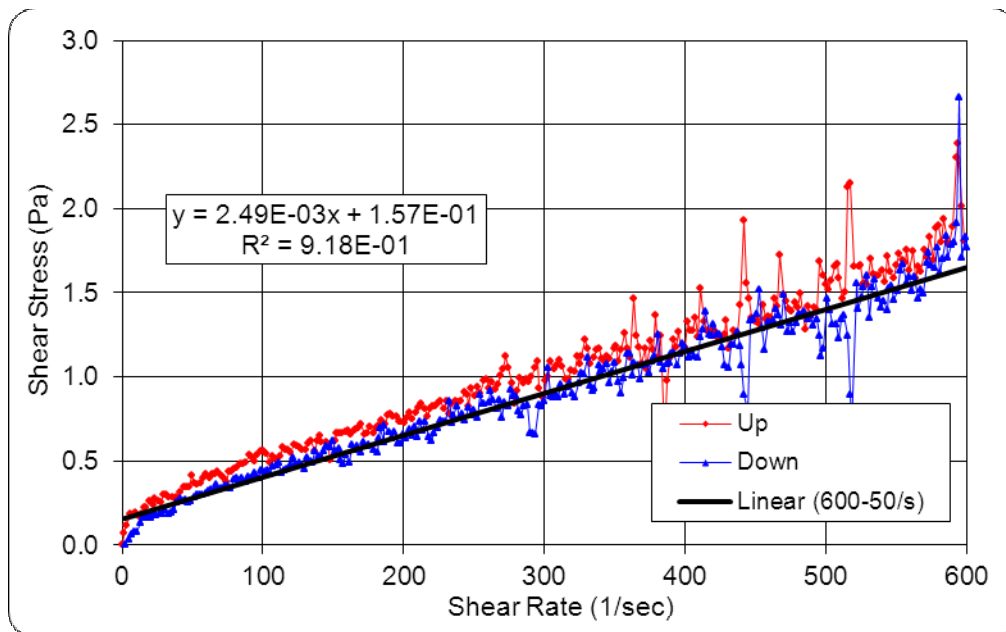


Figure B-24: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 30 °C after 1 Day (Second Replicate)

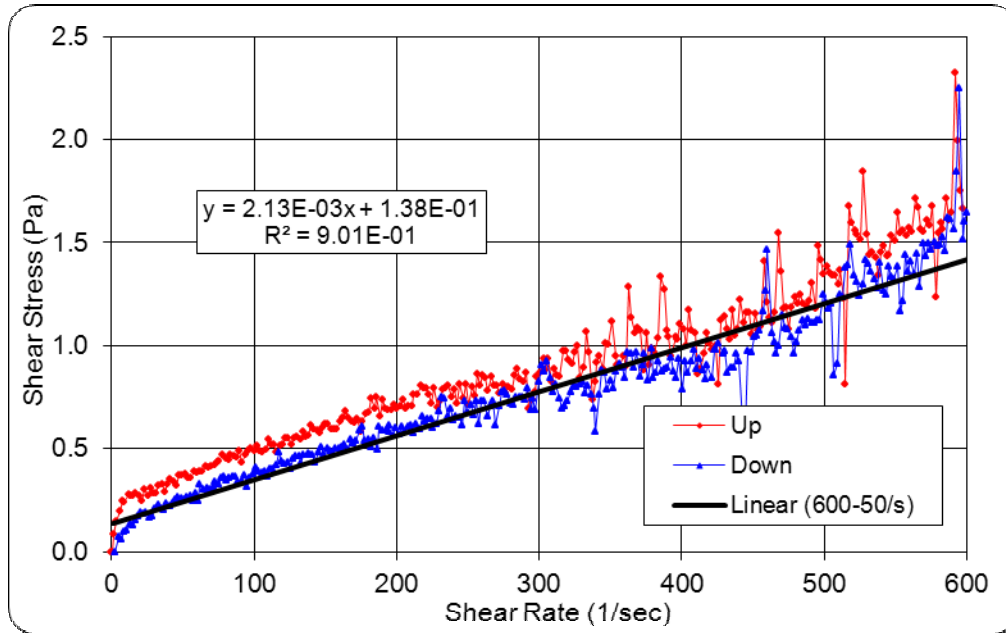


Figure B-25: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 40 °C after 1 Day (First Replicate)

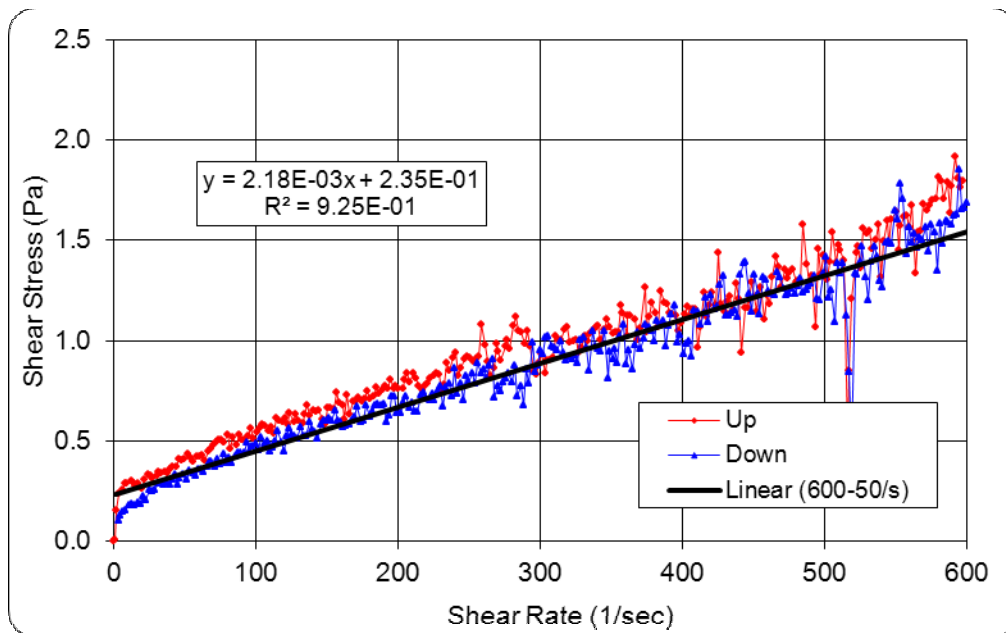


Figure B-26: Stress Versus Strain Curve for Tank 12H Baseline Dilute at 40 °C after 1 Day (Second Replicate)

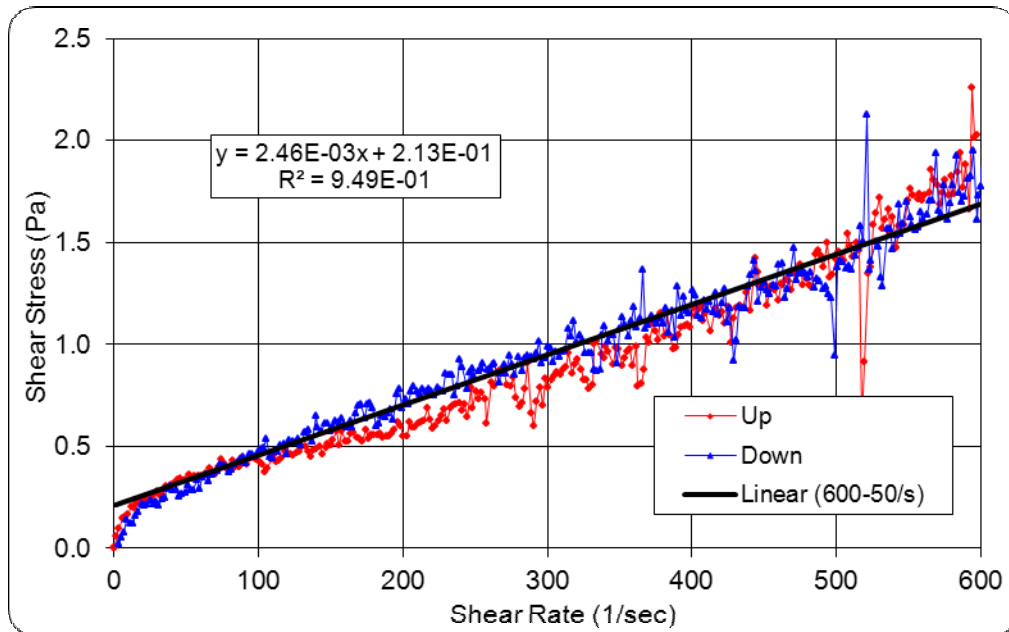


Figure B-27: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 30 °C (First Replicate)

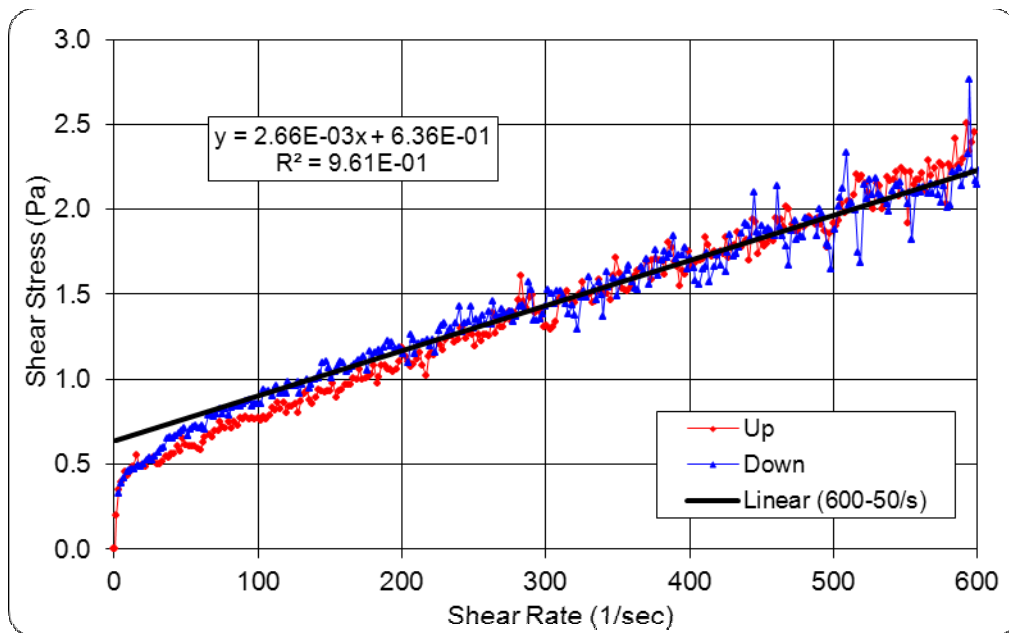


Figure B-28: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 30 °C (Second Replicate)

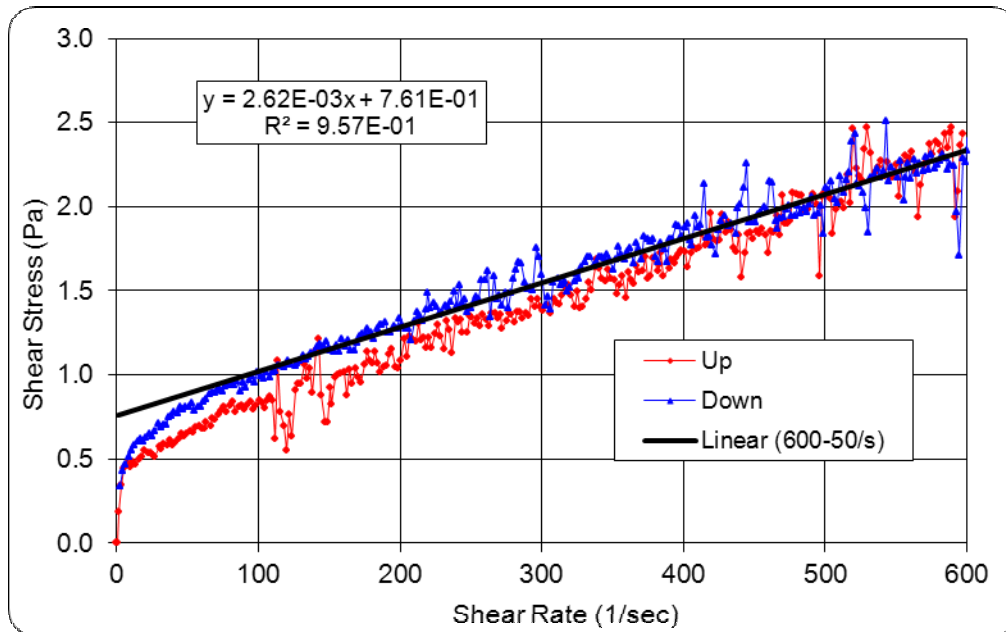


Figure B-29: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 40 °C (First Replicate)

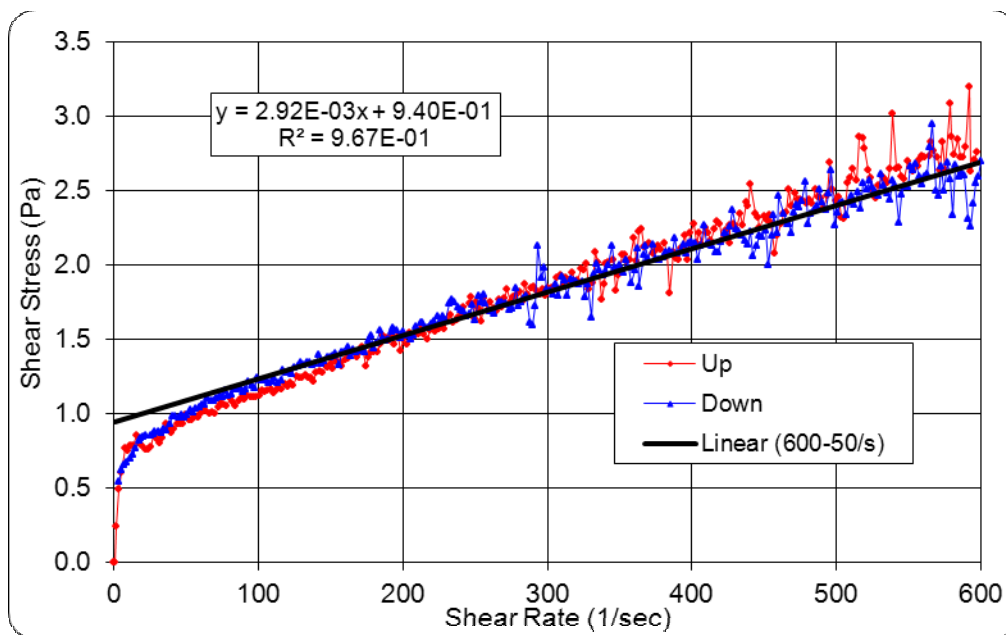


Figure B-30: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 40 °C (Second Replicate)

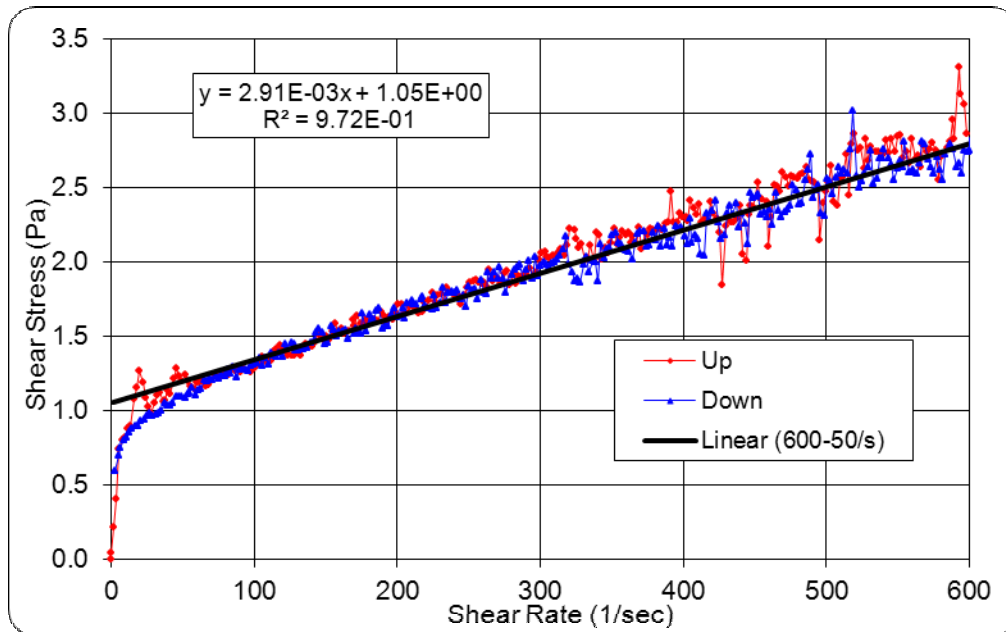


Figure B-31: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 50 °C (First Replicate)

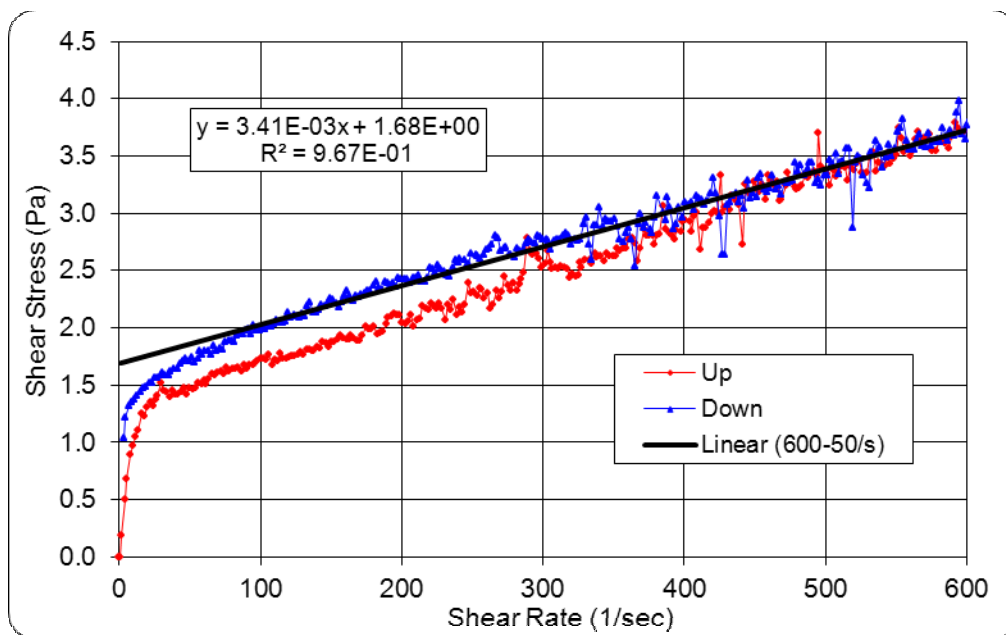


Figure B-32: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 50 °C (Second Replicate)

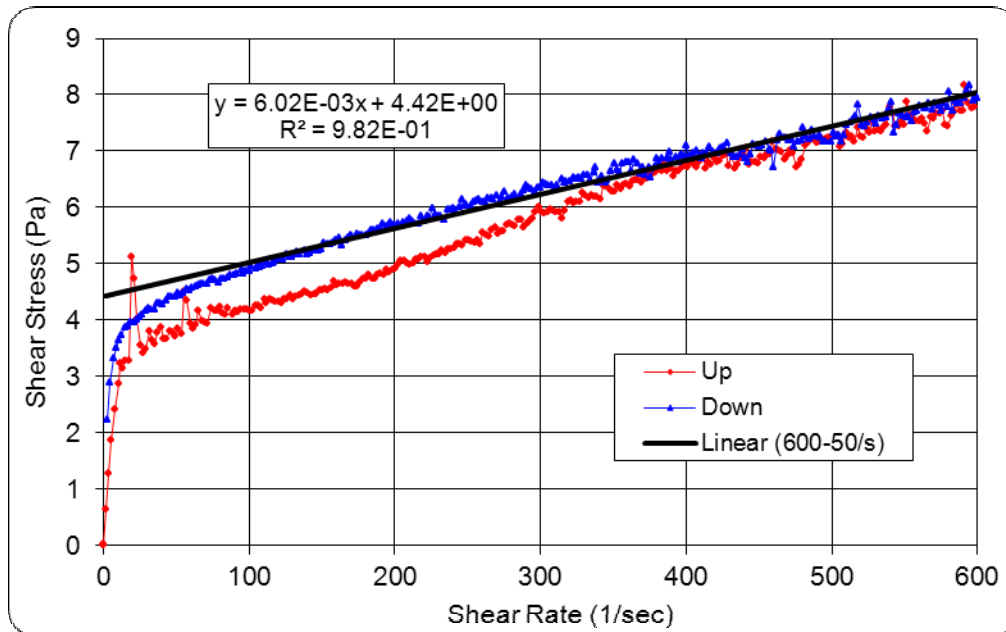


Figure B-33: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 30 °C after 4 Days (First Replicate)

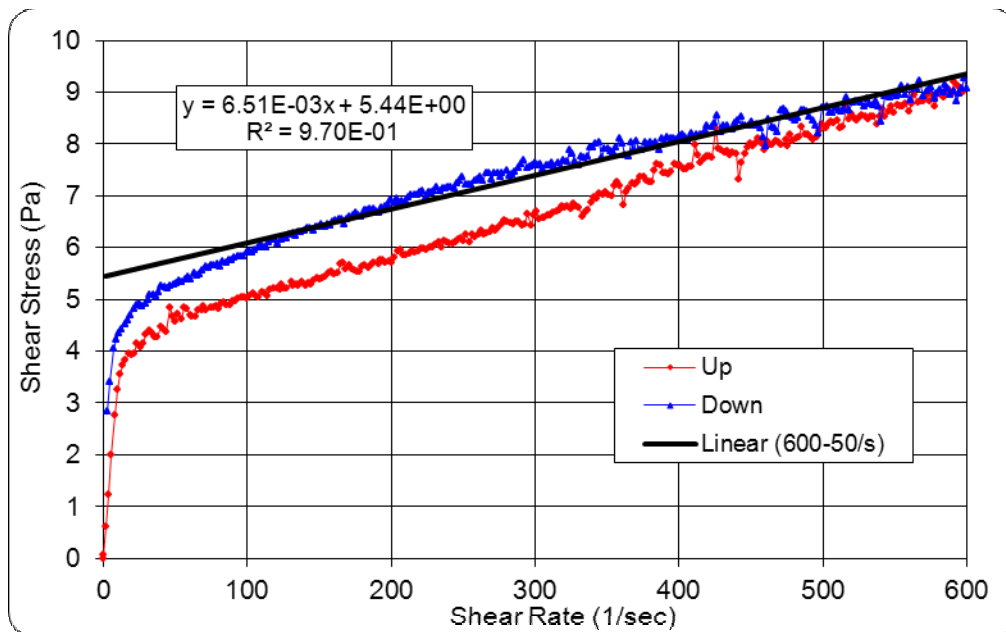


Figure B-34: Stress Versus Strain Curve for Tank 12H Baseline Concentrate at 30 °C after 4 Days (Second Replicate)

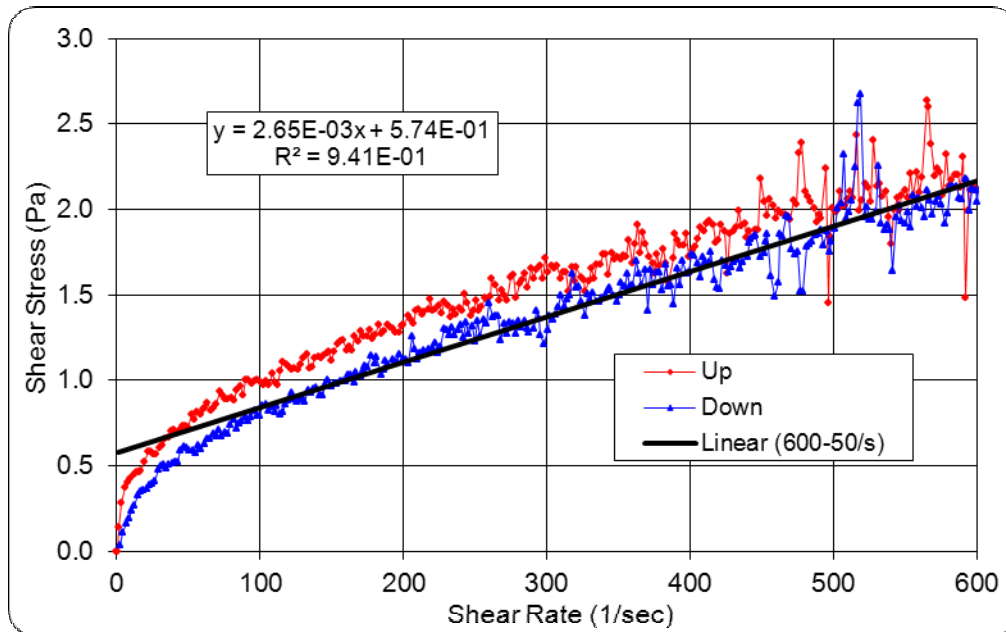


Figure B-35: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 30 °C (First Replicate)

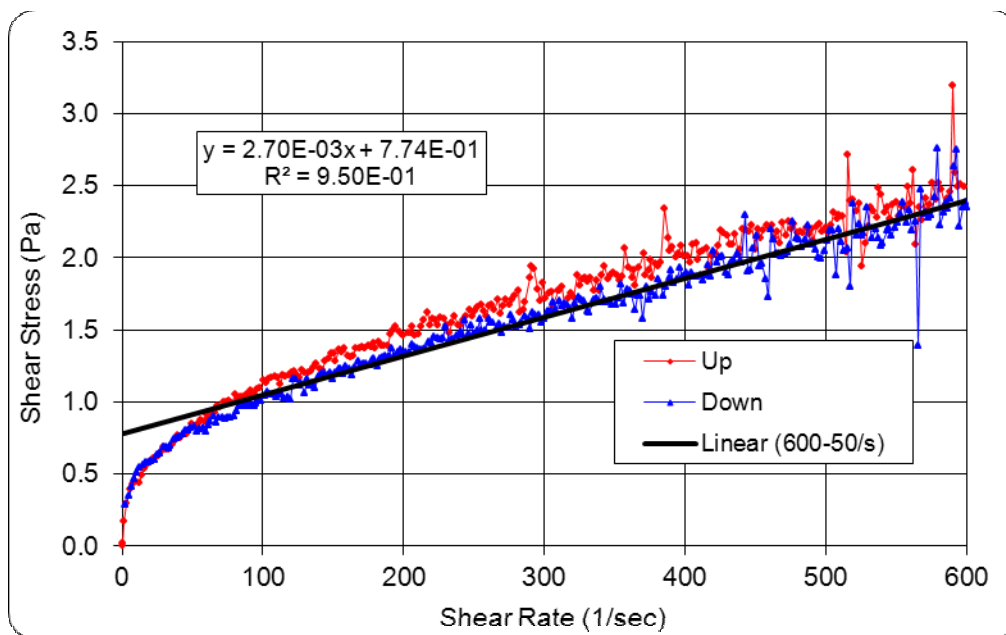


Figure B-36: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 30 °C (Second Replicate)

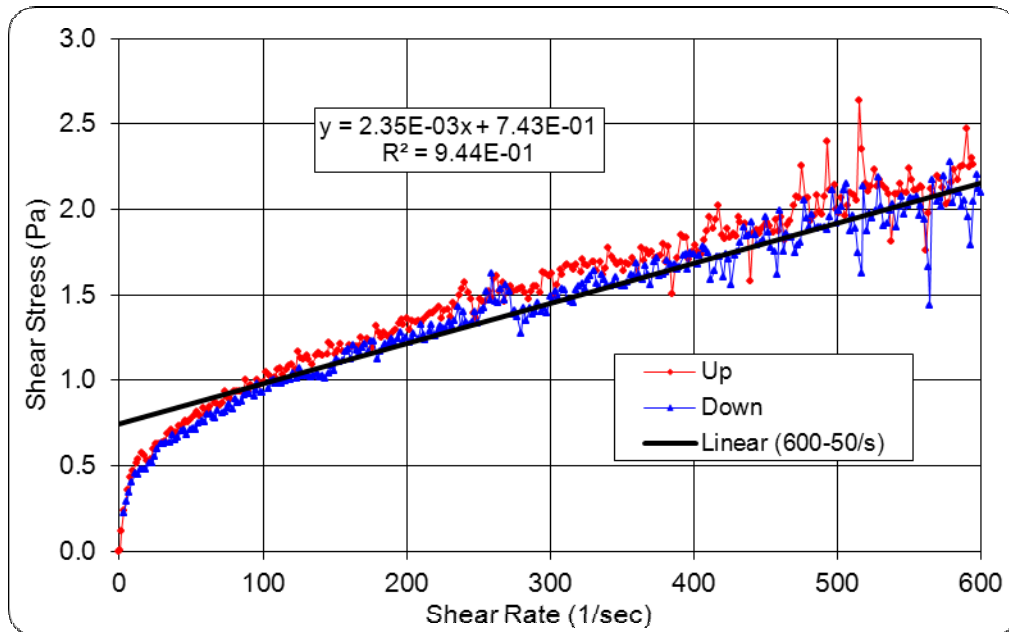


Figure B-37: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 40 °C (First Replicate)

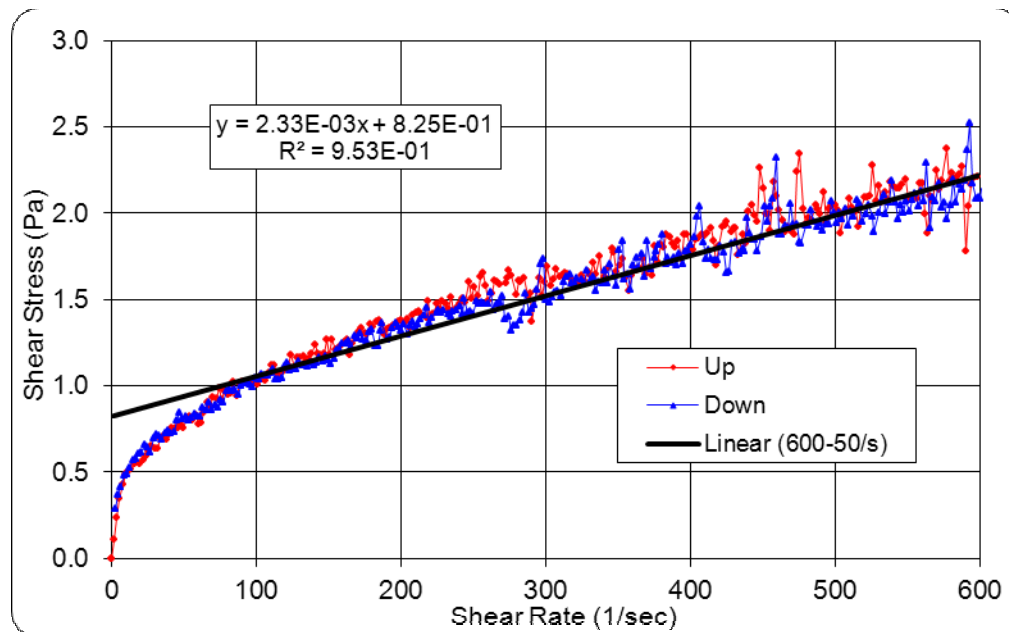


Figure B-38: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 40 °C (Second Replicate)

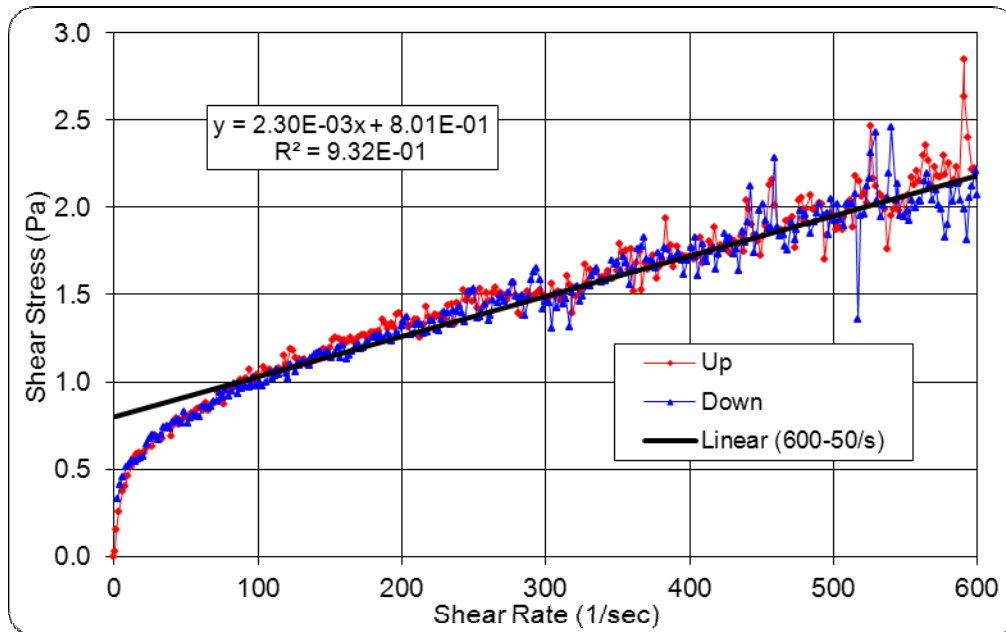


Figure B-39: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 50 °C (First Replicate)

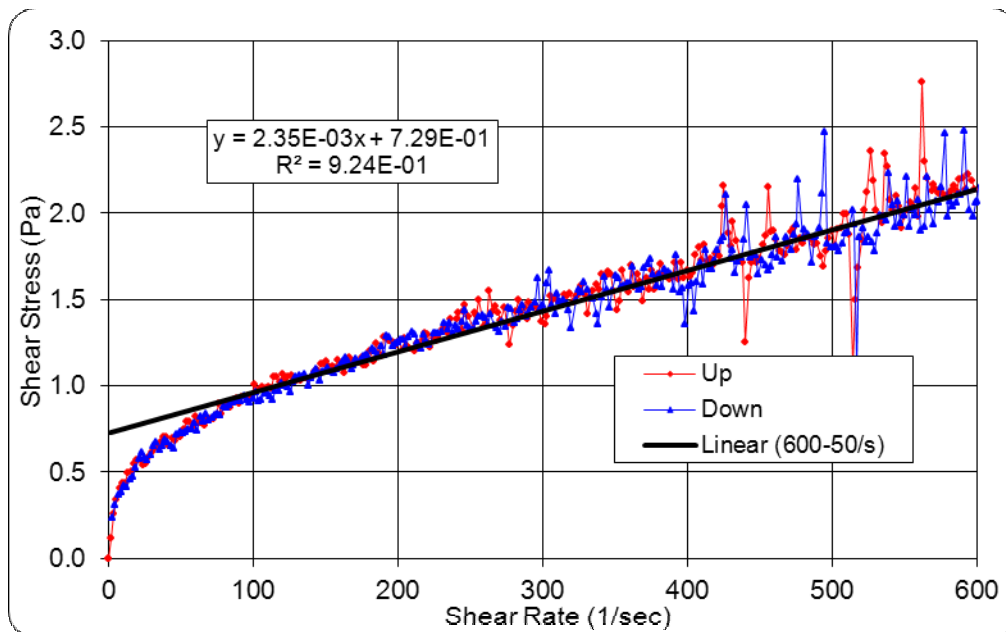


Figure B-40: Stress Versus Strain Curve for Tank 12H Midpoint Dilute at 50 °C (Second Replicate)

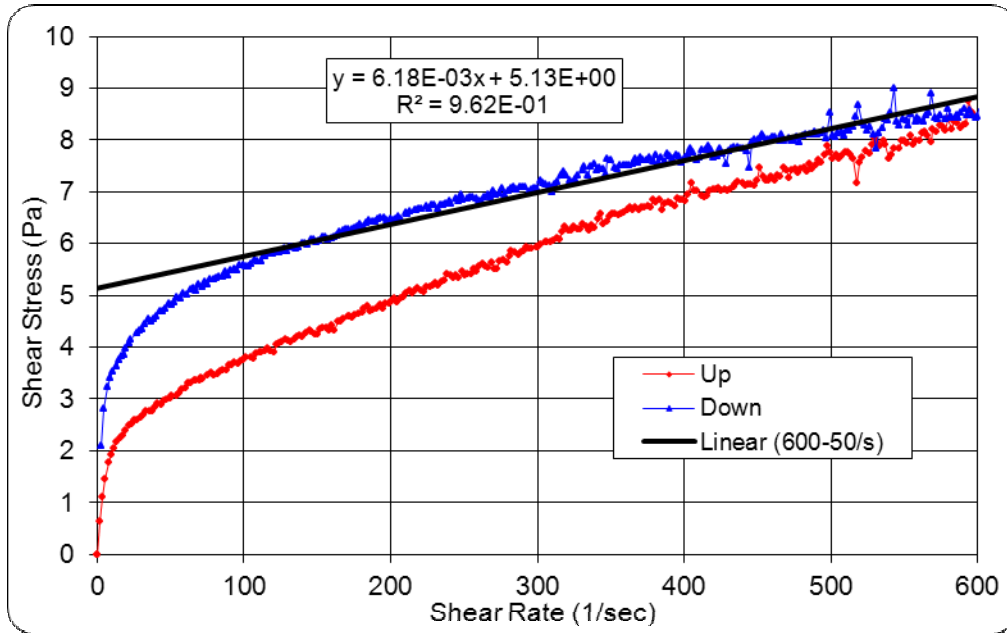


Figure B-41: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 30 °C (First Replicate)

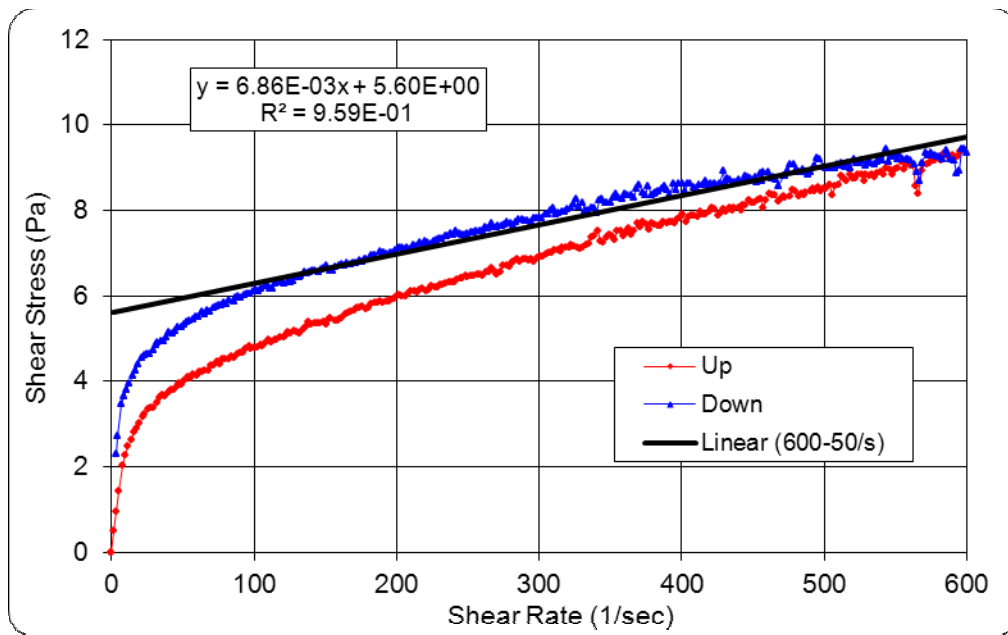


Figure B-42: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 30 °C (Second Replicate)

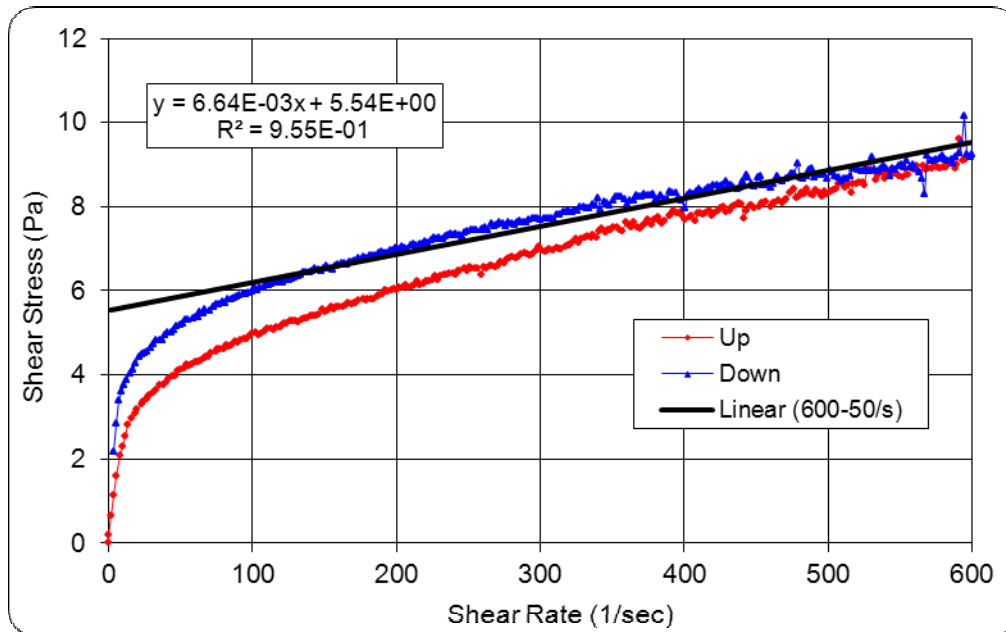


Figure B-43: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 40 °C (First Replicate)

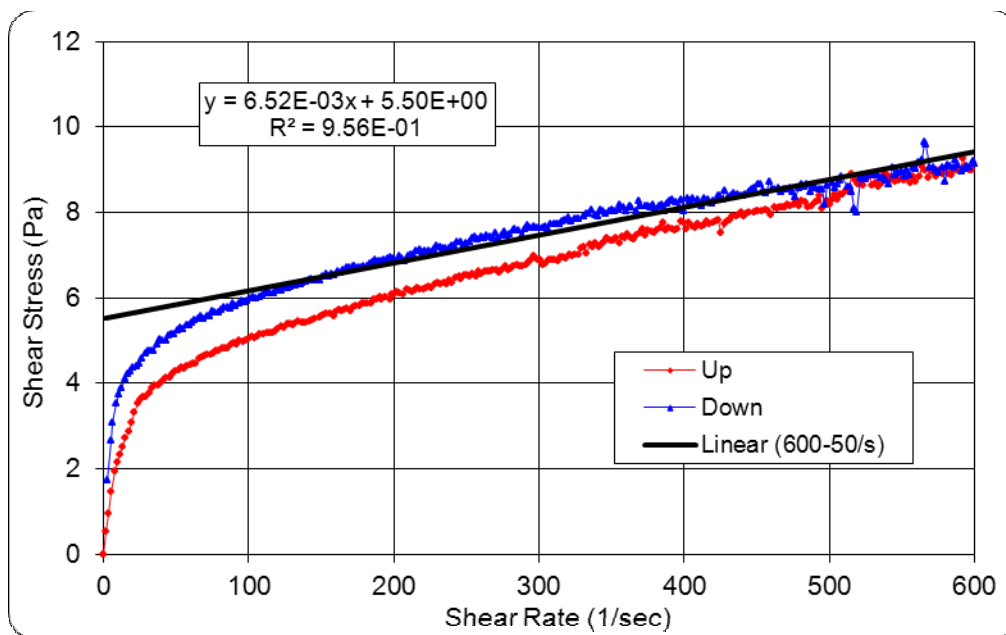


Figure B-44: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 40 °C (Second Replicate)

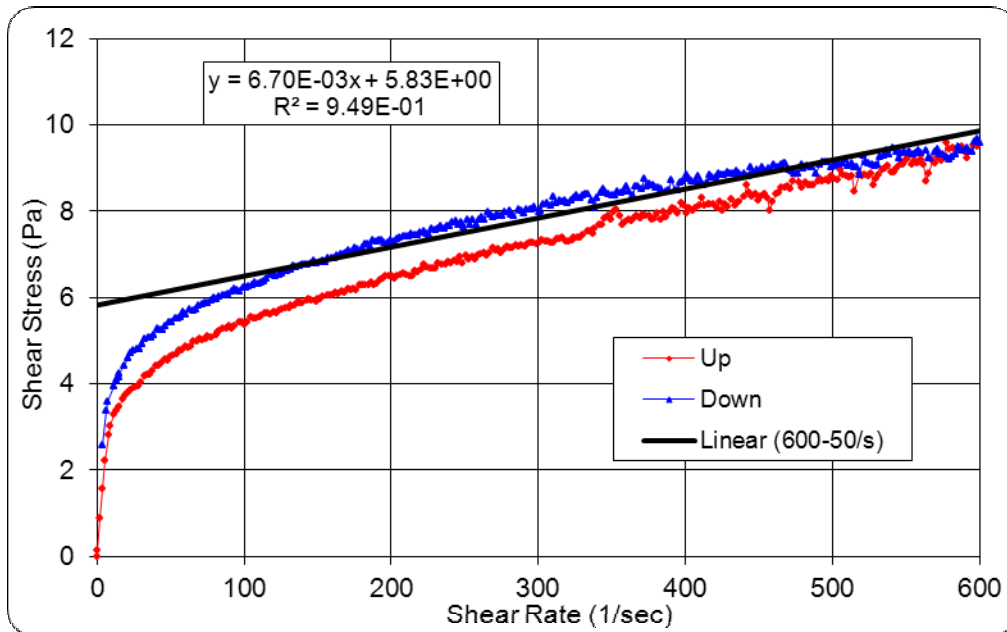


Figure B-45: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 50 °C (First Replicate)

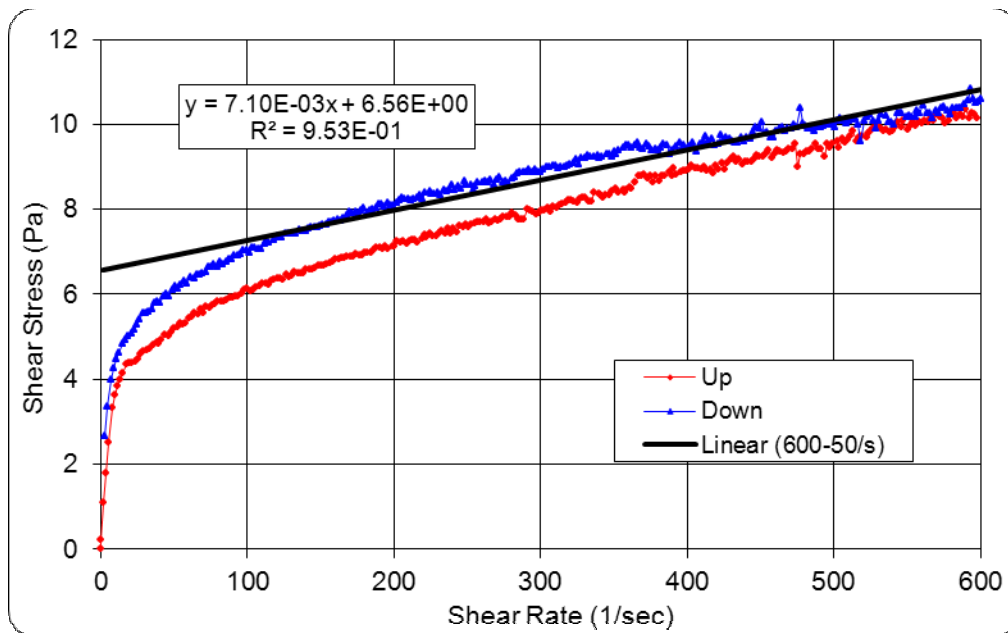


Figure B-46: Stress Versus Strain Curve for Tank 12H Midpoint Concentrate at 50 °C (Second Replicate)

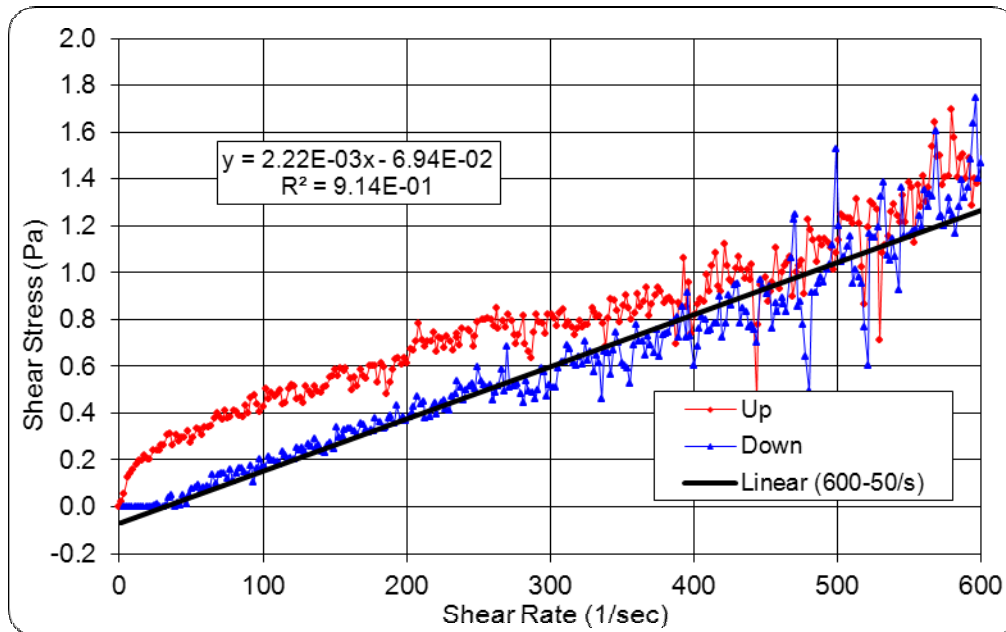


Figure B-47: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 30 °C (First Replicate)

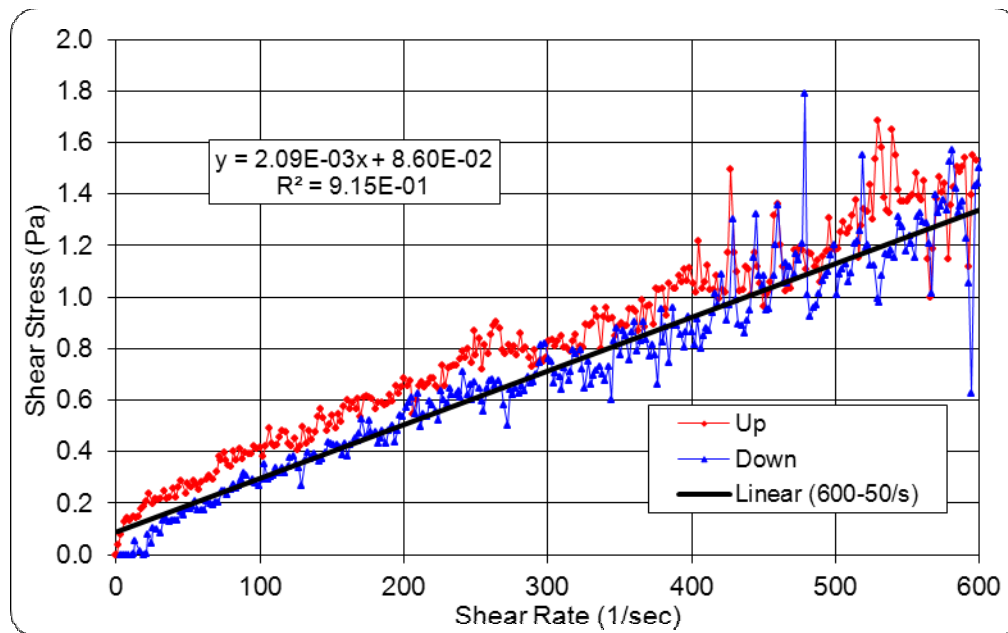


Figure B-48: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 30 °C (Second Replicate)

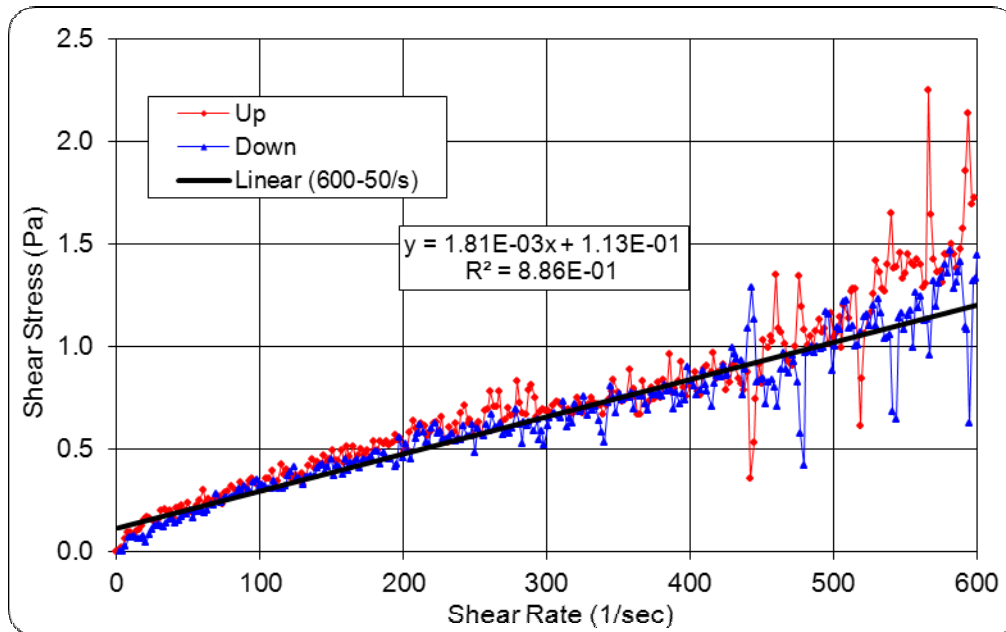


Figure B-49: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 40 °C (First Replicate)

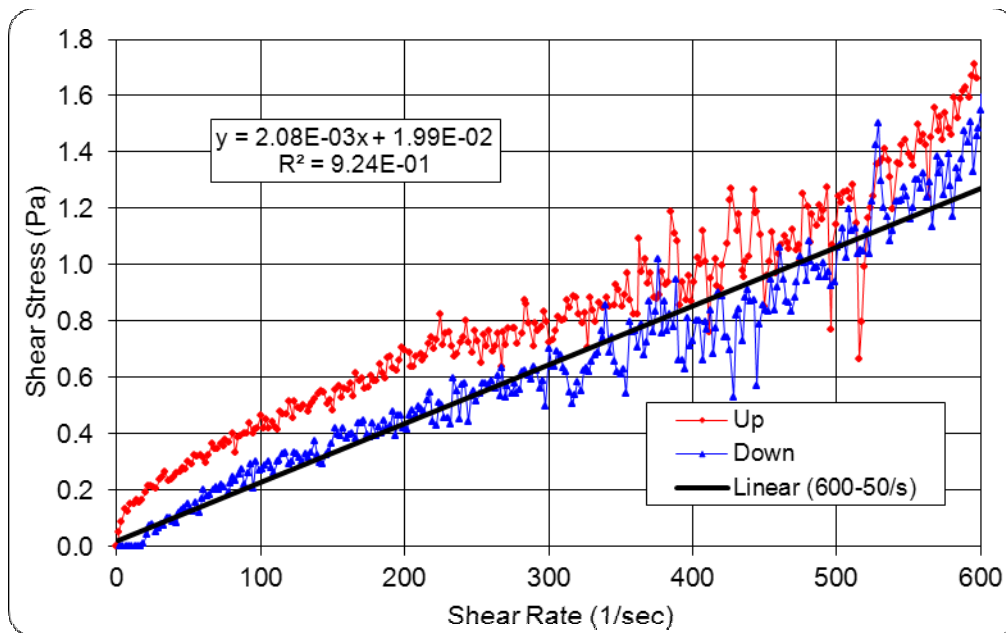


Figure B-50: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 40 °C (Second Replicate)

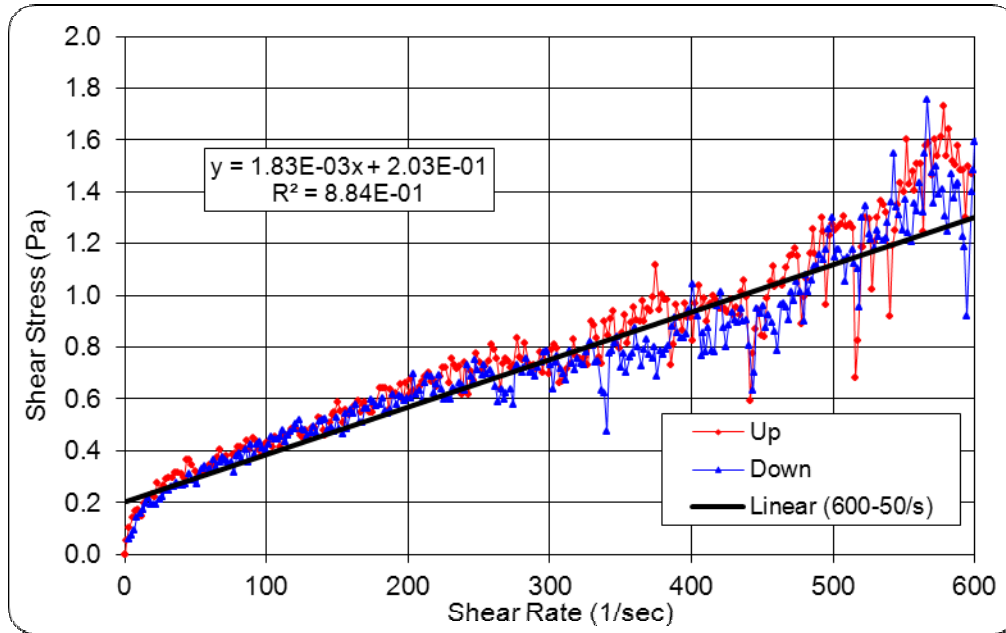


Figure B-51: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 50 °C (First Replicate)

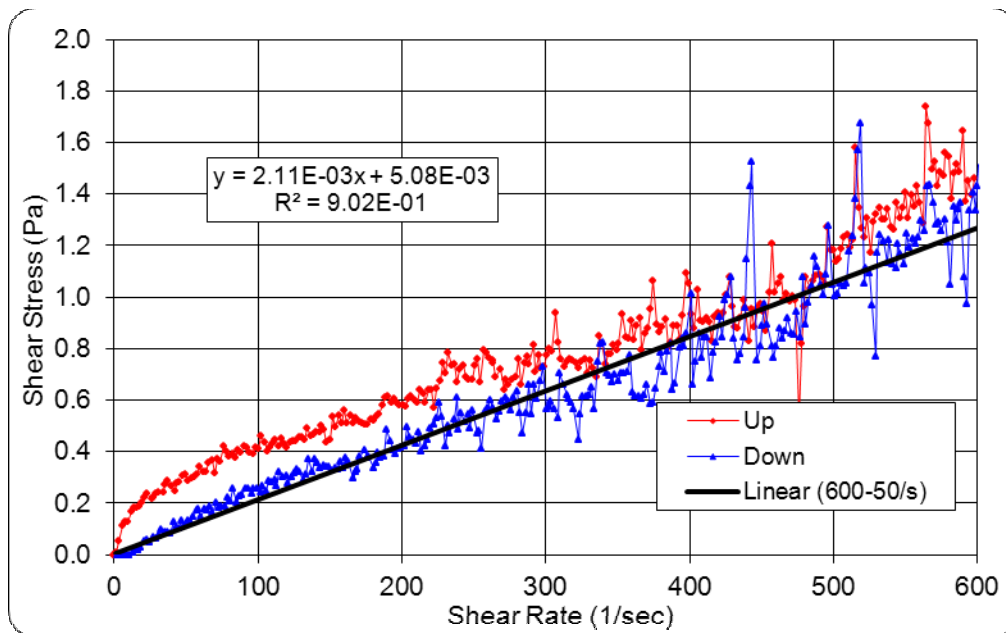


Figure B-52: Stress Versus Strain Curve for Tank 12H Endpoint Dilute at 50 °C (Second Replicate)

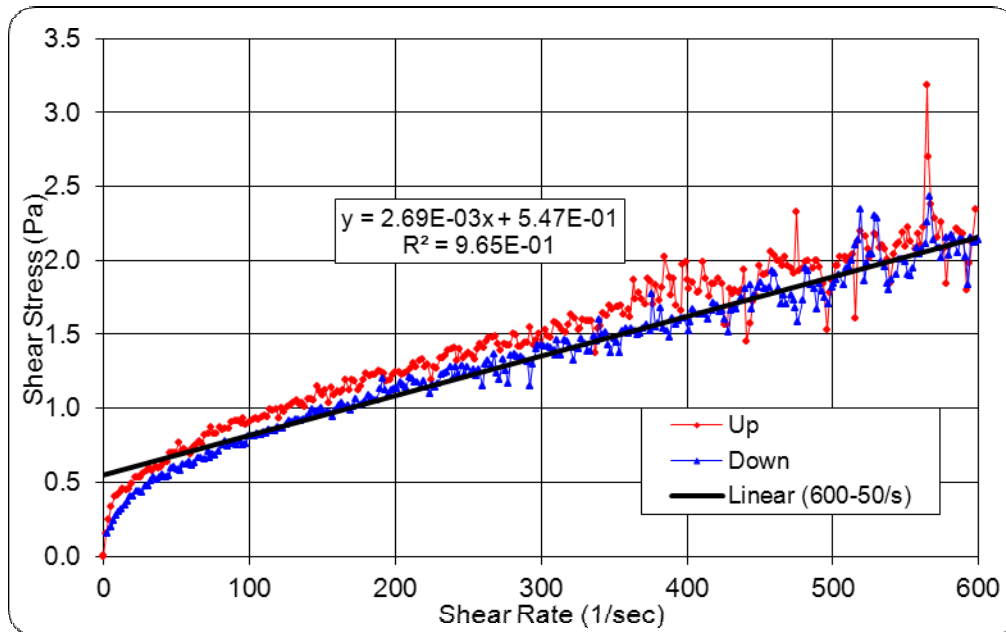


Figure B-53: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 30 °C (First Replicate)

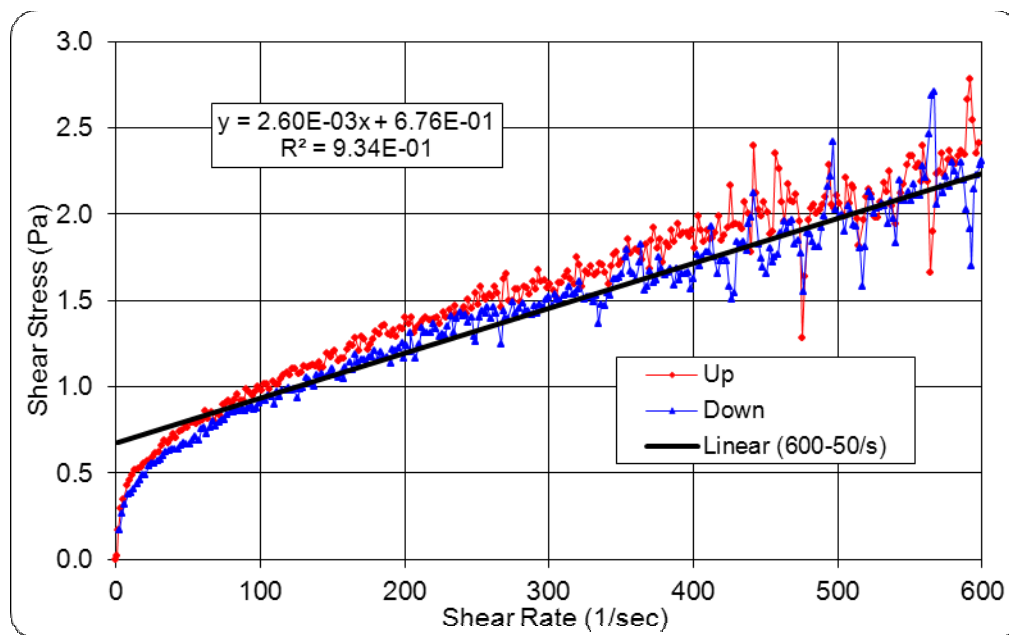


Figure B-54: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 30 °C (Second Replicate)

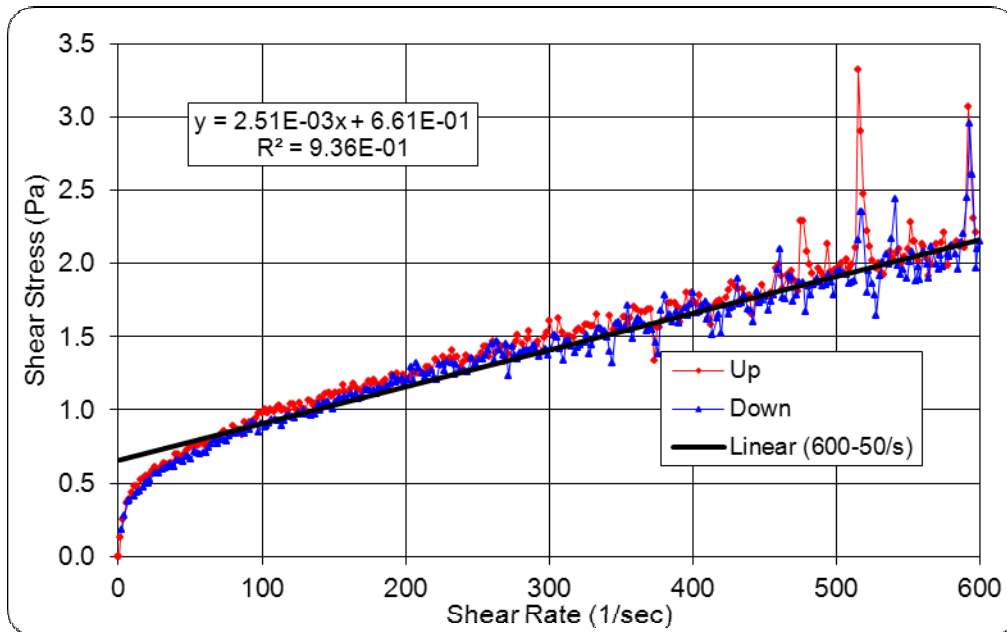


Figure B-55: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 40 °C (First Replicate)

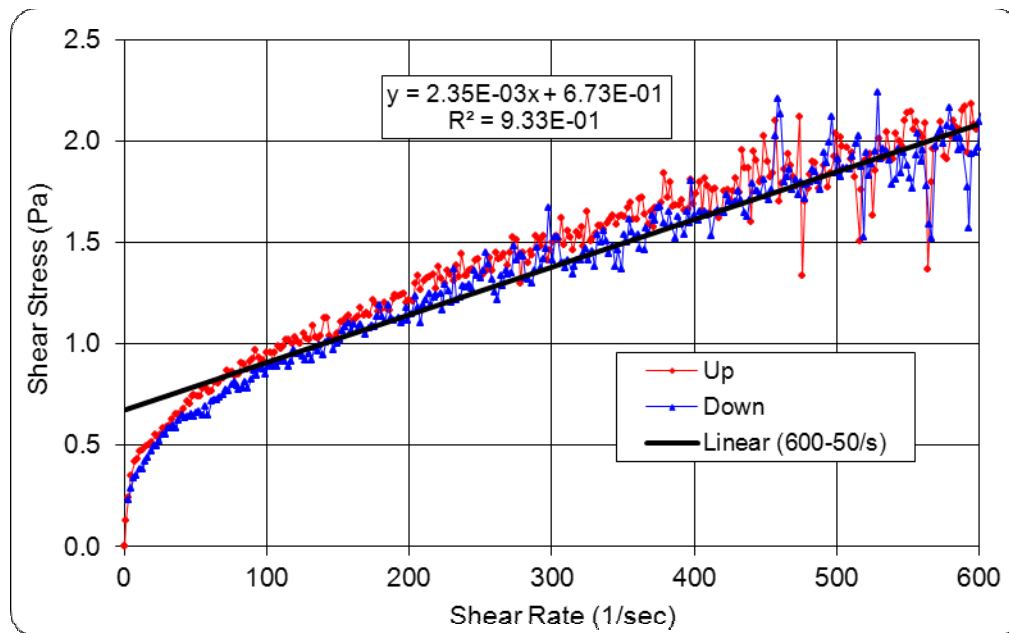


Figure B-56: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 40 °C (Second Replicate)

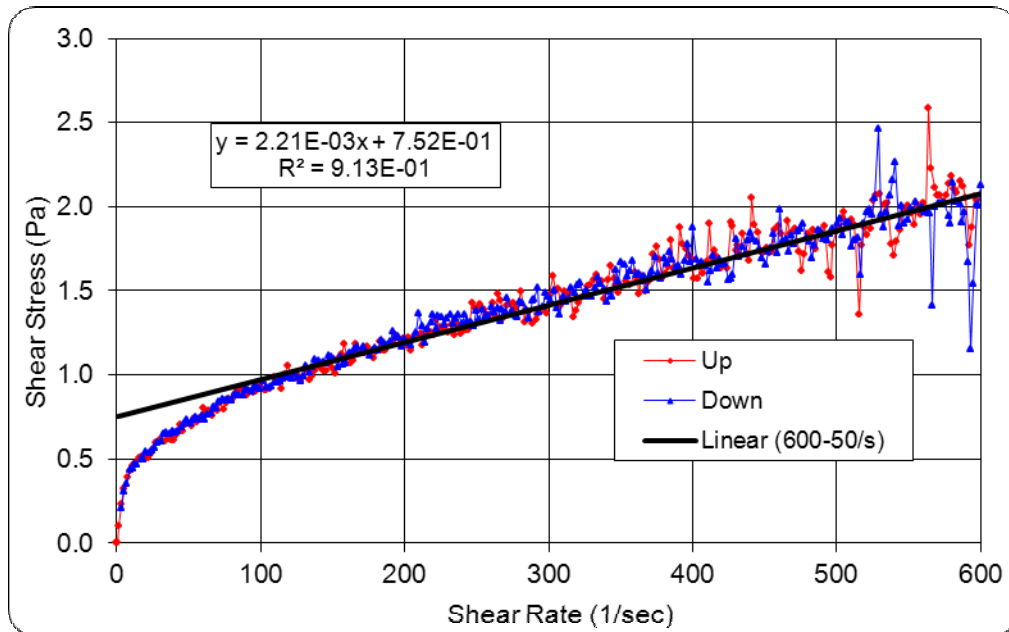


Figure B-57: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 50 °C (First Replicate)

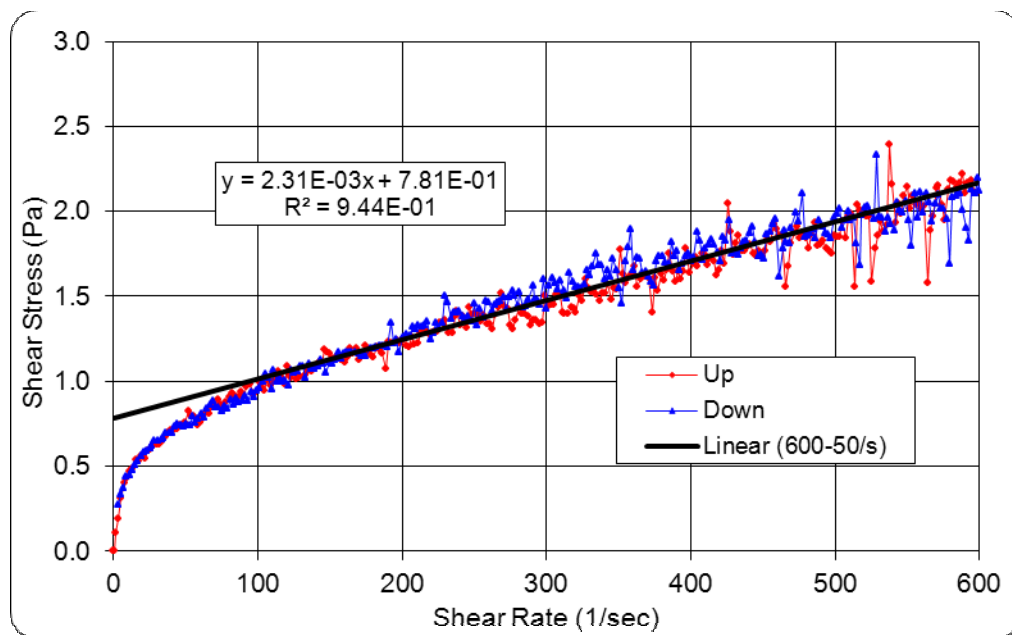


Figure B-58: Stress Versus Strain Curve for Tank 12H Endpoint Concentrate at 50 °C (Second Replicate)

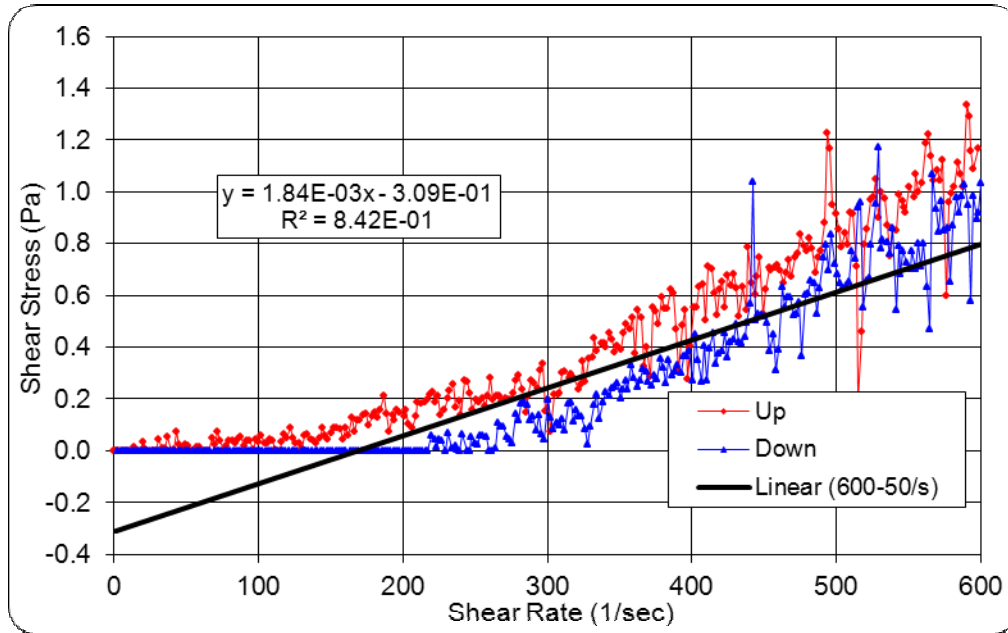


Figure B-59: Stress Versus Strain Curve for Tank 5F No Evaporation Deposition Sample at 30 °C (First Replicate)

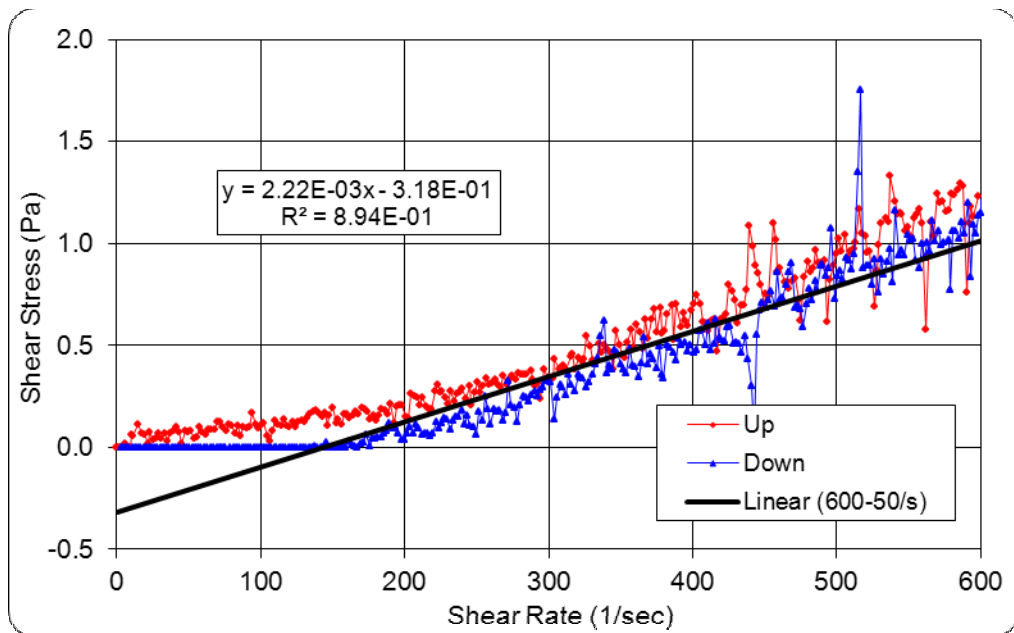


Figure B-60: Stress Versus Strain Curve for Tank 5F No Evaporation Deposition Sample at 30 °C (Second Replicate)

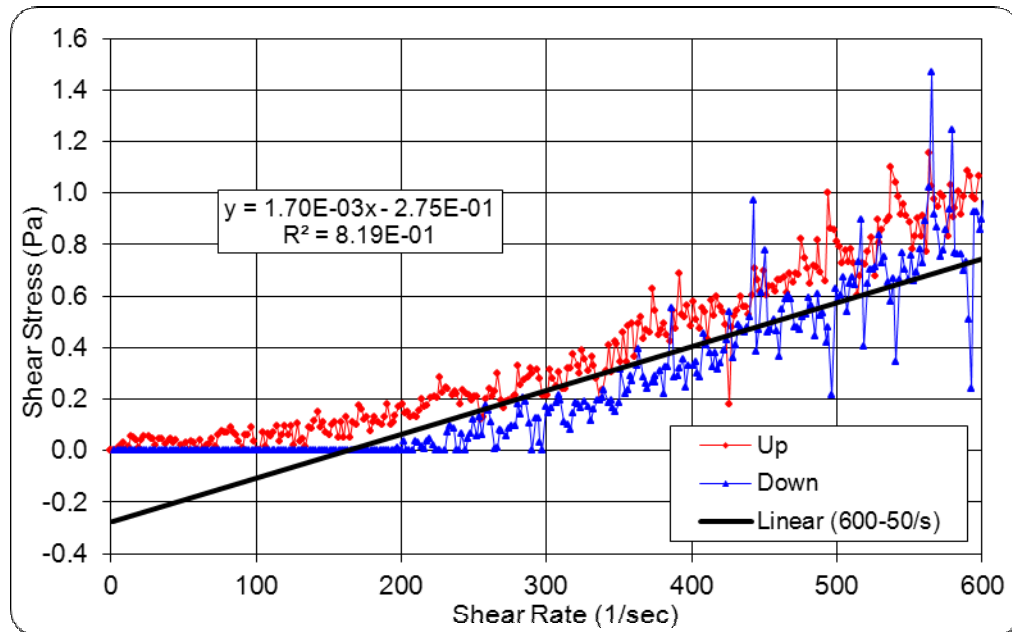


Figure B-61: Stress Versus Strain Curve for Tank 5F No Evaporation Deposition Sample at 45 °C (First Replicate)

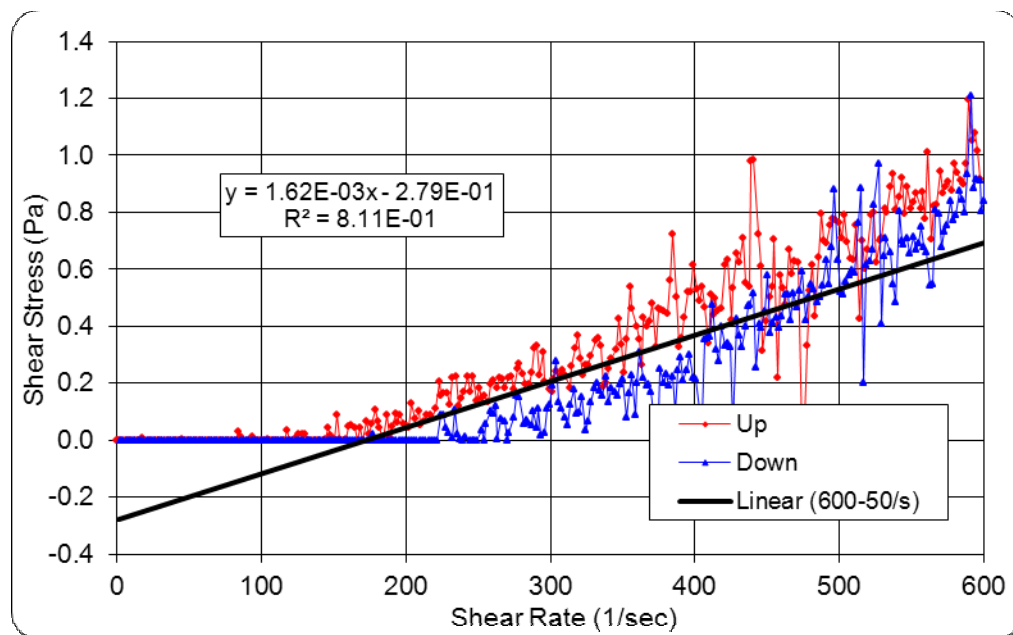


Figure B-62: Stress Versus Strain Curve for Tank 5F No Evaporation Deposition Sample at 45 °C (Second Replicate)

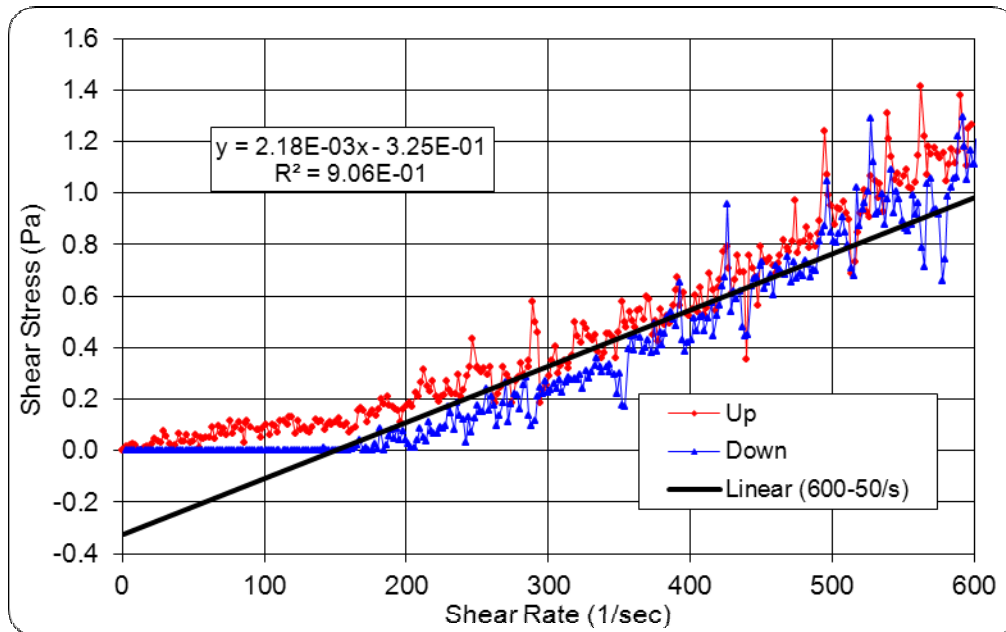


Figure B-63: Stress Versus Strain Curve for Tank 5F Evaporated Deposition Sample at 30 °C (First Replicate)

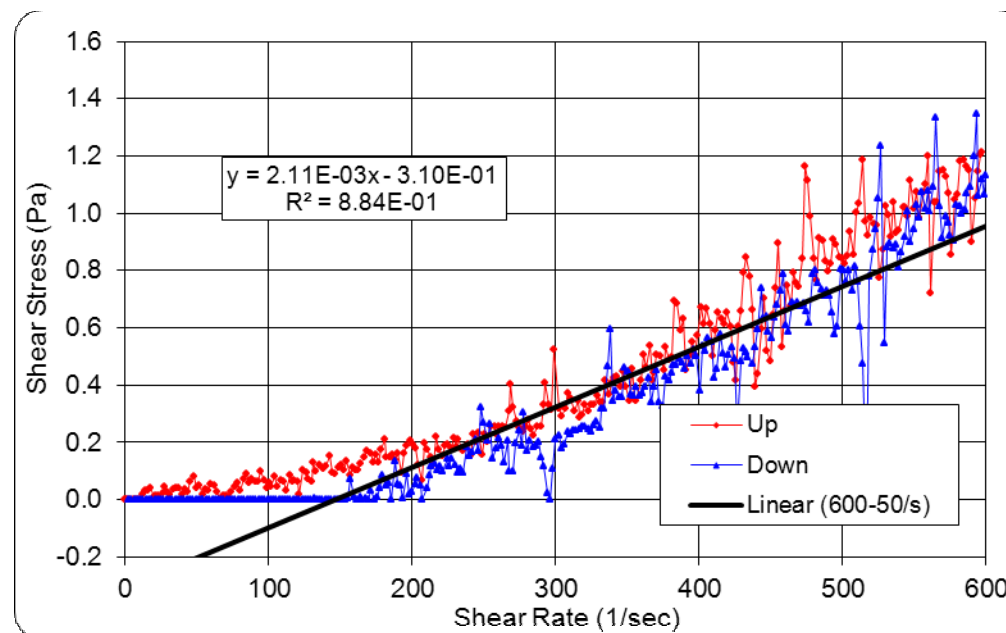


Figure B-64: Stress Versus Strain Curve for Tank 5F Evaporated Deposition Sample at 30 °C (Second Replicate)

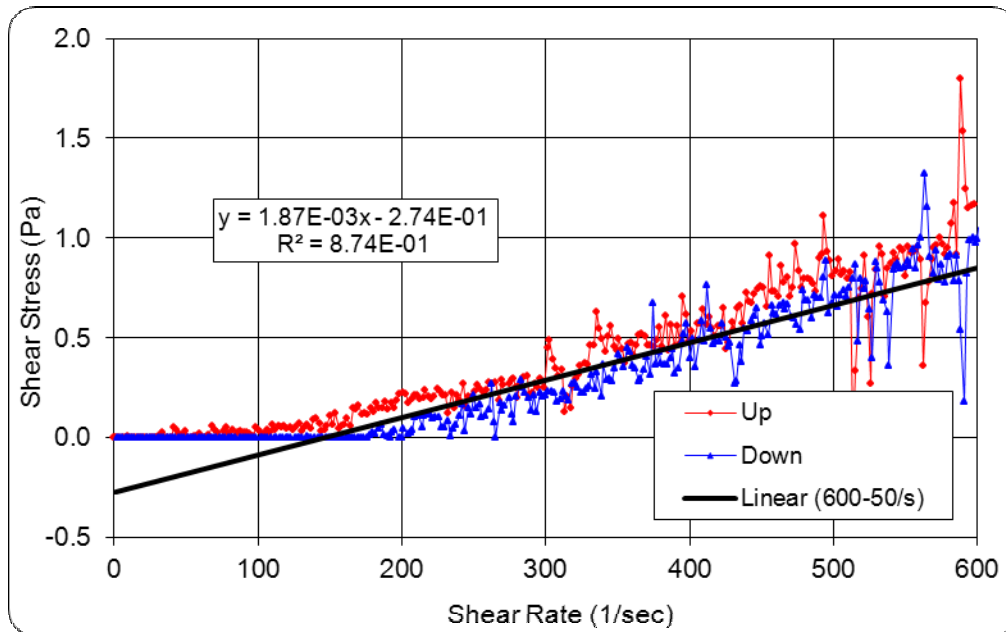


Figure B-65: Stress Versus Strain Curve for Tank 5F Evaporated Deposition Sample at 45 °C (First Replicate)

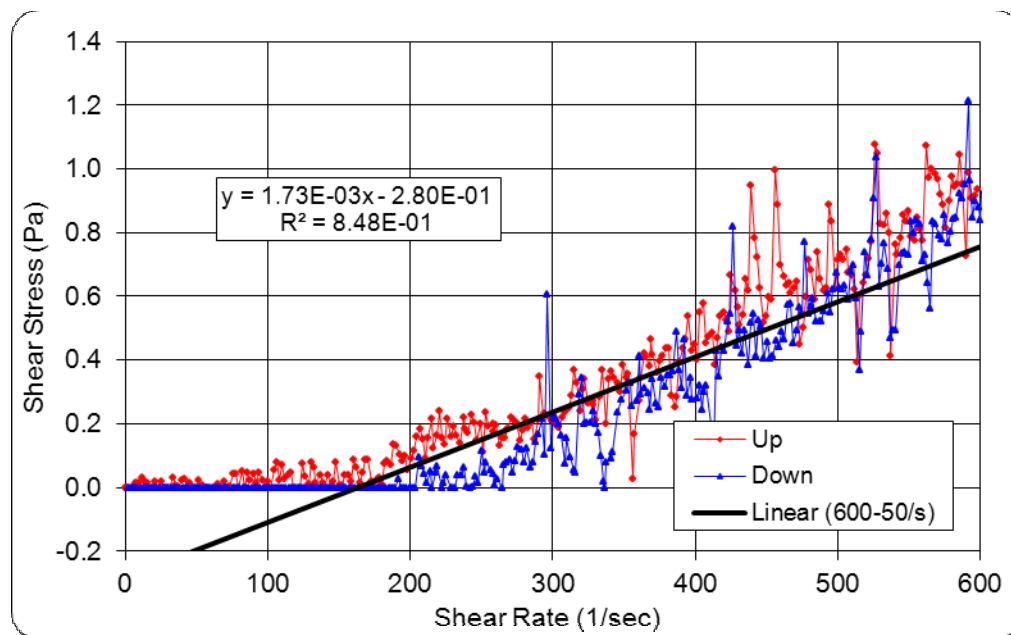


Figure B-66: Stress Versus Strain Curve for Tank 5F Evaporated Deposition Sample at 45 °C (Second Replicate)

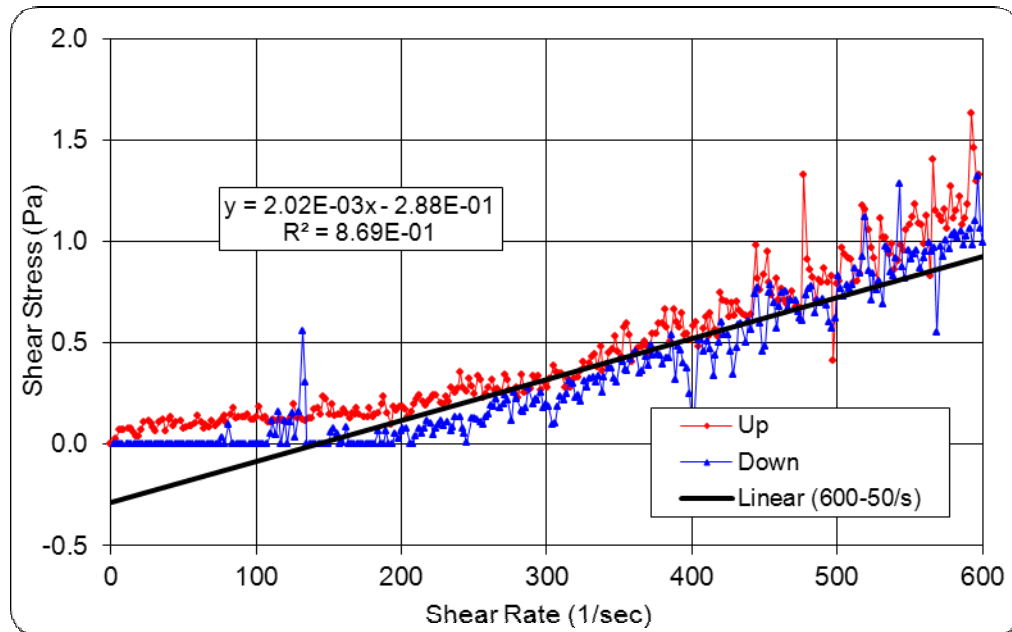


Figure B-67: Stress Versus Strain Curve for Tank 12H No Evaporation Deposition Sample at 30 °C (First Replicate)

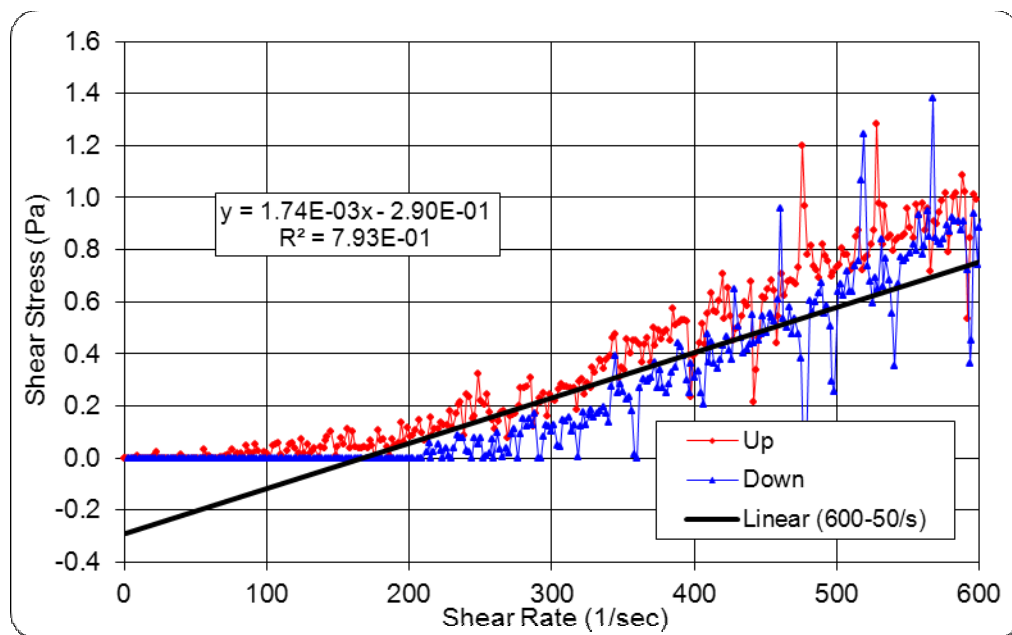


Figure B-68: Stress Versus Strain Curve for Tank 12H No Evaporation Deposition Sample at 30 °C (Second Replicate)

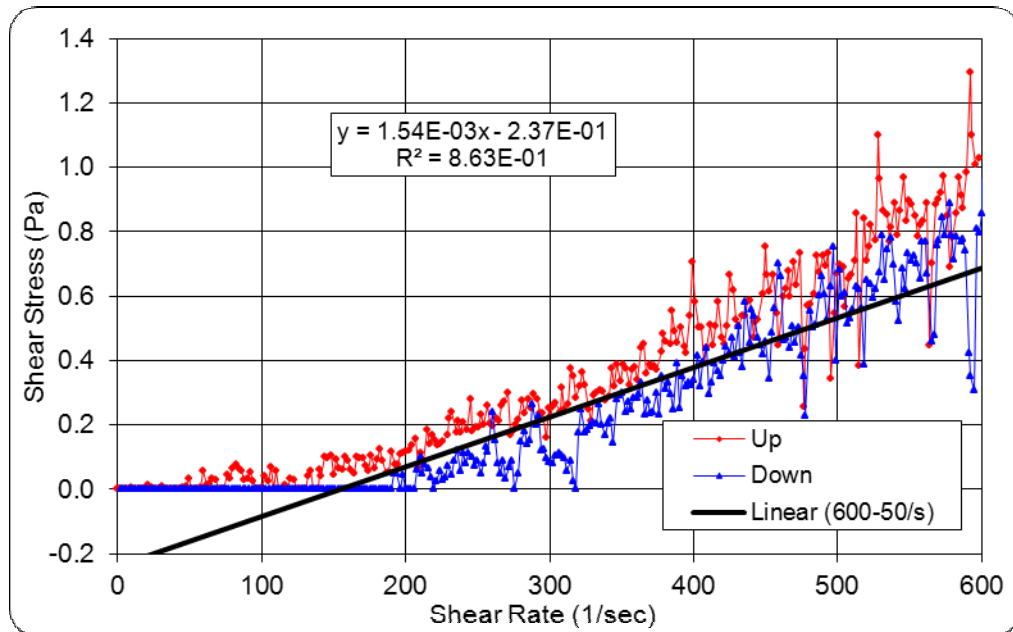


Figure B-69: Stress Versus Strain Curve for Tank 12H No Evaporation Deposition Sample at 45 °C (First Replicate)

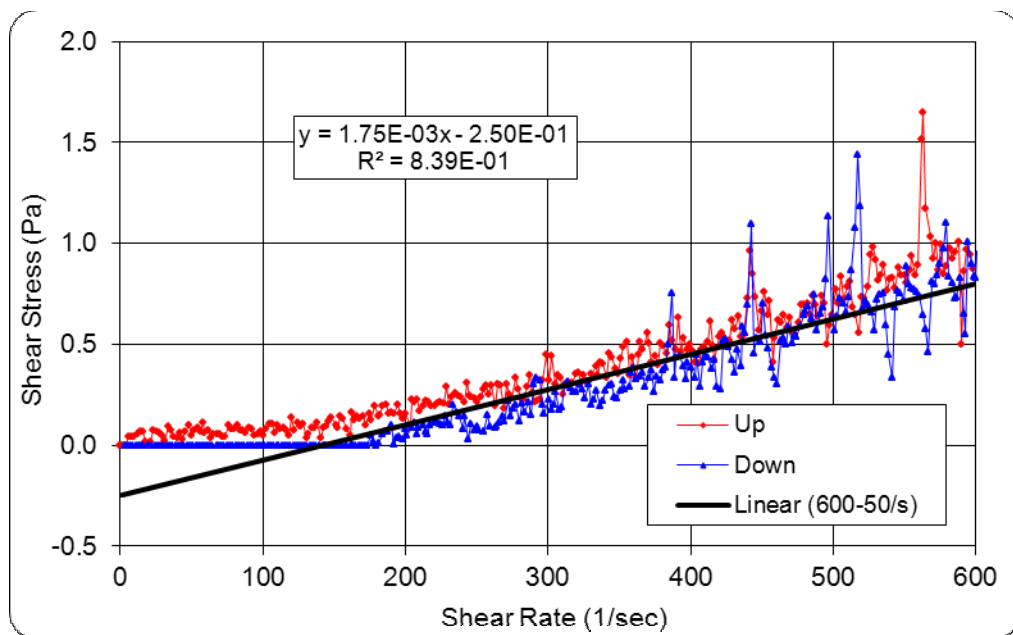


Figure B-70: Stress Versus Strain Curve for Tank 12H No Evaporation Deposition Sample at 45 °C (Second Replicate)

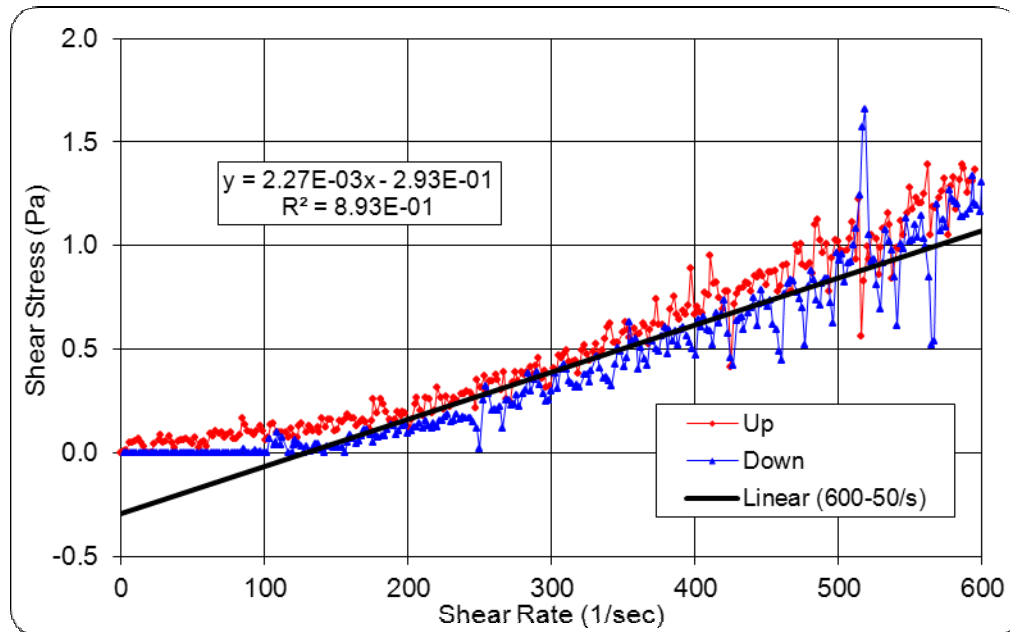


Figure B-71: Stress Versus Strain Curve for Tank 12H Evaporated Deposition Sample at 30 °C (First Replicate)

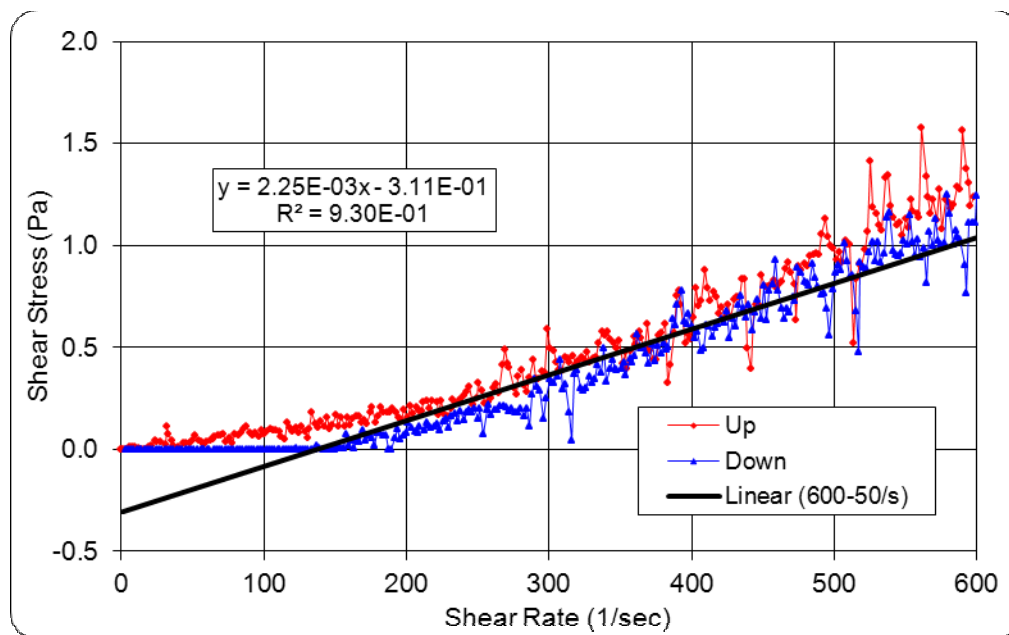


Figure B-72: Stress Versus Strain Curve for Tank 12H Evaporated Deposition Sample at 30 °C (Second Replicate)

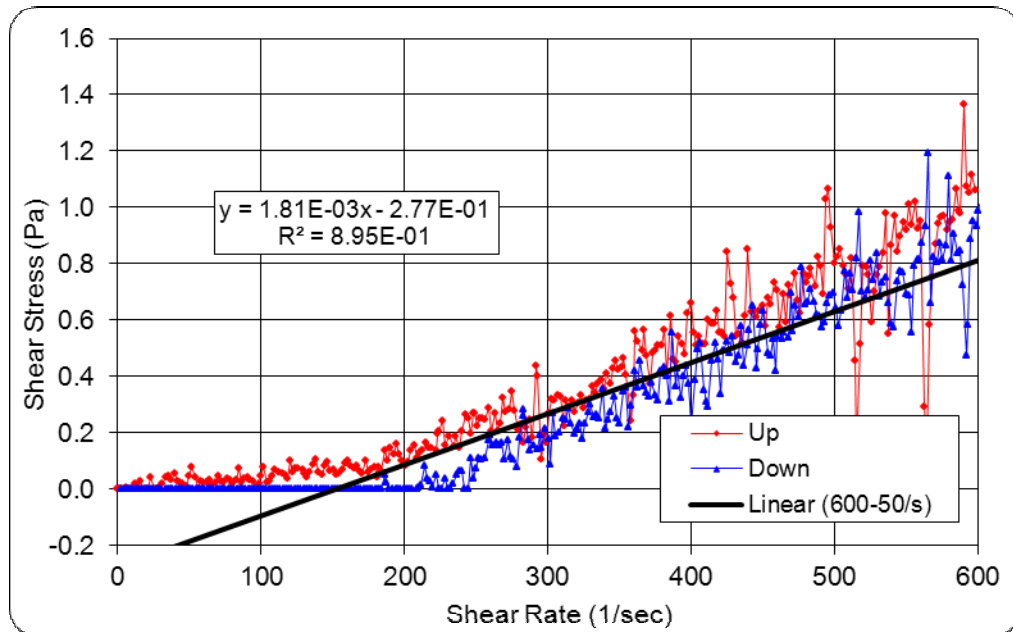


Figure B-73: Stress Versus Strain Curve for Tank 12H Evaporated Deposition Sample at 45 °C (First Replicate)

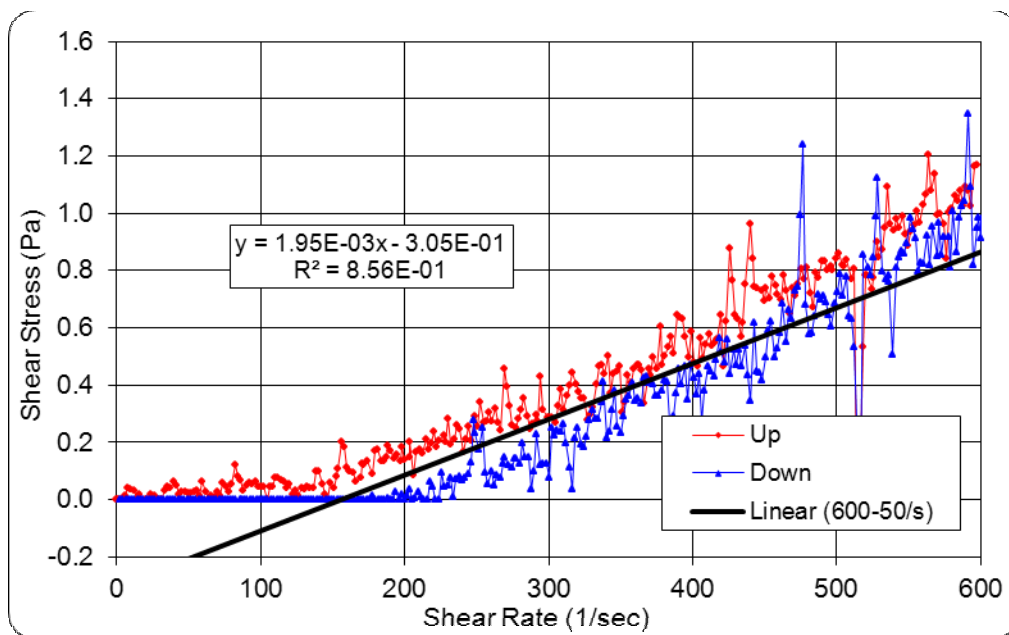


Figure B-74: Stress Versus Strain Curve for Tank 12H Evaporated Deposition Sample at 45 °C (Second Replicate)

Figure B-75: Particle Size Analysis for Tank 5F Sludge Prior to Contact with OA

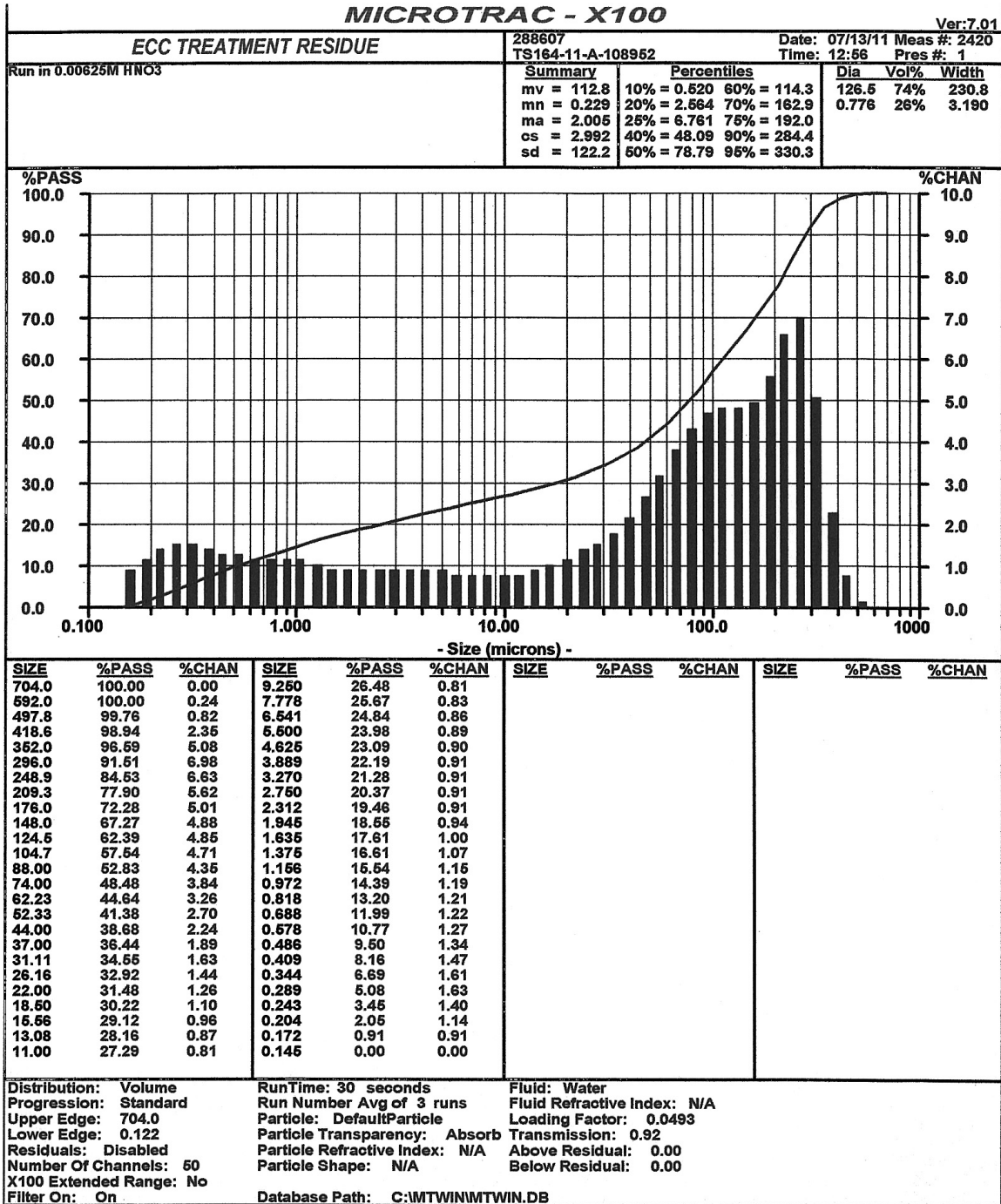


Figure B-76: Particle Size Analysis for Tank 12H Sludge Prior to Contact with OA

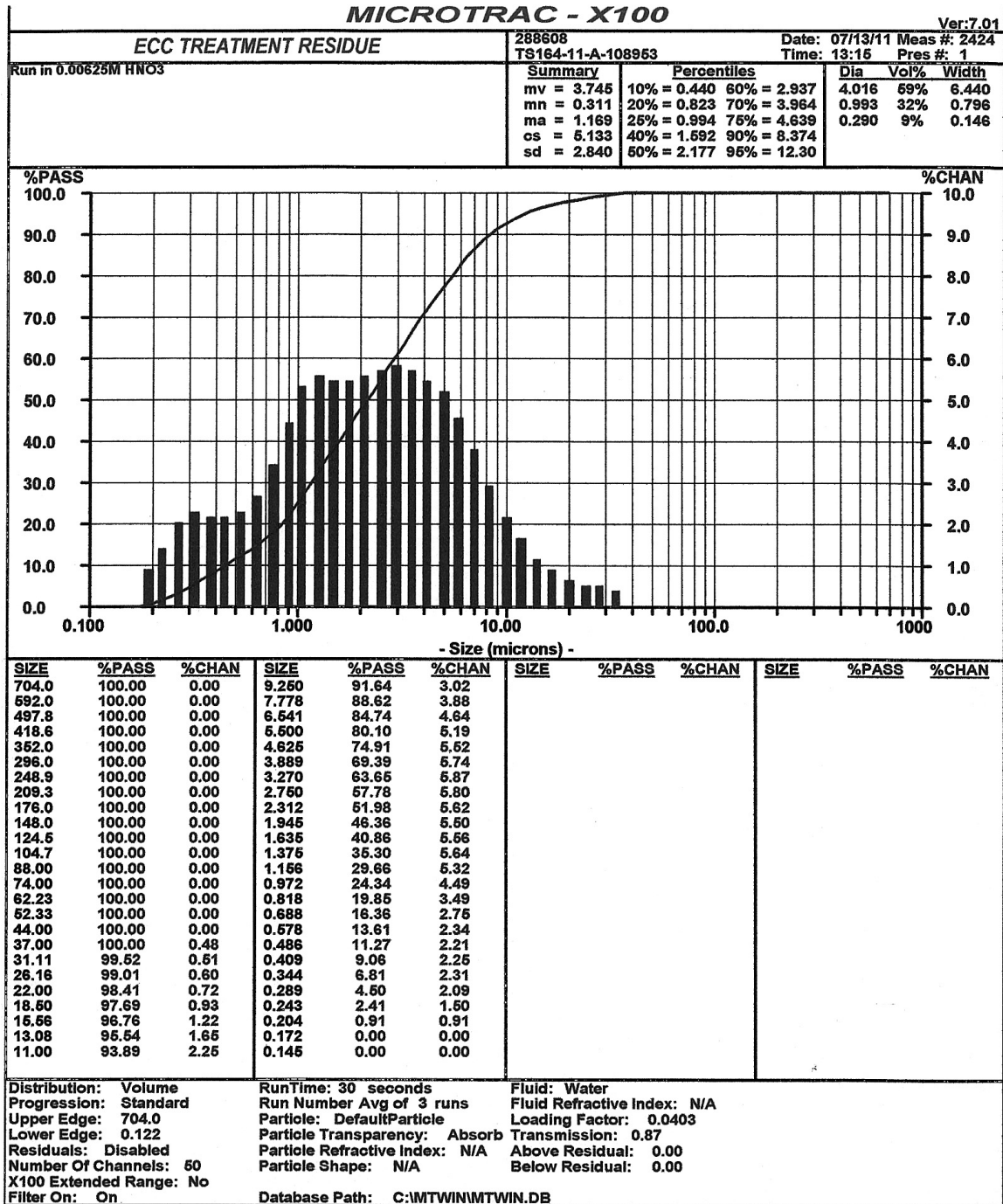


Figure B-77: Particle Size Analysis of Intermediate Treatment Tank Heel after Dissolution of Tank 5F Sludge with One OA Batch

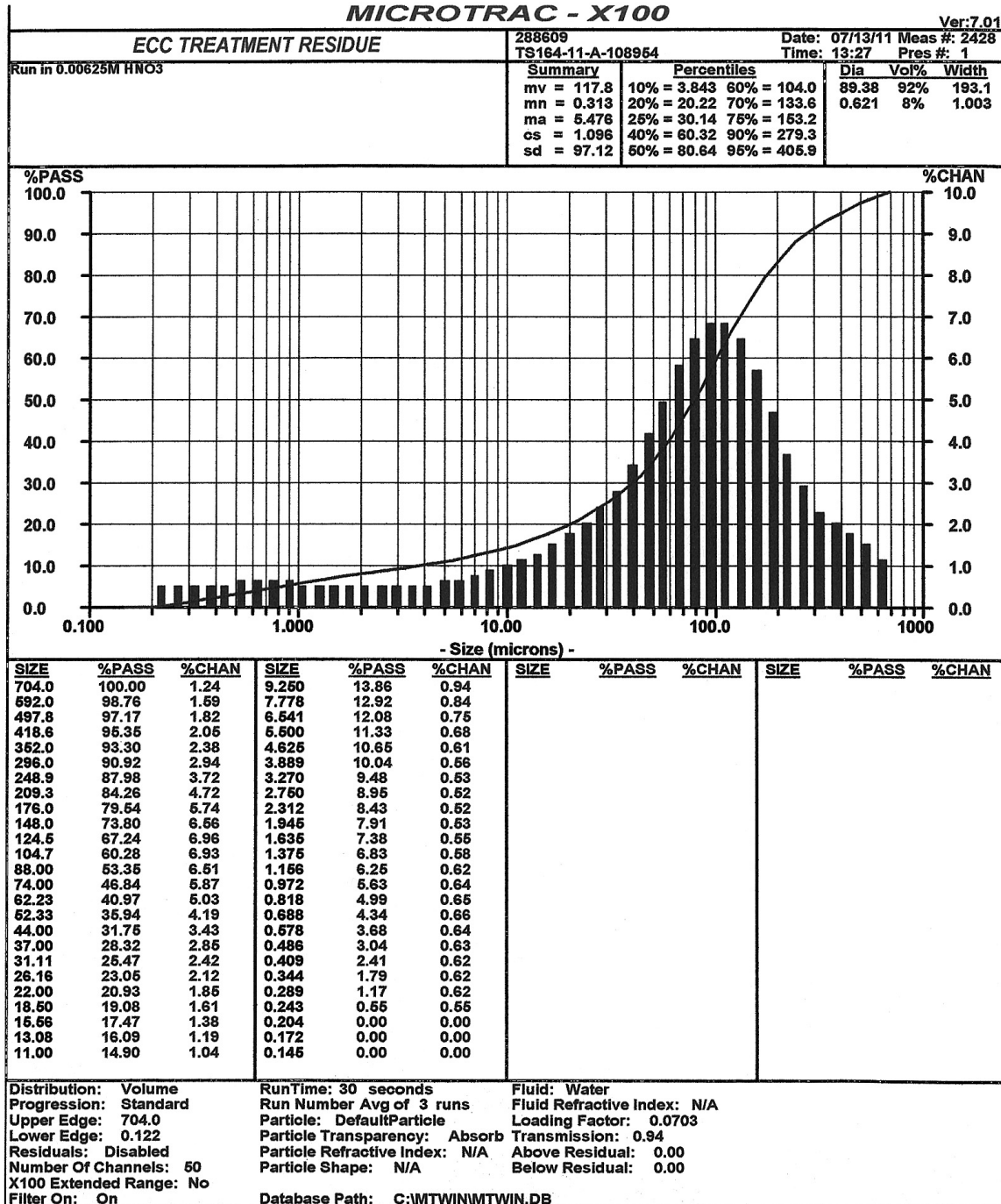


Figure B-78: Particle Size Analysis of Intermediate Treatment Tank Heel after Dissolution of Tank 12H Sludge with One OA Batch

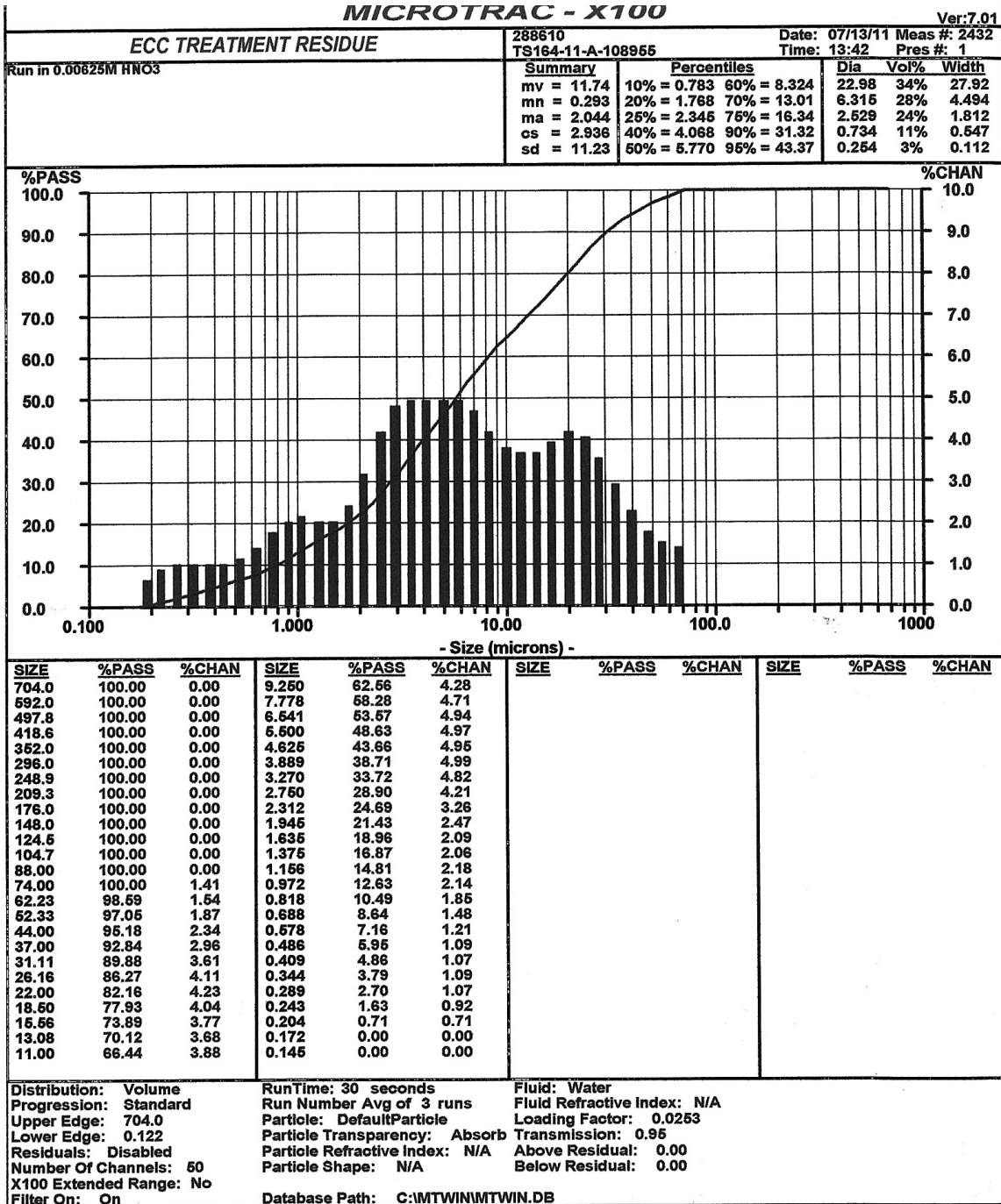


Figure B-79: Particle Size Analysis of Final Treatment Tank Heel after Dissolution of Tank 5F Sludge with Two OA Batches

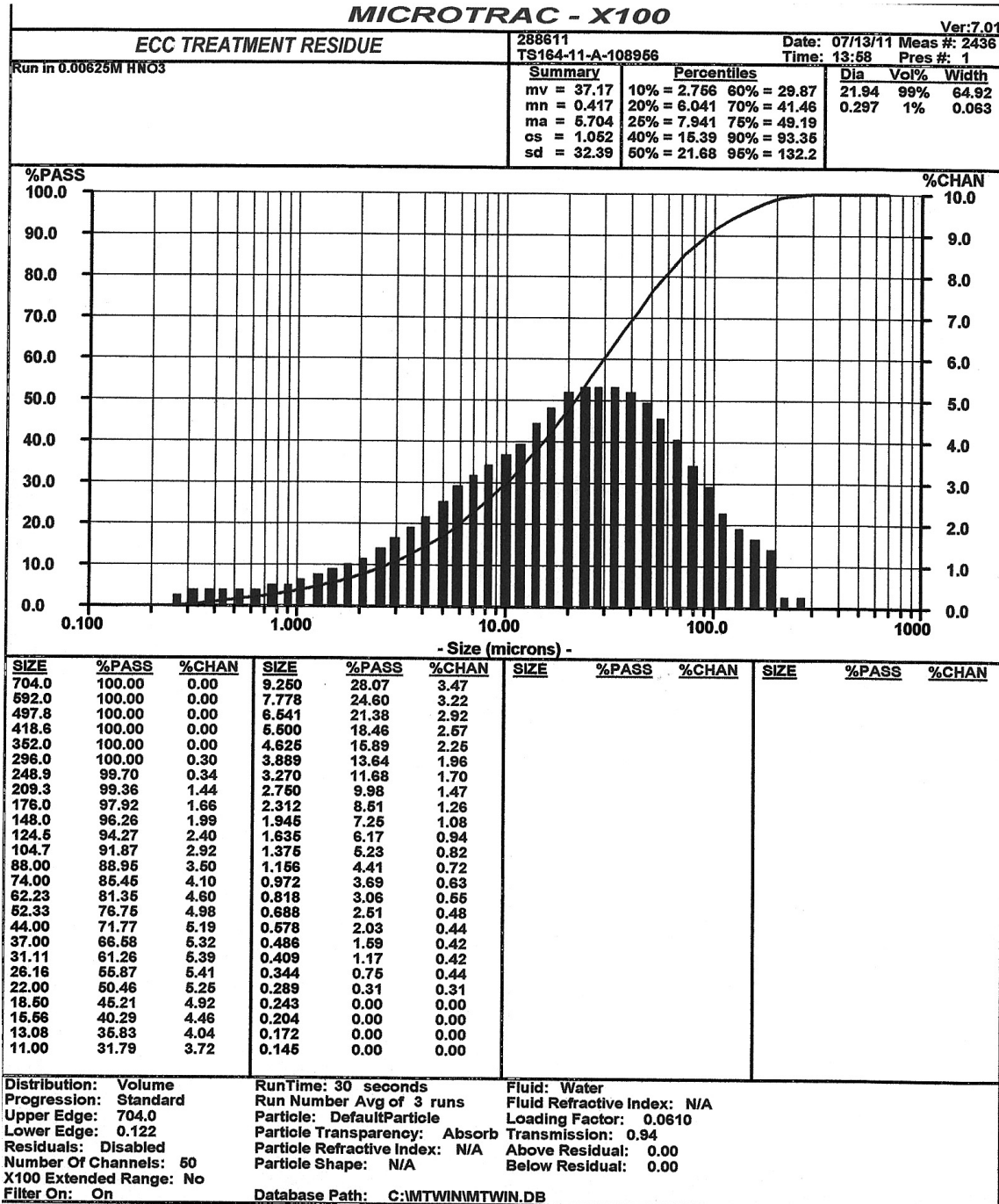


Figure B-80: Particle Size Analysis of Final Treatment Tank Heel after Dissolution of Tank 12H Sludge with Two OA Batches

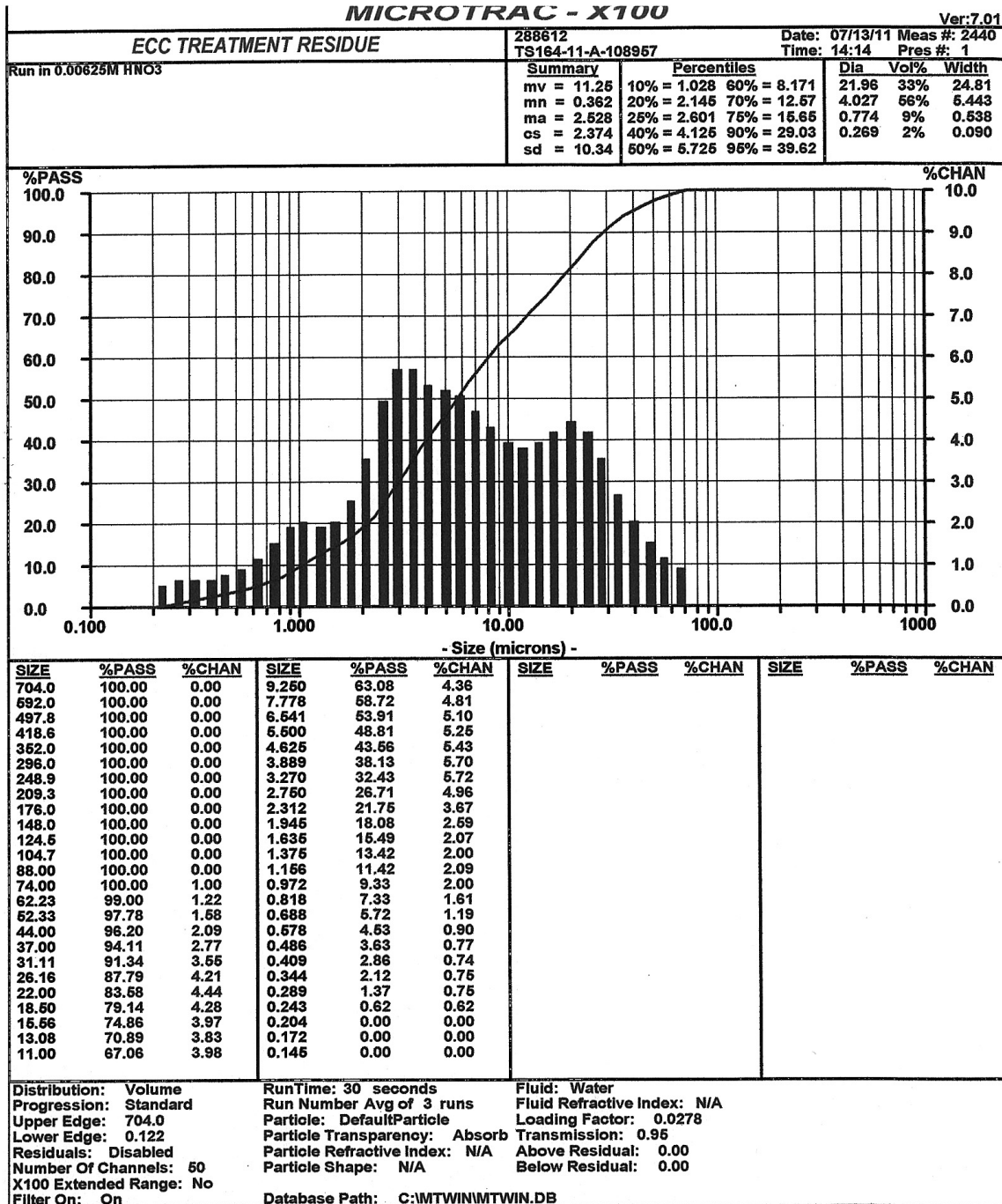


Figure B-81: Particle Size Analysis of Test 1 (Tank 12H) Storage Condition 3 (without evaporation, without pH adjustment, and without sludge heel), representing ECC product prior to pH adjustment and evaporation.

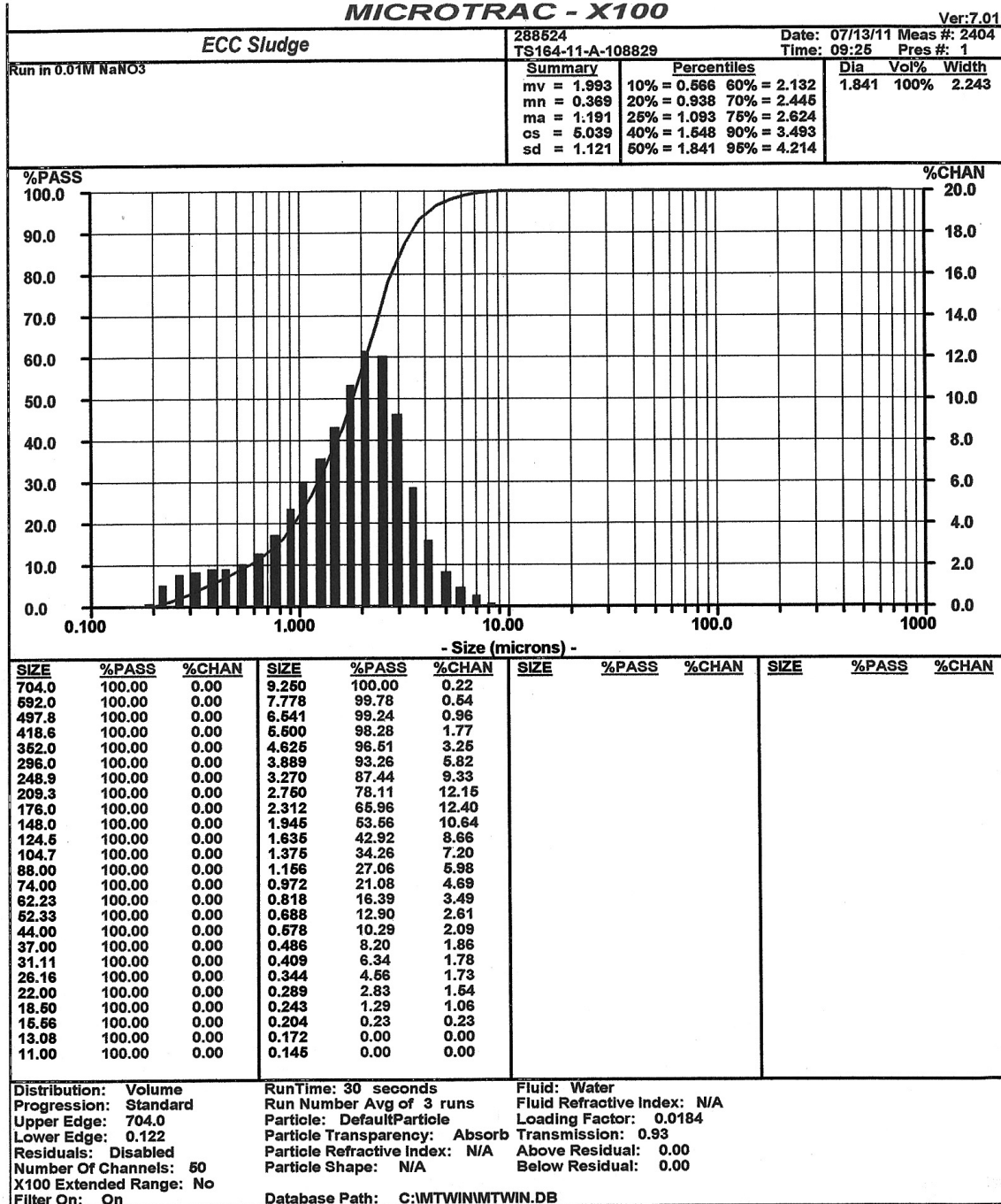


Figure B-82: Particle Size Analysis of Test 1 (Tank 12H) Storage Condition 5 (with evaporation, with pH adjustment, and with sludge heel), representing ECC product after evaporation (H-area baseline) and at storage tank conditions.

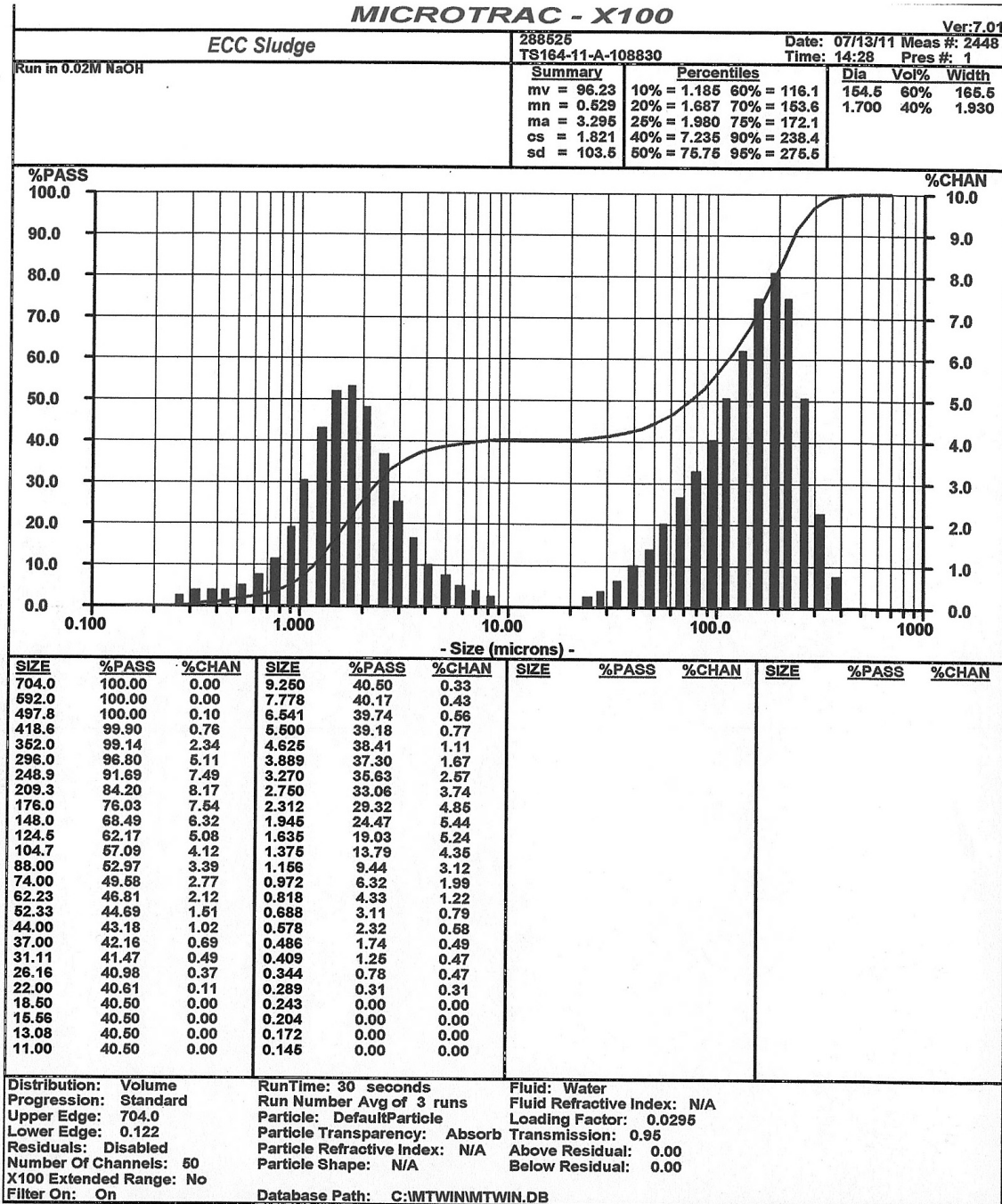


Figure B-83: Particle Size Analysis of Test 1 (Tank 12H) Storage Condition 6 (with evaporation, without pH adjustment, and without sludge heel), representing ECC product just after evaporation and prior to pH adjustment.

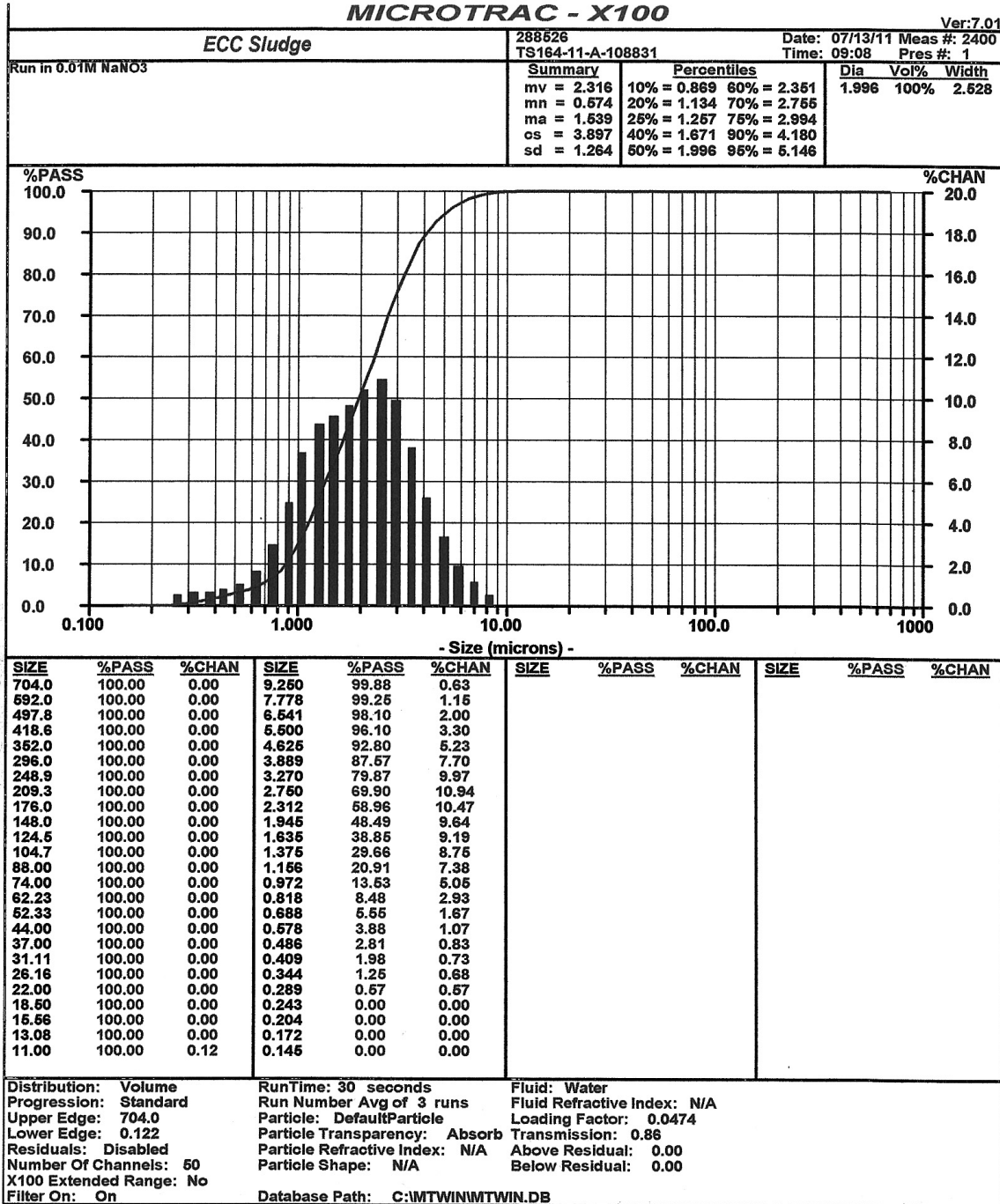


Figure B-84: Particle Size Analysis of Test 1 (Tank 12H) Control (Storage Condition 9) (without evaporation, with pH adjustment, and with sludge heel), representing the original Tank 12H sludge with pH adjustment and heel in deposition tank.

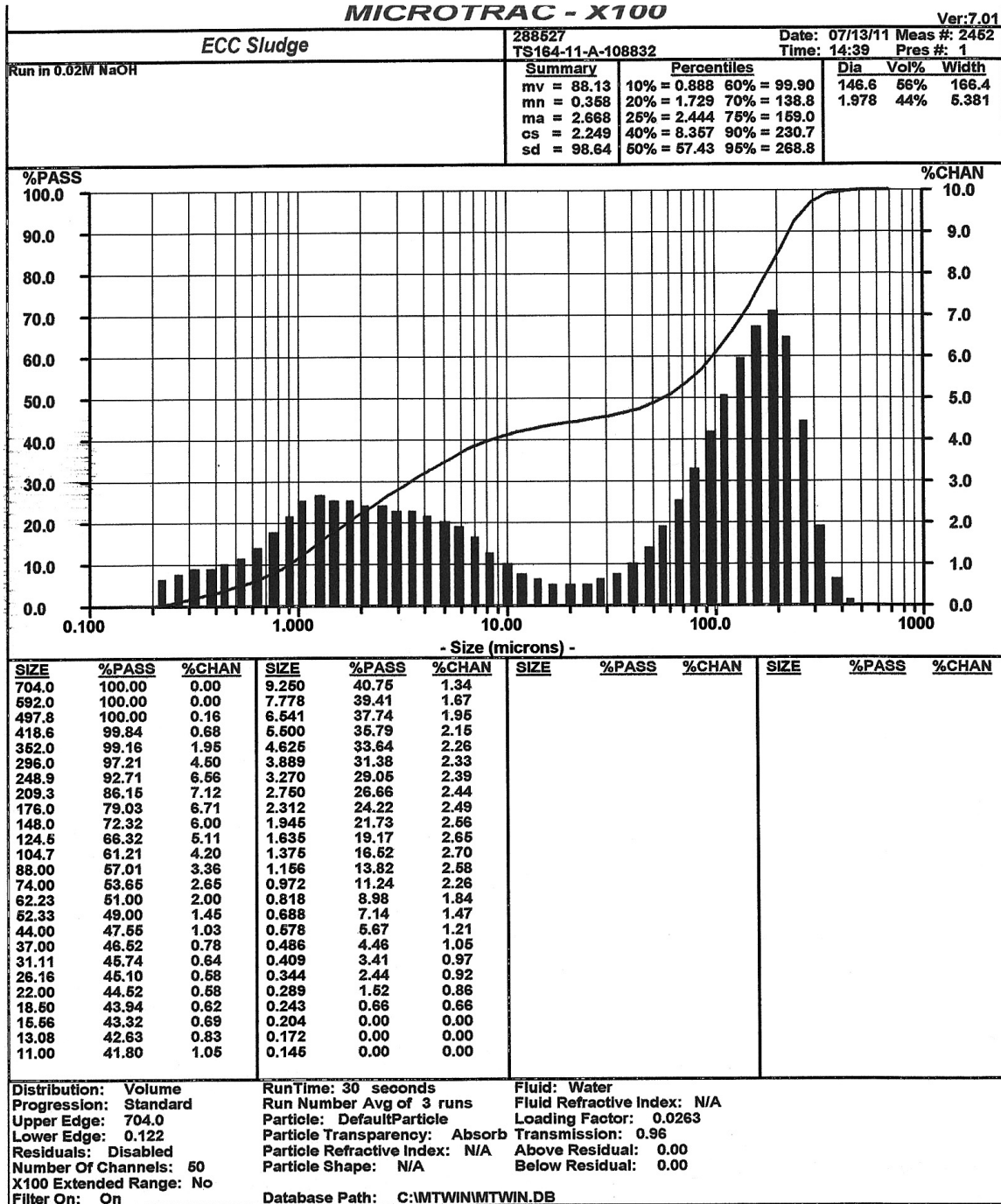


Figure B-85: Particle Size Analysis of Test 2 (Tank 5F) Storage Condition 2 (without evaporation, with pH adjustment, and with sludge heel), representing ECC product without evaporation (F-area baseline) and at storage tank conditions.

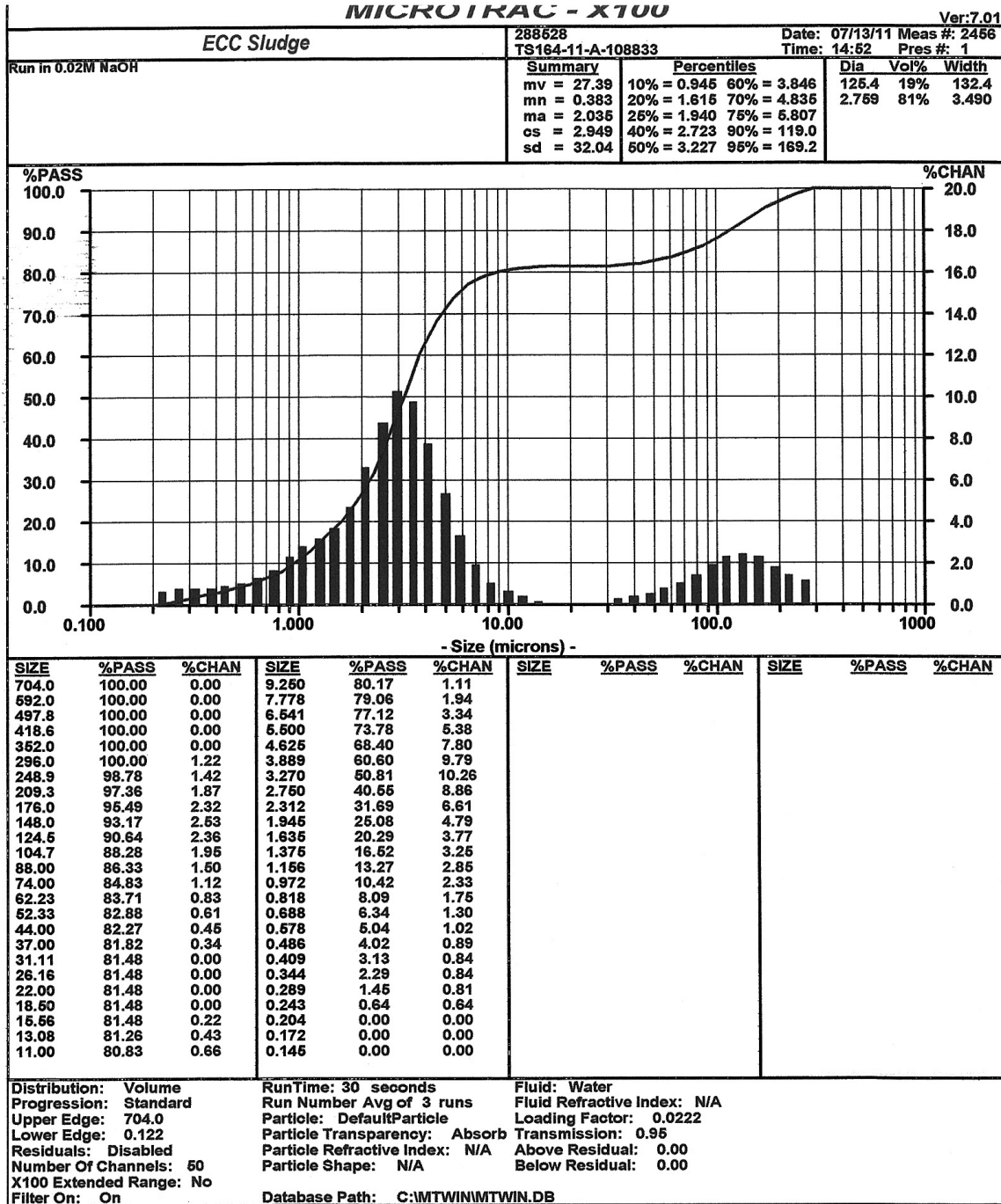


Figure B-86: Particle Size Analysis of Test 2 (Tank 5F) Storage Condition 3 (without evaporation, without pH adjustment, and without sludge heel), representing ECC product prior to pH adjustment and evaporation.

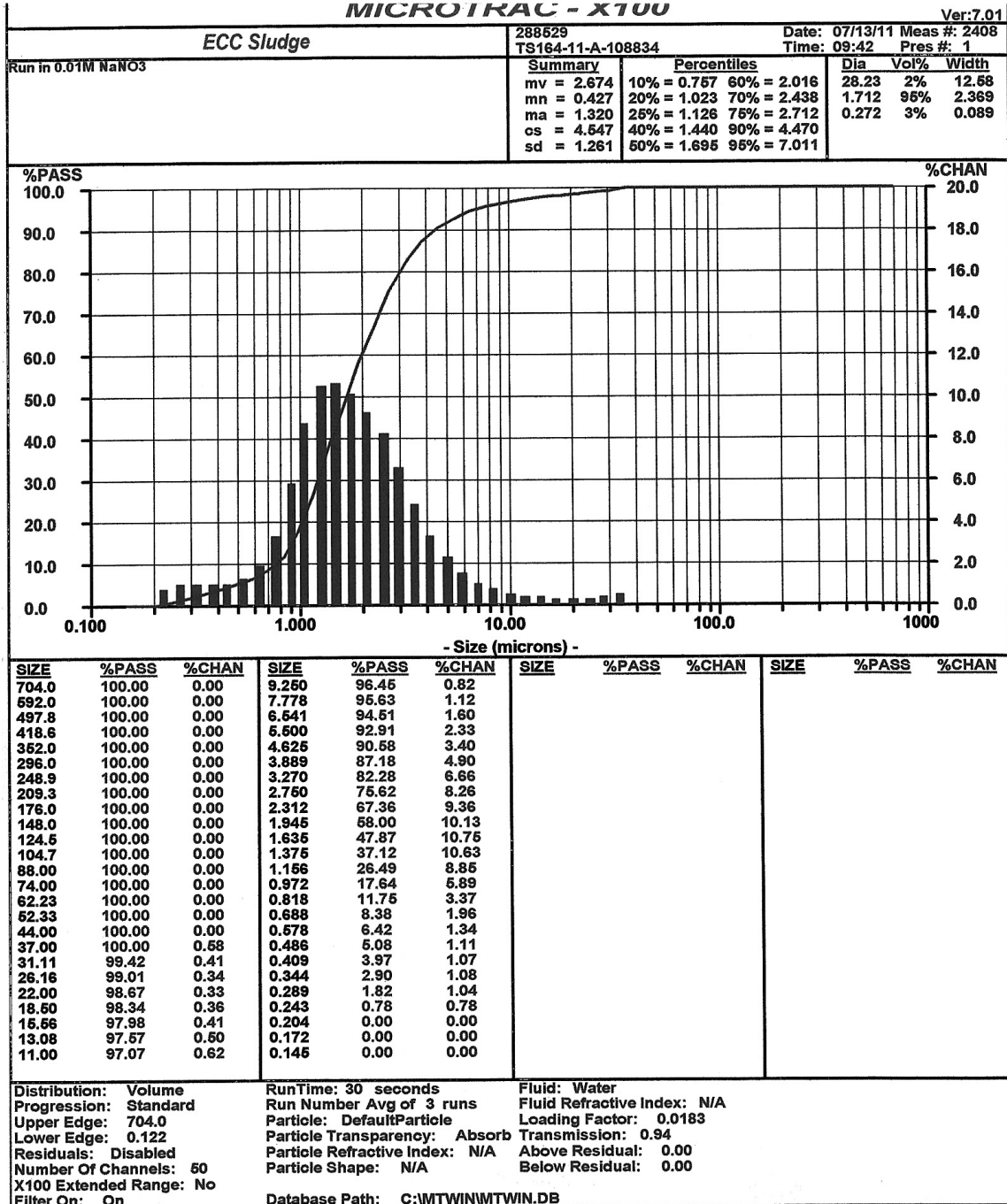


Figure B-87: Particle Size Analysis of Test 2 (Tank 5F) Storage Condition 6 (with evaporation, without pH adjustment, and without sludge heel), representing ECC product just after evaporation and prior to pH adjustment.

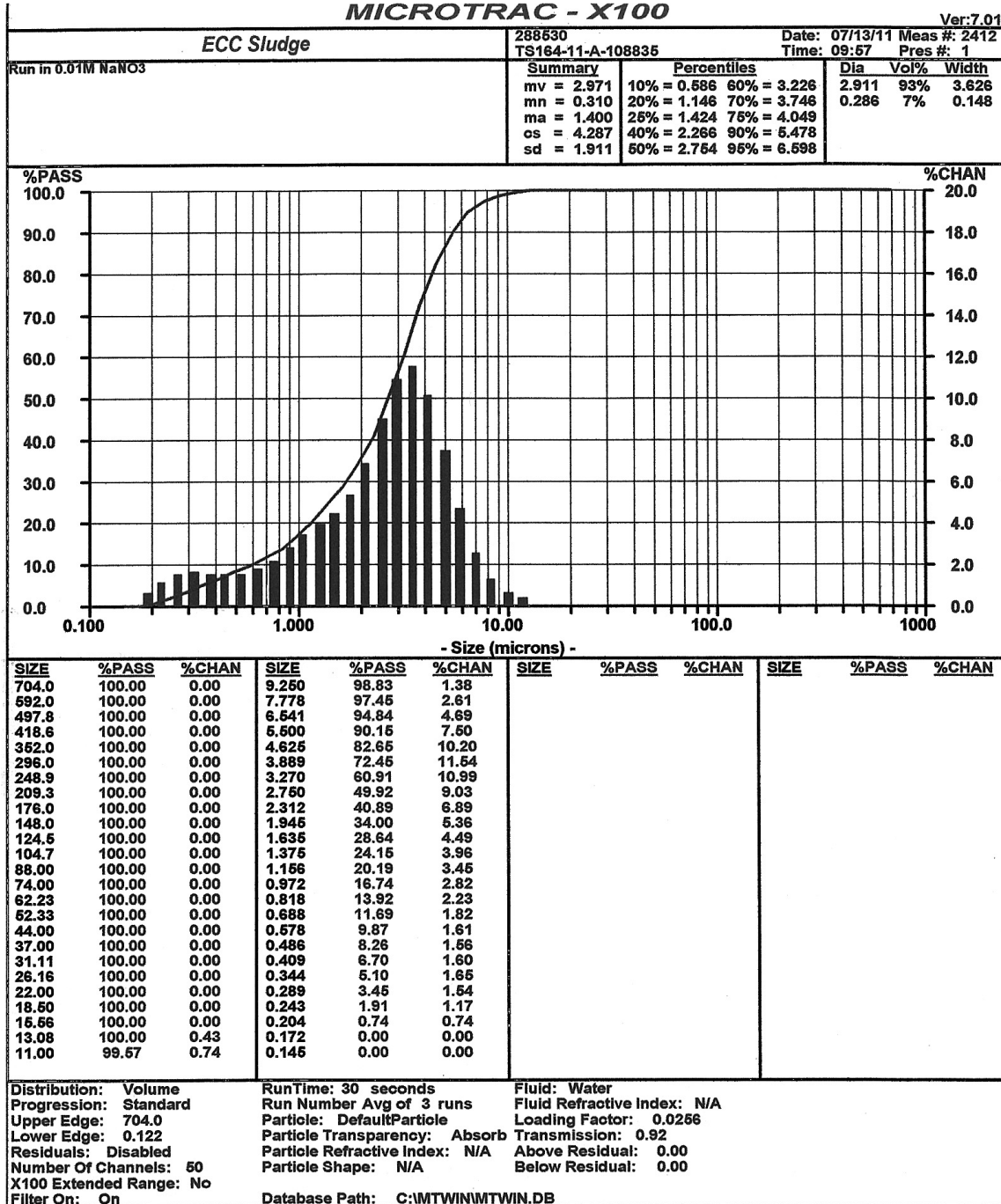
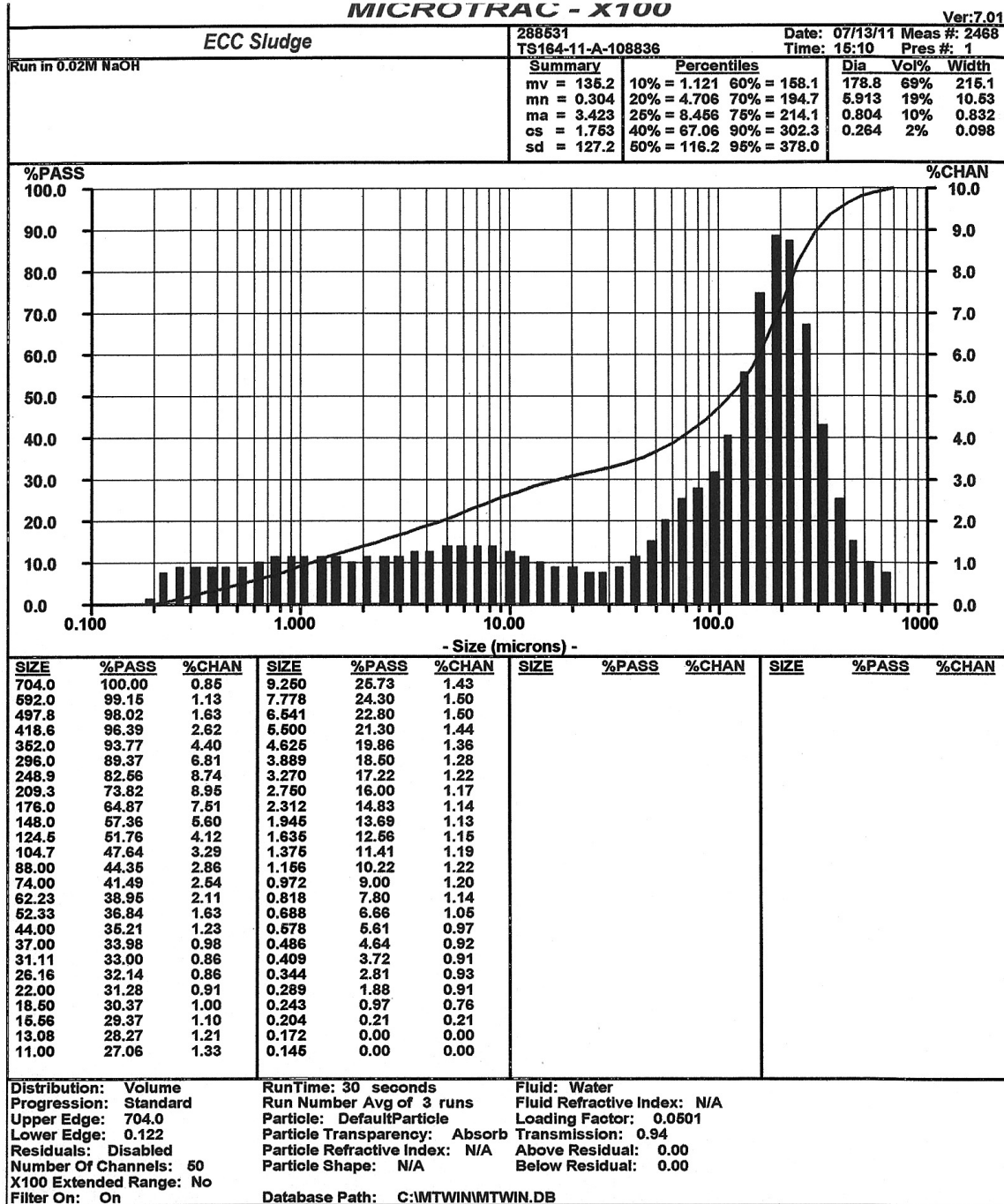


Figure B-88: Particle Size Analysis of Test 2 (Tank 5F) Control (Storage Condition 9) (without evaporation, with pH adjustment, and with sludge heel), representing the original Tank 12H sludge with pH adjustment and heel in deposition tank.



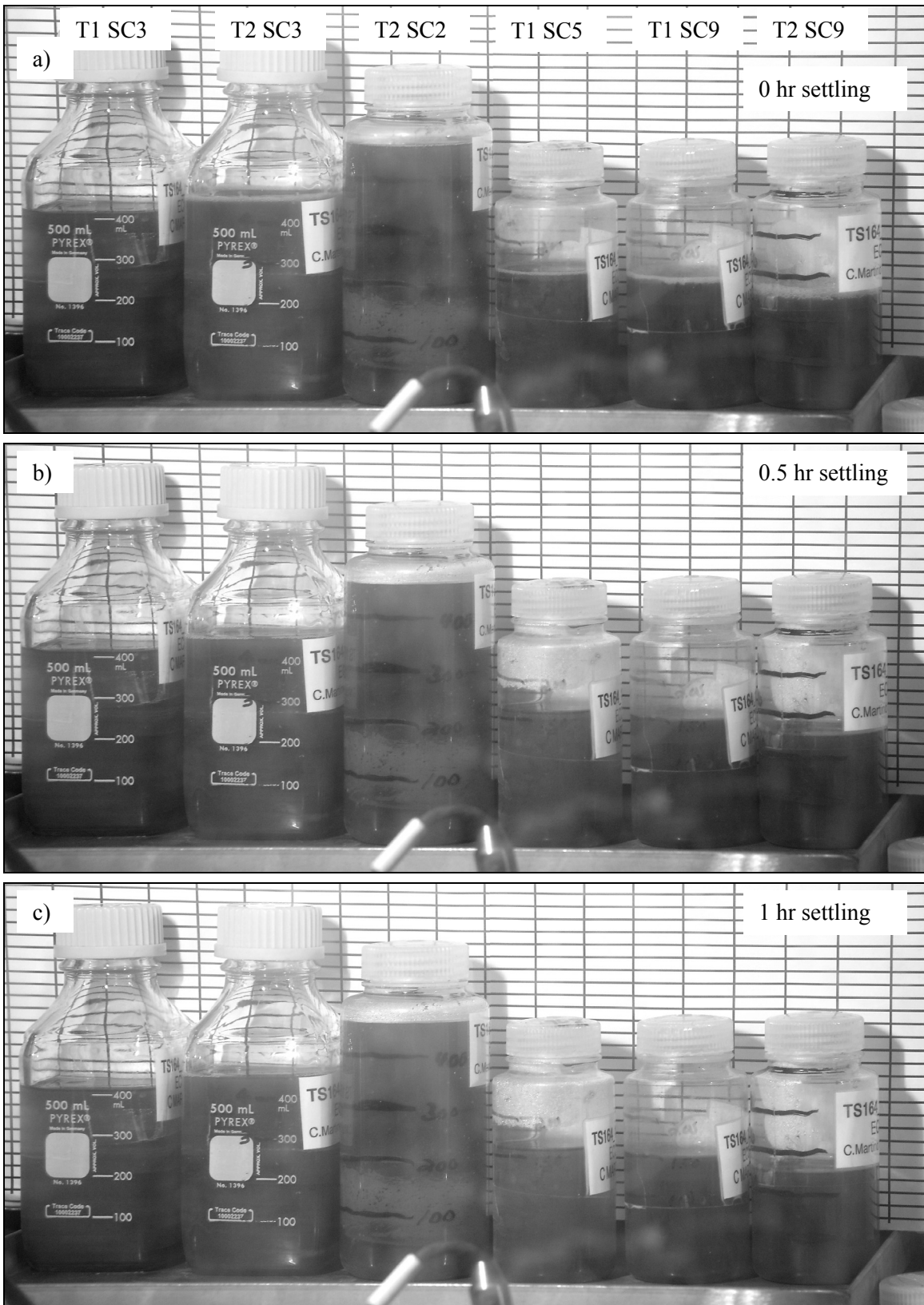


Figure B-89: ECC Product Settling (part 1 of 4)

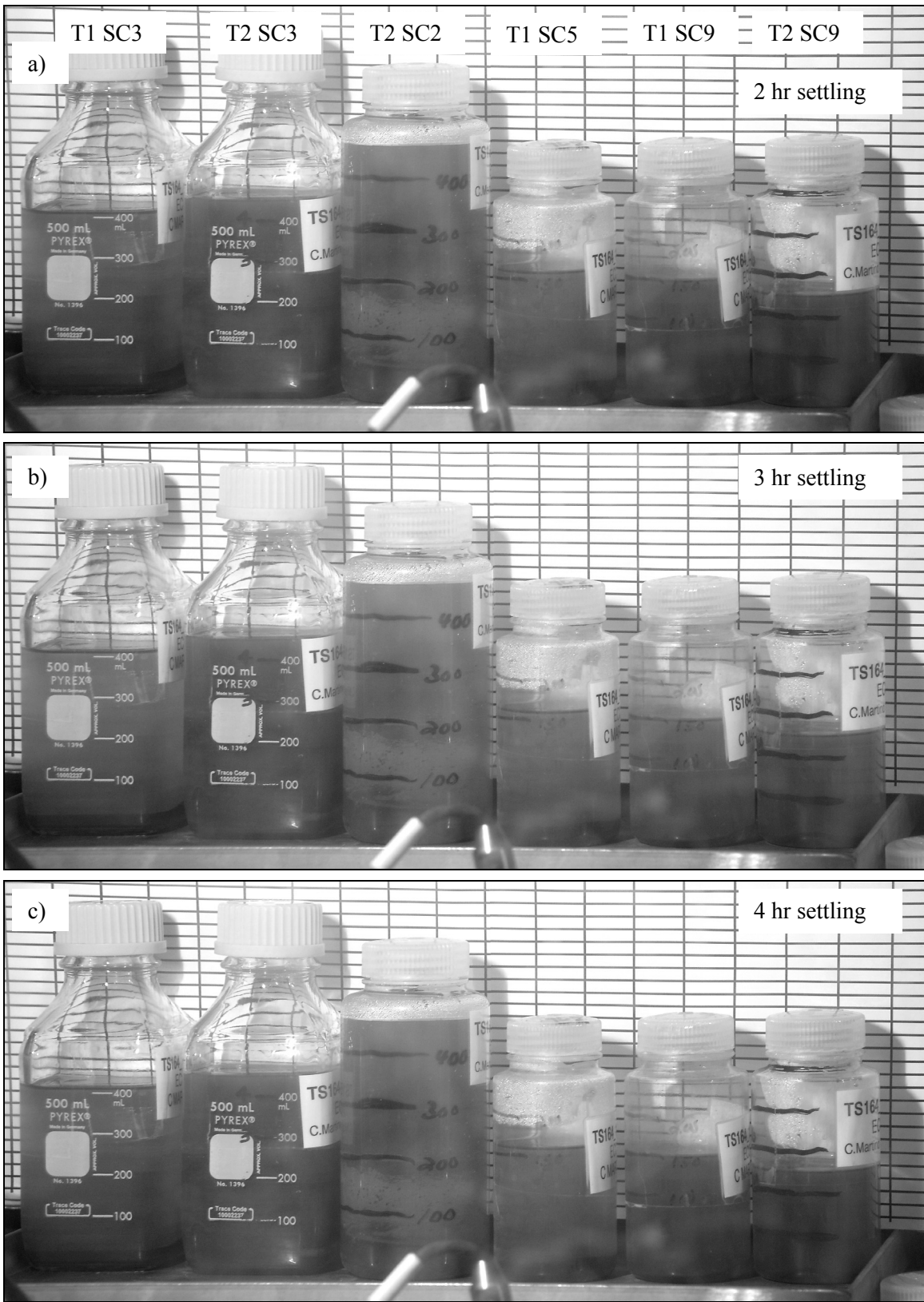


Figure B-90: ECC Product Settling (part 2 of 4)

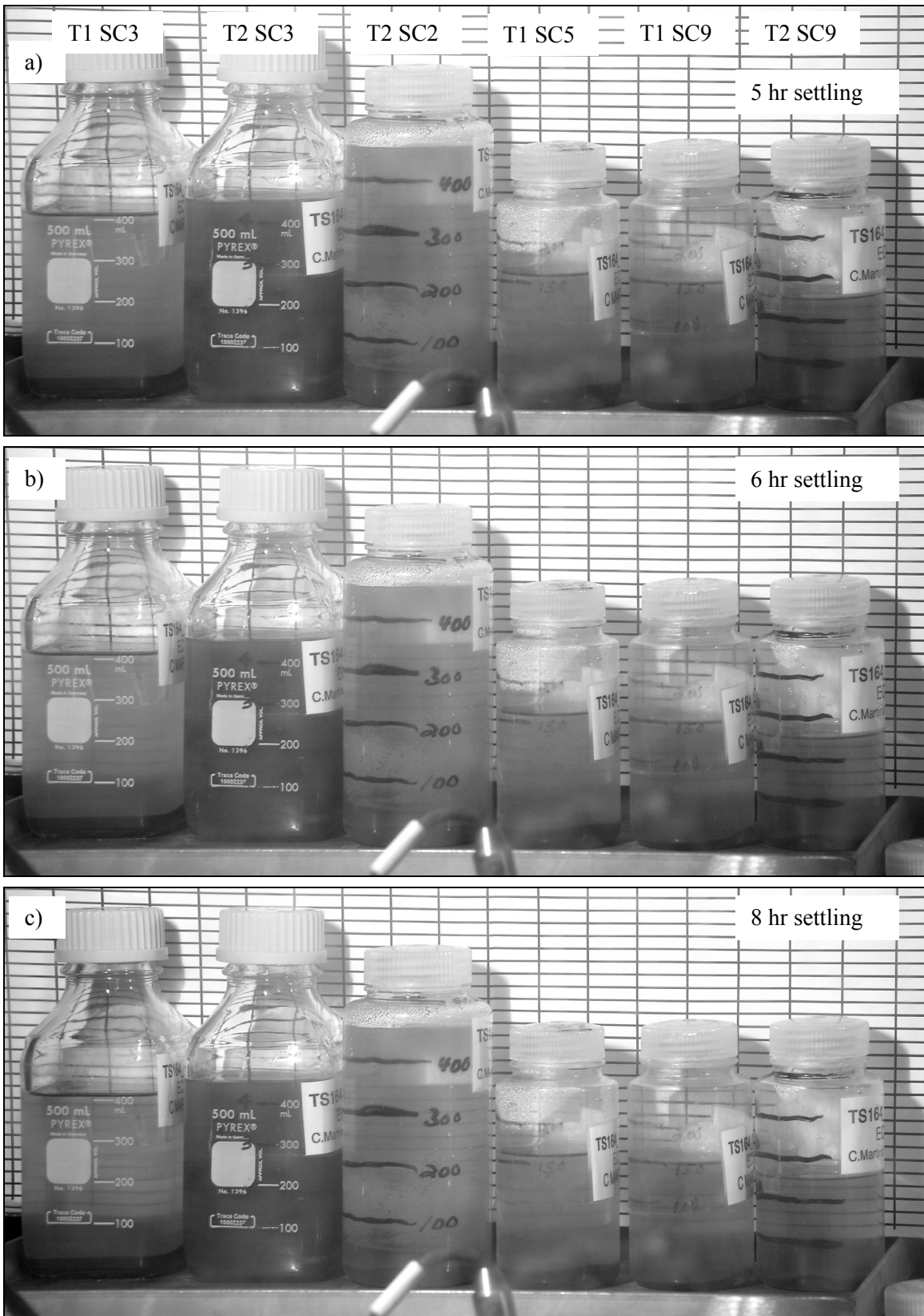


Figure B-91: ECC Product Settling (part 3 of 4)

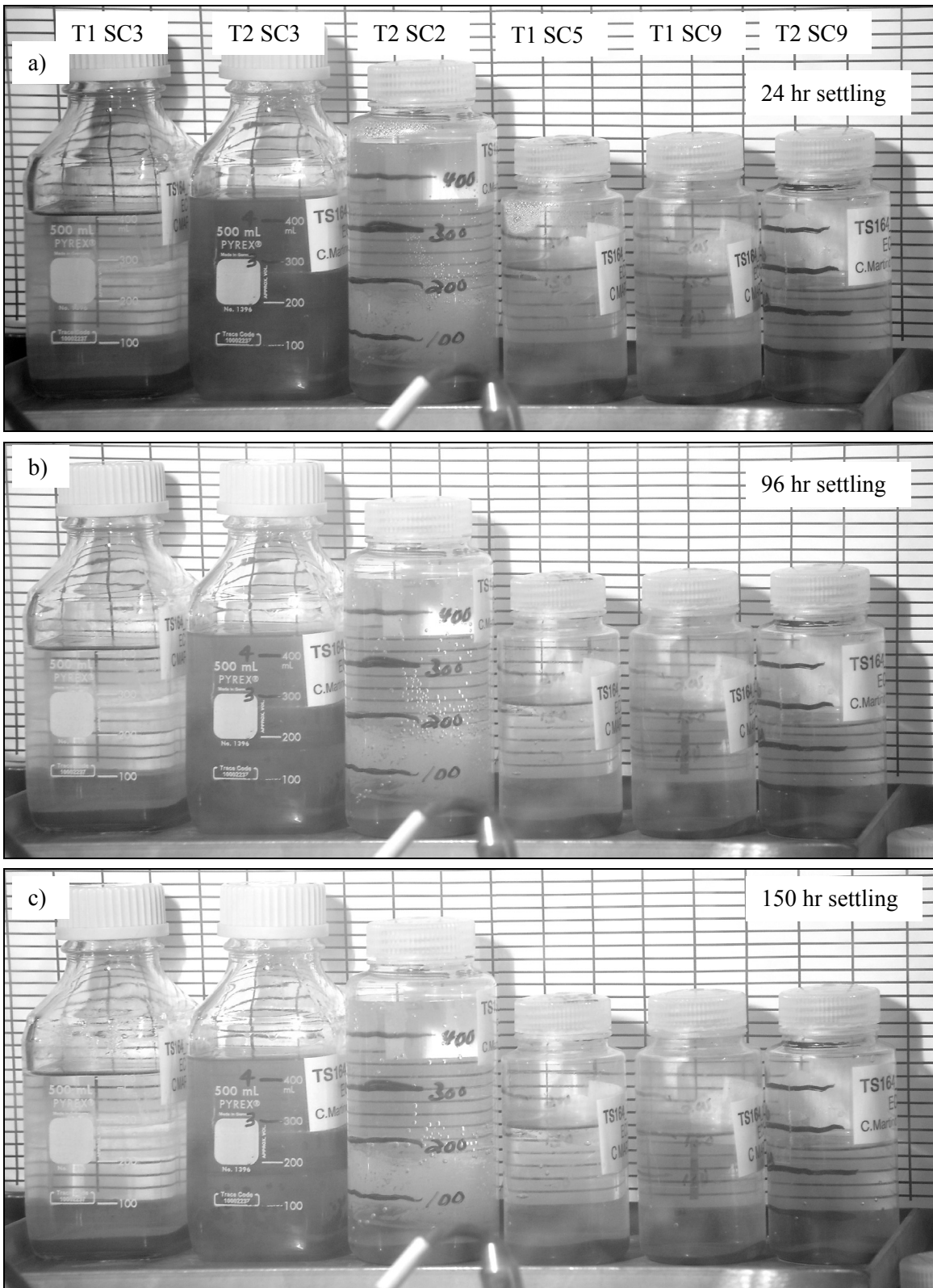


Figure B-92: ECC Product Settling (part 4 of 4)

Distribution:

R. H. Spires, 704-26F
T. M. Punch, 704-71F
C. B. Sherburne, 704-71F
P. E. Carroll, 704-71F
E. T. Ketusky, 704-70F
J. R. Vitali, 704-71F
D. C. Wood, 704-26F
B. J. Rabe, 704-26F
N. R. Davis, 704-26F
W. L. Isom, 704-26F
A. J. Tisler, 704-26F
L. Carey, 742-7G
J. R. Gregory (AREVA), 704-S
C. J. Martino, 773-42A
F. M. Pennebaker, 773-42A
W. D. King, 773-42A
M. S. Hay, 773-42A
B. J. Wiersma, 773-A
J. I. Mickalonis, 773-A
S. H. Reboul, 773-A
A. B. Barnes, 999-W
D. A. Crowley, 773-43A
A. P. Fellingner, 773-41A
S. D. Fink, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
A. M. Murray, 773-A
W. R. Wilmarth, 773-A
P. R. Jackson, DOE-SR, 703-46A
K. H. Subramanian, 766-H