

**This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.**

**DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or any third party's use or the results of such use of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

# DWPF RECYCLE EVAPORATOR SIMULANT TESTS

M. E. Stone

April 2005

Immobilization Technology Section  
Savannah River National Laboratory  
Aiken, SC 29808

---

Prepared for the U.S. Department of Energy Under Contract Number  
DEAC09-96SR18500



**DISCLAIMER**

This report was prepared by Westinghouse Savannah River Company (WSRC) for the United States Department of Energy under Contract No. DE-AC09-96SR18500 and is an account of work performed under that contract. Neither the United States Department of Energy, nor WSRC, nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness, of any information, apparatus, or product or process disclosed herein or represents that its use will not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trademark, name, manufacturer or otherwise does not necessarily constitute or imply endorsement, recommendation, or favoring of same by WSRC or by the United States Government or any agency thereof. The views and opinions of the authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

**Printed in the United States of America**

**Prepared For  
U.S. Department of Energy**

**Key Words:** *DWPF, Recycle,  
Evaporation, Antifoam*

**Retention: Permanent**

# **DWPF RECYCLE EVAPORATOR SIMULANT TESTS**

M. E. Stone

April 2005

Immobilization Technology Section  
Savannah River National Laboratory  
Aiken, SC 29808

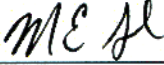
---

Prepared for the U.S. Department of Energy Under Contract Number  
DEAC09-96SR18500





## REVIEWS AND APPROVALS

**AUTHOR(S):**

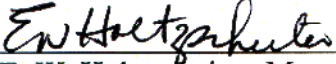
	4/21/05
M. E. Stone, Immobilization Technology Section	Date


**TECHNICAL REVIEWERS:**

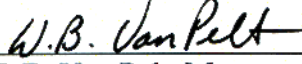
	4-21-05
W. E. Daniel, Immobilization Technology Section	Date

	4-25-2005
T. L. Fellingner, Immobilization Technology Section	Date

**APPROVERS**

	4/25/05
E. W. Holtzscheiter, Manager, Immobilization Technology Section	Date

	4/25/05
S. L. Marra, Manager, Glass Formulation & Process Development	Date

	4-27-05
W. B. Van Pelt, Manager, DWPF Programs Engineering	Date

## EXECUTIVE SUMMARY

Testing was performed to determine the feasibility and processing characteristics of an evaporation process to reduce the volume of the recycle stream from the Defense Waste Processing Facility (DWPF). The concentrated recycle would be returned to DWPF while the overhead condensate would be transferred to the Effluent Treatment Plant.

Various blends of evaporator feed were tested using simulants developed from characterization of actual recycle streams from DWPF and input from DWPF-Engineering. The simulated feed was evaporated in laboratory scale apparatus to target a 30X volume reduction. Condensate and concentrate samples from each run were analyzed and the process characteristics (foaming, scaling, etc) were visually monitored during each run.

The following conclusions were made from the testing:

Concentration of the “typical” recycle stream in DWPF by 30X was feasible. The addition of DWTT recycle streams to the typical recycle stream raises the solids content of the evaporator feed considerably and lowers the amount of concentration that can be achieved.

Foaming was noted during all evaporation tests and must be addressed prior to operation of the full-scale evaporator. Tests were conducted that identified Dow Corning 2210 as an antifoam candidate that warrants further evaluation.

The condensate has the potential to exceed the ETP WAC for mercury, silicon, and TOC. Controlling the amount of equipment decontamination recycle in the evaporator blend would help meet the TOC limits. The evaporator condensate will be saturated with mercury and elemental mercury will collect in the evaporator condensate collection vessel.

No scaling on heating surfaces was noted during the tests, but splatter onto the walls of the evaporation vessels led to a buildup of solids. These solids were difficult to remove with 2M nitric acid. Precipitation of solids was not noted during the testing.

Some of the aluminum present in the recycle streams was converted from gibbsite to aluminum oxide during the evaporation process.

The following recommendations were made:

Recycle from the DWTT should be metered in slowly to the “typical” recycle streams to avoid spikes in solids content to allow consistent processing and avoid process upsets. Additional studies should be conducted to determine acceptable volume ratios for the HEME dissolution and decontamination solutions in the evaporator feed.

Dow Corning 2210 antifoam should be evaluated for use to control foaming. Additional tests are required to determine the concentration of antifoam required to prevent foaming during startup, the frequency of antifoam additions required to control foaming during steady state processing, and the ability of the antifoam to control foam over a range of potential feed compositions. This evaluation should also include evaluation of the degradation of the antifoam and impact on the silicon and TOC content of the condensate.

The caustic HEME dissolution recycle stream should be neutralized to at least pH of 7 prior to blending with the acidic recycle streams.

Dow Corning 2210 should be used during the evaporation testing using the radioactive recycle samples received from DWPF.

Evaluation of additional antifoam candidates should be conducted as a backup for Dow Corning 2210.

A camera and/or foam detection instrument should be included in the evaporator design to allow monitoring of the foaming behavior during operation.

The potential for foam formation and high solids content should be considered during the design of the evaporator vessel.

## TABLE OF CONTENTS

Executive Summary .....	v
Table of Contents .....	vii
List of Figures .....	viii
List of Tables .....	viii
List of Acronyms .....	ix
1.0 Introduction.....	1
2.0 Experimental Methods.....	2
2.1 Recycle Evaporator Feed Blending .....	2
2.2 Recycle Evaporator Feed Compositions .....	3
2.3 Test Apparatus .....	4
2.4 Run Performance .....	5
2.5 Sample Analysis Methods.....	5
2.5.1 pH .....	5
2.5.2 Density.....	6
2.5.3 Solids Content .....	6
2.5.4 Viscosity .....	6
2.5.5 Mobile Laboratory Measurements .....	6
2.5.6 Titrations .....	6
2.5.7 Vapor Pressure .....	6
2.5.8 ADS Measurements.....	7
3.0 Results.....	7
3.1 Foaming .....	7
3.2 Condensate Characterization .....	10
3.3 Concentrate Characterization.....	11
3.4 Precipitation .....	11
3.5 Scaling.....	13
3.6 Evaporator DF.....	13
4.0 Conclusions.....	15
5.0 Recommendations.....	15
6.0 References.....	17
Appendix A. Simulant Recipes.....	18
Appendix B. Equipment Diagrams and Operation Steps.....	20
Appendix C. Sample Results from Boil-Down Tests .....	26
Appendix D. XRD Results.....	30
Appendix E. Condensate pH Profiles .....	40
Appendix F. DF Trending.....	44



## LIST OF FIGURES

Figure 1. Foaming during Initial and Final Stages of Scenario 1, Run 1 Evaporation .....	7
Figure 2. Performance of Antifoams .....	8
Figure 3. Flocculation during Addition of DSP Antifoam .....	9
Figure 4: Foaming During Antifoam Tests with DSP and After 2210 Addition.....	10
Figure 5. XRD from Scenario 1 Feed Sample .....	12
Figure 6. XRD from Scenario 1 Concentrate Sample.....	12
Figure 7: Scale Buildup on Walls during Scenario 1-1 .....	13
Figure 8. DF for Potassium and Molybdenum.....	14

## LIST OF TABLES

Table 1. Simulant Compositions and Physical Properties .....	4
Table 2. Scaling Parameters.....	5
Table 3. Major Species in Recycle Evaporator Condensate .....	10
Table 4. Evaporator DF for Selected Species .....	14

## **LIST OF ACRONYMS**

DWPF	Defense Waste Processing Facility
SMECT	Slurry Mix Evaporator Condensate Tank
OGCT	Off Gas Condensate Tank
DWTT	Decontamination Waste Treatment Tank
RCT	Recycle Collection Tank
SRAT	Sludge Receipt and Adjustment Tank
ETP	Effluent Treatment Plant
SRNL	Savannah River National Laboratory
ITS	Immobilization Technology Section
ACTL	Aiken County Technology Laboratory
ML	Mobile Lab
ADS	Analytical Development Section
DWPF-E	Defense Waste Processing Facility: Engineering
WWCT	Waste Water Collection Tank

## 1.0 INTRODUCTION

DWPF converts the high level waste slurries stored at the Savannah River Site into borosilicate glass for long-term storage. The vitrification process results in the generation of approximately five gallons of dilute recycle streams for each gallon of waste slurry vitrified. This dilute recycle stream is currently transferred to the H-area Tank Farm and amounts to approximately 1,400,000 gallons of effluent per year. Process changes to incorporate salt waste could increase the amount of effluent to approximately 2,900,000 gallons per year.

The recycle consists of two major streams and four smaller streams. The first recycle major recycle stream is condensate from the Chemical Process Cell, and is collected in the Slurry Mix Evaporator Condensate Tank (SMECT). The second major recycle stream is the melter offgas condensate which is collected in the Off Gas Condensate Tank (OGCT). The four smaller streams are the sample flushes, sump flushes, Decon solution, and HEME dissolution solution. These streams are collected in the Decontamination Waste Treatment Tank (DWTT) or the Recycle Collection Tank (RCT). All recycle streams are combined in the RCT and treated with sodium nitrite and sodium hydroxide prior to transfer to the tank farm.

Tank Farm space limitations and previous outages in the 2H Evaporator system due to deposition of sodium alumina-silicates<sup>1</sup> have led to evaluation of alternative methods of dealing with the DWPF recycle. One option identified for processing the recycle was a dedicated evaporator to concentrate the recycle stream to allow the solids to be recycled to the DWPF Sludge Receipt and Adjustment Tank (SRAT) and the condensate to be treated in the Effluent Treatment Plant (ETP).

In order to meet process objectives, the recycle stream must be concentrated to 1/30<sup>th</sup> of the feed volume during the evaporation process. The concentrated stream must be pumpable to the DWPF SRAT vessel and should not precipitate solids to avoid fouling the evaporator vessel and heat transfer coils. The evaporation process must not generate excessive foam and must have a high DF for many species in the evaporator feed to allow the condensate to be transferred to the ETP. An initial scoping study was completed in 2001 to evaluate the feasibility of the evaporator<sup>2</sup> which concluded that the concentration objectives could be met. This initial study was based on initial estimates of recycle concentration and was based solely on OLI modeling of the evaporation process.

SRNL has completed additional studies using simulated recycle streams to evaluate foaming, scaling, and DF using small-scale laboratory simulations of the evaporator. These studies will also be utilized to validate the results from OLI modeling to gain confidence in using the models to further evaluate the evaporator process. This work was in accordance with guidance from DWPF-E<sup>3</sup> and was performed in accordance with the Technical Task and Quality Assurance Plan<sup>4</sup>.

## 2.0 EXPERIMENTAL METHODS

### 2.1 Recycle Evaporator Feed Blending

The recycle evaporator receives feed from multiple sources and the variation in feed composition from the different combinations of streams that could be processed is large. Six scenarios were developed based on input from DWPF Engineering<sup>5</sup> and were designed to represent various operating conditions of the DWPF process. The recycle volumes shown below are the volume ratios for each of the six scenarios tested. Volumes were based on the recycle volumes utilized during the initial feasibility study. Scenario 1 was evaluated in three separate runs to determine the reproducibility of the results. Addition of a silicon reactant was not performed during the testing.

#### Scenario 1: Normal Operation with Sludge (Current Process)

SMECT Volume	2016	ml
OGCT Volume	1721	ml
Sample Flush Volume	5	ml
Drains/Cells Sump Volume	258	ml
Decon Solution Volume	0	ml
HEME Dissolution Volume	0	ml

#### Scenario 2: CPC Operation with Sludge w/ Melter in Idle

SMECT Volume	3539	ml
OGCT Volume	0	ml
Sample Flush Volume	8	ml
Drains/Cells Sump Volume	453	ml
Decon Solution Volume	0	ml
HEME Dissolution Volume	0	ml

#### Scenario 3: Melter Operation with Sludge w/ CPC Idled

SMECT Volume	0	ml
OGCT Volume	4000	ml
Sample Flush Volume	0	ml
Sump Flush Volume	0	ml
Decon Solution Volume	0	ml
HEME Dissolution Volume	0	ml

#### Scenario 4: Normal Operation with Sludge w/ HEME Dissolution

SMECT Volume	1452	ml
OGCT Volume	1240	ml
Sample Flush Volume	3	ml
Drains/Cells Sump Volume	0	ml
Decon Solution Volume	0	ml
HEME Dissolution Volume*	1305	ml

\* The HEME material was neutralized to pH=7 with nitric acid.

Scenario 5: Decon Operation with Sludge w/ Melter Operating

SMECT Volume	0	ml
OGCT Volume	2000	ml
Sample Flush Volume	0	ml
Drains/Cells Sump Volume	0	ml
Decon Solution Volume	2000	ml
HEME Dissolution Volume	0	ml

In addition the five scenarios listed above, the impact of additional condensate generation from the introduction of dilute streams from the planned salt processing was evaluated for normal operation conditions. Scenario 6 was the same as Scenario 1, except that 4000 ml of additional water was added and the required concentration factor for this scenario was 60X, as shown below:

Scenario 6: Normal Operation with Salt Processing Streams

SMECT Volume	2016	ml
OGCT Volume	1721	ml
Sample Flush Volume	5	ml
Drains/Cells Sump Volume	258	ml
Decon Solution Volume	0	ml
HEME Dissolution Volume	0	ml
Additional water from Salt Streams	4000	ml

**2.2 Recycle Evaporator Feed Compositions**

A recipe for each of the streams listed above was developed based on sample results from characterization of DWPF samples<sup>6,7</sup> or process knowledge<sup>8,9</sup>. These recipes were developed by SRNL and reviewed by DWPF-E<sup>10</sup>. The recipe for each recycle stream is shown in Appendix A. Sample results were obtained from each of the simulants and are shown in Table 1.

**Table 1. Simulant Compositions and Physical Properties**

Simulant	Ag	Al	B	Ba	Ca	Cr	Cu	Fe	Gd	Hg	K	Li
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/kg	mg/L	mg/L
SMECT	ltd	0.439	0.215	ltd	0.069	ltd	ltd	ltd	ltd	173	0.234	ltd
OGCT	2.26	555	7.27	10.8	307.5	2.72	17.6	2115	8.785	119	26.65	18.15
DWTT Decon	ltd	0.43	1.97	ltd	24.1	ltd	ltd	1465	ltd	187	2.08	ltd
DWTT HEME	ltd	147	293	0.256	250.5	1.415	ltd	2.825	ltd	ltd	115	ltd
Sample Flush	16.9	15050	30.0	71.5	2115	27.95	151	59200	64.5	nm	217.5	46.85

Simulant	Mg	Mn	Na	Ni	P	Pb	S	Si	Sr	Ti	Zn	Zr
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
SMECT	ltd	ltd	0.494	ltd	ltd	ltd	ltd	1705	ltd	ltd	ltd	ltd
OGCT	383.5	631	2315	93.7	6.1	9.865	41.6	40.1	1.135	1.515	29.7	4.64
DWTT Decon	57.05	127	197.5	42.45	ltd	ltd	20.2	4.005	ltd	ltd	ltd	ltd
DWTT HEME	72.3	ltd	30200	0.0235	0.3995	0.203	1.83	3550	ltd	1.02	0.073	2.43
Sample Flush	5015	9595	15100	2100	78.7	78.05	487	115.5	4.235	21.75	313	39.1

Simulant	NO <sub>2</sub>	NO <sub>3</sub>	SO <sub>4</sub>	HCO <sub>2</sub>	Density		Solids			pH	Acid Equal.	Viscosity @ 25C
					Slurry	Supernate	Total	Soluble	Insoluble			
	mg/L	mg/L	mg/L	mg/L	g/ml	g/ml	wt%	wt%	wt%		Molar	cP
SMECT	ltd	5930	ltd	442.5	nm	1.0009	ltd	ltd	ltd	0.98	nm	nm
OGCT	ltd	1595	1018.5	ltd	1.0010	0.9995	0.37	0.09	0.28	2.15	0.01	0.892
DWTT Decon	ltd	13650	789	8385	1.0140	1.0108	2.46	1.57	0.89	1.68	0.14	0.921
DWTT HEME	201.5	ltd	ltd	ltd	1.0631	1.0613	6.35	6.35	0	13.73	1.39	1.23
Sample Flush	ltd	39550	7575	82100	1.2886	nm	38.22	nm	nm	5	nm	nm

Notes: The DWTT HEME solution was sampled prior to neutralization of the NaOH with nitric acid. The “acid equivalents” shown for this sample is the base equivalents. Denotations “nm” and “ltd” stand for “not measured” and “less than detectable”, respectively.

### 2.3 Test Apparatus

A laboratory scale evaporator was fabricated as shown in Appendix B. The evaporator consists of an evaporation vessel with stir bar, a stirring hotplate, an aluminum heat transfer plate, wire mesh demister, a condenser with recirculating water bath, and a condensate collection vessel. A feed tank and pump was utilized to feed the simulated recycle to the evaporator during the run.

Antifoam tests were conducted using an evaporator with internal heating coil, a hotplate, condenser with recirculating chiller, and a condensate collection tank. This apparatus is also shown in Appendix B. A continuous evaporation was performed with this apparatus by adding a feed system and a condensate removal system.

Scaling parameters for both apparatus are shown in Table 2. The full-scale values are based on a conceptual design proposed by a consultant hired by DWPF to evaluate the feasibility of installing the recycle evaporator in the DWPF Salt Process Cell. The parameters for the DWPF SRAT vessel and 4-L SRAT apparatus are shown for comparison.

**Table 2. Scaling Parameters**

	<b>Full-Scale</b>	<b>DWPF SRAT</b>	<b>Antifoam Test Apparatus</b>	<b>Boil-Down Test Apparatus</b>	<b>4-L SRAT Lab Apparatus</b>
Diameter (inches)	60	144	3.25	2.4	5
Boil-Up Rate (g/min)	28000	38000	14	2.5	4
Flux Rate (g/min-in <sup>2</sup> )	9.20	2.33	1.7	0.6	0.2
Flux Rate (% of full scale)	100	25	17	7	2.2
Flux Rate (% of SRAT)	400	100	73	26	9
Heating Source	Steam	Steam	Hotplate and heating coil	Hotplate w/ aluminum block	Mantle

## 2.4 Run Performance

The boil-down tests were performed in small vessels (~300 ml) to minimize the contribution of splatter and buildup to the material balance. The boil-up rate was approximately 3 ml/min during the water runs, but was reduced during several of the boil-down tests to reduce foam height. The level in the evaporator was maintained as close as possible to 100 ml throughout the run by matching feed additions to the boil-off rate. The process steps utilized during the run are shown in Appendix B.

## 2.5 Sample Analysis Methods

Samples were taken and submitted for analysis as outlined in the Analytical Study Plan.<sup>11</sup> Samples were taken of each simulant and blended feed stream prior to each run. Samples of the concentrate and condensate were taken at the conclusion of each run. Measurements of solids content, density, pH, viscosity, and acid/base titrations were conducted by Immobilization Technology Section (ITS) at the Aiken County Technology Laboratory (ACTL). Sample composition was measured by ITS at the Mobile Lab (ML) for all species except mercury. Mercury and XRD analysis was performed by the Analytical Development Section (ADS). Each analysis was performed as outlined below.

### 2.5.1 pH

An IQ Scientific model IQ150 pH probe was utilized to measure the pH of the samples. This instrument is an ion-selective pH meter with automatic temperature measurement and compensation. The instrument was checked with pH 4, 7, and 10 buffers before and after use.

### **2.5.2 Density**

An Anton Paar DMA-4500 density meter was utilized to determine the density of samples. This instrument utilizes the dampening of a vibrating u-tube to measure density. The instrument was checked with DI water prior to each use.

### **2.5.3 Solids Content**

The solids content was measured with a Mettler-Toledo HR83 halogen moisture meter. The solids content of the slurry and the solids content of supernate were measured. Supernate was prepared by filtration of the slurry through a syringe filter. The insoluble and soluble solids content were calculated from the results. The instrument was checked with standards before and after use.

### **2.5.4 Viscosity**

Viscosity was measured with a Haake RS-600 rheometer using the 0.5 degree, 60 mm cone and plate geometry at a single shear rate (2000 seconds<sup>-1</sup>). The instrument was checked with viscosity standards before each use.

### **2.5.5 Mobile Laboratory Measurements**

The ML performed metals analysis utilizing ICP-ES and anion analysis using IC-Anions. A peroxide fusion digestion was performed on the samples prior to ICP-ES measurements. Standards and blank analysis were performed before and after each set of analytical runs.

### **2.5.6 Titrations**

The initial samples from this study were titrated at ACTL using a Mettler-Toledo DL40GP autotitrator. The standard titration method utilized two milliliters of sample diluted into approximately sixty ml of DI water. 0.1M hydrochloric acid or sodium hydroxide solution was added while the sample was mechanically agitated. pH is continuously monitored during the acid addition. Duplicate analysis was performed on each sample and the results were averaged. Results were rejected if the difference between the duplicate samples was greater than 10%.

The autotitrator failed during the test program, therefore direct titration using manual additions of sodium hydroxide to the sample was utilized. The pH probe utilized during the direct titration tests was the same as for the pH measurements. The direct titration method utilized approximately 5 ml of sample diluted to 20 ml with DI water. The sample was continuously mixed while 0.1 ml additions of sodium hydroxide solution was added. The pH was allowed to stabilize between each addition and was recorded prior to the next addition. A plot of pH versus amount of acid added per ml of sample was generated and the intercept of the pH curve with pH = 7 was determined. Two measurements were taken for each sample and the results checked for consistency.

### **2.5.7 Vapor Pressure**

Vapor pressure measurements were planned using two Grabner MiniVap VOC instruments, but both instruments failed during initial water checks and repairs were not performed. The measurements were intended to help validate the OLI models and to provide temperature data to aid in evaluation of vacuum operation of the evaporator. The OLI validation was completed without the vapor pressure data using the



data from the boil-down tests and vacuum operation is not currently planned, therefore the vapor pressure data is not required to complete the task.

### 2.5.8 ADS Measurements

Mercury was analyzed using atomic adsorption cold vapor methods after digestion with aqua regia. Standards were analyzed before and after each set of analytical runs.

XRD analysis was performed on solids filtered onto filter paper. The supernate from the solids filtration was filtered through a second filter and submitted with the solids to provide a “blank” to determine if solids in the XRD analysis are from the supernate or solids content of the sample.

## 3.0 RESULTS

All six of the scenarios outlined above were tested and Scenario 1 was tested in triplicate<sup>12,13</sup>. The sample results from the tests are shown in Appendix C. DF's were calculated for each species based on the sample results. Foaming was noted in all runs, with severe foaming occurring in some of the runs. Foaming was controlled by reducing the boil-up rate. No evidence of precipitation was noted during any of the runs. Scaling below the liquid surface was not noted, but splattering of particulate onto the vessel led to some solids deposition on the vessel walls above the foam layer.

### 3.1 Foaming

Foaming during the evaporation process was noted to some extent during all runs. Foam was controlled during the tests by reducing the boil-up rate. The reduced boil-up rate lowered the vapor flux rate at the liquid surface and significantly reduced the amount of foam in the evaporation vessel. The flux rate for the boil-down tests was less than 10% of the flux rate in the conceptual design for the evaporator proposed by Bennett, as shown above in Table 2. Higher flux rates would have led to increased foaming, therefore the foaming noted during the boil-down tests was noted as a concern for operation of the recycle evaporator. The maxima in foam height were observed for the runs at the onset of boiling with a gradual decrease in foam height noted as the runs progressed, as shown in Figure 1. Foaming was least in the run during evaporation of Scenario 3 (OGCT only) recycle.



**Figure 1. Foaming during Initial and Final Stages of Scenario 1, Run 1 Evaporation**

Scaling of foaming phenomena is difficult from tests conducted in small-scale apparatus due to the effects of the vessel walls in stabilizing foam lamellas. The “wall effect” is appreciable in the small-scale tests but disappears on scale-up. The wall effect is especially pronounced if the diameter of the bubbles in the foam lamella are greater than 1/3 the diameter of the vessel. As shown in the above figures, bubbles during the boil-down tests were much smaller than the vessel and the amount of foam noted was not attributed to wall effects.

Additional tests were conducted to study the potential for foam formation and to evaluate the performance of antifoams recommended by Dow Corning and the antifoams identified for use in the Hanford RPP project (Dow Corning Q2-3183A). These tests were conducted in a larger diameter vessel with increased flux rates to better simulate the full-scale process. The tests were conducted by charging 700 ml of feed to the vessel and evaporating until 400 ml of condensate had been collected. Antifoam (1000 ppm) was added prior to the heating the vessel to boiling.

A water run was conducted to determine the maximum boil-up rate and the height of vapor disengagement in the vessel with a non-foaming liquid. During this run, full power (1000 watts) on the coil and a temperature setting of 500° C on the hotplate yielded a boil-up rate of 25 grams per minute. Vapor disengagement amounted to approximately 100 ml during the run. A baseline run without antifoam was then conducted. The foam quickly rose in the vessel to a height greater than 2000 ml (height of last graduation line) and would likely have foamed over in the condensate collection vessel if the coil power had not been reduced to 600 watts. The foam level remained above 2000 ml until the conclusion of the run. All subsequent tests were conducted at 600 watts to allow direct comparison to this test and prevent foam-over if the antifoam tested was ineffective.

The next test was conducted with Dow Corning Q2-3183A. The foam height at the onset of boiling was significantly reduced, but the foam level was judged to be excessive and an additional 1000 ppm of the antifoam was added. Foam control during this run was not deemed adequate, and additional tests were performed with antifoams recommended by Dow Corning (1430, 2210, and DSP). As shown in Figure 2, foam control with these antifoams was much improved over the Q2 3183A antifoam and all three antifoams performed similarly.

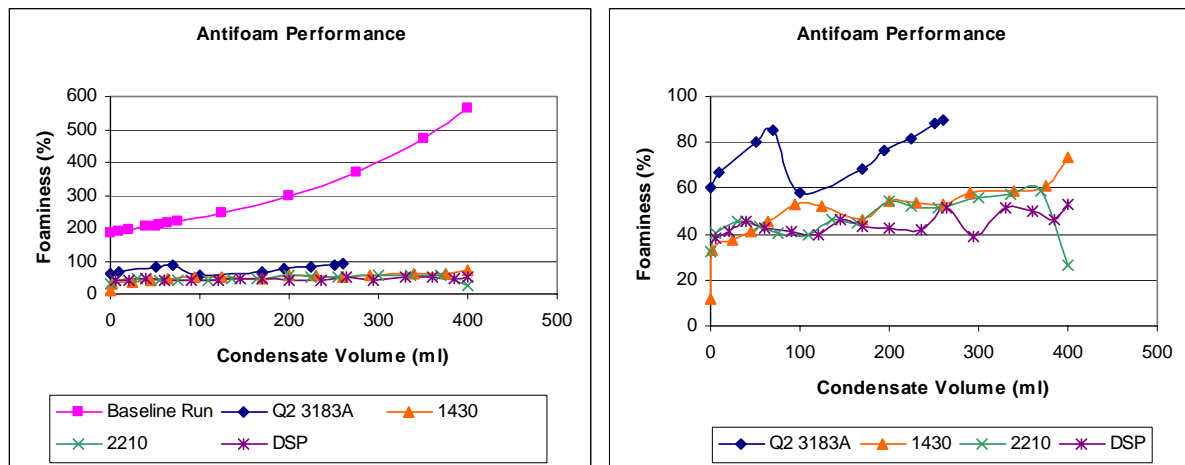
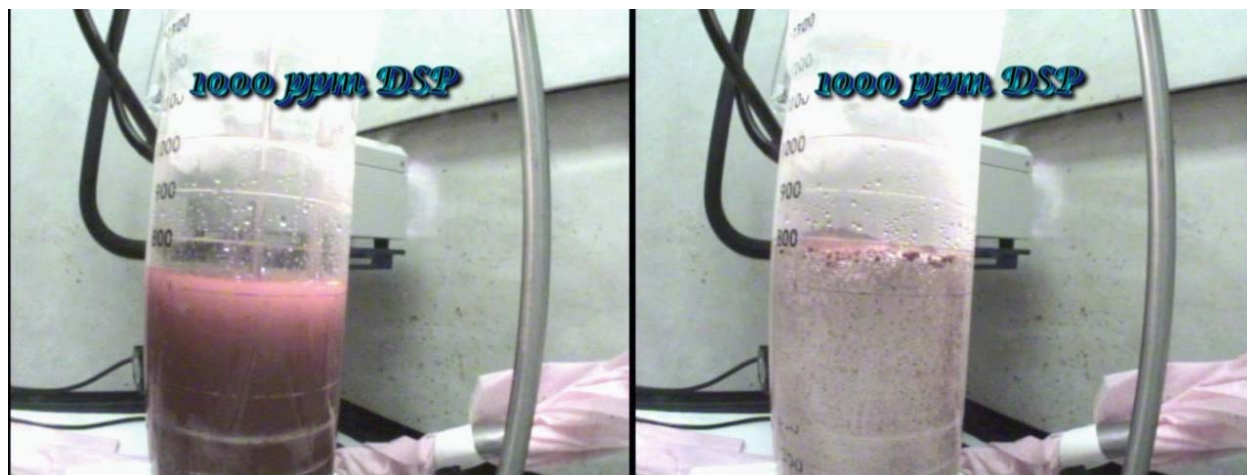


Figure 2. Performance of Antifoams

Severe flocculation of the feed material was noted in both the 2210 and DSP antifoam tests, as shown in Figure 3. The flocculation occurred just prior to the onset of boiling and resulted in the solution

appearance changing from a red, opaque slurry to a clear liquor containing large red chunks. A significant portion of the solids were trapped in a surface film. This film was agitated and mixed into the body of the solution at the onset of boiling. Antifoams often contain flocculating agents to aid in foam control, therefore the severe flocculation noted could have been the result of the addition of more antifoam than required. As the run progressed, the solids began to break apart and the appearance of the slurry at the conclusion of the tests was similar to the baseline run.



**Figure 3. Flocculation during Addition of DSP Antifoam**

A test run was performed with DSP antifoam to determine the concentration of the antifoam required to control the foam and to determine how often additional antifoam would be required during evaporator operation. The antifoam test apparatus was modified to allow continuous operation and the test was performed. An initial charge of 700 ml was added to the evaporation vessel and the run was started. Feed material was added to match the boil off amounts to maintain the level in the evaporator at 700 ml and the feed was concentrated 30X.

Antifoam was added in small amounts until the foam height was less than 1200 ml, with approximately ~700 ppm required. Additional 100 ppm antifoam additions were required after approximately 30 minutes to keep the foam height below 1200 ml. As the initial foaming subsided, less antifoam was required. While the foam was stable, an additional charge of DSP antifoam was added to determine if the foam height would be reduced. The foam height increased significantly after addition, and 100 ppm 2210 antifoam was added to control the foam, as shown in Figure 4. No additional antifoam was required, but an additional charge of 2210 antifoam was added to determine if the stable foam height could be reduced. Very little reduction in the foam height was noted, but the foam height did not increase as was noted with the DSP antifoam.



Figure 4: Foaming During Antifoam Tests with DSP and After 2210 Addition

### 3.2 Condensate Characterization

The condensate was sampled at the conclusion of the test run, but this sample did not represent an aggregate sample of all condensate from the run. Condensate was collected continuously during the test run, but was removed batch wise when the condensate collection tank was filled. A small heel was left in the collection vessel during each transfer. The evaporator contents at the start of the run matched the feed composition, therefore the condensate generated during the initial portion of the run would not be representative. The first three liters of feed were required to reach steady-state concentration in the evaporator. An additional liter of feed was fed during steady-state operation; condensate from this stage of operation was contained in the sample at the conclusion of the run. The Scenario 4 condensate sample represents the last sample pulled prior to the foam over. All condensate bottles were analyzed for pH, results are shown in Appendix E. The sample results obtained for the condensate are shown in Appendix C and were compared to the ETP WAC for the Waste Water Collection Tank (WWCT)<sup>14</sup>. Major species noted in the condensate are shown in Table 3.

Table 3. Major Species in Recycle Evaporator Condensate

Scenario #	Description	Hg	Si	Nitrate	Formate	TOC*	pH
ETP WAC Acceptance Limits for the WWCT		40.6	99.2	1180		385	1-12.5
		ppm	ppm	ppm	ppm	ppm	
1-1	"Typical" Operation	152	408	429	361	96	2.2
1-2	"Typical" Operation	12	67	284	398	106	2.4
1-3	"Typical" Operation	77	67	420	434	116	2.3
2	Melter Idled	17	1120	703	819	218	1.9
3	CPC Idled	0	0	0	0	0	3.0
4	HEME Dissolution	36	28	840	0	0	2.0
5	Decon Operation	20	0	1080	3015	804	2.0
6	Salt Operations	23	9.5	172	177	47	2.5

\* Calculated from formate concentration.

The WAC for mercury, silicon, and organic carbon was exceeded during at least one of the test runs. The mercury results contained a significant amount of scatter and are discussed below. The silicon noted for Scenario 1-1 may be an outlier, but the result from Scenario 2 is well above the limits and is consistent with the larger amounts of silicon-based antifoam that would be present during a run that only contains material from the CPC operations. The run with high TOC contained a large amount of formate in the SME solids dissolved in the decon solution. Limiting the amount of decon solution in the evaporator feed would likely prevent formate from exceeding the limit.

Beads of mercury were noted in the condensate collection vessel for all runs except Scenario 3 (OGCT only). Based on the presence of elemental mercury beads in the condensate, the condensate was saturated with Hg. Typically, the saturation point of a solution is pH dependant. However, the concentration of Hg in the condensate samples varied from run to run at the same pH. These data suggest that the saturation point of mercury in the condensate samples was not merely a function of the pH. A possible explanation for the different concentrations noted for the three runs of Scenario 1, could be due to the reducing and oxidizing agents present in the samples. The presence of oxidizing and reducing agents is known to impact the solubility of mercury.

A “silicon reactant” was shown on several draft versions of the evaporator flowsheets prepared by DWPF-E. This reactant was developed to lower the risk of sodium alumino-silicate formation in waste evaporators in the tank farm<sup>15</sup>. The reactant (ferric nitrate) was to be added to recycle after caustic adjustment and would co-precipitate with the sodium meta-silicate in solution. The reactant would not function in an acidic stream (as planned for the recycle evaporator) and would likely not react with the siloxanes in the recycle stream from antifoam degradation products.

### 3.3 Concentrate Characterization

The concentrate from each boil-down test was sampled and characterized and are shown in Appendix C. A concentration factor was calculated for each species in the concentrate by dividing the concentrate concentration by the feed concentration and these values are also shown in Appendix C. The concentrate from Scenario 4 was lost when the evaporator foamed over into the condensate.

The concentration factors vary somewhat from the concentration target due to uncertainties in the sample analysis and the errors in determining the solution height during the run. The foaming in the evaporator led to difficulty in the determination of the liquid height during the run. During steady state operation, errors in the level would have resulted in removal of concentrate that was too dilute or too concentrated. These errors led to some variability in the actual concentration of the final sample.

All runs except Scenario 4 were able to achieve the concentration target, within the uncertainties described above and resulted in concentrate that was free flowing. Solids contents ranged from 3 to 30%, dependent on the solids content of the incoming feed, with a nominal value of 10% for runs without DWTT content. The solids content of the DWTT stream raises the solids content of the feed considerably. The amount of HEME dissolution material in Scenario 4 resulted in a feed that would have exceeded 100% solids if concentrated 30X, resulting in a foam over when the feed became too concentrated for the evaporator apparatus. The run with coil decontamination solution from the DWTT reached 30% solids content.

### 3.4 Precipitation

No precipitation of solids was noted during the runs, based on visual observations during the runs and XRD results. Results from the solids analysis were inconclusive due to the small amount of solids

originally present. Figure 5 and Figure 6 are typical XRD results for the feed and concentrate samples and are from Scenario 1. The remaining XRD diagrams are shown in Appendix D. Note that much of the gibbsite noted in the feed is converted to aluminum oxide during the evaporation process.

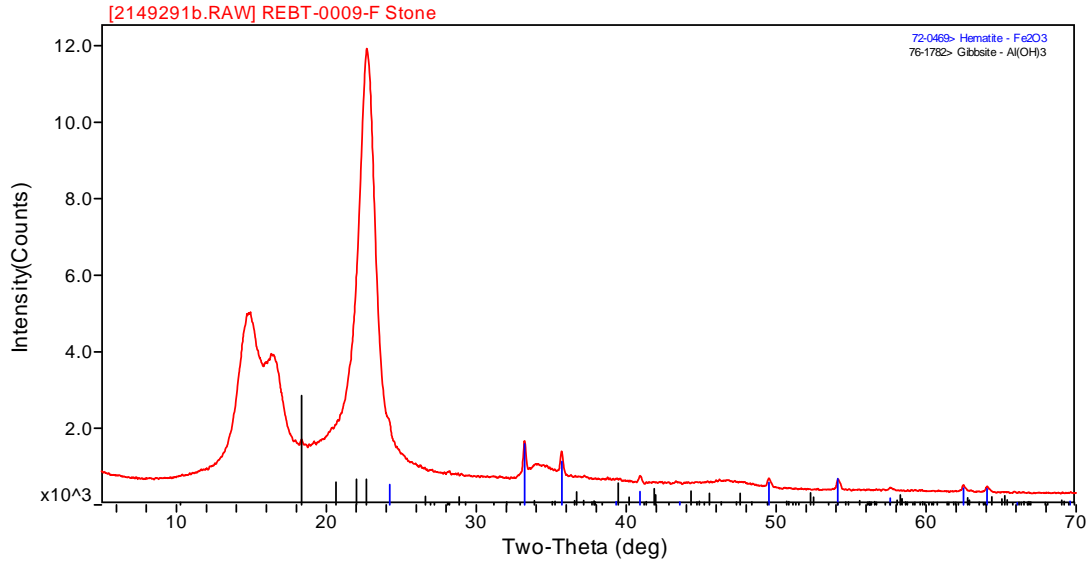


Figure 5. XRD from Scenario 1 Feed Sample

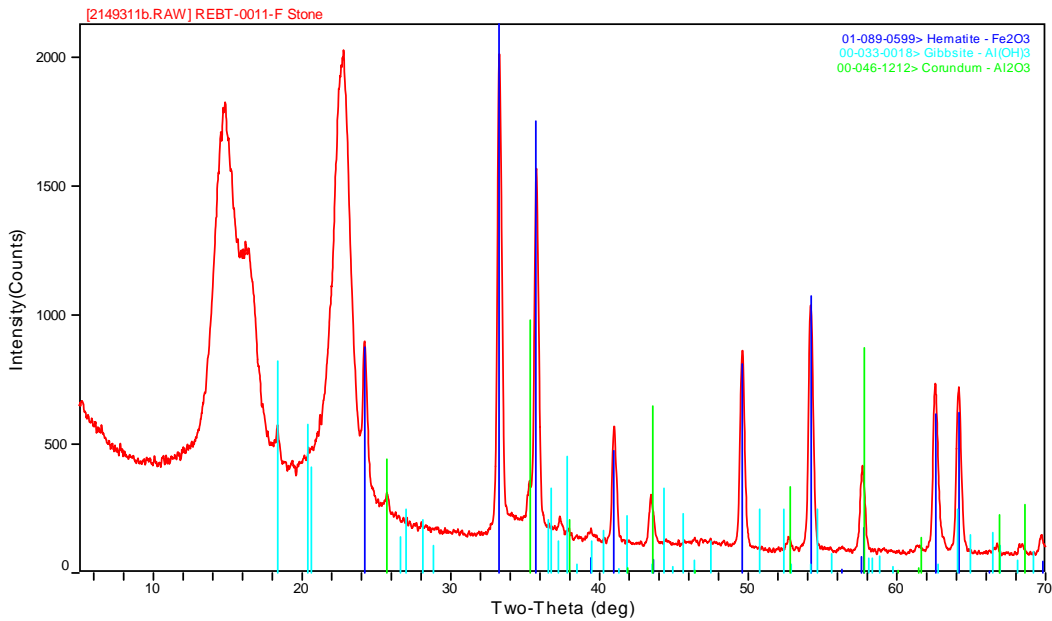


Figure 6. XRD from Scenario 1 Concentrate Sample



### 3.5 Scaling

Visual observations of the evaporation apparatus after each test were conducted to determine if scaling was occurring. During all runs, the boiling surface was vigorous and caused some splatter of solids onto the vessel walls. These solids tended to stick to the walls, as shown in Figure 7 and were not washed off by the limited amount of internal reflux that occurred during the runs. The solids deposited on the walls were not easily removed by 2M nitric acid rinses. Two of the antifoams (Q2-3183A and 1430) led to a film formation above the foam line during antifoam testing. This film was easily cleaned from the vessel with water. The flocculation caused by 2210 and DSP antifoams led to larger particles being deposited on the walls, but these particles were easily removed during cleaning.



**Figure 7: Scale Buildup on Walls during Scenario 1-1**

During antifoam testing, a stainless steel heating coil was utilized. No significant deposition was noted on the heating surface during the tests.

### 3.6 Evaporator DF

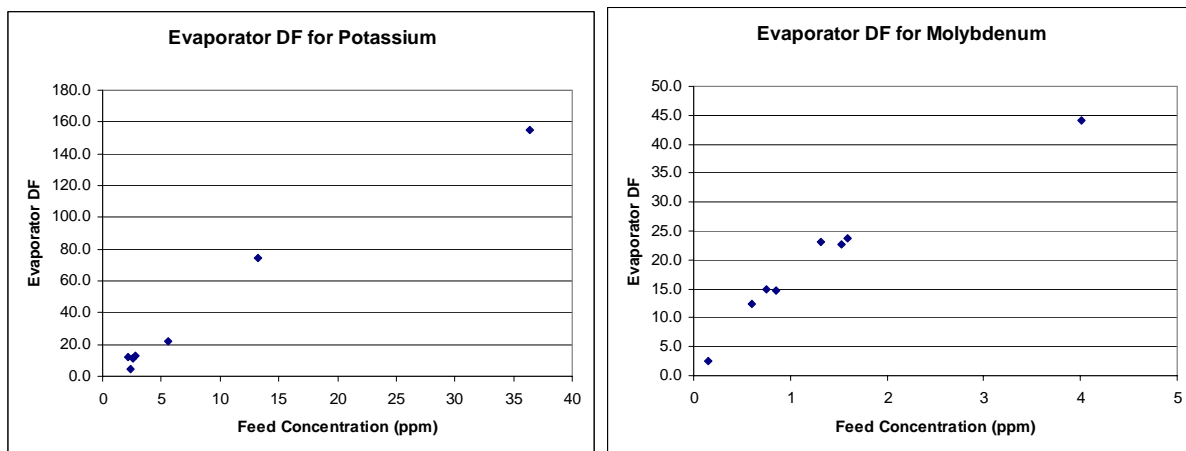
The evaporator DF was calculated for each species present in the feed samples based on the sample results from the feed and condensate for each run. When the condensate results indicated less than detectable amounts, the detection limit was utilized to determine the DF. The average results along with the lowest and highest value are shown in Table 4 for selected species. All results are shown in Appendix F. Values shown in italics in Table 4 and Appendix F are based on the sample detection limits.

**Table 4. Evaporator DF for Selected Species**

	Sample Detection Limit	Low	Average	High
Al	0.1	5	355	2255
B	n/a	1.2	10	22
Fe	0.04	2663	22138	46750
K	n/a	4	38	155
Hg	2.5	1.0	13	64
Mo	n/a	2	20	44
Si	n/a	0.05	8	33
NO <sub>3</sub>	n/a	7	13	29
HCO <sub>2</sub>	n/a	0.80	1.08	1.39

Several issues with the DF's should be noted. The small evaporator size utilized during the boil-down tests could have impacted the results and no attempts were made to make the vent system "prototypical". A crude demister was utilized during the test which consisted of a steel mesh wrapped into a cylinder and forced into the offgas line. This demister was likely less efficient than a commercially designed unit. Deposition of selected species on the walls of the offgas system could have raised the apparent DF for that species.

The DF for each element in the table above was evaluated graphically to determine if trends existed in the DF of the component and the feed concentration. The graphs indicate that DF is a function of feed composition for selected species and that higher feed concentrations led to higher DFs, as shown in Figure 8. Graphs for other species, except formate, are shown in Appendix F.



**Figure 8. DF for Potassium and Molybdenum**



Other species had DF's that were not a function of feed composition. These elements included silicon whose DF was a function of the type of silicon present, not the amount. Other elements such as boron also did not indicate a trend with feed composition, but the cause is not known. The detection limit was utilized to calculate the DF for most species, therefore trending could not be performed.

A review of the results for selected species was performed to determine the magnitude of the impacts of the issues listed above. The relatively high DF values for iron indicate that entrainment of drops into the offgas system was not excessive, therefore, foaming is likely not the cause of the low DF's seen for some species. When the DF for silicon is examined, runs with large amounts of frit had much higher DF's than runs with larger amounts of siloxanes from the antifoam. The presence of elemental mercury beads in the condensate collection indicates that the holdup of mercury on vessel and tubing walls did not affect the amount of mercury seen in the condensate.

## 4.0 CONCLUSIONS

Concentration of the "typical" recycle stream in DWPF by 30X was feasible. The addition of DWTT recycle streams to the typical recycle stream raises the solids content of the evaporator feed considerably and lowers the amount of concentration that can be achieved.

Foaming was noted during all evaporation tests and must be addressed prior to operation of the full-scale evaporator. Tests were conducted that identified Dow Corning 2210 as an antifoam candidate that warrants further evaluation.

The condensate has the potential to exceed the ETP WAC for mercury, silicon, and TOC. Controlling the amount of equipment decontamination recycle in the evaporator blend would help meet the TOC limits. The evaporator condensate will be saturated with mercury and elemental mercury will collect in the evaporator condensate collection vessel.

No scaling on heating surfaces was noted during the tests, but splatter onto the walls of the evaporation vessels led to a buildup of solids. These solids were difficult to remove with 2M nitric acid. Precipitation of solids was not noted during the testing.

Some of the aluminum present in the recycle streams was converted from gibbsite to aluminum oxide during the evaporation process.

## 5.0 RECOMMENDATIONS

Recycle from the DWTT should be metered in slowly to the "typical" recycle streams to avoid spikes in solids content to allow consistent processing and avoid process upsets. Additional studies should be conducted to determine acceptable volume ratios for the HEME dissolution and decontamination solutions in the evaporator feed.

Dow Corning 2210 antifoam should be evaluated for use to control foaming. Additional tests are required to determine the concentration of antifoam required to prevent foaming during startup, the frequency of antifoam additions required to control foaming during steady state processing, and the ability of the antifoam to control foam over a range of potential feed compositions. This evaluation should also include evaluation of the degradation of the antifoam and impact on the silicon and TOC content of the condensate.

The caustic HEME dissolution recycle stream should be neutralized to at least pH of 7 prior to blending with the acidic recycle streams.

Dow Corning 2210 should be used during the evaporation testing using the radioactive recycle samples received from DWPF.

Evaluation of additional antifoam candidates should be conducted as a backup for Dow Corning 2210.

A camera and/or foam detection instrument should be included in the evaporator design to allow monitoring of the foaming behavior during operation.

The potential for foam formation and high solids content should be incorporated into the design of the evaporator vessel.

## 6.0 REFERENCES

- <sup>1</sup> W. R. Wilmarth, “Results of Aluminosilicate Formation Testing”, WSRC-TR-2001-00330, September 11, 2001.
- <sup>2</sup> D. P. Lambert, C. D. Barnes, S. G. Phillips, “DWPF Recycle Evaporator – Feasibility Study”, WSRC-RP-2001-01101, January 18, 2002.
- <sup>3</sup> A. V. Staub, “Process Development for DWPF Recycle Acid Evaporator”, HLW/DWPF/TTR-04-0020, Rev. 1., October 18, 2004.
- <sup>4</sup> M. E. Stone, “DWPF Recycle Evaporator Boil-Down Tests”, WSRC-RP-2004-00648, September 24, 2004.
- <sup>5</sup> J. E. Occhipinti and A. V. Staub, “Estimated Stream Compositions for DWPF Recycle Influent”, CBU-WSE-2004-00177, September 27, 2004.
- <sup>6</sup> T. L. Fellingner, and N. E. Bibler, “Results for the DWPF Slurry Mix Evaporator Condensate Tank, Off Gas Condensate Tank, and Recycle Collection Tank (U)”, WSRC-TR-2004-00577, November 19, 2004.
- <sup>7</sup> T. L. Fellingner, and N. E. Bibler, “Results for the DWPF Decontamination Waste Treatment Tank Samples (U)”, WSRC-TR-2005-00036, January 25, 2005.
- <sup>8</sup> C. A. Cicero, “HEME and HEPA Filter Element Dissolution Process (U)”, WSRC-TR-92-549, November 23, 1992.
- <sup>9</sup> J. E. Occhipinti and A. V. Staub, “Estimated Stream Compositions for DWPF Recycle Influent”, CBU-WSE-2004-00177, September 27, 2004.
- <sup>10</sup> M. E. Stone, “Simulant Recipes for DWPF Recycle Evaporator Tests (U)”, SRNL-GPD-2004-00061, November 8, 2004.
- <sup>11</sup> M. E. Stone, “Analytical Study Plan for DWPF Acid Evaporator Samples (U)”, SRNL-GPD-2004-00058, October 26, 2004.
- <sup>12</sup> Laboratory Notebook, “DWPF Recycle Evaporatory Boil-Down Tests”, WSRC-NB-2004-00151
- <sup>13</sup> Laboratory Notebook, “DWPF Recycle Evaporation Boil-Down Tests 2”, WSRC-NB-2005-00026
- <sup>14</sup> A. W. Wiggins, “F/H Effluent Treatment Project Waste Acceptance Criteria (U)”, X-SD-H-00009, December, 2004.
- <sup>15</sup> W. R. Wilmarth, et al, “Removal of Silicon from High Level Waste Streams via Ferric Flocculation”, WSRC-MS-2003-00759, June 25, 2003.

**APPENDIX A. SIMULANT RECIPES**

Table A-1. DWTT Decon Solution

Deionized Water	985.7	grams
70% Nitric Acid	14.29	grams
Dried SB3 SME Solids	50	grams
Mercuric Oxide	0.23	grams

Table A-2. DWTT HEME Dissolution\*

Deionized Water	997.5	grams
Sodium Hydroxide	52.5	grams
HEME filter media	6.55	grams

\* Solution was boiled until HEME filter disintegrated. Solution was neutralized with nitric acid prior to use.

Table A-3. SMECT Condensate Simulant

Addition Chemical	Amount (grams)
70% Nitric acid	7.2238
Mercuric nitrate hydrate	0.3416
90% Formic acid	0.3237
1:10 757 Antifoam	3.7297
Deionized Water	Add to 1 liter

Table A-4. OGCT Simulant Recipe

Addition Chemical	Amount (grams)
70% Nitric Acid	1.5532
97.5% Sulfuric Acid	0.0639
10% Hydrofluoric Acid	0.2000
Sodium Nitrate	0.6153
Calcium Nitrate Tetrahydrate	0.1356
Aluminum oxide	0.4001
Ferric oxide	2.9011
Magnesium Oxide	0.0945
Manganese Oxide	0.1559
Nickel Oxide	0.0674
Mercuric Oxide	0.2154
Leached Frit 418	0.1767
DI Water	Fill to 1 liter mark

#### Sample Flush Recipe

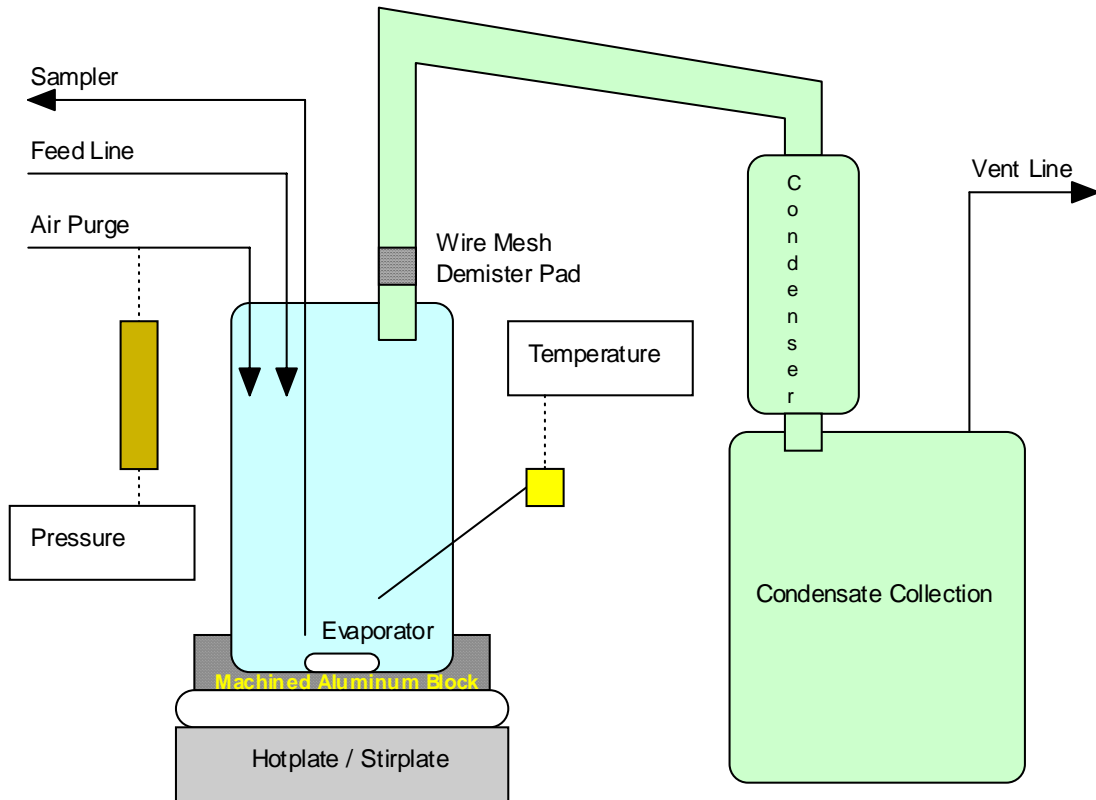
The sample flush was a 50-50 blend of SRAT and SME product from SB3 simulant tests.

#### Other Streams

All other streams were assumed to be primarily water and was added as deionized water during the evaporation testing.

APPENDIX B. EQUIPMENT DIAGRAMS AND OPERATION STEPS

Evaporator Volume: 300 ml  
Evaporator Diameter: 6 cm  
Top Ports: 4 #7 Ace Threads  
1 24/40 ground glass



Not Shown: Feed tank and pump  
Concentrate tank and pump  
Offgas composition instruments  
Water bath and lines

Figure B-1. Diagram of Evaporator for Boil Down Tests.



Figure B-2. Boil-Down Evaporator Photograph

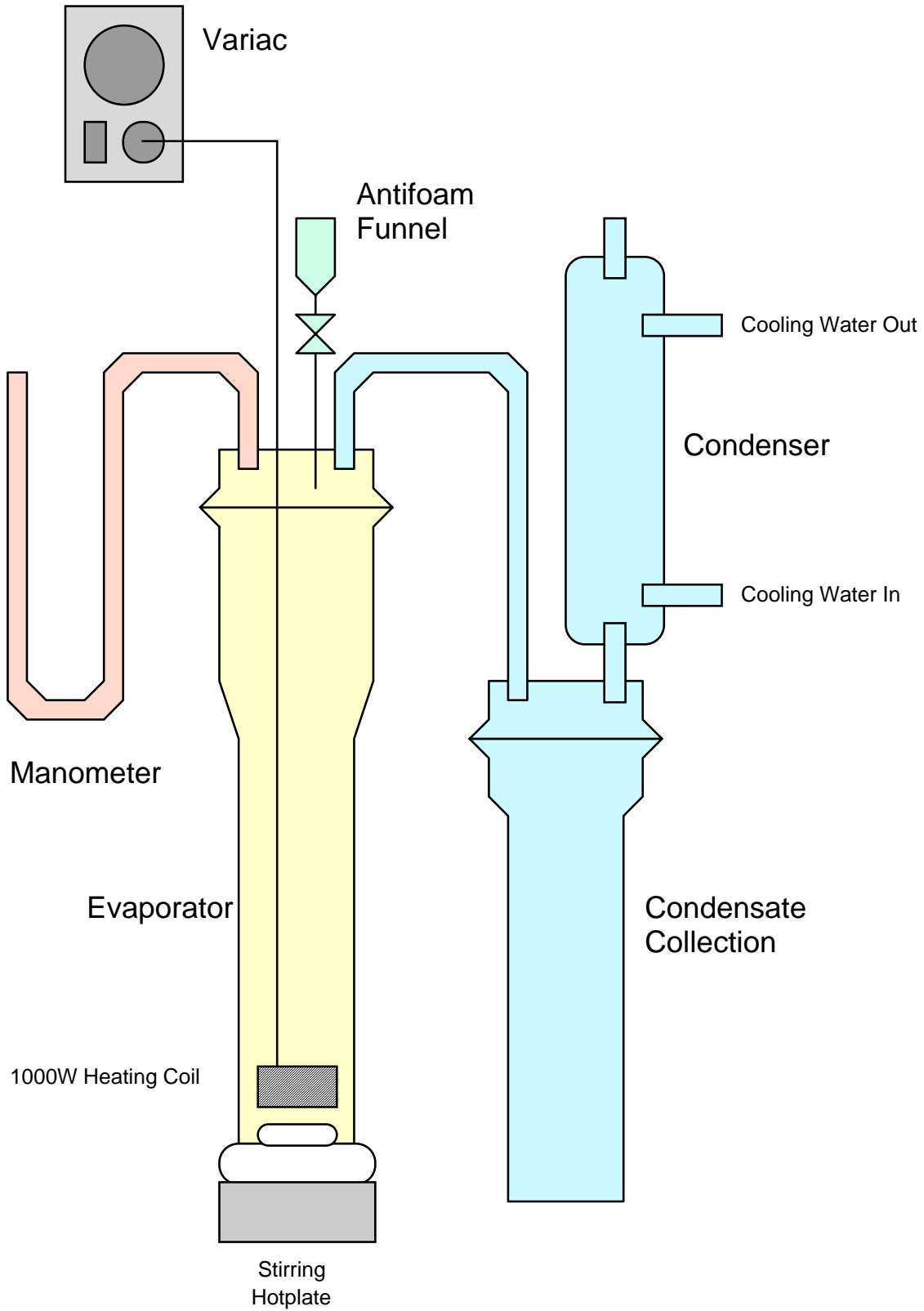


Figure B-3. Diagram of Evaporator for Antifoam Tests





Figure B-4. Antifoam Evaporator Photograph

### Operating Steps for Boil-Down Tests

- 1) Ensure rig has been assembled and leaked checked.
- 2) Ensure that feed pump has been checked per instructions from PI or alternate.
- 3) Ensure that all M&TE to be utilized is recorded in laboratory notebook and Usage Log for the lab module.
- 4) Ensure that water bath contains adequate water and start bath with a temperature setpoint of 10° C.
- 5) Verify that water bath hoses and fittings are not leaking.
- 6) Charge evaporator vessel with approximately 130 ml of feed solution.
- 7) Start air purge at 50 ml/min.
- 8) Begin stirring as specified by Principle Investigator.
- 9) Ensure pressure and temperature indication are operating.
- 10) Turn on hotplate and adjust power to 500 watts.
- 11) Record time that vessel reaches boiling.
- 12) When vessel volume decreases to 100 ml, start feed pump at 2.9 ml/min.
- 13) Adjust feed rate as required to maintain 100 ml in evaporator vessel.
- 14) Condensate should be removed from the condensate collection vessel as required to prevent overfilling. The condensate collection tank should be emptied as much as possible during each transfer. Care should be taken to avoid removal of any solids that may accumulate on the bottom of the vessel. The condensate should be placed in a tared polybottle and kept separate from condensate removed at different times. The condensate removed should be weighed and labeled “DWPF Recycle Evaporator Condensate– Scenario 1” along with sample time.
- 15) When 3 liters have been fed, the feed rate should be increased and concentrate removal started via the sampler as specified by the PI. The feed rate increase and concentrate removal rate will be calculated based on the boilup rate achieved during the test.
- 16) The feed solution should be transferred to smaller bottles as required to allow all the contents to be fed to the evaporator.
- 17) When the feed is exhausted, the run should be stopped by turning off the hotplate.
- 18) When the evaporator contents have cooled to 50° C, then all equipment can be turned off.
- 19) The evaporator contents should be placed in the concentrate collection bottle. This bottle should be labeled with REBT-0011, date, researcher name, “DWPF Recycle Evaporator Concentrate– Scenario 1”.
- 20) The condensate in the collection vessel should be collected and labeled with REBT-0010, date, researcher name, “DWPF Recycle Evaporator Condensate– Scenario 1”.
- 21) All offgas components should be visually inspected for elemental mercury.

**Operating Steps for Antifoam Tests**

- 1) Ensure rig has been assembled and leak checked.
- 2) Ensure that all M&TE to be utilized is recorded in laboratory notebook and Usage Log for the lab module.
- 3) Ensure that water bath contains adequate water and start bath with a temperature setpoint of 10° C.
- 4) Verify that water bath hose and fitting are not leaking.
- 5) Charge evaporator vessel with 700 ml of feed solution.
- 6) Begin stirring as specified by Principle Investigator (PI). Record final speed of mixer.
- 7) Charge vessel with 7 grams of the 2210 antifoam (1000 ppm).
- 8) Rinse antifoam addition funnel with 10 grams of DI water.
- 9) Turn on digital hotplate with a setpoint of 500° C and turn on heating coil to 600 watts.
- 10) Record time that vessel reaches boiling.
- 11) Record foam height and condensate amount every minute during the run until directed by PI to reduce recording to once every 5 minutes.
- 12) Add additional antifoam to vessel as directed by the PI.
- 13) Dewater 400 ml of condensate.
- 14) When dewater is complete, turn off hot plate and heating coil.
- 15) Allow vessel to cool to 50° C.

## APPENDIX C. SAMPLE RESULTS FROM BOIL-DOWN TESTS

Table C-1. Sample Results from Feed Slurries

Sample ID		REBT 0009	REBT 0027	REBT 0030	REBT 0012	REBT 0015	REBT 0018	REBT 0021	REBT 0024
Description	Units	Scenario 1-1 Feed	Scenario 1-2 Feed	Scenario 1-3 Feed	Scenario 2 Feed	Scenario 3 Feed	Scenario 4 Feed	Scenario 5 Feed	Scenario 6 Feed
Ag	mg/L							0.415	
Al	mg/L	10	9.37	8.135	15.4	0.507	52.9	225.5	4.61
B	mg/L	12.18	1.535	0.7685	6.445	4.22	92.45	5.425	1.525
Ba	mg/L	0.479			0.5475			5.315	
Ca	mg/L	15.9	16.4	16.2	9.685	23.85	106	162.5	8.46
Cr	mg/L							0.4145	
Cu	mg/L	0.2665	0.442	0.373	0.498		0.3025	8.865	0.1695
Fe	mg/L	1070	841	870.5	106.5	1280	652.5	1870	393.5
Gd	mg/L							4.545	
K	mg/L	5.585	2.58	2.555	2.83	2.19	36.4	13.2	2.405
Li	mg/L							9.03	
Mg	mg/L	31.15	31.8	31.5	10.1	55.5	52.15	218	15.35
Mn	mg/L	62.15	63.35	64	19.5	121.5	45.45	372.5	32.35
Mo	mg/L	0.1515	1.59	0.858	0.7535	1.32	4.015	0.6	1.525
Na	mg/L	148	127.5	137.5	61.2	2065	9270	1215	60.7
Ni	mg/L	18.5	14.65	13.55	3.57	31.05	13.35	66.5	5.675
P	mg/L				0.3615			2.83	
Pb	mg/L							4.88	
S	mg/L	9.925	10.2	10.01	49.95	19.55	8.83	30.05	5.205
Sb	mg/L	1.695	1.795						
Si	mg/L	30.25	77.3	78.65	58.05	5.415	176	17.35	41.6
Ti	mg/L							0.612	
Zn	mg/L	0.134			0.4705			15.4	
Zr	mg/L						0.6685	0.301	
NO3	mg/L	3880	3657.5	3862	5465	1605	23935	7675	1886.5
PO4	mg/L								
SO4	mg/L	487	360.5	379.5	165.5	1300	196	815	212.5
HCO2	mg/L	380.5			654.5			4195	
Hg	mg/kg	159	151	201	146	159	119	107	76.1
Slurry Density	g/ml	1.0012	1.00127	1.001335	1.00095	1.000795	1.023775	1.007955	0.99515
Supernate Density	g/ml	1.00026	1.00025	1.00026	1.000855	0.99949	1.0215	1.00506	0.99905
Total Solids	wt%	0.27	0.14	0.34	0.16	0.36	3.85	1.31	0.04
Soluble Solids	wt%	0.1	0.11	0.09	0.04	0.06	3.44	0.78	0.06
Insoluble Solids	wt%	0.17	0.03	0.25	0.12	0.29	0.41	0.53	0
pH		1.29	1.34	1.31	1.02	2.1	1.6	1.91	1.64
Titration	Molar	0.048	0.047	0.049	0.078	0.010	0.028	0.071	0.023
Supernate Viscosity	cP	0.884	0.894	0.888	0.885	0.871	0.904	0.893	0.895

Note: Blanks indicate less than detectable amounts in sample. Detection limits shown in Table F-1.

Table C-2. Sample Results from Condensates

Sample ID	Units	WAC Limits	REBT 0010	REBT 0028	REBT 0031	REBT 0013	REBT 0016	REBT 0019	REBT 0022	REBT 0025
Description			Scenario 1-1	Scenario 1-2	Scenario 1-3	Scenario 2	Scenario 3	Scenario 4	Scenario 5	Scenario 6
Al	mg/L	1080	0.0105	0.235	0.205			0.231		0.2525
B	mg/L	6.92	0.9475	0.762	0.664	0.4205	0.254	16.45	0.2415	0.789
Ca	mg/L		0.1445			0.027			0.058	
Cr	mg/L	8.40								2.64
K	mg/L		0.2515	0.2325	0.2315	0.2235	0.1775	0.235	0.1765	0.539
Mn	mg/L	6.92							0.0535	
Mo	mg/L		0.061	0.067	0.058	0.0505	0.057	0.091	0.048	0.067
Na	mg/L	1240		0.3945	0.62			0.5865		0.3515
S	mg/L					0.2115				
Si	mg/L	90.2	408	66.5	66.9	1120	0.2535	28.05	0.5285	9.41
	mg/L									
NO3	mg/L	1180	428.5	284	420	702.5		839.5	1080	172
SO4	mg/L	133		397.5	434			162.5	103	176.5
HCO2	mg/L		361			818.5			3015	
Hg	mg/kg	40.6	152	12	77.1	17.1	0	36.3	20	22.6
Supernat Density	g/ml		0.99812	0.99806	0.99812	0.99828	0.99789	0.99828	0.99870	0.99800
Total Solids	wt %		0.02	0	0	0	0	0	0	0
pH			2.2	2.42	2.27	1.92	3.01	1.95	3.01	2.46
Titration	Molar		nm	nm	nm	nm	nm	0.01	nm	nm
Supernat Viscosity	cP		nm	nm	nm	nm	nm	0.890	nm	0.897

Note: Blanks indicate less than detectable amounts in sample. Detection limits shown in Table F-1. “nm” indicates not measured.

Table C-3. Sample Results from Concentrates

Sample ID	Units	REBT 0011	REBT 0029	REBT 0032	REBT 0014	REBT 0017	REBT 0023	REBT 0026
Description		Scenario 1-1	Scenario 1-2	Scenario 1-3	Scenario 2	Scenario 3	Scenario 5	Scenario 6
Ag	mg/L	0.625	0.9185	0.769	1.16		44.8	0.5555
Al	mg/L	555	330	496.5	655.5	47.7	5275	367.5
B	mg/L	18.65	3.3	2.585	8.49	23.75	105	2.715
Ba	mg/L	8.635	8.84	9.74	15.55		114	7.34
Ca	mg/L	463.5	467	552.5	288	622.5	4535	433.5
Ce	mg/L	0.139				0.4595	4.1	
Cr	mg/L	1.455	3.47	2.965	1.88	0.7765	7.545	
Cu	mg/L	11.6	12.5	13.5	19.2	0.1775	279.5	9.915
Fe	mg/L	26300	24150	32350	3320	41850	52250	24800
Gd	mg/L	8.055	8.1	9.825	7.975	5.995	147.5	7.56
K	mg/L	22	21.55	24.7	26.65	4.375	515.5	18.35
La	mg/L					0.3085	1.155	
Li	mg/L	6.62	4.645	5.1	6.32		319.5	3.505
Mg	mg/L	830.5	834.5	958	316	1335	6600	831
Mn	mg/L	1865	1870	2195	581	2750	12450	1885
Mo	mg/L	0.705	1.0145	0.7155	0.732	0.6245	0.8205	1.07
Na	mg/L	3515	3770	4385	2025	4970	35800	3455
Ni	mg/L	583	195	529	126	1085	1565	282.5
P	mg/L	6.03	5.205	5.33	10.15		81.85	3.995
Pb	mg/L	6.715	6.765	7.625	11.2	2.27	156	5.89
S	mg/L	300.5	295	361	61.1	538	918.5	288.5
Sb	mg/L	5.725						
Si	mg/L	263	294.5	344	251.5	16.1	75.8	276
Sn	mg/L			0.763			5.08	0.7035
Sr	mg/L	1.285	1.185	1.225	1.115	1.08	8.895	1.13
Ti	mg/L	1.48	1.49	1.74	2.715		22.3	1.175
Zn	mg/L	26.3	24.6	29.6	38.25	8.425	486.5	21.7
Zr	mg/L	0.462	3.26	3.02	0.5455	0.6915	2.685	2.23
F	mg/L						231.5	
NO2	mg/L						182	
NO3	mg/L	109500	100500	119600	183000	40650	209000	95280
SO4	mg/L	1450	355	470.5	3425	1675	1295	221
HCO2	mg/L	392.5			852		3205	
Hg	mg/kg	2480	2980	3380	2780	3930	77	2460
Slurry Density	g/ml	1.09455	1.088335	1.1119	1.08455	1.096055	1.255955	1.09363
Supernate Density	g/ml	1.06686	1.06711	1.07693	1.08406	1.0387	1.1762	1.06477
Total Solids	wt %	8.73	7.18	9.13	3.37	11.14	28.71	7.71
Soluble Solids	wt %	4.43	4.2	4.72	3.3	4.53	21.04	4.09
Insoluble Solids	wt %	4.3	2.97	4.41	0.07	6.61	7.67	3.62
pH		2.68	2.65	2.58	2.33	0.71	2.6	2.76
Titration	molar	1.12	1.16	1.36	2.05	0.20	0.21	1.16
Supernate Viscosity	cP	1.021	1.029	1.035	1.045	0.939	1.387	1.022

Note: Blanks indicate less than detectable amounts in sample. Detection limits shown in Table F-1.

Table C-4. Concentration Factors for Each Scenario

Element	Scenario 1-1	Scenario 1-2	Scenario 1-3	Scenario 2	Scenario 3	Scenario 5	Scenario 6
Al	55.5	35.2	61.0	42.6	94.1	23.4	79.7
B	1.5	2.1	3.4	1.3	5.6	19.4	1.8
Ba	18.0			28.4			
Ca	29.2	28.5	34.1	29.7	26.1	27.9	51.2
Cr						18.2	
Cu	43.5	28.3	36.2	38.6		31.5	58.5
Fe	24.6	28.7	37.2	31.2	32.7	27.9	63.0
Gd						32.5	
K	3.9	8.4	9.7	9.4	2.0	39.1	7.6
Li						35.4	
Mg	26.7	26.2	30.4	31.3	24.1	30.3	54.1
Mn	30.0	29.5	34.3	29.8	22.6	33.4	58.3
Mo	4.7	0.6	0.8	1.0	0.5	1.4	0.7
Na	23.8	29.6	31.9	33.1	2.4	29.5	56.9
Ni	31.5	13.3	39.0	35.3	34.9	23.5	49.8
P				28.1		28.9	
Pb						32.0	
S	30.3	28.9	36.1	1.2	27.5	30.6	55.4
Si	8.7	3.8	4.4	4.3	3.0	4.4	6.6
Ti						36.4	
Zn	196.3			81.3		31.6	
NO3	28.2	27.5	31.0	33.5	25.3	27.2	50.5
SO4	3.0	1.0	1.2	20.7	1.3	1.6	1.0
HCO2	1.0			1.3		0.8	
Hg	15.6	19.7	16.8	19.0	24.7	0.7	32.3
K	2.0					3.8	
Total Solids	32.3	51.3	26.9	21.1	30.9	21.9	192.8
Soluble Solids	44.3	38.2	52.4	82.5	75.5	27.0	68.2
Insoluble Solids	25.3	99.0	17.6	0.6	22.8	14.5	N/A

APPENDIX D. XRD RESULTS

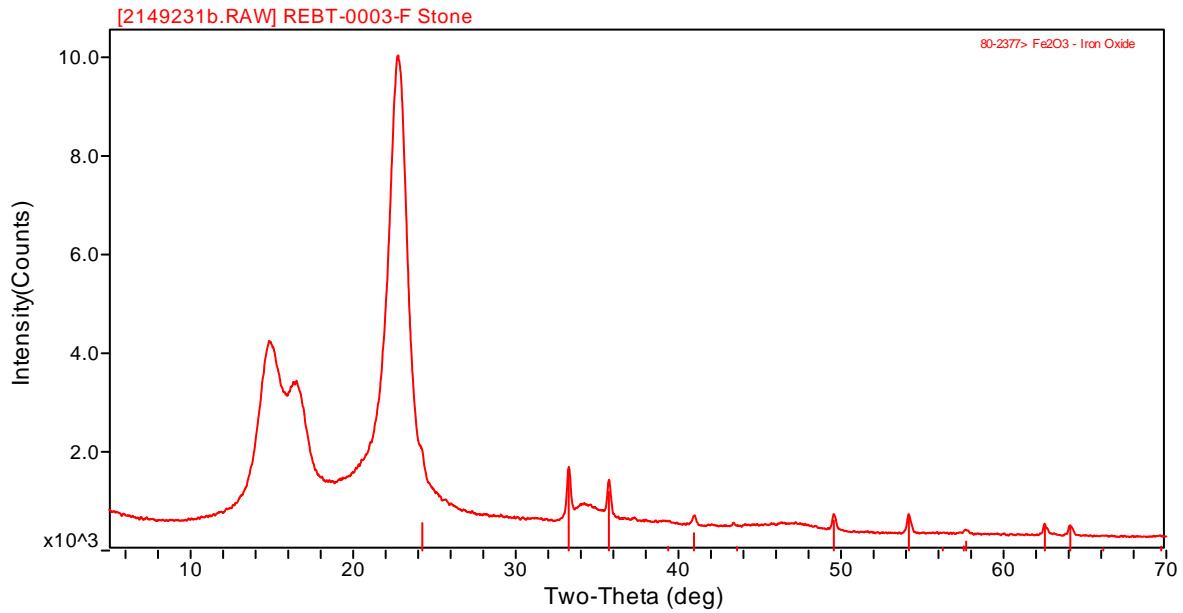


Figure D-1. OGCT Simulant XRD

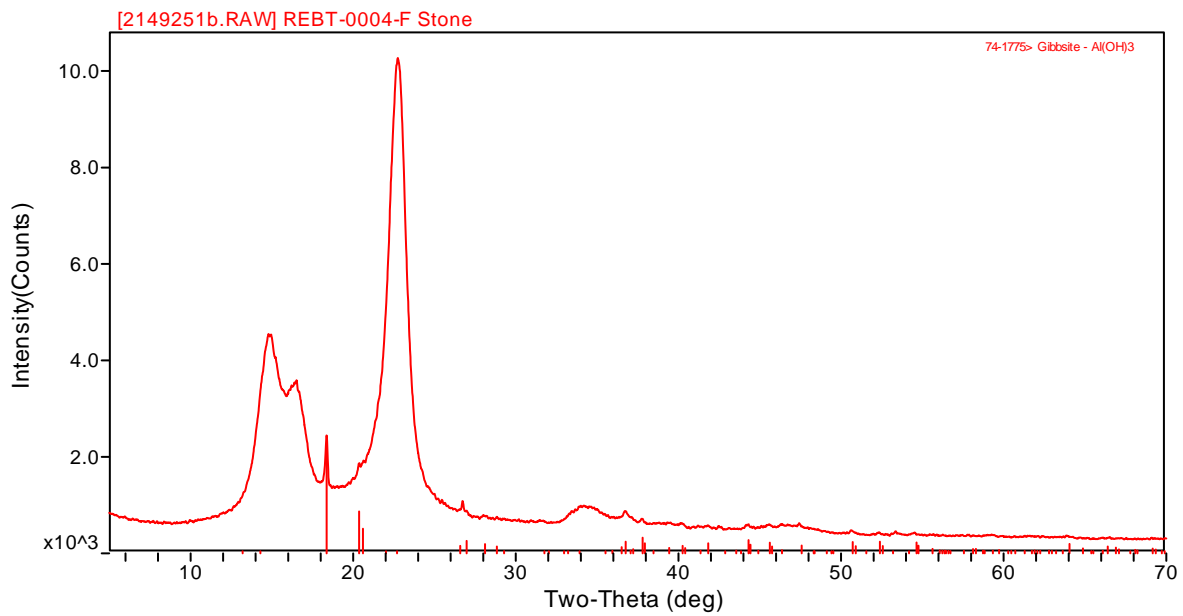


Figure D-2. Decon Solution Simulant XRD



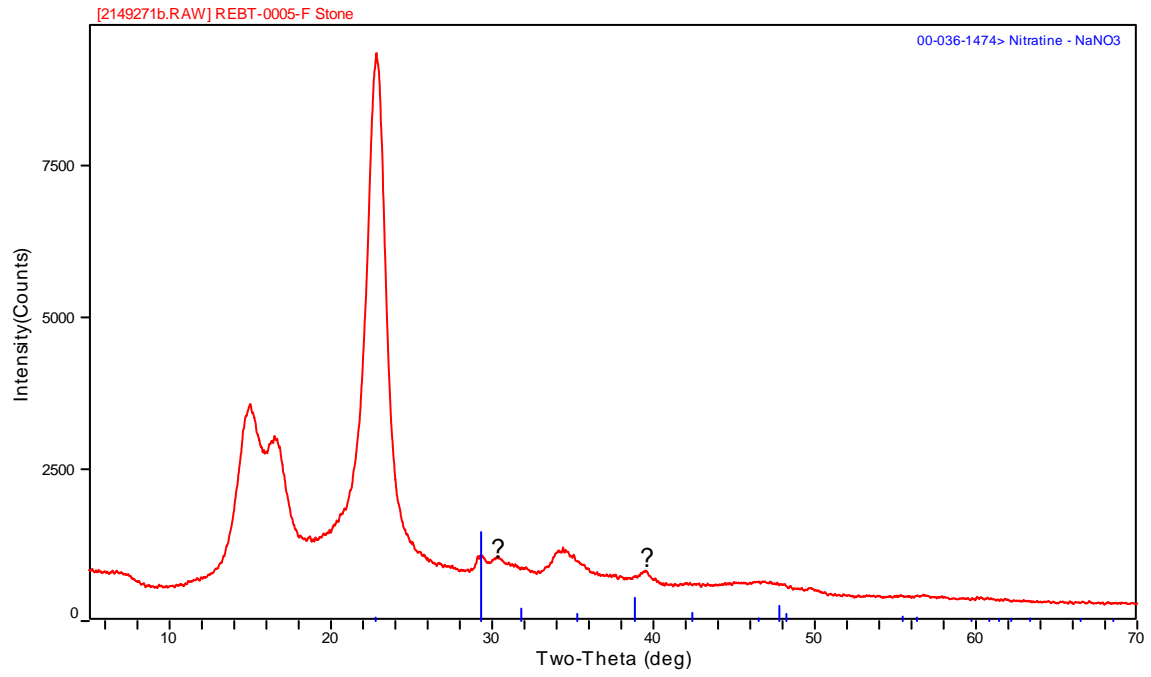


Figure D-3. HEME Dissolution Simulant XRD

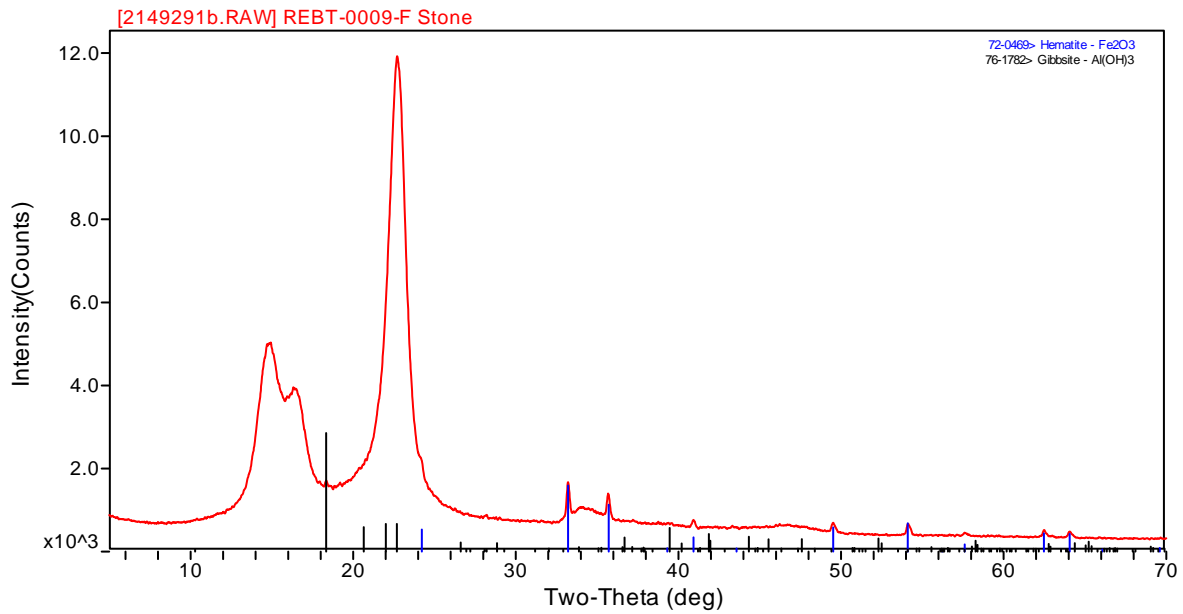


Figure D-4. Scenario 1-1 Feed XRD

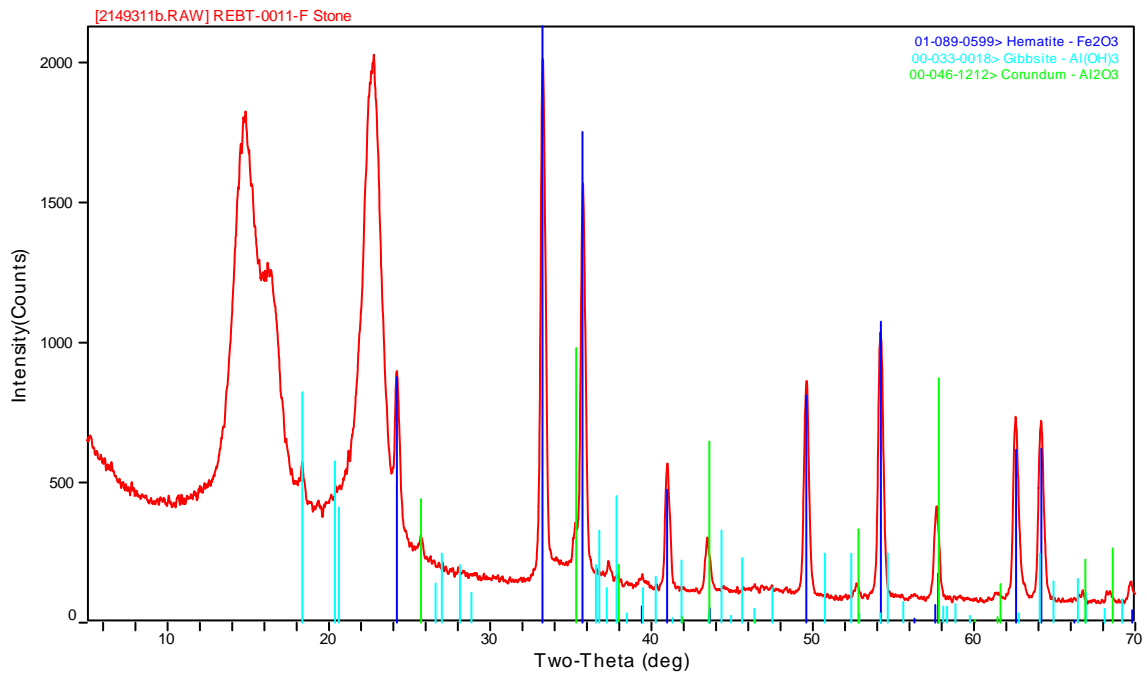


Figure D-5. Scenario 1-1 Concentrate XRD

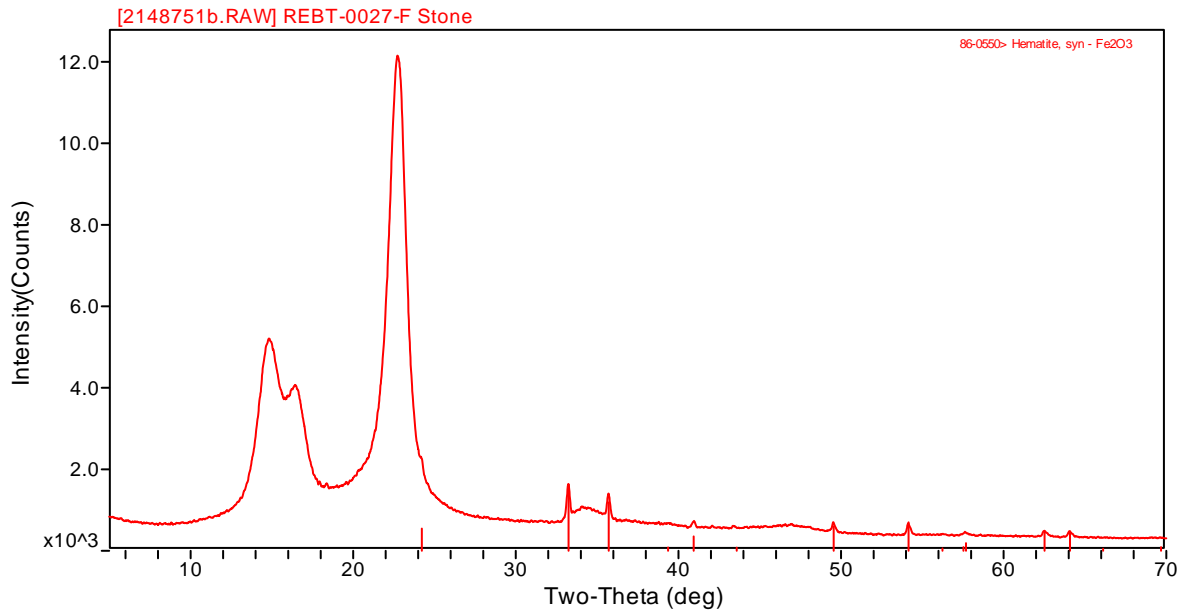


Figure D-6. Scenario 1-2 Feed XRD

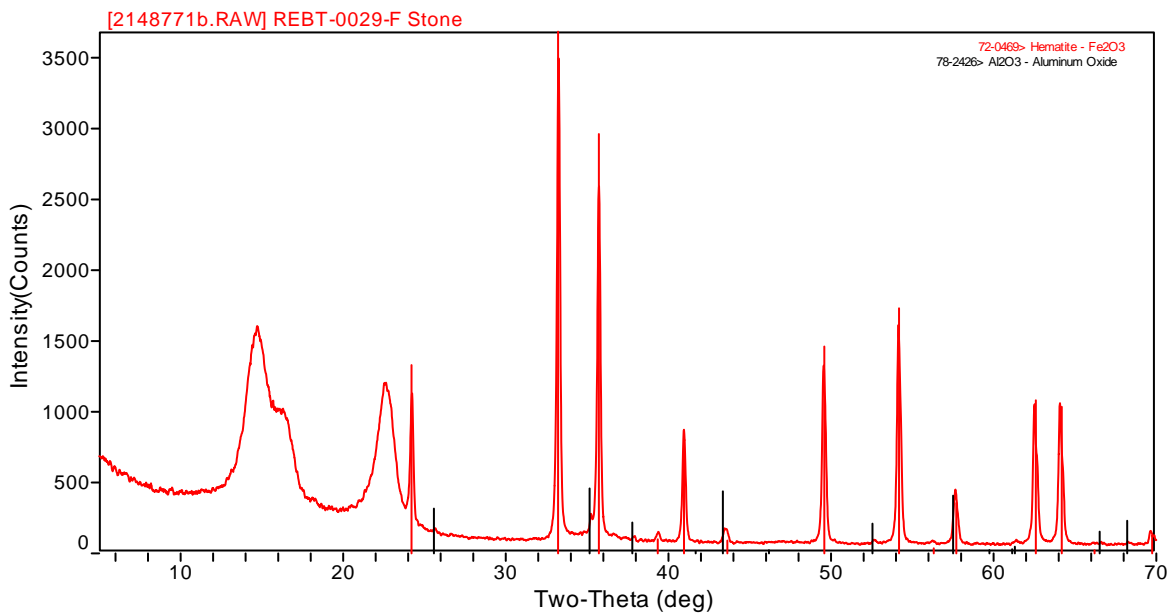


Figure D-7. Scenario 1-2 Concentrate XRD

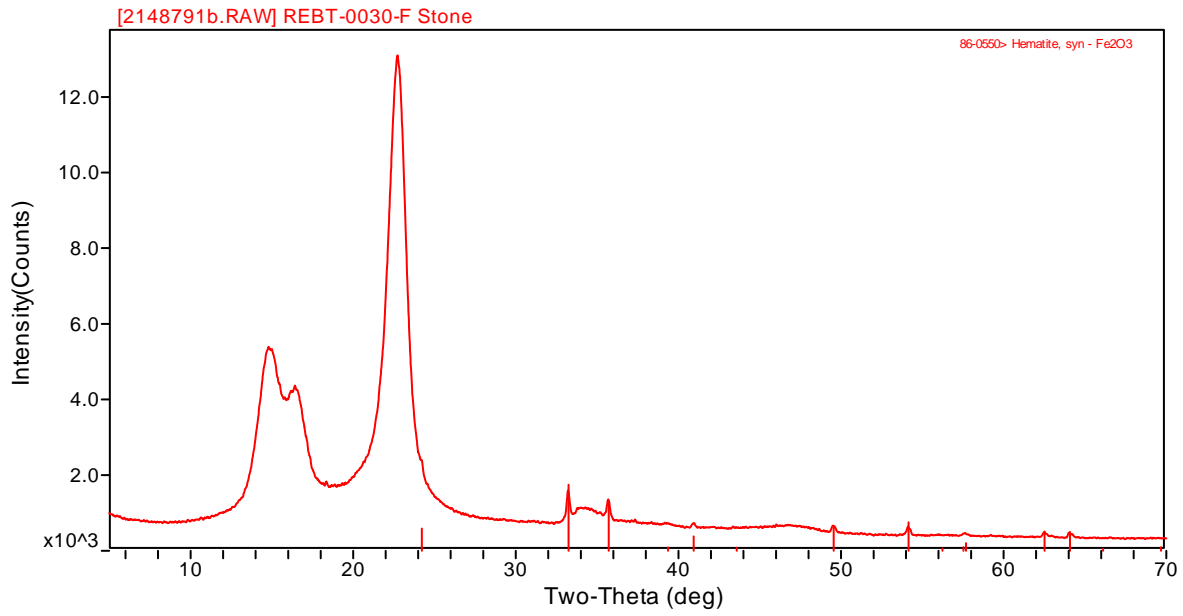


Figure D-8. Scenario 1-3 Feed XRD

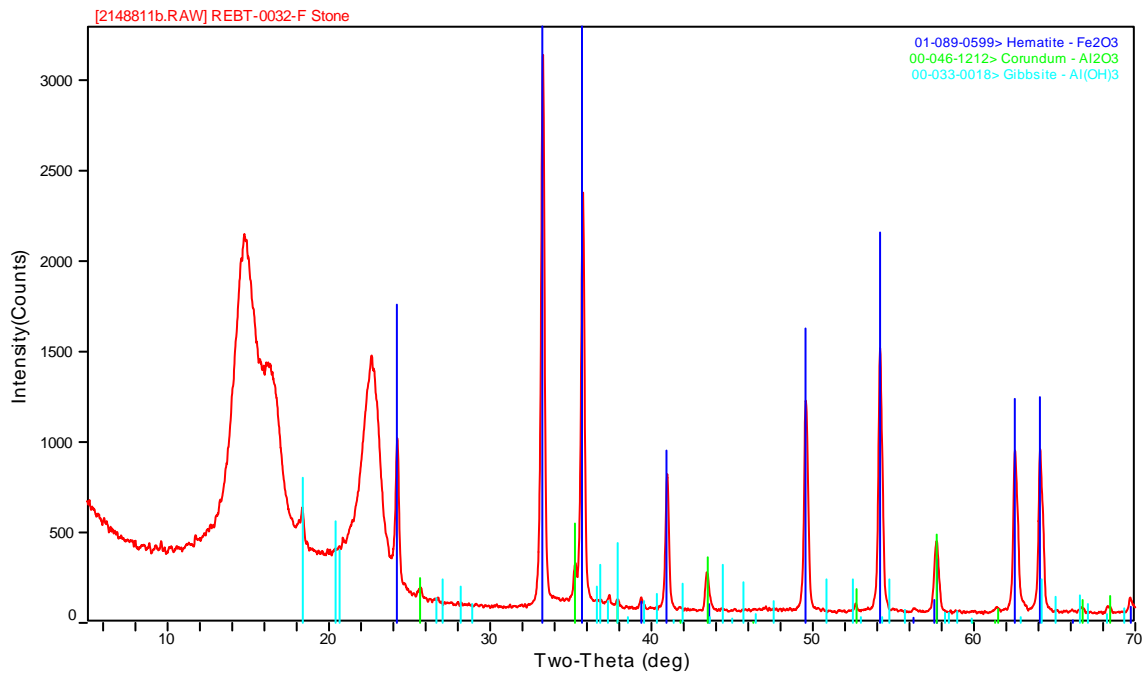


Figure D-9. Scenario 1-3 Concentrate XRD

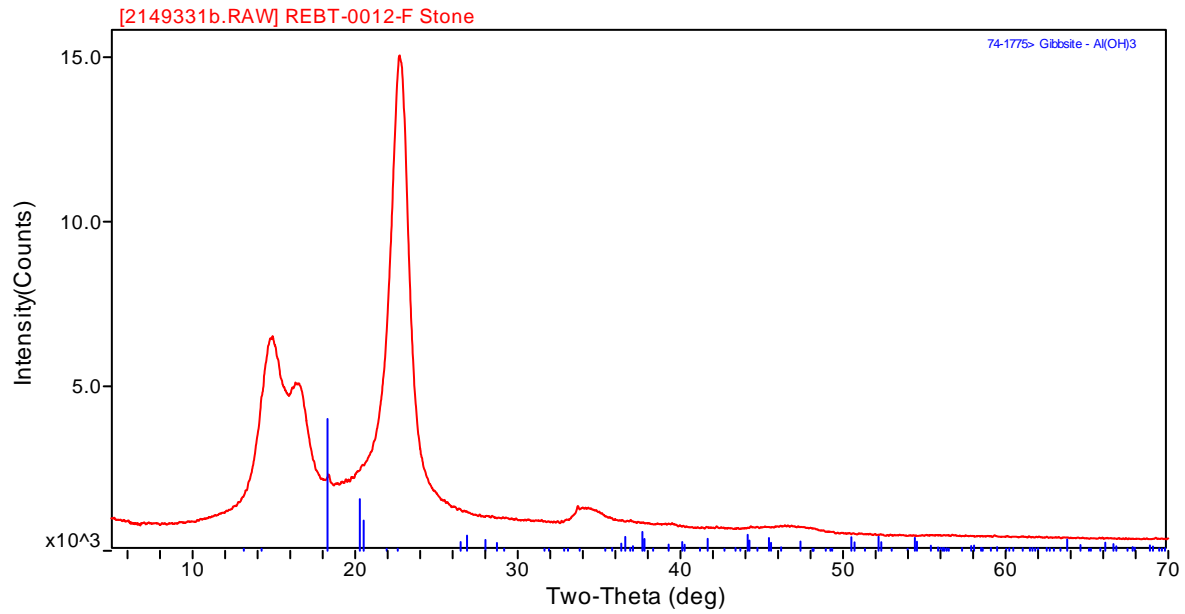


Figure D-10. Scenario 2 Feed XRD

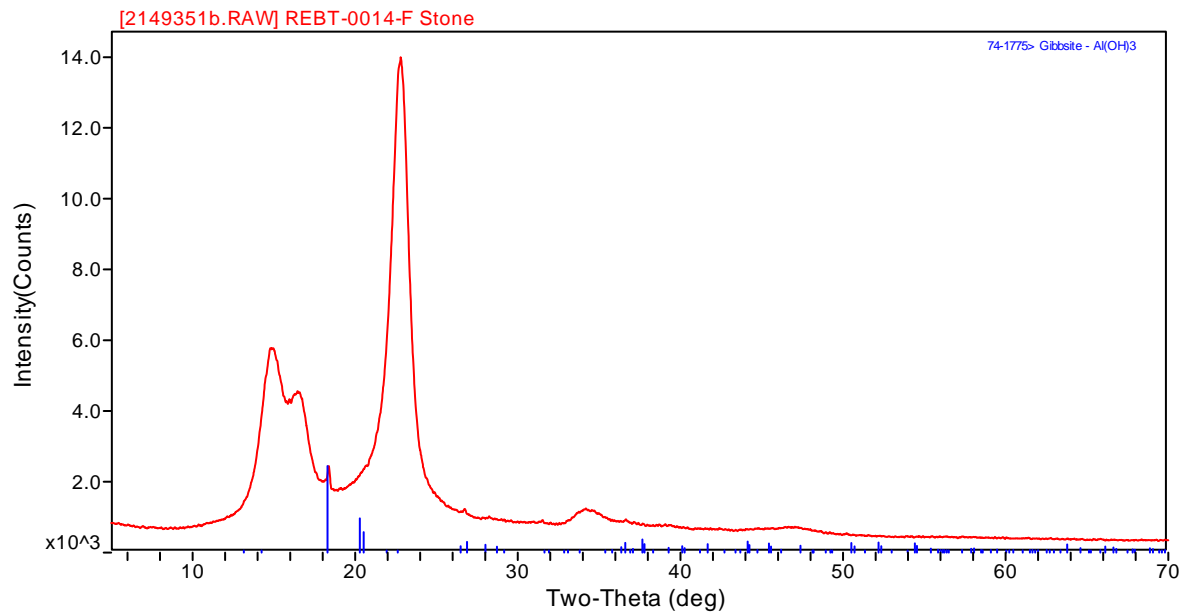


Figure D-11. Scenario 2 Concentrate XRD

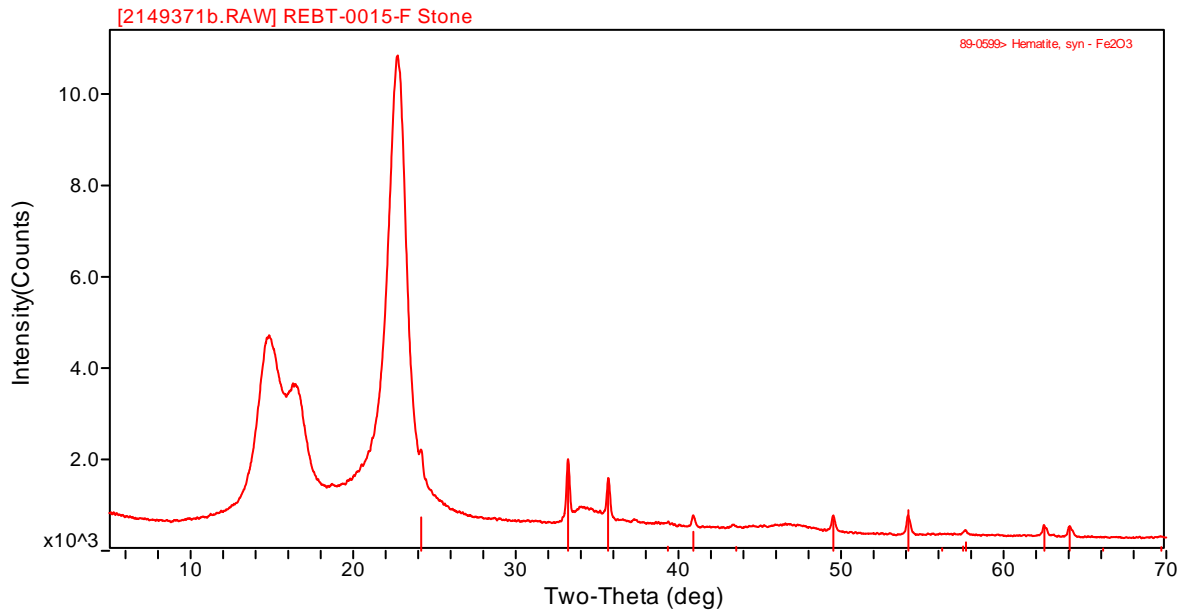


Figure D-12. Scenario 3 Feed XRD

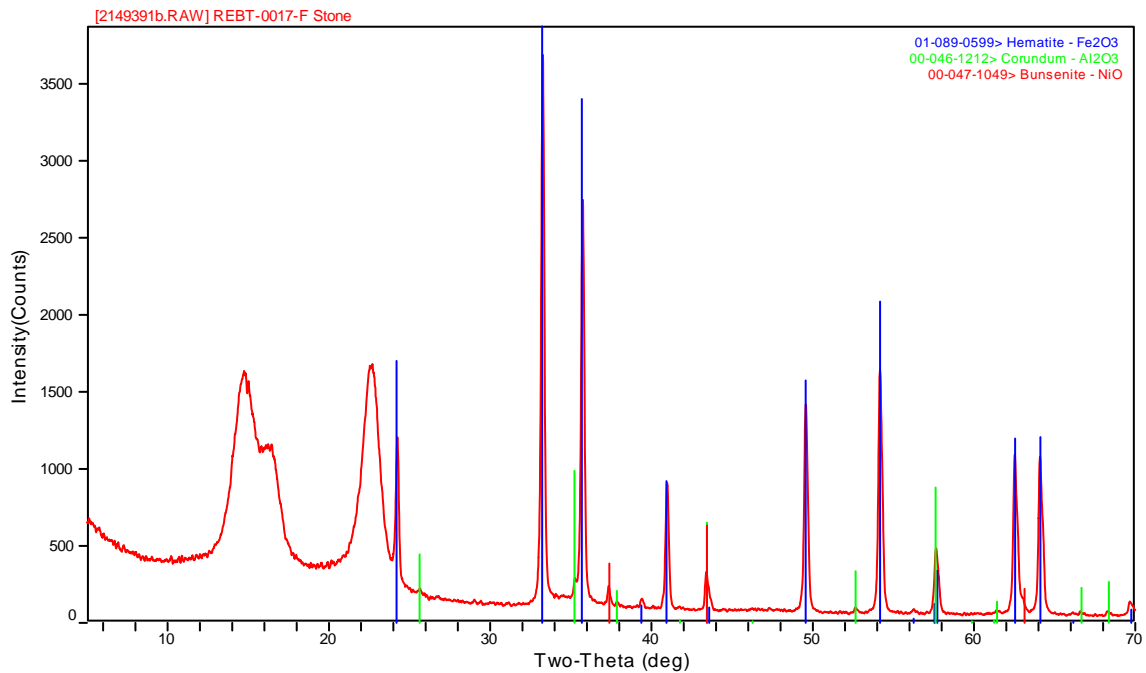


Figure D-13. Scenario 3 Concentrate XRD

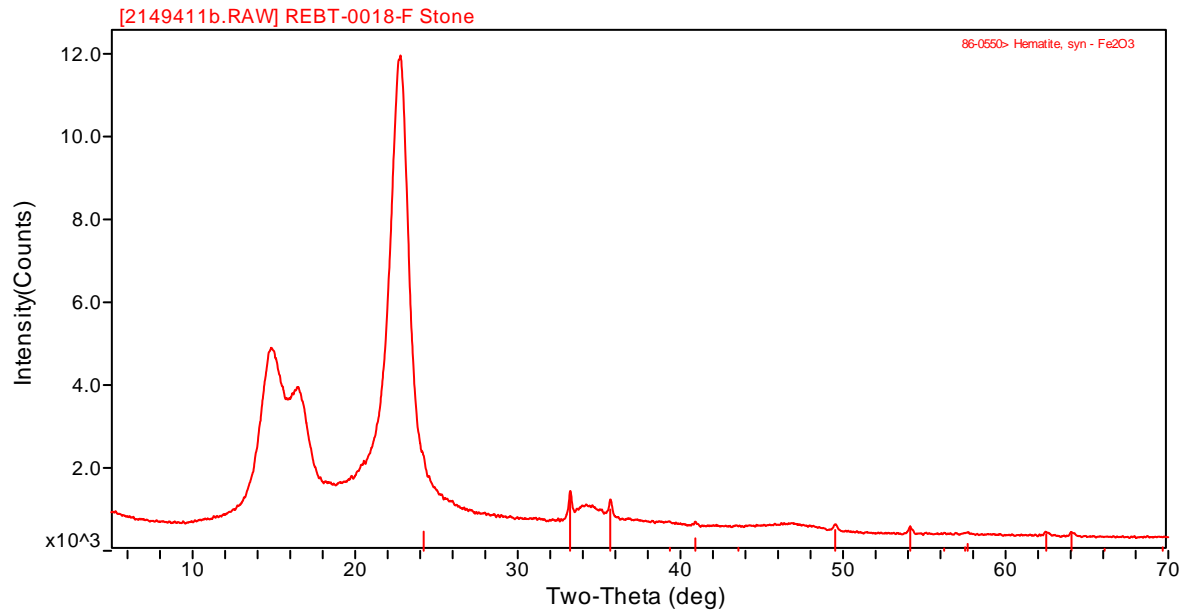


Figure D-14. Scenario 4 Feed XRD

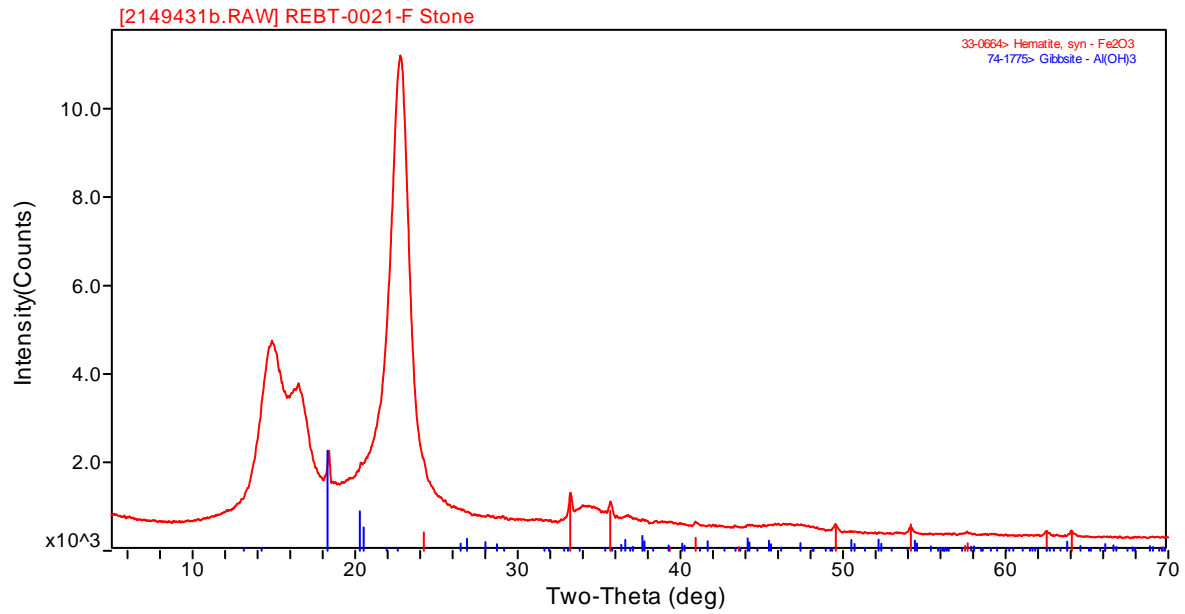


Figure D-15. Scenario 5 Feed XRD

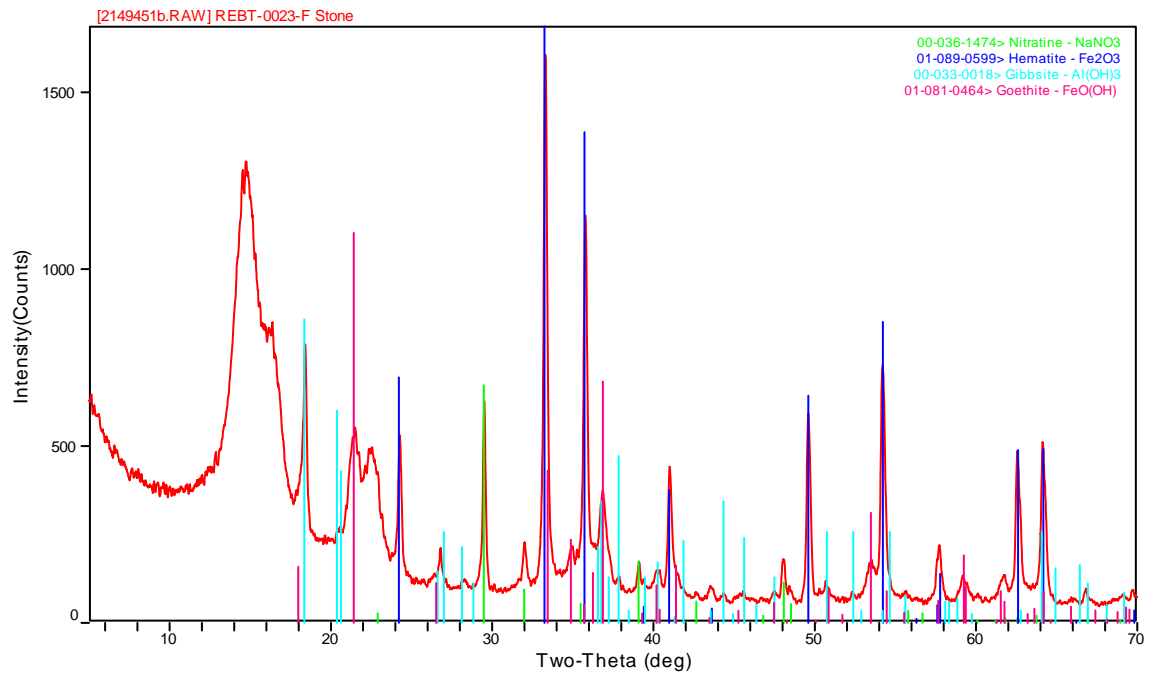


Figure D-16. Scenario 5 Concentrate XRD



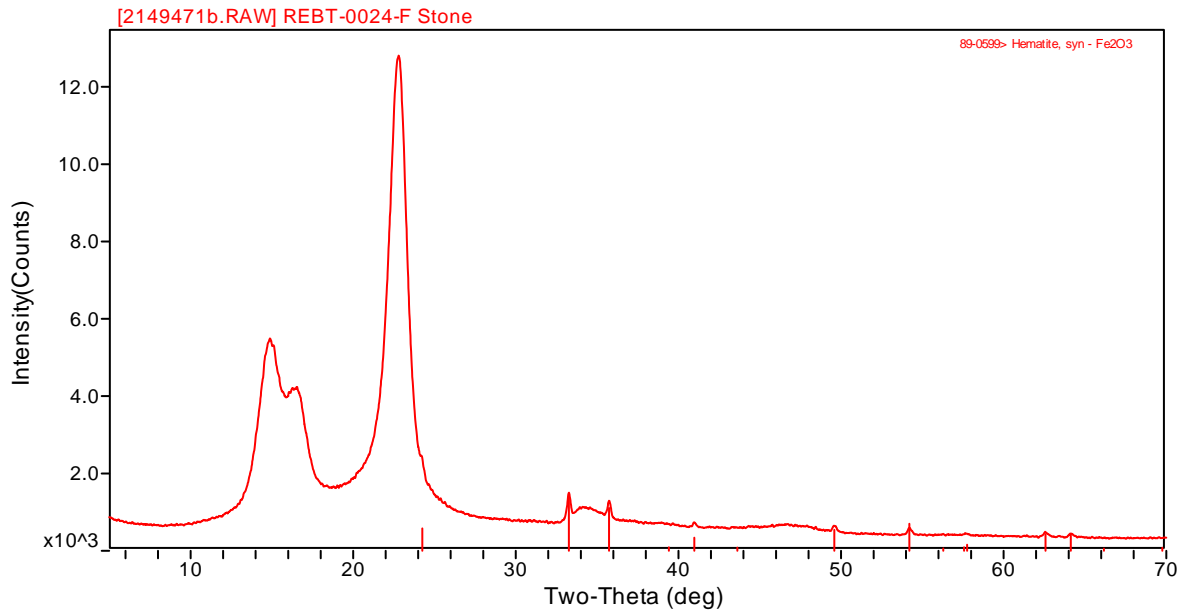


Figure D-17. Scenario 6 Feed XRD

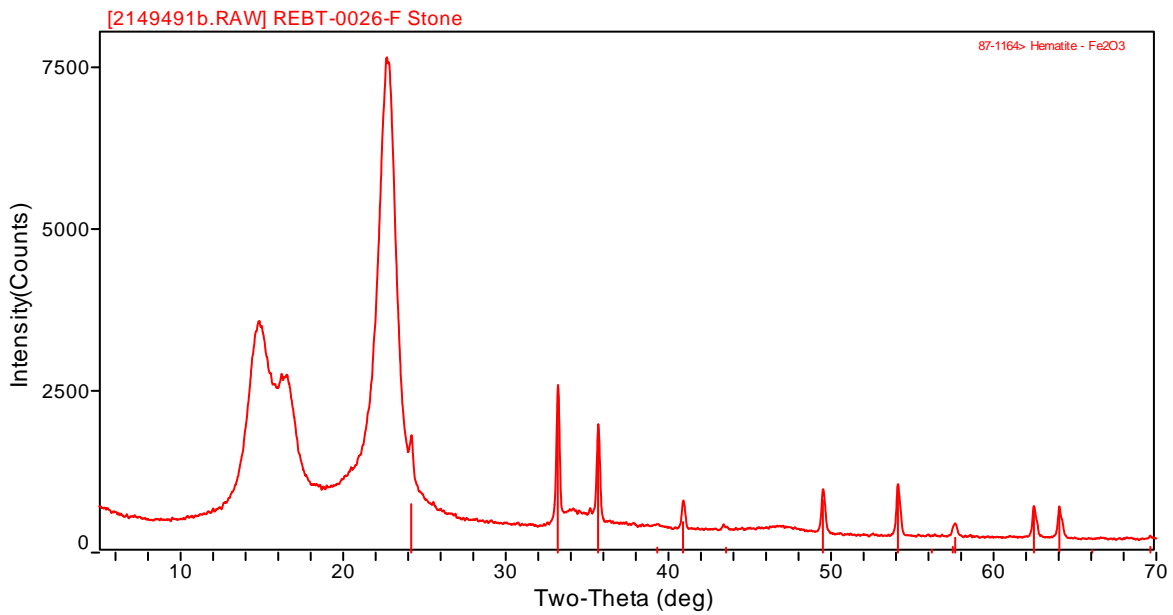


Figure D-18. Scenario 6 Concentrate XRD

APPENDIX E. CONDENSATE PH PROFILES

Table E-1. Condensate pH Measurements

Bottle #	Scenario 1-1	Scenario 1-2	Scenario 1-3	Scenario 2	Scenario 3	Scenario 4	Scenario 5	Scenario 6
1	3.07	3.04	2.94	2.84	2.64	3.05	2.27	3.18
2	2.86	2.88	2.85	2.71	2.97	2.74	2.12	3.15
3	2.78	2.84	2.77	2.57	2.96	2.46	2.00	3.04
4	2.66	2.80	2.69	2.43	2.95	2.29	2.02	2.70
5	2.09	2.74	2.57	2.20	2.94	2.11	1.98	3.11
6	2.15	2.65	2.54	2.07	2.94	1.95	1.99	3.13
7	2.17	2.52	2.43	1.95	2.94	n/a	n/a	3.09
8	2.01	2.37	2.31	2.16	2.94	n/a	n/a	3.00
9	2.33	n/a	n/a	n/a	n/a	n/a	n/a	2.89
10	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.93
11	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.84
12	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.86
13	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.64
14	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.62
15	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.44
16	n/a	n/a	n/a	n/a	n/a	n/a	n/a	2.60

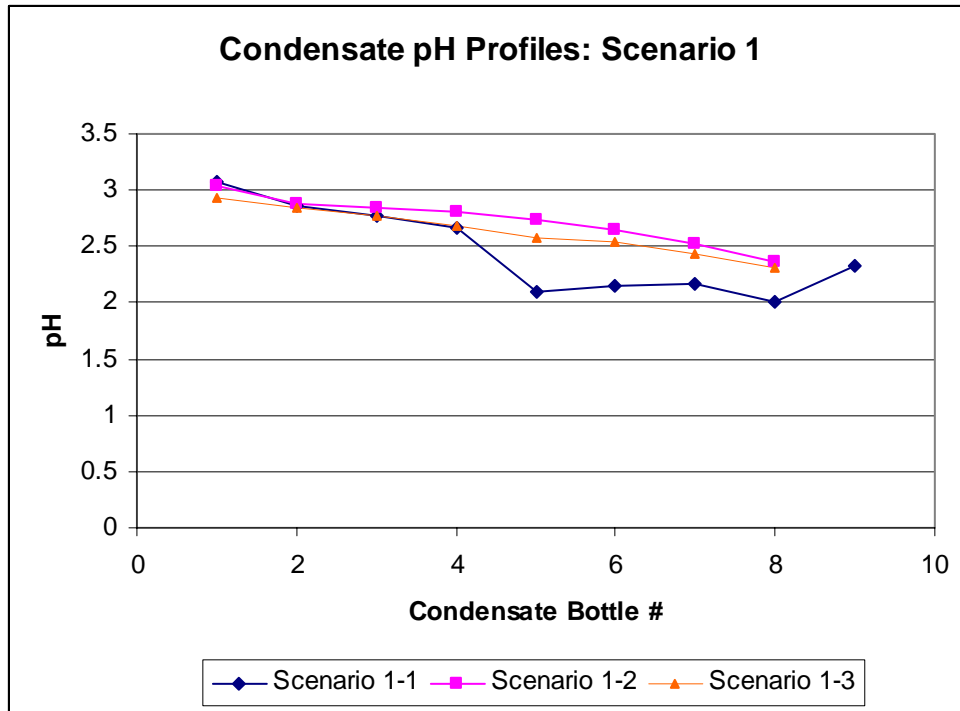


Figure E-1. pH profile for Scenario 1-1, 1-2, and 1-3

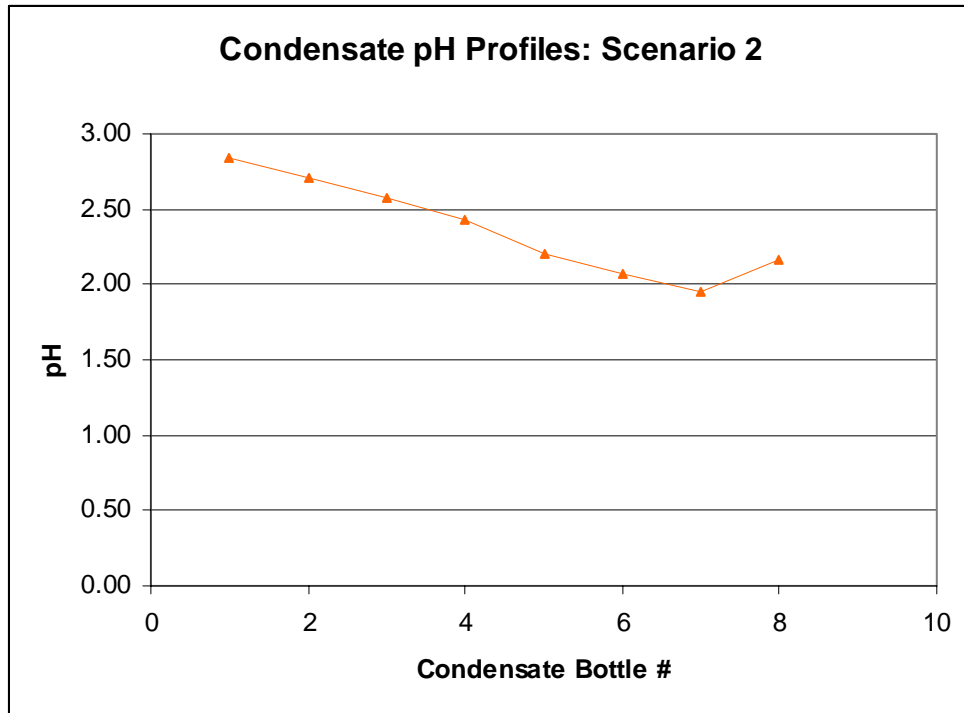


Figure E-2. pH profile for Scenario 2

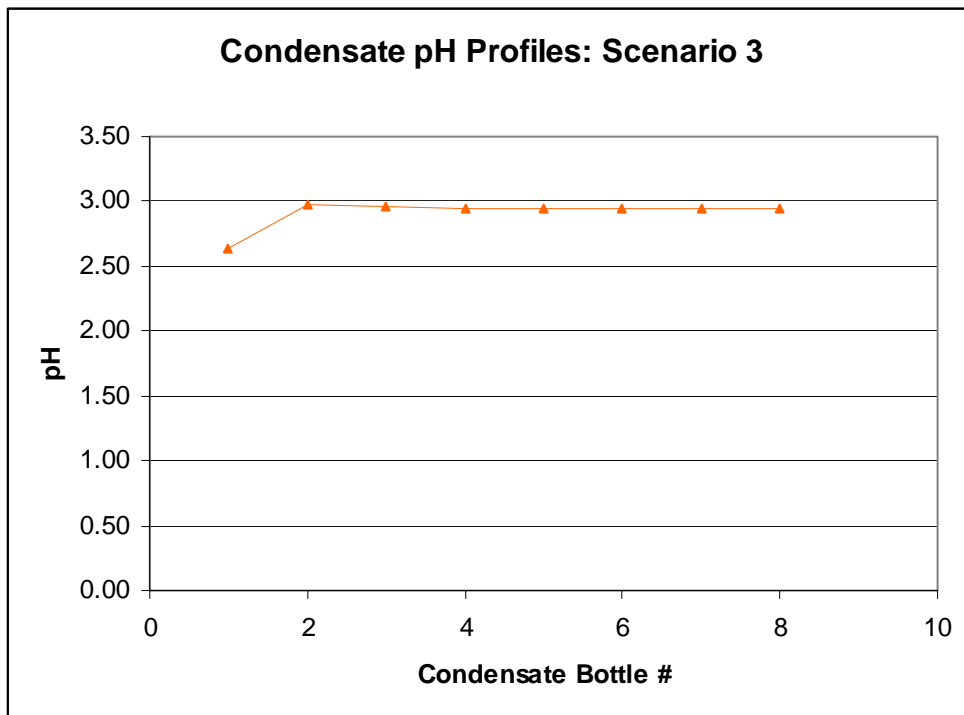


Figure E-3. pH profile for Scenario 3

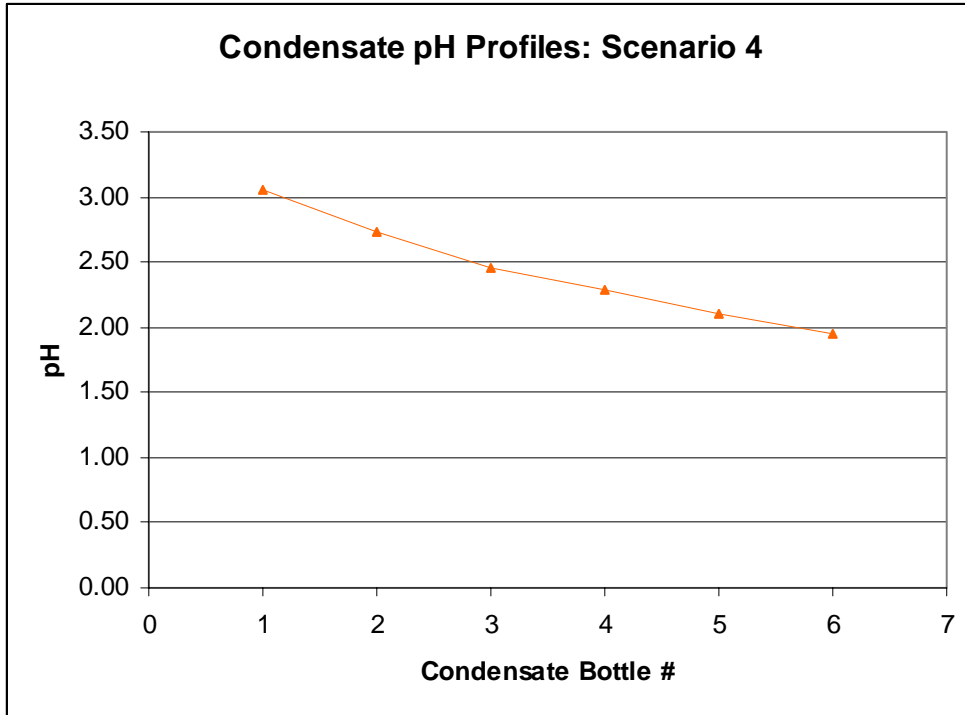


Figure E-4. pH profile for Scenario 4

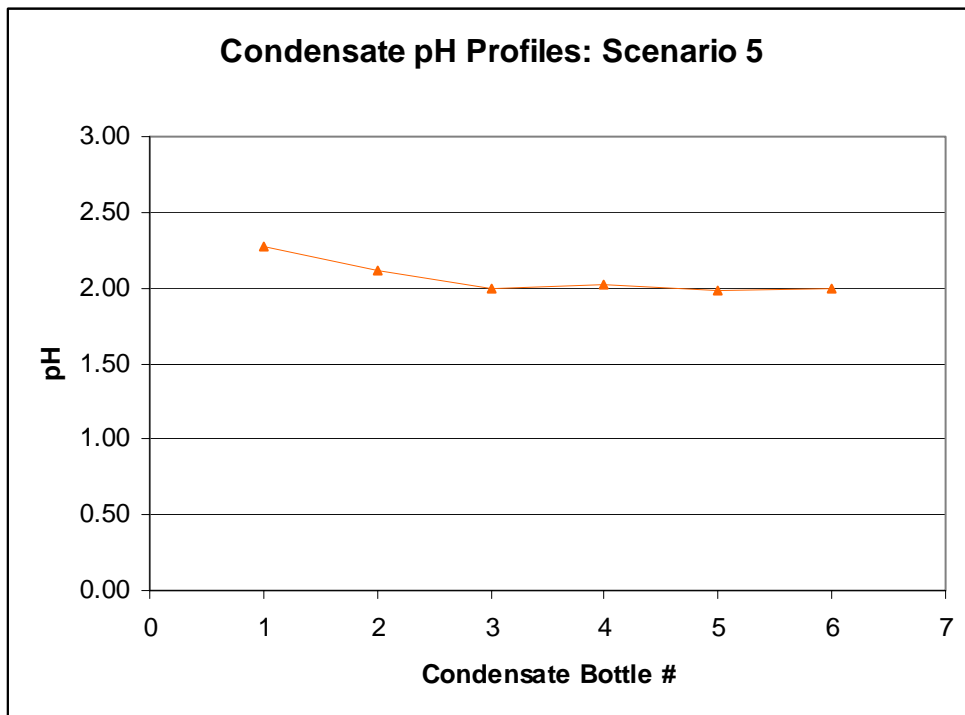


Figure E-5. pH profile for Scenario 5

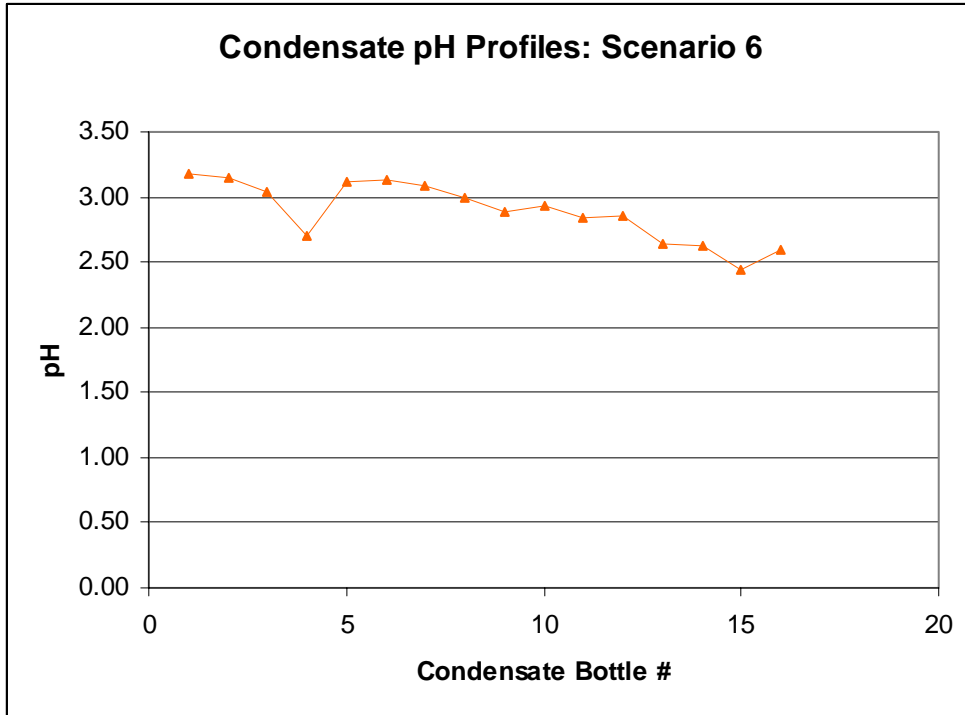


Figure E-6. pH profile for Scenario 6

APPENDIX F. DF TRENDING

Table F-1. DF Factors for Recycle Evaporation

	Sample Detection Limit	Scenario 1-1	Scenario 1-2	Scenario 1-3	Scenario 2	Scenario 3	Scenario 4	Scenario 5	Scenario 6	Low	Average	High
Ag	0.1	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>4.15</i>	<i>0</i>	<i>nm</i>	<i>4.15</i>	<i>nm</i>
Al	0.1	<i>100</i>	<i>40</i>	<i>40</i>	<i>154</i>	<i>5.07</i>	<i>229</i>	<i>2255</i>	<i>18</i>	<i>5</i>	<i>355</i>	<i>2255</i>
B		<i>12.85</i>	<i>2.01</i>	<i>1.16</i>	<i>15.33</i>	<i>16.61</i>	<i>5.62</i>	<i>22.46</i>	<i>1.93</i>	<i>1.2</i>	<i>10</i>	<i>22</i>
Ba	0.01	<i>47.9</i>	<i>0</i>	<i>0</i>	<i>54.8</i>	<i>0</i>	<i>0</i>	<i>532</i>	<i>0</i>	<i>48</i>	<i>211</i>	<i>532</i>
Ca	0.01	<i>110</i>	<i>1640</i>	<i>1620</i>	<i>359</i>	<i>2385</i>	<i>10600</i>	<i>2802</i>	<i>846</i>	<i>110</i>	<i>2545</i>	<i>10600</i>
Cr	0.12	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>3.45</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>3</i>
Cu	0.01	<i>26.7</i>	<i>44.2</i>	<i>37.3</i>	<i>49.8</i>	<i>0.0</i>	<i>30.3</i>	<i>886.5</i>	<i>17.0</i>	<i>17</i>	<i>156</i>	<i>887</i>
Fe	0.04	<i>26750</i>	<i>21025</i>	<i>21763</i>	<i>2663</i>	<i>32000</i>	<i>16313</i>	<i>46750</i>	<i>9838</i>	<i>2663</i>	<i>22138</i>	<i>46750</i>
Gd	0.03	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>151.5</i>	<i>0</i>	<i>nm</i>	<i>152</i>	<i>nm</i>
K		<i>22.2</i>	<i>11.1</i>	<i>11.0</i>	<i>12.7</i>	<i>12.3</i>	<i>154.9</i>	<i>74.8</i>	<i>4.5</i>	<i>4</i>	<i>38</i>	<i>155</i>
Li	1	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>9.0</i>	<i>0</i>	<i>nm</i>	<i>9</i>	<i>nm</i>
Mg	0.01	<i>3115</i>	<i>3180</i>	<i>3150</i>	<i>1010</i>	<i>5550</i>	<i>5215</i>	<i>21800</i>	<i>1535</i>	<i>1010</i>	<i>5569</i>	<i>21800</i>
Mn	0.01	<i>6215</i>	<i>6335</i>	<i>6400</i>	<i>1950</i>	<i>12150</i>	<i>4545</i>	<i>6963</i>	<i>3235</i>	<i>1950</i>	<i>5974</i>	<i>12150</i>
Mo		<i>2.5</i>	<i>23.7</i>	<i>14.8</i>	<i>14.9</i>	<i>23.2</i>	<i>44.1</i>	<i>12.5</i>	<i>22.8</i>	<i>2</i>	<i>20</i>	<i>44</i>
Na	1	<i>148</i>	<i>323</i>	<i>222</i>	<i>61.2</i>	<i>2065</i>	<i>15806</i>	<i>1215</i>	<i>173</i>	<i>61</i>	<i>2502</i>	<i>15806</i>
Ni	0.017	<i>1088</i>	<i>862</i>	<i>797</i>	<i>210</i>	<i>1826</i>	<i>785</i>	<i>3912</i>	<i>334</i>	<i>210</i>	<i>1227</i>	<i>3912</i>
P	0.26	<i>0</i>	<i>0</i>	<i>0</i>	<i>1.39</i>	<i>0</i>	<i>0</i>	<i>10.9</i>	<i>0</i>	<i>1</i>	<i>nm</i>	<i>11</i>
Pb	0.02	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>244</i>	<i>0</i>	<i>nm</i>	<i>244</i>	<i>nm</i>
S	0.1	<i>99</i>	<i>102</i>	<i>100</i>	<i>236</i>	<i>196</i>	<i>88</i>	<i>301</i>	<i>52</i>	<i>52</i>	<i>147</i>	<i>301</i>
Si		<i>0.074</i>	<i>1.162</i>	<i>1.176</i>	<i>0.052</i>	<i>21.361</i>	<i>6.275</i>	<i>32.829</i>	<i>4.421</i>	<i>0.05</i>	<i>8</i>	<i>33</i>
Ti	0.01	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>61</i>	<i>0</i>	<i>nm</i>	<i>61</i>	<i>nm</i>
Zn	0.01	<i>13.4</i>	<i>0</i>	<i>0</i>	<i>47</i>	<i>0</i>	<i>0</i>	<i>1540</i>	<i>0</i>	<i>13</i>	<i>533</i>	<i>1540</i>
Zr	0.01	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>0</i>	<i>67</i>	<i>30</i>	<i>0</i>	<i>30</i>	<i>nm</i>	<i>67</i>
NO3	100	<i>9.1</i>	<i>12.9</i>	<i>9.2</i>	<i>7.8</i>	<i>16.1</i>	<i>28.5</i>	<i>7.1</i>	<i>11.0</i>	<i>7</i>	<i>13</i>	<i>29</i>
SO4	100	<i>4.87</i>	<i>0.91</i>	<i>0.87</i>	<i>1.66</i>	<i>13</i>	<i>1.21</i>	<i>7.91</i>	<i>1.20</i>	<i>0.9</i>	<i>4</i>	<i>13</i>
HCO2	100	<i>1.05</i>	<i>0</i>	<i>0</i>	<i>0.80</i>	<i>0</i>	<i>0</i>	<i>1.39</i>	<i>0</i>	<i>0.80</i>	<i>1.08</i>	<i>1.39</i>
Hg	2.5	<i>1.0</i>	<i>12.6</i>	<i>2.6</i>	<i>8.5</i>	<i>63.6</i>	<i>3.3</i>	<i>5.4</i>	<i>3.4</i>	<i>1.0</i>	<i>13</i>	<i>64</i>

NOTE: Italics represent DF's calculated based on the detection limits in the condensate.

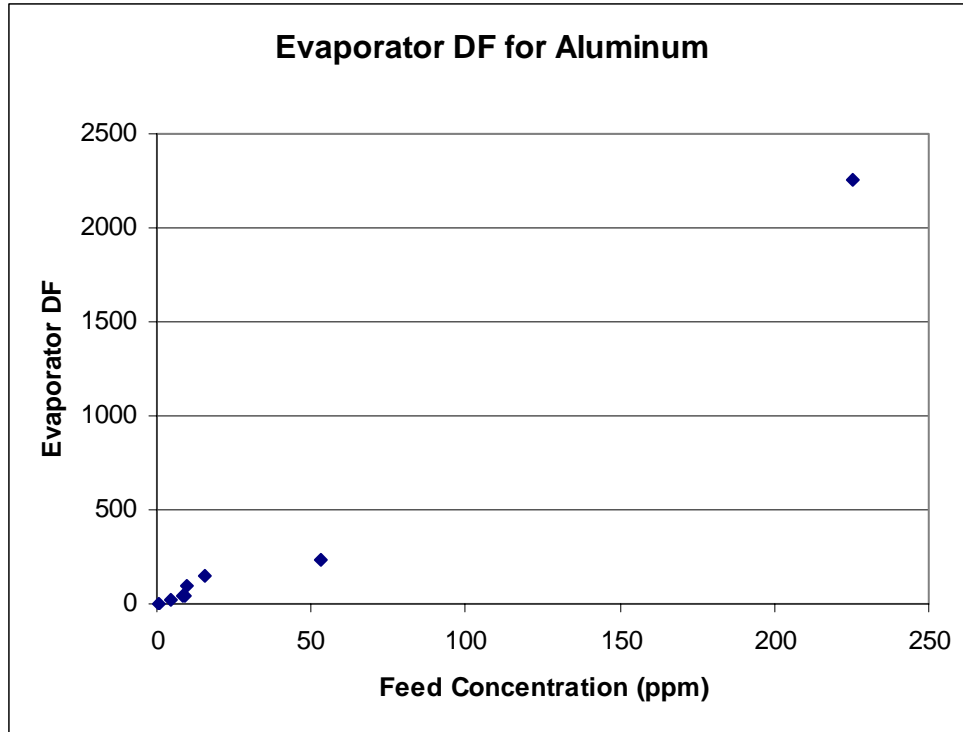


Figure F-1. DF Trend for Aluminum

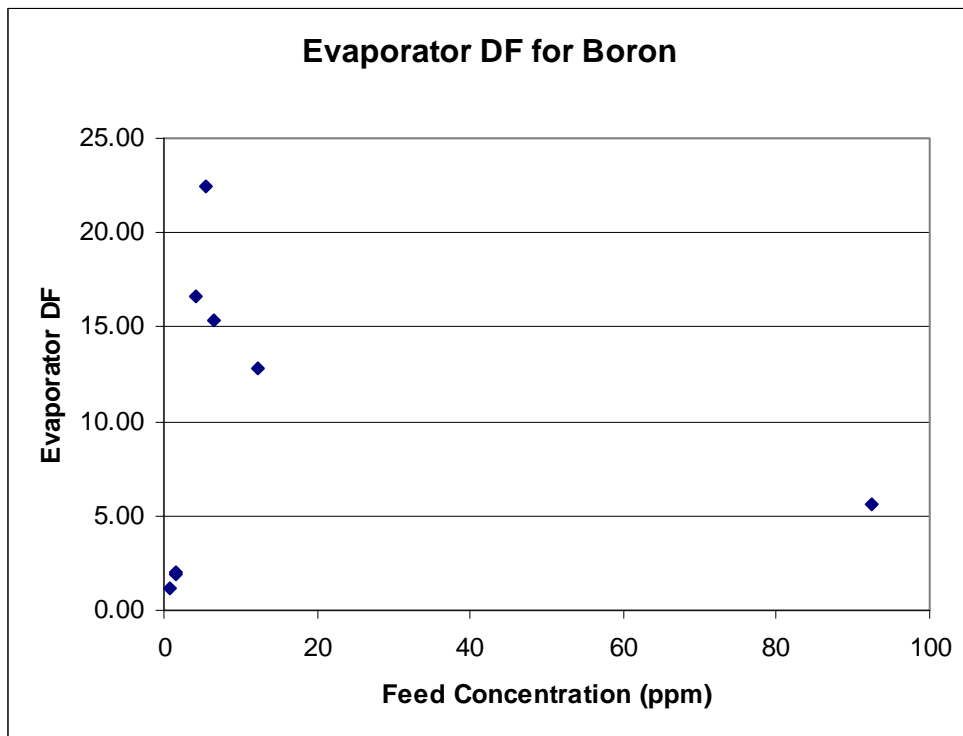


Figure F-2. DF Trend for Boron

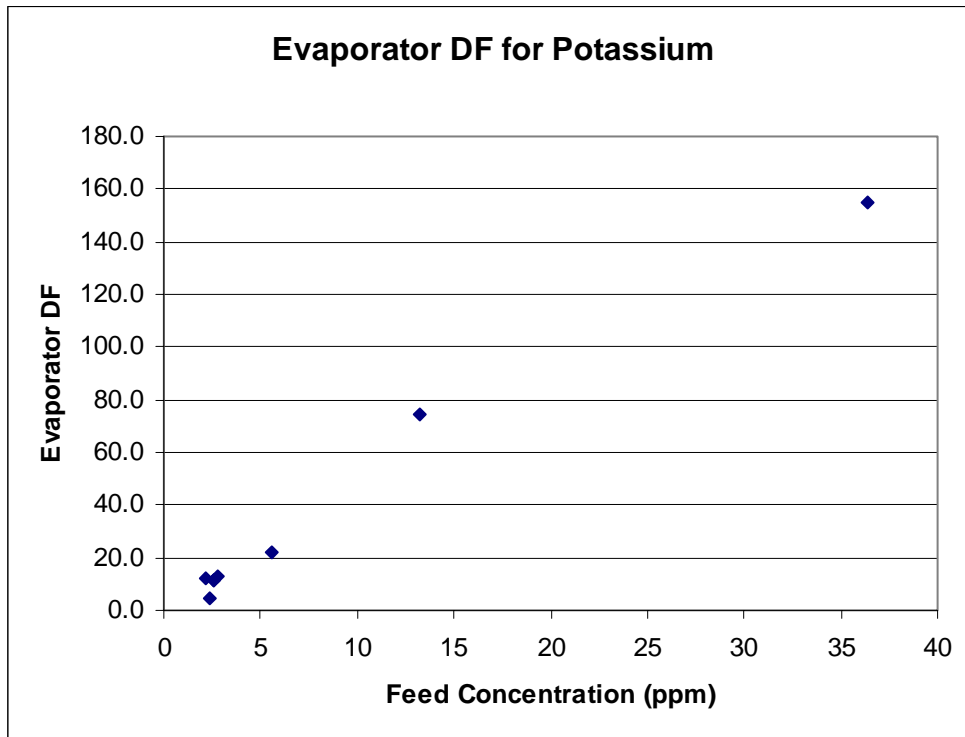


Figure F-3. DF Trend for Potassium

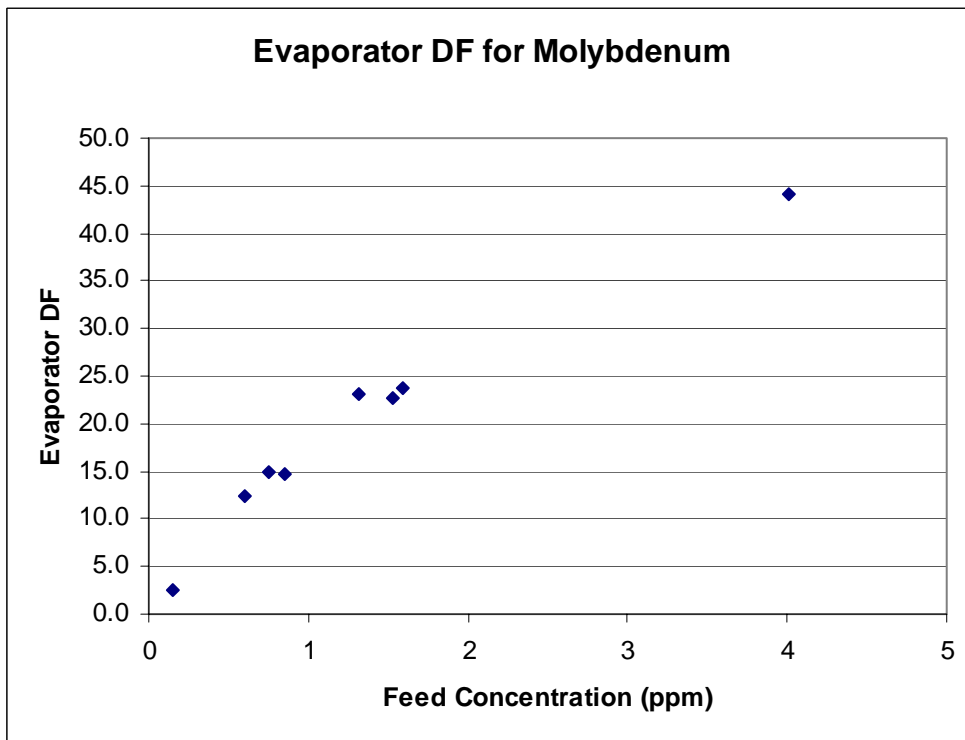


Figure F-4. DF Trend for Molybdenum



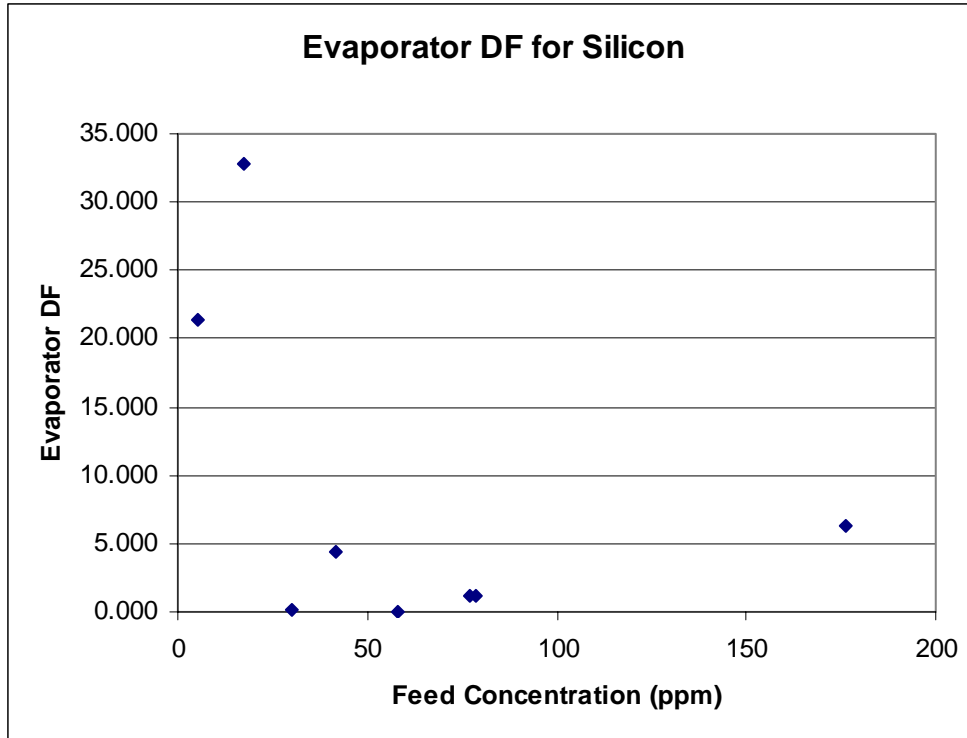


Figure F-5. DF Trend for Silicon

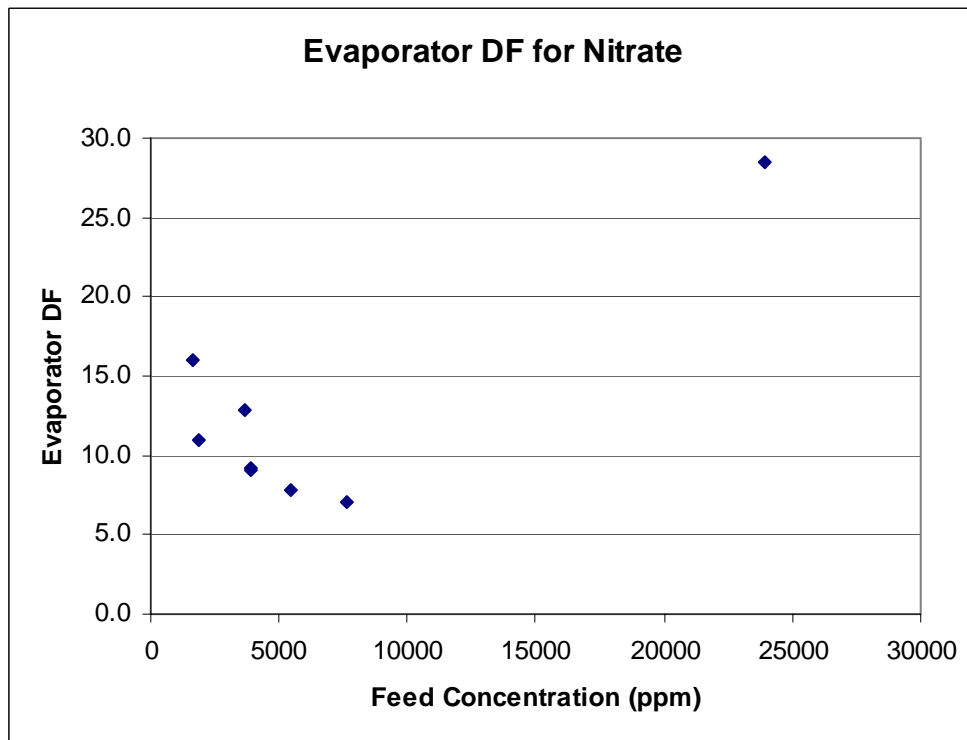


Figure F-6. DF Trend for Nitrate

This page intentionally left blank.

**Distribution:**

E. W. Holtzscheiter, 773-A  
D. A. Crowley, 999-W  
S. L. Marra, 999-W  
T. B. Calloway, 999-W  
N. E. Bibler, 773-A  
C.M. Jantzen, 773-A  
J. R. Harbour, 773-42A  
G. G. Wicks, 773-A  
W. E. Daniel, 999-W  
T. L. Fellingner, 773-A  
C.C. Herman, 773-42A  
D. K. Peeler, 999-W  
M. E. Smith, 773-42A  
D. C. Koopman, 773-42A  
T. B. Edwards, 773-42A  
D. T. Herman, 773-A  
M. S. Miller, 704-S  
J. E. Occhipinti, 704-S  
R. M. Hoeppe, 704-27S  
H. H. Elder, 703-H  
J. F. Iaukea, 704-30S  
J. W. Ray, 704-S  
F. A. Washburn, 704-27S  
W. B. Van-Pelt, 704-S  
S. G. Phillips, 704-27S  
A. V. Staub, 704-27S