WSRC-TR-2003-00056, REV. 0
SRT-RPP-2002-00231, REV. 0

AN-102 SIMULANT SR/TRU PRECIPITATION AND ULTRAFILTRATION (U)

February 10, 2003

Savannah River Technology Center
Waste Treatment Technology
Waste Processing Technology Section
Immobilization Technology Section

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

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

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, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness 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. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available for sale to the public, in paper, from: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161,
phone: (800) 553-6847,
fax: (703) 605-6900
email: orders@ntis.fedworld.gov
online ordering: http://www.ntis.gov/help/index.asp

Available electronically at http://www.osti.gov/bridge
Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from: U.S. Department of Energy, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062,
phone: (865)576-8401,
fax: (865)576-5728
email: reports@adonis.osti.gov

WSRC-TR-2003-00056, REV. 0
SRT-RPP-2002-00231, REV. 0

Key Words:
Envelope C
Cells Unit Filter
Slurry Rheology
Permeate Flux

Retention: Permanent

Key WTP R&T References:

24590-WTP-TSP-RT-01-019, Rev. 0
SRT-RPP-2002-00007, Rev. 0
Test Scoping Statement S-43

AN-102 SIMULANT SR/TRU PRECIPITATION AND ULTRAFILTRATION (U)

John R. Zamecnik
Mark A. Baich
Erich K. Hansen
Michael R. Poirier

February 10, 2003

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DE-AC09-96SR18500

TABLE OF CONTENTS

LIST OF TABLES vii
LIST OF ACRONYMS viii
TERMS AND DEFINITIONS ix
1.0 SUMMARY OF TESTING 1
  1.1 OBJECTIVES 1
  1.2 CONDUCT OF TESTING 2
    1.2.1 Tests with First Remediation (R1) Simulant 2
    1.2.2 Tests with Second Remediation (R2) Simulant 3
  1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES 5
    1.3.1 Remediation 1 Baseline Conditions 5
    1.3.2 Remediation 2 with Newly Optimized Conditions and Baseline Conditions . 6
  1.4 QUALITY REQUIREMENTS 7
  1.5 ISSUES 7
2.0 CD-ROM ENCLOSURES 8
3.0 DISCUSSION 8
  3.1 EXPERIMENTAL DETAIL 8
    3.1.1 Simulant Remediation 1 Precipitation at Baseline Conditions 11
    3.1.2 Simulant Remediation 2 and Newly Optimized Conditions Precipitations . 13
    3.1.3 Simulant Remediation 2 and Baseline Conditions 14
  3.2 RESULTS 15
    3.2.1 Simulant Formulations 15
      3.2.1.1 Simulant Remediation 1 15
      3.2.1.2 Simulant Remediation 2 16
      3.2.1.3 Precipitations 18
    3.2.2 Concentration of Precipitated Simulants – Composition and Properties 21
      3.2.2.1 Remediation 1 – Baseline Conditions 21
      3.2.2.2 Remediation 2 – Newly Optimized Conditions Run 2 (R2NOC2) 24
      3.2.2.3 Remediation 2 – Baseline Conditions 26
    3.2.3 R1BC Washing and Re-Concentration 28
    3.2.4 Rheological Measurements 28
    3.2.5 Heat Transfer 33
    3.2.6 Particle Size Distributions 34
    3.2.7 Feed Decontamination – R1BC and R2NOC2 Data 42
    3.2.8 Filtration – Remediation 1 Baseline Conditions 49
      3.2.8.1 Clean Water and Strontium Carbonate Fluxes 49
      3.2.8.2 Factorial Design Tests – R1BC 52
      3.2.8.3 Flux versus Insoluble Solids Concentration 57
    3.2.9 Filtration – Remediation 2 Runs 62
4.0 FUTURE WORK 65
5.0 REFERENCES 66
APPENDIX A1. GRAPHICAL FLUX DATA 68
APPENDIX A2. RAW FLUX DATA 77
APPENDIX A3. ANALYTICAL DATA 81
APPENDIX A4. RHEOLOGICAL DATA 88

APPENDIX A5. JMP CURVE FITTING DATA 101
APPENDIX A6. SIMULANT RECIPES 108
APPENDIX A7. PARTICLE SIZE DISTRIBUTION DATA 113
APPENDIX A8. EMAIL FROM TOWNSON TO ZAMECNIK 123

LIST OF FIGURES

Figure 1 Precipitator Equipment....9
Figure 2 Cold Cells Unit Filter (Cold CUF) System....10
Figure 3 Schematic of CUF System....10
Figure 4 Slurry Consistencies....30
Figure 5 Slurry Yield Stresses....31
Figure 6 Feed Tank Sketch Showing Possible Stagnation....32
Figure 7 CUF Feed Slurry Mixing at Approximately 15-17 wt% Insoluble Solids....32
Figure 8 Reynolds Number in Filter Tube versus Insoluble Solids Content....33
Figure 9 Heat Transfer During Concentration of R2NOC2 Slurry....34
Figure 10 Particle Size Volume Distribution for R1BC Slurry....36
Figure 11 Particle Size Volume Distribution for R2NOC Pilot Scale Slurry....37
Figure 12 Particle Size Volume Distribution for R2NOC2 Slurry....38
Figure 13 Particle Size Volume Distribution for R2BCL Slurry....39
Figure 14 Volume Distributions of Concentrated Slurries by the X100 Absorptive Method....41
Figure 15 Volume Distributions of Concentrated Slurries by the SRA150 Reflective Method....41
Figure 16 Volume Distributions of Initial Precipitates by the X100 Absorptive Method....43
Figure 17 SEM Photo of Filtered R1BC & R2NOC1 Slurries....43
Figure 18 Definitions for Percent Retained and Decontamination Factor....45
Figure 19 Clean Water Fluxes – R1BC Run....51
Figure 20 Clean Water and $SrCO_3$ Fluxes – R1BC Run....52
Figure 21 Factorial Design Arrangement: Low and High Solids Concentrations....53
Figure 22 Low Solids Factorial: Flux versus Time at 11 fps Velocity.....53
Figure 23 Low Solids Factorial: Flux versus Time at 30 psi TMP.....54
Figure 24 Low Solids Factorial: Flux versus Time at 11 fps Velocity, 40 psi TMP.....54
Figure 25 Comparison of Fluxes for this Work, AN-102 Small C, and AN-107 Pilot Simulant Filtration....55
Figure 26 Predicted Flux versus Time for Concentration of Unwashed Slurry....57
Figure 27 Flux During Concentration Showing Flux Decline....58
Figure 28 Comparison of Fluxes During R1BC Concentration....58
Figure 29 Fluxes for R1BC Concentration and Re-Concentration of Washed Slurry....59
Figure 30 Predicted Flux versus Insoluble Solids Content....60
Figure 31 Predicted Flux versus Time for Concentration of Unwashed & Washed Slurry....61
Figure 32 Flux Corrected for Time & Viscosity....62
Figure 33 R2NOC Filtration Fluxes versus Insoluble Solids....63
Figure 34 Baseline Conditions Fluxes versus Insoluble Solids....64
Figure 35 Flux Comparison of CUF Concentrations with Large C....64

LIST OF TABLES

Table 1 Summary of Conditions for Precipitation Runs 5
Table 2 Approved Solids Simulant Formulation........16
Table 3 Calculated and Measured Composition of R1BC Precipitate Slurry Feed Batch #1........17
Table 4 Composition of Batches 3B and 3C Simulant........18
Table 5 Summary of Reagents for R1BC (per 5L of Simulant)........19
Table 6 Precipitation Reagent Quantities for R1BC 20
Table 7 Summary of Reagents for RNOC1 and R2NOC2........20
Table 8 Summary of Reagents for R2BCL........21
Table 9 Solids Content of R1BC Precipitate Batches and Concentrated Slurry........21
Table 10 R1BC Measured and Calculated Solids Content and Volume 22
Table 11 R1BC Permeate Compositions During Concentration........23
Table 12 Turbidity of Permeates........24
Table 13 Summary of R2NOC2 Solids Contents........24
Table 14 R2NOC Slurry and Permeate Compositions 25
Table 15 Summary of R2BCL Solids Contents........26
Table 16 Composition of R2BCL Precipitate and Concentrated Slurry........27
Table 17 R1BC Composition of Unwashed and Washed Slurry and Permeate........29
Table 18 Rheological Properties of Initial Precipitates, Concentrated Slurries, and Permeates........30
Table 19 Microtrac Particle Size Analyzer Methods........35
Table 20 Comparison of Permeate and Precipitate Compositions 44
Table 21 Decontamination Factors and Fraction Retained 47
Table 22 Measured and Calculated Concentrated Washed Slurry Concentrations........48
Table 23 Permeate and Concentrate Decontamination Factors........49
Table 24 Factorial Design Test Conditions for R1BC 50

LIST OF ACRONYMS

µm	micrometer
AA	atomic absorption
ADS	Analytical Development Section (SRTC)
cp	centipoise
CUF	Cells Unit Filter
DF	decontamination factor
EDL	Engineering Development Laboratory (SRTC)
fps	feet per second
gpm	gallons per minute
HLW	high level waste
IS	insoluble solids
L	liter
LAW	low activity waste
M	molar
NA or na	not applicable or not available
NC	not calculated
NIST	National Institute of Standards and Testing
NM	not measured
NQA	Nuclear Quality Assurance
NTU	nephelometric turbidity unit
psi	pounds per square inch
QA	quality assurance
R & T	Research & Technology
RPP	River Protection Project
SRTC	Savannah River Technology Center
TMP	transmembrane pressure
TRU	transuranic
TS	total solids
WSRC	Westinghouse Savannah River Company
wt%	weight percent
WTP	Waste Treatment Plant

TERMS AND DEFINITIONS

suspended solids or insoluble solids	Solids that are not dissolved in solution; these solids would be filterable; often called undissolved solids
R1	First remediation of Optima simulant
R2	Second remediation of Optima simulant; has more carbonate
R1BC	First remediation simulant, large batch, precipitated at the baseline conditions: 0.9M added hydroxide, 0.075M Sr, 0.05M Mn, 50°C
R2NOC1	Second remediation simulant, first large batch (115L), precipitated at the "newly optimized conditions" (NOC): no added hydroxide, 0.03M Sr, 0.03M Mn, 25°C
R2NOC small	Second remediation simulant, small batch, precipitated at the "newly optimized conditions" (NOC): no added hydroxide, 0.03M Sr, 0.03M Mn, 25°C
R2NOC2	Second remediation simulant, second large batch, precipitated at the "newly optimized conditions" (NOC): no added hydroxide, 0.03M Sr, 0.03M Mn, 25°C
R2BC	Second remediation simulant, small batch, precipitated at the baseline conditions: 0.9M added hydroxide, 0.075M Sr, 0.05M Mn, 50°C
R2BCL	Second remediation simulant, small batch, precipitated at the baseline conditions (slightly modified): 0.8M added hydroxide (to give 1.0M hydroxide total), 0.075M Sr, 0.05M Mn, 50°C

1.0 SUMMARY OF TESTING

1.1 OBJECTIVES

The objectives of these tests were specified in R&T Test Scoping Statement S-43, Test Specification 24590-WTP-TSP-RT-01-019, Rev. 0[1], and SRTC Task Technical & Quality Assurance Plan SRT-RPP-2002-00007, Rev. 0.[2] The test included gathering data on performance of the single-tube crossflow ultrafilter unit to de-water the simulant precipitate derived from a project approved tank 241-AN-102 simulant.

The initial task objectives and requirements are as follows:

1. Perform a de-watering test (details described later) to estimate the removal efficiency for soluble species.
2. Using a filtration test matrix, measure indicative data on equipment performance (permeate flux, back pulse efficiency) with the initial precipitate and also at approximately 15 wt% insoluble solids.
3. Compare filtration flux data with the work done using active waste[3] to validate the simulant. Validation of the simulant was verbally agreed upon to mean that if the fluxes determined during concentration of the precipitate were within ±25% of the Large C radioactive demonstration fluxes, the simulant would be validated. Validation of the simulant will then allow work to be conducted at a larger scale on the (multi-tube) filtration pilot plant. (This pilot scale work was covered under separate Test Specifications.[4,5])
4. Show that the mean flux throughout the complete de-watering cycle is greater than 0.02 gpm/ft^2.
5. After completion of filtration, chemically clean the filter system and measure the clean water fluxes to establish that the fluxes can be returned to pre-operation (clean) levels.
6. Determine if a final insoluble solids concentration of 15 wt% can be improved.

A general requirement that applied to all of this work was that no solids must pass into the ultrafiltration permeate. The work listed above was done with a remediation of a purchased simulant; this remediation was called “R1”.

Upon completion of the above objectives with the approved R1 simulant, the simulant specification was changed and additional work at modified precipitation conditions was requested. The revised simulant formulation was called “R2”. The additional requirements were:

7. With “newly optimized” precipitation conditions and the R2 simulant recipe, perform a de-watering test and show that the time-averaged flux is above 0.02 gpm/ft^2.
8. Perform a precipitation with the original baseline conditions and the new simulant recipe and show that the time-averaged flux is above 0.02 gpm/ft^2.

1.2 CONDUCT OF TESTING

For the tests described below, the following equipment and sequence of work was performed for each set of tests. Crossflow filtration testing of the feed samples was then conducted on a Cells Unit Filter (CUF, shown later in Figure 2). The CUF consisted of a single Mott industrial grade 0.1 µm tube filter module 24" long, with a 3/8" inside diameter tube. The concentrate slurry and permeate flows, concentrate temperature, tube inlet, outlet, and permeate pressure, volumes collected, and time were measured with calibrated devices.

Before filtering the simulant slurry, the CUF was cleaned with 2M HNO_3, and then rinsed with inhibited water (0.01M NaOH). Clean water flux measurements at 10, 20, and 30 psi transmembrane pressure (TMP) and a velocity of 11 feet/sec (fps) were then performed. These same conditions were duplicated with a 5.0 wt% slurry of granular strontium carbonate. The unit was rinsed with inhibited water, then briefly with 2M HNO_3 to dissolve residual carbonate, then rinsed again with inhibited water. The clean water fluxes were then measured again.

The permeate fluxes were expressed in terms of flux corrected to 25°C by the use of a temperature correction factor specified by WTP.[1] Samples of the slurry and permeate were taken throughout this task as specified by WTP. These analyses included particle size distribution, rheology, total solids, insoluble solids, specific gravity, elemental analysis by ICPES, ion chromatography, and turbidity.

Following completion of the tests, the CUF was cleaned with inhibited water to establish the cleaning efficiency for water alone prior to chemical cleaning with 2M nitric acid. The system was then flushed with inhibited water, followed by repeating the inhibited water, strontium carbonate, and second inhibited water flux tests.

1.2.1 Tests with First Remediation (R1) Simulant

The Envelope C AN-102 simulant solution developed by SRTC (WTP level IV activity ID 2BPR1SC202) was specified to be used for this work. This simulant recipe included both a supernate simulant recipe and a solids simulant recipe. The amount of solids simulant added to the supernate simulant was 0.1 wt%.[6] This simulant recipe has been designated "AN102R1" or just "R1" for remediation 1 since it was the result of remediating a purchased simulant prepared by Optima Chemicals. The remediation was required because the simulant recipe was changed after purchase of the simulant. This simulant recipe and a second remediation recipe are shown in the Appendix.

The Test Specification gave the Sr/TRU precipitation recipe to be used for this work.[5] This recipe specified first diluting the simulant with water to achieve 6.0M sodium, addition of NaOH so that the total (not specified as <u>added</u> hydroxide) hydroxide was 1.0M, addition of $Sr(NO_3)_2$ at 50°C to give a final Sr concentration of 0.075M, and addition of $NaMnO_4$ at 50°C to give an added permanganate concentration of 0.05M. The recipe subsequently published by SRTC specified that the <u>added</u> hydroxide should be 1.0M.[7] However, in a follow-up email from Townson[8] regarding an error in the Test Specification recipe, the added hydroxide was specified as 0.9M rather than 1.0M; this value was subsequently used for the R1BC precipitation. (See Section 3.2.1.3 for additional discussion.)

The specific amounts of reagents called for in the recipe were adjusted for the actual reagent concentrations to give the same number of moles. Nine batches of simulant precipitate, each starting from 5 liters of simulant, were made; the final volume of each batch of precipitate was about 6.5 liters. This set of experiments has been designated "AN102R1BC" or "R1BC", which means the first remediation simulant precipitated at the baseline conditions.

The precipitate generated from the steps above resulted in a slurry with approximately 1.45-1.60 wt% insoluble solids. The flux was then measured for a 17-point modified factorial matrix of TMPs and axial velocities to determine the optimum set of conditions and compare to the results of the active waste tests. Each set of conditions was run for one hour, with multiple backpulses of permeate between each set of conditions. The slurry temperature in all tests was maintained at 25±5°C.

Eight additional batches of precipitate were then made over the course of several days and each was fed into the CUF system. The dewatering test was conducted at 50 psi TMP and 12 fps velocity. These are the same conditions used during most of the first dewatering in the active waste run.[3] Once all precipitate batches were added, the final concentration step was performed, reducing the total volume below the initial 6.5 liters. The slurry was concentrated to about 4.5 liters volume, or about 19.3 wt% insoluble solids. Permeate was added back into the slurry and reduced the insoluble solids concentration to approximately 17.2 wt% versus the target of 15 wt%. Samples of slurry and permeate were taken at approximately 13.3 wt% and at 17.2 wt% insoluble solids in the slurry.

The approximately 17.2 wt% insoluble solids slurry was subjected to the same set of factorial experiments described above for the dilute slurry. The total volume of permeate collected during concentration was about 54 liters. The final concentrated slurry volume was about 4.5 liters.

After concentrating to approximately 17.2 wt% insoluble solids, and performing the factorial experiments, the solids were washed with a single equal volume of inhibited water. The slurry was then re-concentrated to approximately 20.6 wt% insoluble solids.

1.2.2 Tests with Second Remediation (R2) Simulant

After completion of the tests with the R1 formulation and baseline conditions, the customer determined that the carbonate concentration in the simulant specification should have been higher. This revised simulant recipe is designated "AN102R2" or "R2". In addition, the Sr/TRU precipitation recipe was also changed. Washing and re-concentration of the slurry was not performed for any of the runs with the R2 simulant.

The “newly optimized conditions” (NOC) targets for precipitation were specified by WTP as:

1. No added caustic
2. 0.03M Sr final concentration added as $Sr(NO_3)_2$
3. 0.03M Mn final concentration added as $NaMnO_4$
4. Precipitation temperature of 25°C

The first precipitation with R2 simulant was conducted with a single large batch of 115L of R2 simulant; this material is designated “AN102R2NOC1” or “R2NOC1”. The insoluble solids content of this batch as about 0.74-1.19 wt%. Immediately upon starting the filtration, the flux decreased below 0.02 gpm/ft^2, so this test was stopped.

Per project direction, this precipitation was repeated with 5L of R2 simulant (R2NOC small) and filtered. The initial fluxes measured were significantly higher that found during R2NOC1, so another 115L batch (R2NOC2) was prepared at the same precipitation conditions. The initial fluxes were again higher (0.119 gpm/ft^2), so this material was concentrated to 18.5 wt% insoluble solids. The initial precipitate insoluble solids was 1.32 wt% before concentration. Although the initial fluxes were acceptable, the flux dropped below the minimum 0.02 gpm/ft^2 at about 8 wt% insoluble solids. The time-averaged flux was 0.013 gpm/ft^2.

After extensive discussions with Project personnel, the path forward was changed to go back to the baseline conditions for precipitation. A small batch of R2 simulant was precipitated at the baseline conditions (R2BC small) and filtered. The initial filtration fluxes were much higher than those found for the NOC recipe. A large batch was then planned with the following conditions:

1. Add caustic to give 1.0M hydroxide (rather than added caustic equal to 1.0M)
2. 0.075M Sr added as $Sr(NO_3)_2$
3. 0.05M Mn added as $NaMnO_4$
4. Precipitation temperature of 50°C

Note that this modification of the baseline conditions with a smaller hydroxide additional is still referred to herein as “baseline conditions” since it is much closer to the true baseline conditions than the NOC conditions.

This precipitation was performed on a large batch (approx. 880L) in the Engineering Development Laboratory, with a small quantity then sent for use in the CUF. The initial insoluble solids content was 1.60 wt%. Filtration of this batch gave acceptable flux results and it was concentrated to 18.8 wt% insoluble solids. The time-averaged flux was 0.060 gpm/ft^2.

The tests at the various conditions are summarized in Table 1. The hydroxide was added at no particular rate or temperature, while the $Sr(NO_3)_2$ and $NaMnO_4$ reagents were each added over approximately 20 minutes at the specified temperature, with a 30 minute waiting period both before the $Sr(NO_3)_2$ addition and the $NaMnO_4$ addition. The hold time at temperature after completion of the $NaMnO_4$ addition was four hours (or a wait time before filtration of four hours for those runs at 25°C).

Table 1 Summary of Conditions for Precipitation Runs

Experiment	Batch Source*	Remediation	OH- Added (M)	Sr (M)	Mn (M)	Temp. (°C)	Batch Volume (L)
R1BC	Small remediation of Optima simulant	R1	0.9	0.075	0.05	50	115
R2NOC1	Optima remediation batch 3C by EDL	R2	0	0.03	0.03	25	115
R2NOC small	Optima remediation batch 3C by EDL	R2	0	0.03	0.03	25	5
R2BC	Optima remediation batch 3C by EDL	R2	0.9	0.075	0.05	50	5
R2NOC2	Optima remediation batch 3C by EDL	R2	0	0.03	0.03	25	115
R2BCL	Optima remediation batch 3B by EDL	R2	0.8 (to 1.0 total)	0.075	0.05	50	880 (approx. 60 to CUF)

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

Prior to and after all filtration work, the clean water and $SrCO_3$ fluxes were measured. The before and after fluxes were found to be essentially identical for both water and $SrCO_3$, indicating that no irreversible changes in the filter occurred. The $SrCO_3$ fluxes were about 30% lower than the clean water fluxes. These results complete Objective 5: *After completion of filtration, chemically clean the filter system and measure the clean water fluxes to establish that the fluxes can be returned to pre-operation (clean) levels.*

1.3.1 Remediation 1 Baseline Conditions

Nine 5L batches of simulant were prepared and precipitated using the Sr/TRU precipitation recipe that was the latest baseline formulation at the time of the test (as specified by the Test Specification).

The optimum dewatering conditions for the initial (approximately 1.45-1.60 wt% insoluble solids) slurry were determined by performing the specified factorial experimental design. The experimental design data was analyzed and fit to a mathematical model.

Maximum flux is predicted to occur at the maximum velocity and TMP. Of the conditions actually tested, the optimum was a velocity of 15 fps and a TMP of 40 psi. The permeate flux was found to decrease with time at the same process conditions, which may indicate pore plugging or reduced efficiency due to decreasing particle size versus time.

The slurry was successfully dewatered to greater than 15 wt% insoluble solids. The highest concentration achieved was 19.3 wt% insoluble solids. This slurry was diluted with permeate to 17.2 wt% and a factorial design matrix was performed. The optimum conditions were the highest velocity; pressure had no significant effect. The permeate flux was again found to decrease with time at the same process conditions. These results complete Objective 2: *Using a filtration test matrix, measure indicative data on equipment performance with the initial precipitate and also at approximately 15 wt% insoluble solids*.

Concentration of the slurry from the initial solids loading to 19.3 wt% was conducted at the specified conditions. The fluxes achieved were shown to be linearly dependent on the insoluble solids content when the effect of time was included. The mean permeate flux during dewatering was 0.036 gpm/ft^2 compared to the average design flux of 0.02 gpm/ft^2. This completes Objectives 4: *Show that the mean flux throughout the complete de-watering cycle is greater than 0.02 gpm/ft*^{2}.

For the fluxes measured, 73% were within ±25% and the remaining fluxes were within 32% of the fluxes determined during the initial concentration step of the Large C active waste filtration work.[3] Although not meeting Objective 3 exactly, the results are sufficiently close that we consider the simulant to be validated. (Objective 3: *Compare filtration flux data with the work done using active waste to validate the simulant*. *Validation meant that the fluxes determined during concentration were within ±25% of the Large C fluxes.)*

The concentrated slurry was subjected to a 1:1 volumetric water wash and then re-concentrated to 20.6 wt% insoluble solids. The flux during re-concentration was predicted well by the previously determined model, with the addition of a term for the permeate viscosity. Concentration of both the unwashed and washed slurry meets Objective 6: *Determine if a final insoluble solids concentration of 15 wt% can be improved*.

The decontamination factors for removal of soluble components from the concentrated slurry ranged form 1.61 to 28.4 for soluble or partially soluble species. Greater than 95% of the Al and Na were removed from the slurry. These results complete Objective 1: *Perform a de-watering test (details described later) to estimate the removal efficiency for soluble species.*

Permeate samples analyzed all had turbidities of less than 5 NTU, which indicates that no breakthrough of solids, due to a crack or similar failure, occurred during filtration. After standing for several days, all permeates had formed a brown coating on all container surfaces, indicating post precipitation. However, the liquid was still transparent, as indicated by the turbidity results.

1.3.2 Remediation 2 with Newly Optimized Conditions and Baseline Conditions

For the succeeding experiments, the full factorial matrices were not requested for either the dilute initial precipitate or the concentrated slurry. A few selected factorial points were run for several of the experiments. The objectives for each run were determined both before and during the run, depending on the results found.

The filtration for both runs with the R2 remediation and the NOC conditions showed that the desired fluxes could not be maintained. This result shows that the NOC conditions result in unacceptable performance. These runs also neither validate nor invalidate the R2 simulant since the precipitation conditions were also changed. The NOC2 run flux, above an insoluble solids concentration of about 4 wt%, ranged from about 50-65% of the Large C active waste fluxes. The pilot filtration fluxes from the EDL for this same case were even lower. These results showed that Objective 7 could not be met. (Objective 7: *With "newly optimized" precipitation conditions and the new simulant recipe, perform a de-watering test and show that the time-averaged flux is above 0.02 gpm/ft^2.*)

The R2 run with the baseline conditions resulted in an time-averaged flux of 0.060 gpm/ft^2, which was about two times greater than those measured for the R1BC run and the Large C active waste run. This result completes Objective 8: *Perform a precipitation with the original baseline conditions and the new simulant recipe and show that the time-averaged flux is above 0.02 gpm/ft^2.*

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO MOSRLE60. Researchers followed the WSRC Quality Assurance Program, which has been approved by WTP, and the WSRC Quality Assurance Management Plan (WSRC-RP-92-225). SRTC has provided matrices to WTP demonstrating compliance of the SRTC QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRTC QA program with NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices. This program will apply the appropriate quality assurance requirements for this task, as indicated by the QA Plan Checklist in Section VIII.

Analytical sample labeling and tracking complied with established procedures (WSRC Manual L1, Procedure 7.15). The SRTC Analytical Development Section (ADS) conducted all analyses using the routine level QA program. Calibrated measuring and test equipment were utilized for all flow rate, pressure, and temperature measurements on the CUF unit.

The Task Technical & Quality Assurance Plan provided the quality requirements for this work.[2] NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 were applied as appropriate.

Calibrated balances were checked with calibrated weights that were traceable to NIST before use. The accuracy was ±1%. Pressures were measured with calibrated pressure gauges that had an accuracy of ±2 psi. The flow meters were calibrated and verified to have an accuracy of ±10%. The thermocouples used to measure temperature were checked against calibrated thermometers and had an accuracy of ±1°C. Calibrated stopwatches were used to measure the filtrate flow rate and had an accuracy of ±1%.

1.5 ISSUES

The slurries for all three runs that were concentrated to higher than 15 wt% insoluble solids were very difficult to mix in the slurry feed tank. The small agitator used provided little agitation; a larger agitator used in the later runs was somewhat more effective. This problem became even worse with the washed concentrated (20.6 wt%) slurry. The slurry samples above 13 wt% insoluble solids were all found to be non-Newtonian, with the specific behavior being close to a Bingham plastic. The measured consistencies and yield stresses of these slurries put these slurries in the laminar flow region for both tank agitation and pipe flow. These results indicate that the best equipment for mixing would be laminar mixers, and for pumping, would be positive displacement pumps, possibly with screw suction.

In addition to affecting fluid transport, the high consistencies and non-Newtonian behavior increased the heat load on the heat exchanger. Significantly more energy was input into the fluid by the pump when the slurry was more concentrated. At the highest concentrations, there was insufficient heat transfer area to maintain the slurry temperature within the required range. The overall heat transfer coefficients decreased only about 20%, so the main factor in heat removal appears to be assuring sufficient heat exchange area.

The "newly optimized" precipitation conditions did not result in a filterable precipitate. Future optimization of the precipitation reactions should be coupled with crossflow filter tests to assure that the precipitate will be filterable. These tests should take the precipitate to at least 10 wt% insoluble solids, and preferably beyond.

During the precipitations, it was noted that ammonia gas was formed. Ammonia evolution may be a flammability, emissions, or ammonium nitrate formation concern for the WTP.

2.0 CD-ROM ENCLOSURES

The enclosed CD-ROM contains two movies:

1. Resuspension of a settled precipitate batch: "Slurry-Resuspension.mpg"
2. Mixing of concentrated slurry in feed tank: "Feed-Mixing.mpg"

These movies can be viewed using the Windows Media Player or other multimedia software.

An electronic copy of this report is also included on the CD-ROM. Adobe Acrobat Reader is required to view this document.

3.0 DISCUSSION

3.1 EXPERIMENTAL DETAIL

The experimental work will be discussed in chronological order. The precipitator equipment used for the 5L precipitations is shown in Figure 1.

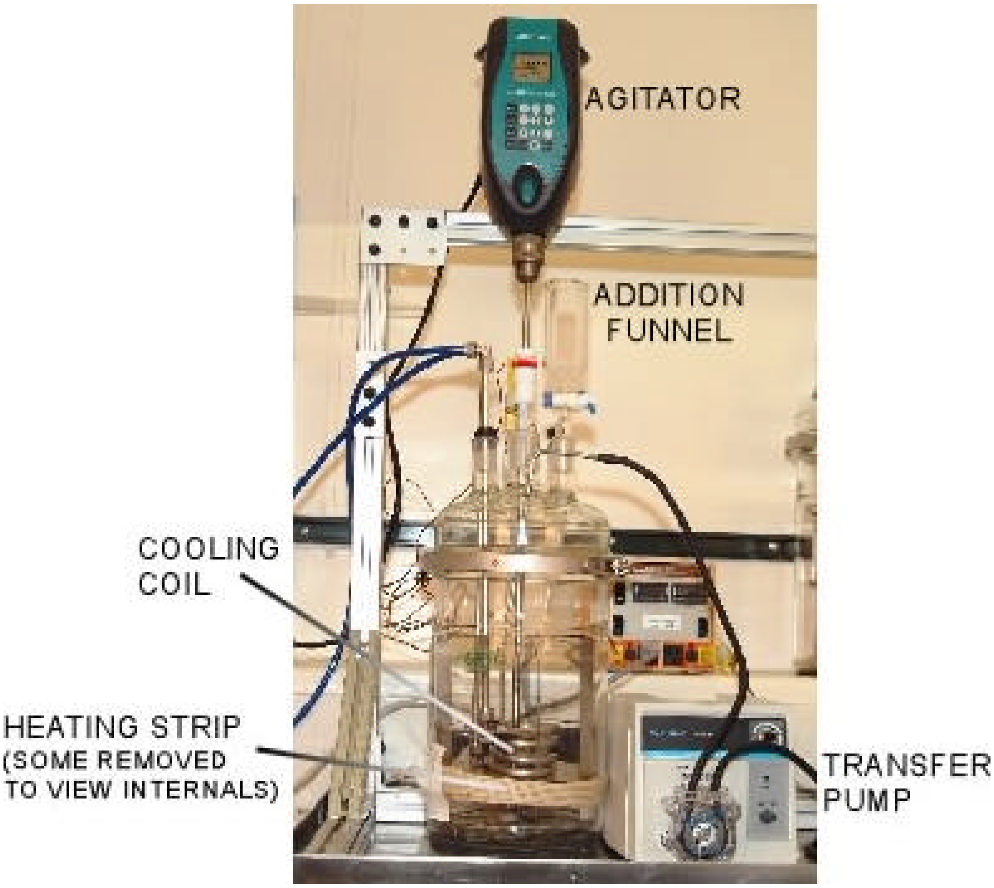


Figure 1 Precipitator Equipment

Crossflow filtration testing of the feed samples was then conducted on a Cells Unit Filter (CUF). The CUF consisted of a single Mott industrial grade 0.1 μm tube filter module 24” long, with a 3/8” inside diameter tube. A photo of the CUF is shown in Figure 2 and a piping diagram in Figure 3. The concentrate flow and temperature, and tube inlet, outlet, and permeate pressure were all measured with calibrated measurement devices. The flowrate of permeate was measured by determining the amount of time to collect a specific amount of permeate, which was 40 ml during tests where the permeate was recycled, 250 or 500 ml during concentration, and 250 ml during clean water flux tests. Calibrated stopwatches and graduated cylinders were used.

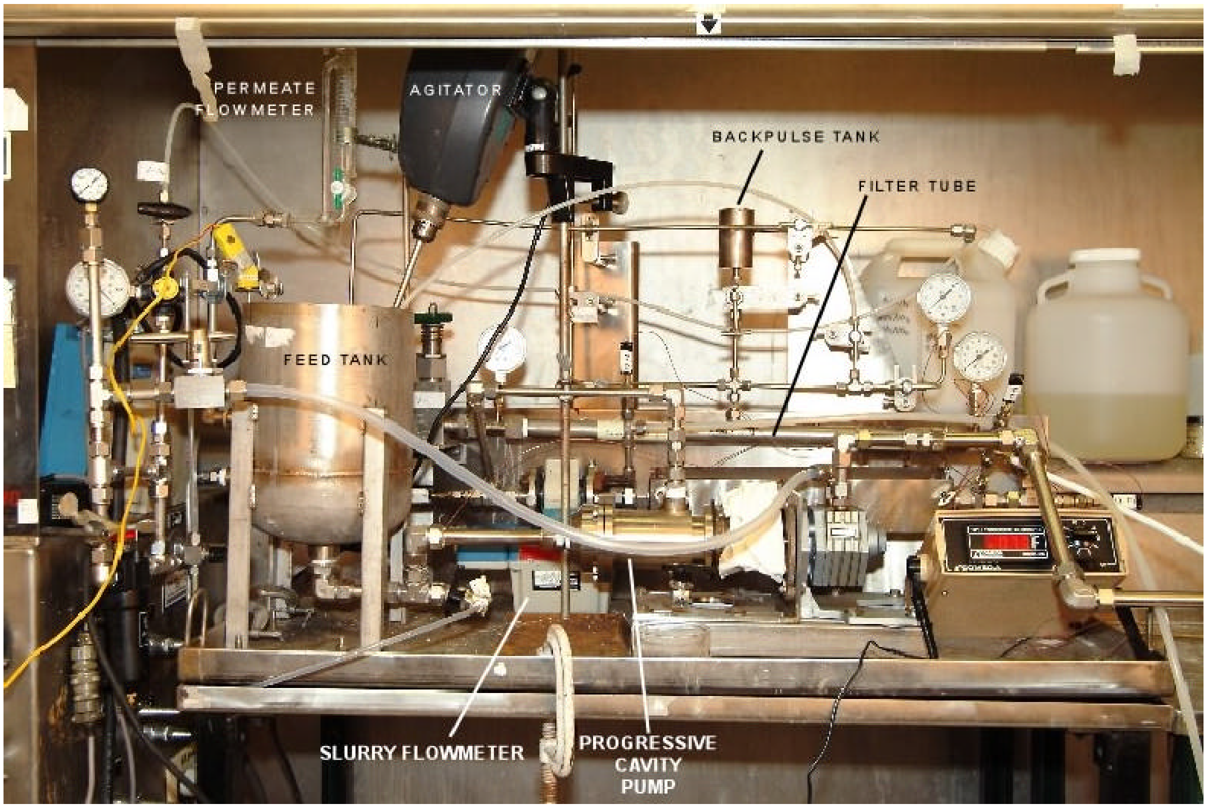


Figure 2 Cold Cells Unit Filter (Cold CUF) System

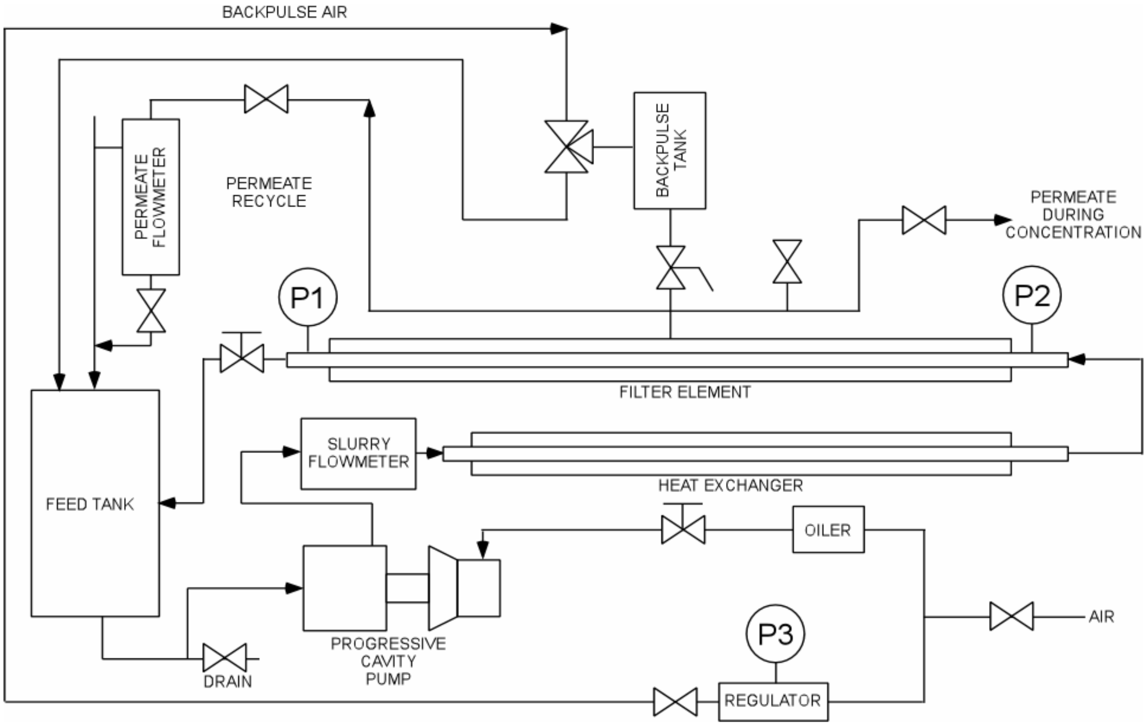


Figure 3 Schematic of CUF System

Before filtering the simulant slurry, the CUF was cleaned with 2M HNO_3, then rinsed with inhibited water (0.01M NaOH). “Clean water flux” measurements at 10, 20, and 30 psi transmembrane pressure (TMP) and a velocity of 11 feet/sec (fps) were then performed. Note that “clean water flux” actually uses inhibited water. After measuring these fluxes, the same conditions were duplicated with a 5.0 wt% solution of granular strontium carbonate. This strontium carbonate was purchased as nominal 1 µm material; particle size distribution analysis showed a relatively Gaussian distribution centered on 4 µm. After the strontium carbonate fluxes were measured, the unit was rinsed with inhibited water, then briefly with 2M HNO_3 to dissolve residual carbonate, then rinsed again with inhibited water. The clean water fluxes were then measured again.

3.1.1 Simulant Remediation 1 Precipitation at Baseline Conditions

The Envelope C AN-102 simulant solution developed by SRTC (WTP level IV activity ID 2BPR1SC202) was specified to be used for this work.[6] This simulant recipe included both a supernate simulant recipe and a solids simulant recipe. Both of these recipes were received via email from the SRTC researcher after verbal approval by Project personnel (Townson). The amount of solids simulant to use in the supernate simulant was also verbally communicated; this value was 0.1 wt%. The supernate simulant for this portion of the task was made by remediating simulant prepared for SRTC by Optima Chemicals. This remediation, called “R1”, was performed to give 50L of remediated simulant. The target and measured compositions of the simulant, as well as other samples, are shown and discussed more fully in Section 3.2.1.

The Test Specification gave the Sr/TRU precipitation recipe to be used for this work.[5] This recipe specified first diluting the simulant with water to achieve 6.0M sodium, addition of NaOH so that the total (not specified as added hydroxide) hydroxide was 1.0M, addition of $Sr(NO_3)_2$ at 50°C to give a final Sr concentration of 0.075M, and addition of $NaMnO_4$ at 50°C to give an added permanganate concentration of 0.05M. However, in a follow-up email from Townson[8] (see Appendix) regarding an error in the Test Specification recipe, the added hydroxide was specified as 0.9M rather than 1.0M; this value was subsequently, and erroneously, used for the R1BC precipitation. (See Section 3.2.1.3 for additional discussion.)

The specific amounts of reagents called for in the recipe were adjusted for the actual reagent concentrations to give the same number of moles. Per 1-liter, the amounts of each reagent were specified to be added within specific tolerances. The actual initial concentration of Na, the concentration of the NaOH reagent, and the concentration of the $NaMnO_4$ reagent differed from those specified (which was acceptable), so the actual amounts added were adjusted to give the same number of moles. The “per liter of simulant” tolerances specified were maintained. All water used during the course of this task for reagent preparation was deionized and filtered with a 0.1 µm filter. Nine batches of simulant precipitate, each starting from 5 liters of simulant, were made; the final volume of each batch of precipitate was about 6.5 liters.

The first and fifth precipitate batches were analyzed; the first was analyzed after the filtration factorial experiments (described below), while the fifth batch was analyzed directly from the precipitator. The first batch analysis should show if there are any effects of the pumping operation on the particle size distribution.

The precipitate generated from the steps above resulted in a slurry with 1.45-1.60 wt% insoluble solids. The flux was then measured for a 17-point modified factorial matrix of TMPs (20-70 psi) and velocities (7-15 fps) to determine the optimum set of conditions and compare to the results of the active waste tests. Each set of conditions was run for one hour, with multiple backpulses of permeate between each set of conditions. The approximate steady state flux for each set of conditions was determined by first visually examining the graph of flux versus time and then taking the average of 1 to 3 of the last points (40, 50, 60 min), per the researchers judgment. During the time interval of 60 minutes for each set of conditions, the flux did not reach as steady state value in most cases, but continued to drop slightly. The slurry temperature in all tests was maintained at 25±5°C.

Eight additional batches of precipitate were then made over the course of several days and each was fed into the CUF system. The level in the CUF feed tank was maintained approximately constant by adding precipitate at the same rate that permeate was drawn off, thus concentrating the slurry. Each test was conducted at the conditions of 50 psi TMP and 12 fps velocity used during the active waste run.[3] Each added batch increased the insoluble solids concentration of the slurry by about 1.45-1.60 wt%. Slight variations from this value occurred due to the removal of slurry samples.

Once all precipitate batches were added, the final concentration step was performed, thus reducing the total volume of slurry below the initial 6.5 liters. Backpulses performed between each batch required successively longer time to perform because the decreased flux increased the amount of time necessary to fill the backpulse tank. The amount of time between a backpulse and the resumption of permeate collection also increased; the backpulses introduced air into the permeate side of the filter element which then had to be displaced before the flow measurement could resume. Note that it is the measurement of permeate flow that is delayed and not the actual resumption of permeate flow. For these reasons, the first flux reading after backpulsing is actually lower than the true initial flux.

The total volume of permeate collected during concentration was about 54 liters. The final concentrated slurry volume was 4 liters, or about 19.3 wt% insoluble solids. The amount of permeate to be removed was calculated to be larger than necessary due to assuming the precipitate batches were 1.0 wt% insoluble solids rather than 1.45-1.60 wt%. When filtration became very difficult, a slurry sample was taken and analyzed for total solids. The total solids analysis indicated the insoluble solids were approximately 16-18 wt% rather than 15 wt%, so a calculated amount of permeate was added back into the slurry. At this point, the insoluble solids content was not measured; later analyses showed that the slurry had actually been concentrated to about 19.3 wt% insoluble solids and then diluted to about 17.2 wt%. Samples of slurry and permeate were taken at approximately 13.3 wt% and at 17.2 wt% insoluble solids in the slurry.

The approximately 17.2 wt% insoluble solids slurry was subjected to the same set of factorial experiments described above for the dilute slurry. The amount of time required for the permeate to start flowing in the flowmeter, due to the air in the permeate side of the filter, resulted in the first permeate flow (flux) measurements actually occurring at about 20 minutes after backpulsing rather than only several seconds to at most a minute for the low solids factorials. For this reason, most of the flux versus time curves for approximately 17.2 wt% insoluble solids do not show the initial flux decline after backpulsing. The typically seen flux decline is in general due to buildup of the solids on the filter membrane.

The permeate fluxes were expressed in terms of flux corrected to 25°C by the use of a temperature correction factor specified by WTP: $\exp\left(2500\left(\frac{1}{273+T}-\frac{1}{298}\right)\right)$, where T is in °C. This temperature correction factor corrects flux back to an equivalent flux at 25°C and accounts for changes in fluid viscosity and surface tension. The equation shown above was reported by WTP to have been derived at Oak Ridge National Laboratory, but no reference for this equation was supplied.

After concentration to approximately 17.2 wt% insoluble solids, and performing the factorial experiments, the solids were washed with a single equal volume of inhibited water. The slurry was then re-concentrated back to approximately 20.6 wt% insoluble solids.

Following completion of these tests, the CUF was cleaned with only inhibited water to establish the cleaning efficiency for water alone prior to performing chemical cleaning with 2M nitric acid. After chemical cleaning, the system was then flushed with inhibited water, followed by repeating the inhibited water, strontium carbonate, and second inhibited water flux tests.

Samples of the slurry and permeate were taken throughout this task as specified by WTP. A complete listing of these samples and results are given later.

3.1.2 Simulant Remediation 2 and Newly Optimized Conditions Precipitations

After completion of the R1BC run, Project personnel determined that the composition of the supernate simulant was incorrect. The carbonate concentration should have been higher than what was used. A new remediation recipe was devised (R2). At the same time, optimization work on the precipitation process indicated that lower reagent additions could accomplish the required decontamination,[9] so an additional run at these conditions was requested.

Approximately 115L of R2 remediated simulant was precipitated with no added hydroxide and final Sr and Mn concentrations of 0.03M each. The temperature was room temperature, which ranged from 19°C to 23°C, compared to the nominal 25°C requested. Heating of the drum used for the precipitation was not possible. The agitation during the precipitation was moderate. An 8” diameter marine propeller was used without baffles. The agitator speed had to be limited due to splashing and significant vortex formation. Stringy globs of the reacting reagents could be seen in the liquid for up to 30-60 seconds as the reagents were being added onto the surface of the liquid. Eventually, these strings mixed into the bulk liquid.

The $Sr(NO_3)_2$ and $NaMnO_4$ additions were each done in about 17 minutes. A 30-minute waiting period between the Sr and Mn additions was observed. After adding the $NaMnO_4$, the solution was mixed for four hours.

The precipitated simulant was transported to the CUF location and filtration was begun five days after completion of the precipitation. Within two hours, the flux had dropped from an initial 0.033 to 0.015 gpm/ft^2, which was below the plant minimum average flux. The CUF unit was then emptied, cleaned, and the clean water and $SrCO_3$ fluxes were determined. These fluxes were acceptable. The R2NOC1 precipitate was added back into the CUF feed tank and filtered again. The flux again dropped quickly, from an initial 0.047 to 0.019 gpm/ft^2 after only 30 minutes.

At this time, SRTC and Project personnel discussed possible paths forward. Two potential causes for the low flux results were proposed: 1) the delay in beginning filtration (aging); 2) the less than “vigorous” agitation. Therefore, a small (5L) batch precipitation (R2NOC small) was performed with vigorous mixing and no time delay between the end of the precipitation and the filtration. The flux measured ranged from 0.046 to 0.067 gpm/ft^2 at TMPs of 40 and 50 psi and a velocity of 12 fps. Based on these encouraging results, a large batch precipitation was planned.

The next precipitation (R2NOC2) was a repeat of R2NOC1. Vigorous agitation was maintained by adding baffles and operating the propeller agitator at a higher speed (220 rpm versus the previous approximately160 rpm or less). The quantities of reagents added were the same as for R2NOC1, with adjustments for the reagent concentrations. Immediately upon completion of the precipitation four hour hold time, filtration was begun. The initial flux was 0.119 gpm/ft^2, which was acceptable. However, the flux dropped very quickly to less than 0.03 gpm/ft^2, and was less than 0.02 gpm/ft^2 at 23 hours of processing. This batch was concentrated to a final insoluble solids concentration of 18.5 wt%. The time-averaged flux was 0.013 gpm/ft^2. A parallel precipitation and filtration was conducted in the pilot unit. The results from this run were even worse than the CUF results; within several hours, the flux had dropped below the minimum.

3.1.3 Simulant Remediation 2 and Baseline Conditions

The poor results for R2NOC2 prompted Project personnel to go back and try the baseline conditions with the R2 remediation. An 880L precipitation was performed at the EDL with the following conditions:

1. Caustic added to give 1.0M free hydroxide before Sr and Mn additions (0.8M added).
2. Sr added to 0.075M
3. Mn added to 0.050M
4. Temperature of 50°C

About 41L of precipitate was then processed in the CUF. The initial average flux measured over the collection of the first 3L of permeate was 0.192 gpm/ft^2. Extrapolation of the flux measurements back to the starting time gives an estimated initial flux of 0.25 gpm/ft^2. This material was concentrated to about 18.8 wt% in about 14 hours. The final flux was 0.018 gpm/ft^2 and the time-averaged flux was 0.060 gpm/ft^2.

3.2 RESULTS

3.2.1 Simulant Formulations

The initial experiments for this work used the WTP verbally approved simulant for the supernate and solids. Subsequent work on the supernate simulant formulation resulted in changes to the formulation. Optimization work on the Sr/TRU precipitation led the WTP to request additional filtration work be done with the updated supernate simulant and the "newly optimized conditions" Sr/TRU precipitation.

3.2.1.1 Simulant Remediation 1

The simulant formulations used for this work are summarized in Table 2 and Table 3.[6] The simulant recipe on a per liter basis is shown in the Appendix. The supernate simulant used was a remediation of simulant purchased from Optima Chemicals. The remediation calculations were based on the recipe for the purchased simulant rather than on analytical measurements. Table 3 shows the predicted composition of a precipitated simulant batch and the measured composition of the first batch. This prediction is based on the simulant recipes, the amounts of precipitating reagents used, and assumed precipitation reactions. The elemental compositions were determined from a sodium peroxide fusion/ HCl uptake and aqua regia dissolution.

The raw analytical data was generally about 10% lower than the expected values for the elemental, IC, and carbon analyses, which suggests that the sample may have been more dilute than expected. However, the solids analyses matched well. To compare on a relative basis, the measured values were normalized so that the measured Na concentration equaled the calculated value. Good agreement was then found for Al, Ca, Ni, Cd, Cr, NO_3^-, NO_2^-, Cl^-, SO_4^{-2}, and formate. Both Mn and Sr were 15-20% lower than expected based on the reagent additions. Phosphorus and phosphate were low by about 33 and 24%, which indicates that the phosphate was precipitated from the simulant <u>prior</u> to the precipitation. These concentrations could be low due to formation of insoluble sodium fluorophosphate ($Na_7F(PO_4)_2$) that was identified as a precipitate during simulant development.[6] The fluoride concentration was high by about 33%, but this value is inconsistent with the calculated value, values from later simulant remediations (see Table 4), and permeate compositions (see Table 11). The fluoride concentrations measured in the permeates tend to indicate that the fluoride content of the simulant may have actually been less than the calculated value, which would be consistent with the formation of the fluorophosphate. If phosphate and fluoride precipitated from the simulant and were not quantitatively transferred with the bulk of the liquid, low values would result.

Iron was about six times higher than expected. Potassium measurements on dissolved samples by ICPES were unreliable, so the measured value should be discarded. The test specification called for potassium analysis by ICPES, but SRTC would recommend for future work that it be measured by atomic absorption (AA). The higher than expected barium concentration is probably due to a contamination problem in the preparation of the samples for analysis; higher than expected values have been seen in other samples in other programs. The high value for oxalate is probably the result of the permanganate oxidation of the organic materials; oxalate is a likely product of these reactions. Duplicate analyses for total and insoluble solids were performed. The calculated insoluble solids (IS) in the table was based on the assumption that all added Sr formed $SrCO_3$ and all $NaMnO_4$ formed MnO_2. This assumption should give an approximate upper bound for the amount of anhydrous solids formed during the precipitation; hydration of the precipitated material and also the initial insoluble solids may account for the measured values (1.60, 1.88 wt%) being higher than the calculated value (1.45 wt%). The formation of heavier Sr compounds such as $SrHPO_4$, $SrFPO_4$, or $SrSO_4$ would be consistent with the higher measured solids values. However, no measurements were made to determine if these compounds actually were formed.

The solids simulant composition is shown in Table 2. The solids simulant was added to the supernate simulant to give 0.1 wt% of solids simulant. Some of the solids were purchased material and most of the oxalates and the FeOOH were made in the laboratory from reagent grade chemicals. This solids simulant formulation was used for all of the runs.

Table 2 Approved Solids Simulant Formulation

Solid	g/100 g solids	Source
Al_2O_3	15.12	Reagent
$BaSO_4$	0.02	$Ba(NO_3)_2 + Na_2SO_4$
$CaC_2O_4.H_2O$	0.13	$Ca(NO_3)_2 + Na_2C_2O_4$
$CaWO_4$	0.11	Reagent
$Na_2CO_3.H_2O$	42.71	Reagent
$Ce_2(C_2O_4)_3.9H_2O$	0.02	$Ce(NO_3)_4 + Na_2C_2O_4$
Cr_2O_3	0.93	Reagent
NaF	3.15	Reagent
FeOOH	0.68	$Fe(NO_3)_3$ + NaOH
$La_2(C_2O_4)_3.9H_2O$	0.02	$La(NO_3)_3 + Na_2C_2O_4$
$PbSO_4$	0.08	Reagent
MnO_2	0.15	Reagent
$Nd_2(C_2O_4)_3.10H_2O$	0.04	$Nd(NO_3)_3 + Na_2C_2O_4$
NiO	0.01	Reagent
$Na_2C_2O_4$	16.1	Reagent
$Na_3PO_4.12H_2O$	12.28	Reagent
SiO_2	0.05	Reagent
Na_2SO_4	8.35	Reagent
$ZnC_2O_4.2H_2O$	0.02	Reagent
ZrO_2	0.02	Reagent
	99.99	

3.2.1.2 Simulant Remediation 2

The R2 remediation formulation is given in the Appendix. Several measurements of the composition of this remediated material were made by the EDL and are shown in Table 4. Remediation batch “3C” from the EDL was used for the R2NOC CUF runs (and also R2BC small). Batch “3B” was used for the R2BCL CUF run.

Table 3 Calculated and Measured Composition of R1BC Precipitate Slurry Feed Batch #1

		Calculated from Batching	Measured	Measured / Calculated
ICPES (mg/L)	Al	7670	7540	0.98
	B	22.9	<92	<4.02
	Ba	0.1	189	1630
	Ca	306	306	1.00
	Cd	37.9	36.1	0.95
	Co	2.1	<35	<16.8
	Cr	160	162	1.02
	Cu	15.1	<35	<2.32
	Fe	30.2	196	6.50
	Li	0	<47	<47.0
	Mg	0	<41	NA
	Mn	2770	2320	0.84
	Mo	28.1	<69	<2.46
	Na	135000	135000	1.00
	Ni	260	270	1.04
	P	1140	768	0.67
	Pb	116	<302	<2.61
	Si	6.5	<69	<10.7
	Sn	0	<163	NA
	Sr	6570	5360	0.81
	Ti	0	<69	NA
	V	0	<69	NA
	Zn	3.2	<163	<50.9
	Zr	8.5	<23	<2.72
	La	9.9	<324	<32.6
	K	1270	293	0.23
	S	2640	NM	NA
	Nd	19.9	<232	<11.7
	Ce	0.1	NM	NA
	Cs	9.9	NM	NA
	Rb	5.2	NM	NA
	W	105	NM	NA
Ion Chrom. (mg/L)	NO_2^-	48100	50900	1.06
	NO_3^-	137000	134000	0.98
	Cl-	2980	3030	1.02
	F-	1080	1440	1.33
	PO_4^{-3}	3500	2680	0.76
	SO_4^{-2}	7910	7590	0.96
	formate	5240	5770	1.10
	oxalate	397	1960	4.94
Carbon (mg/L)	Total Organic Carbon	NC	6220	NA
	Total Inorganic Carbon	NC	4950	NA
	Total Carbon	NC	11100	NA
	Total Solids (wt%)	34.1-34.5	33.1, 33.6	0.96-0.99
	Insoluble Solids (wt%)	1.45	1.60, 1.88	0.77-0.91

NM: not measured NA: not available NC: not calculated
<: measurement below detection limit

Table 4 Composition of Batches 3B and 3C Simulant

		Batch 3C	Batch 3B
ICPES (mg/L)	Al	9110	9880
	B	39.1	37.5
	Ba	<0.01	0.45
	Ca	395	418
	Cd	38.5	33.8
	Ce	28.7	28.9
	Co	1.11	1.76
	Cr	154	158
	Cu	7.3	7.1
	Fe	33.4	27.8
	K	2530	3180
	La	26.7	21.9
	Mg	0.53	1.48
	Mn	18.6	14.6
	Mo	31.6	29.8
	Na	134000	140000
	Nd	33.4	47.1
	Ni	258	272
	P	748	643
	Pb	68.3	58.4
	S	3200	3550
	Sr	0.52	5.35
	Si	42.6	37.7
	W	131	142
	Zn	4.9	4.0
	Zr	9.2	7.0
Ion Chrom. (mg/L)	NO_2^-	54500	54500
	NO_3^-	137000	137000
	Cl-	3700	3690
	F-	921	920
	PO_4^{-3}	1970	1970
	SO_4^{-2}	10400	10300
	formate	7350	7340
	oxalate	343	<1000
	Free OH- (M)	0.228	0.203
	Total Base (M)	1.61	1.70
	Specific Gravity	1.32	1.32

<: measurement below detection limit

3.2.1.3 Precipitations

The batching calculations for the precipitation reactions are summarized in Table 5. A total of nine precipitation batches were prepared. The initial volume of a precipitation batch was 5.0L. As discussed in Section 1.2.1, the added hydroxide concentration target used was 0.9M rather than 1.0M. In addition, an error in batching the NaOH was made. The added NaOH was based on increasing the hydroxide concentration from zero M to 0.9M, but more accurately should have been based on increasing it from 0.3M to 1.2M (0.9M added). The initial free hydroxide concentration was assumed to be about 0.3M, based on the formulation. Free hydroxide concentration in the simulant was not measured, so the final free hydroxide concentration is only an estimate. Table 6 shows a comparison of the actual and target reagent use. These errors introduced by the calculations should be smaller than the uncertainty in the initial hydroxide concentration. The final calculated free hydroxide concentration was 1.13M versus the target of 1.20M; the correct final value would be 1.3M based on 1M added hydroxide. The concentrations of Sr, Mn, and Na were correct to two decimal places. The final volume of the precipitate was about 6.5 liters.

Table 5 Summary of Reagents for R1BC (per 5L of Simulant)

Initial Volume (L)	5.0	Final Na (M)	5.93
Initial Na (M)	6.53	Final OH (M)	1.13 **
Estimated Initial OH- (M)	0.300	Final Sr (M)	0.075
$Sr(NO_3)_2$ reagent (M)	1.00	Final NaMnO4 (M)	0.050
$NaMnO_4$ reagent (M)	1.128		
NaOH reagent (M)	19.17	Water added (mL)	439.2
		NaOH added (mL)	306.4
Target Na after water addition (M)	6.00	$Sr(NO_3)_2$ added (mL)	489.3
Target final OH- (M)	1.20	$NaMnO_4$ added (mL)	289.2
Target final Sr (M)	0.075	Total volume added (mL)	1524.1
Target final NaMnO4 (M)	0.050		
		Final volume (L)	6.55
Actual Na after water addition (M)	6.00		
		Estimated density (kg/L)	1.268
		Estimated Total Solids (wt%)	34.07

** Target was 1.20; value low due to calculation error.

One precipitate batch was allowed to settle overnight. The volume of the settled solids was about 30% of the total volume. The liquid above the solids was very clear. If the liquid above the settled slurry solids was decanted, an immediate concentration of the slurry from about 1.5 wt% insoluble solids to about 4.5 wt% could have been achieved. The decanted liquid should filter essentially like clear permeate would. A movie of the re-suspension of the settled batch is contained in the report CD-ROM (“Slurry-Resuspension.mpg”).

During several of the precipitations, evolution of ammonia gas was noted by its odor. Later work on precipitation of a 115L batch confirmed that ammonia was formed. Ammonia is likely formed from the oxidation by the permanganate of the disodium ethylenediamine tetraacetate (NaEDTA), n-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), and ammonium acetate. The odor was quite strong, but it only takes about 10 ppm of ammonia to be detected by its odor. Since the amount of ammonia generated was not determined, the WTP may want to consider quantifying the amount formed or determining an upper bound for its formation. Ammonia is flammable, with a lower flammable limit of about 15% and an upper flammable limit of about 25%. By itself, ammonia is rarely flammable due to its high auto-ignition temperature of about 650°C. However, in the presence of hydrogen from radiolysis, it may be a concern. Alternatively, the evolution of ammonia into the WTP vent system may need to be accounted for to meet environmental release limits or for permitting.

Table 6 Precipitation Reagent Quantities for R1BC

	Correct Batching	Batching Calculation Used	Actual Batching Used
Final Na (M)	6.00	5.95	5.95
Final OH (M)	**1.20**	0.900	**1.13**
Final Sr (M)	—	0.0750	—
Final NaMnO4 (M)	—	0.0500	—
Water added (mL)	439.2	439.2	439.2
NaOH added (mL)	**332.0**	306.4	**306.4**
$Sr(NO_3)_2$ added (mL)	**491.5**	489.3	**489.3**
$NaMnO_4$ added (mL)	**290.5**	289.2	**289.2**
Final volume, estimated (L)	6.55	6.52	6.52

The R2NOC runs all used the same precipitation recipe, as shown in Table 7. The Batch 3C simulant for from the EDL contained about 0.23M hydroxide before dilution and entrained solids addition.

Table 7 Summary of Reagents for RNOC1 and R2NOC2

Initial Volume (L)	115	Final Na (M)	5.65
Initial Na (M)	6.53	Final OH (M)	0.258
Estimated Initial OH- (M)	0.300	Final Sr (M)	0.030
$Sr(NO_3)_2$ reagent (M)	1.00	Final NaMnO4 (M)	0.030
$NaMnO_4$ reagent (M)	0.976		
NaOH reagent (M)	19.17	Water added (L)	10.10
		NaOH added (L)	0
Target Na after water addition (M)	6.00	$Sr(NO_3)_2$ added (L)	4.01
Target final OH- (M)	NA	$NaMnO_4$ added (L)	4.11
Target final Sr (M)	0.030	Total volume added (L)	18.21
Target final NaMnO4 (M)	0.030		
		Final volume (L)	133.6
Actual Na after water addition (M)	6.00		
		Estimated density (kg/L)	1.270
		Estimated Total Solids (wt%)	34.26

The R2BCL run precipitation was performed by the EDL on approximately 880L of Batch 3B simulant. The precipitation recipe is shown in Table 8.

Table 8 Summary of Reagents for R2BCL

Initial Volume (L)	880	Final Na (M)	5.94
Initial Na (M)	6.0	Final OH (M)	0.866
Estimated Initial OH- (M)	0.20	Final Sr (M)	0.030
$Sr(NO_3)_2$ reagent (M)	1.00	Final NaMnO4 (M)	0.030
$NaMnO_4$ reagent (M)	1.00	Water added (L)	0
NaOH added as solid	NA	NaOH added (kg)	28200
		$Sr(NO_3)_2$ added (L)	75.43
Target final OH- (M) (prior to Sr & Mn additions)	1.00	$NaMnO_4$ added (L)	49.06
Target final Sr (M)	0.075	Final volume (L)	1006
Target final NaMnO4 (M)	0.050		
		Estimated density (kg/L)	1.30
		Estimated Total Solids (wt%)	37.0

3.2.2 Concentration of Precipitated Simulants – Composition and Properties

3.2.2.1 Remediation 1 – Baseline Conditions

The Sr/TRU precipitates from precipitation batches #1 and #5 were analyzed for rheology, total solids, suspended solids, and particle size distribution. Batch #1 was also analyzed for elements, anions, and carbon as previously described. The major difference between batch #1 and batch #5 is that batch #1 had been subjected to the entire factorial matrix of filtration tests, whereas batch #5 was fresh material from the precipitator. A comparison of the solids concentrations is shown in Table 9.

Table 9 Solids Content of R1BC Precipitate Batches and Concentrated Slurry

	Total Solids (wt%)	Insoluble Solids (wt%)			
Simulant (Supernate + Solids)	not meas.	0.10 from batching	0.31 measured	0.49 calc.	0.85 calc.
Calculated Precipitate Batch	34.5	1.29	1.45		
Measured Precipitate Batch #5 (fresh precipitate)	33.6			1.60	
Measured Precipitate Batch #1 (subjected to filtration)	33.1				1.88
Measured Concentrated Slurry	42.1	13.33			
Measured Concentrated Slurry	45.0	17.17			
Measured Washed Concentrated Slurry	35.1	20.63			

The estimated and measured total solids concentrations agree well. There are four columns for insoluble solids. The first shows that the expected insoluble solids in the precipitate batch was 1.29 wt% which is based on the addition of 0.1 wt% sludge solids to the supernate simulant. However, the supernate simulant actually contained some insoluble solids; the measured value was 0.31 wt%, which results in a calculated insoluble solids after precipitation of 1.45 wt%. The measured insoluble solids in the precipitate (1.60, 1.88 wt%)

were used to back-calculate the incoming insoluble solids concentrations, which were 0.49 and 0.85 wt%. Based on these data, a reasonable estimate of the insoluble solids concentration in the simulant is 0.31-0.49 wt% and in the precipitate it is 1.45-1.60 wt%.

To determine if the measured total and insoluble solids contents of the concentrated slurries were consistent, they were checked using the starting total and insoluble solids concentrations in the precipitate batches and calculating the increase in solids as these precipitate feed batches were added and concentrated. Based on a precipitate insoluble solids content of 1.60 wt%, the volume and total solids content of the concentrated slurry was calculated for insoluble solids concentrations of 13.33 and 17.17 wt%. This same calculation was also performed for the washing and re-concentration step. The results of these calculations are shown in Table 10. The total solids concentrations and concentrated slurry volumes calculated match the measured values very well, so the data is consistent.

Note that the initial goal for the dewatering step was 15 wt% insoluble solids, but the actual value reached initially was about 19.3 wt%. The overshoot occurred because of the assumption that the precipitate feed batches would be approximately 1 wt% rather than 1.45-1.60 wt%. After it was apparent that the solids concentration was higher than expected, the slurry was diluted with permeate back to 17.2 wt% (the goal here was again 15 wt%, but the actual wt% insoluble solids was not known, so the dilution was based on an estimate).

Table 10 R1BC Measured and Calculated Solids Content and Volume

		Typical Batch **	Nine Batches Concentrated to 13.3 wt% IS		Concentrated to 17.2 wt% IS		Wash Water Added	Concentrated to 20.63 wt% IS	
			Calculated	Measured	Calculated	Measured		Calculated	Measured
Volume	L	6.55	6.44	approx. 6.5	4.69	approx. 4.6	9.19	4.26	approx. 4.2
Estimated Density	kg/L	1.27	1.36		1.40		1.19	1.28	
Insoluble Solids	g	130	1169	**	1124		1124	1124	
Total Solids	g	2778	3677		2912		2912	1903	
Total Mass	g	8153	8770		6544		11044	5446	
Insoluble Solids	wt%	1.60	13.33	13.33	17.17	17.17	10.17	20.63	20.63
Total Solids	wt%	34.07	41.93	42.14	44.50	44.98	26.37	34.94	35.06

** 0.125 to 0.250 L samples removed from each batch

Permeate samples were taken during the initial factorial experiments (at 1.45-1.60 wt% insoluble solids), during concentration at approximately 13.3 and 17.2 wt% insoluble solids, and during concentration of the washed slurry. The compositions of the permeate samples (except the washed slurry) should be essentially identical and the soluble components should be similar to the precipitate composition. The permeate concentrations are summarized in Table 11. To within the accuracy of the measurements, the permeate composition in R1BC did not change as the slurry was concentrated.

Table 11 R1BC Permeate Compositions During Concentration

		Measured			Measured	
		Batch 1	At Approx. 13 wt% Insoluble Solids in Slurry	At Approx. 17 wt% Insoluble Solids in Slurry	Mean	2 * Standard Deviation
ICPES (mg/L)	Al	7350	7540	7700	7530	350
	B	22.4	23.5	24.2	23.4	1.9
	Ba	<0.02	<0.02	<0.02	<0.02	NA
	Ca	91.7	95.8	102	96.5	10.4
	Cd	34.5	35.6	38.1	36.1	3.7
	Co	<0.1	<0.1	<0.1	<0.1	NA
	Cr	149	153	137	146	16.7
	Cu	3.5	3.7	4.15	3.8	0.7
	Fe	0.5	0.5	0.6	0.5	0.2
	Li	0.2	0.3	0.2	0.2	0.04
	Mg	<0.2	<0.2	<0.2	<0.2	NA
	Mn	0.2	0.3	0.5	0.3	0.3
	Mo	28.4	28.5	29.0	28.6	0.7
	Na	133000	138000	142000	138000	9020
	Ni	195	200	201	199	6.4
	P	573	577	553	568	25.7
	Pb	36.2	39.9	42.1	39.4	5.9
	Si	15.3	15.2	16.2	15.5	1.1
	Sn	<0.7	<0.7	<0.7	<0.7	NA
	Sr	28.0	26.1	26.4	26.8	2.0
	Ti	<0.3	<0.3	<0.3	<0.3	NA
	V	<0.3	<0.3	<0.3	<0.3	NA
	Zn	3.0	2.2	2.4	2.5	0.8
	Zr	0.5	0.4	0.5	0.5	0.10
	La	<1.4	<1.4	<1.4	<1.4	NA
	K	1770	2150	2380	2100	616
	S	2750	2820	2910	2830	160
	Nd	0.6	1.2	0.8	0.9	0.6
	Ce, Cs, Rb, W	Not Measured				
Ion Chrom.	NO_2^-	44700	46100	41100	44000	5159
	NO_3^-	126000	129000	124000	126300	5033
	Cl^-	2770	2790	2770	2777	23
	F^-	733	732	672	712	70
	SO_4^{-2}	7080	7400	6540	7007	260
	PO_4^{-3}	2360	2240	2500	2367	869
	formate	4880	4820	4900	4867	83
	oxalate	702	712	523	679	98

The permeate samples from the concentration of the unwashed R1BC slurry were all initially yellowish in color, with no discernable orange or red tint. The permeates produced from the R2NOC precipitates all were much more orange-red in tint and were much darker. The R2BC permeates were yellowish like R1, but a with little bit of an orange tint.

After about 2-4 days, the color of the R1BC permeate changed to a greenish tint. The other permeates also showed similar color changes, but the greenish color was not as intense. For all samples, within about one week, a dark brown precipitate coated the bottle surfaces, but the permeate liquid remained transparent. Table 12 shows the turbidity measured for several permeate samples. The samples were not shaken before measurements were made because the initial permeates were transparent and shaking would have released the post-precipitated solids from the bottle surfaces. Measurement of the turbidity immediately after filtration would have been ideal, but was not possible. All permeate samples had turbidities of less than 4 NTU, even after standing. At SRS, turbidities of less than 5 NTU are considered to indicate that no breakthrough of particles has occurred in the filter.

Table 12 Turbidity of Permeates

Run		Turbidity (NTU)
R1BC	Permeate from Batch #1	<1
	Permeate at 13.3 wt% IS	1.34
	Permeate at 17.2 wt% IS	3.80
	Permeate from Washed Slurry	1.80
R2NOC1	Initial Permeate	0.09
R2NOC2	Initial Permeate	0.40
	Intermediate Permeate	0.86
	Intermediate Permeate	1.00
	Permeate at 18.4 wt% IS	1.69
R2BCL	Initial Permeate	0.35
	Permeate at 14.5 wt% IS	0.30
	Permeate at 18.8 wt% IS	0.36

3.2.2.2 Remediation 2 – Newly Optimized Conditions Run 2 (R2NOC2)

The R2NOC2 precipitate was concentrated from an initial 1.32 wt% insoluble solids to a final 18.48 wt%. Table 13 summarizes the measured and calculated solids concentrations and specific gravities.

Table 13 Summary of R2NOC2 Solids Contents

	Total Solids (wt%)	Insoluble Solids (wt%)	Specific Gravity	Volume in Feed Tank (L)
Simulant: Measured	35.87	0.2-0.62	NM	
Simulant: Calculated	36.98	0.86	1.30	
Precipitate: Measured	33.60	1.32	1.26	
Precipitate: Calculated *	33.6	1.32	1.26	
Concentrated Slurry: Measured	42.50	12.30	1.37	approx. 6
Concentrated Slurry: Calculated	40.99	12.30 *	1.35	6.00
Concentrated Slurry: Measured	47.36	18.48	NM	approx. 4
Concentrated Slurry: Calculated	45.15	18.48 *	1.41	3.84

* Calculated set equal to measured. NM = not measured.

In the calculations, the initial measured precipitate solids contents were used to predict the concentrated slurry. The calculated total solids for the concentrated slurries were 1-2 wt% low when the calculated insoluble solids concentrations were set to equal the measured values. The volume calculated for the final slurry (3.84L) matches the observed approximately 4L value.

The measured compositions of the precipitate slurry and permeate generated are given in Table 14. The precipitate from R2NOC1 is also shown. Elemental analysis measurements from the peroxide fusion (Na_2O_2) and aqua regia dissolutions agree well.

Table 14 R2NOC Slurry and Permeate Compositions

		Slurries				Permeate
		R2NOC1		R2NOC2		R2NOC2
	Dissolution:	Na_2O_2	Aqua Regia	Na_2O_2	Aqua Regia	
ICPES (mg/L)	Al	8880	8810	8850	86010	8980
	B	66.7	74.2	<93	<38	28
	Ba	<6	<6	40.3	19.9	<0.02
	Ca	508	376	1110	296	89
	Cd	42.4	43.4	46.6	44.4	41
	Co	<13	<13	<21	<13	<0.2
	Cr	308	186	195	152	146
	Cu	22.2	21.0	<21	16.2	7
	Fe	531	49.7	202	77.7	3
	Li	<26	<26	<42	<25	<0.2
	Mg	18.0	19.9	<36	19.8	<0.2
	Mn	1680	1730	1930	1800	12
	Mo	42.6	29.2	41.2	34.6	35
	Na	NA	134000	NA	129000	136000
	Ni	391	305	341	307	275
	P	2790	2250	1390	1040	775
	Pb	<195	<195	<296	<252	47
	Si	48.9	<26	149	27.0	27
	Sn	<65	<65	<127	<63	1
	Sr	2290	2730	3750	2670	23
	Ti	<26	<26	<42	<25	<0.3
	V	<26	<26	78.7	<25	<0.3
	Zn	<65	<65	<106	<63	2
	Zr	NA	<130	NA	<10	3
	La	<130	<130	<296	<126	3
	K	NM	1640	1540	1370	1840
	S	NM	NM	NM	NM	3180
	Nd	<65	<65	137	81.8	6

NM: not measured NA: not available <: measurement below detection limit

Table 14 R2NOC Slurry and Permeate Compositions (continued)

	R2NOC1 Slurry Duplicate Samples		R2NOC2 Slurry		Permeate R2NOC2
Ion Chrom (mg/L) NO_2-	54400	63300	48500		50500
NO_3^-	138000	160000	116000		124000
Cl^-	3550	4340	3140		3150
F^-	1180	784	1220		1180
PO_4^{-3}	4160	4890	3650		3310
SO_4^{-2}	8560	11400	8300		8460
formate	6290	6130	5680		5860
oxalate	1600	1830	1590		1020
Carbon (mg/L) TOC	10500	10100	NM		NM
TIC	7370	9610	NM		NM
TC	17900	19700	NM		NM
Carbonate (M)	0.55		NM		NM
Carbonate (mg/L)	33000		NM		NM
Free OH^- (M)	0.15		NM		NM
Total Base (M)	1.50		NM		NM
Total Solids (wt%)	33.6		33.2		35.43
Insoluble Solids (wt%)	1.32		0.74-1.33		0
Specific Gravity	1.26		1.27		NM

NM: not measured

3.2.2.3 Remediation 2 – Baseline Conditions

The solids contents and specific gravities for the R2BCL run are shown in Table 15. The agreement between the values calculated by material balance match the measured values reasonably well, except for the total solids concentrations. The calculated values are substantially higher than the measured values. These calculated values were determined by a material balance on the volumes of precipitate added and permeate removed. Note that for this type of calculation, the cumulative errors build up since the permeate collected was measured incrementally. The composition of the R2BCL precipitate slurry is shown in Table 16. The permeate composition was not measured.

Table 15 Summary of R2BCL Solids Contents

	Total Solids (wt%)	Insoluble Solids (wt%)	Specific Gravity	Volume in Feed Tank (L)
Simulant: Measured	NM	NM	1.32	
Simulant: Calculated	0.30	37.0	1.31	
Precipitate: Measured	32.6-33.7	1.3-1.6	1.26-1.28	
Precipitate: Calculated	33.8	1.60*	1.265	
Concentrated Slurry: Measured	42.93	14.85	1.38	approx. 4
Concentrated Slurry: Calculated	46.71	14.51	1.43	3.92
Concentrated Slurry: Measured	45.43	18.83	NA	approx. 3
Concentrated Slurry: Calculated	51.36	19.15	1.49	2.92

* Calculated set equal to measured. NM = not measured

Table 16 Composition of R2BCL Precipitate and Concentrated Slurry

		Initial Precipitate	Concentrated Slurry	
Dissolution:		Aqua Regia	Na_2O_2	Aqua Regia
ICPES (mg/L)	Al	7870	9220	8340
	B	27.2	<169	24.0
	Ba	34.5	616	567
	Ca	483	4267	3490
	Cd	22.1	68.7	71.5
	Co	<1.0	<35	30.9
	Cr	122	275	214
	Cu	17.4	190	186
	Fe	32.8	781	630
	Li	NM	<70	<4
	Mg	<0.1	187	204
	Mn	2420	36800	34800
	Mo	NM	<70	30.9
	Na	138000	NM	154000
	Na (by AA)	NM	NM	148000
	Ni	214	875	800
	P	874	5060	6230
	Pb	92.6	1440	1240
	Si	33.7	236	1630
	Sn	NM	<197	<10
	Sr	7090	74600	80300
	Ti	NM	<99	6.24
	V	NM	<99	<5
	Zn	<0.1	<282	28.6
	Zr	2.8	NA	101
	La	24.6	<493	318
	K	2730	1580	1380
	K (by AA)	NM	NA	1260
	S	2460	3160	2900
	Nd	42.4	614	600
Ion Chrom (mg/L)	NO_2^-	38200	37500	
	NO_3^-	105000	98300	
	Cl^-	2650	2800	
	F^-	1020	2210	
	PO_4^{-3}	2800	17200	
	SO_4^{-2}	7570	7270	
	formate	5340	5820	
	oxalate	1400	6200	
Carbon (mg/L)	TOC	NM	9300	
	TIC	NM	12000	
	TC	NM	21300	
Total Solids (wt%)		32.66-33.65	45.43	
Insoluble Solids (wt%)		1.60	18.83	
Specific Gravity		1.28	1.41 (estimated)	

NM: not measured NA: not available <: measurement below detection limit

3.2.3 R1BC Washing and Re-Concentration

The slurry at 17.2 wt% insoluble solids was washed with an equal volume (not mass) of inhibited water and then re-concentrated to the original volume. By material balance, the estimated and measured total solids content and the slurry volume agreed very well when calculated assuming the measured insoluble solids content, as shown in Table 10. Table 17 shows comparisons of the compositions of the unwashed and washed slurries and permeates. The soluble components Al, Na, NO_2^-, NO_3^-, Cl-, F-, PO_4^{-3}, SO_4^{-2}, and formate in the washed slurry were approximately one-half the unwashed slurry as expected; the same ratio is found for the permeate. Note that the insoluble solids content of the washed slurry is greater than before washing. This occurred since the wash was on a volumetric basis, whereas the insoluble solids content is on a mass basis.

3.2.4 Rheological Measurements

All rheological measurements shown in Table 18 were taken at 25°C and the flow curves were not corrected for non-Newtonian behavior (slip, geometry, etc.), if applicable, and are consistent with how data is interpreted for RPP-WTP related reports. For the slurry samples, the mean results of the up curve would typically be used for startup of pipe flow or agitator startup and the mean results of the down curve would be used for typical steady pipe flow calculations. The shear rate range for the Newtonian samples was 0-500 or 0-550 s^{-1} and for the non-Newtonian samples it was 50-1000 s^{-1}.

These results show that the initial precipitate slurries and permeates were Newtonian, while the concentrated slurries were all non-Newtonian. Figure 4 shows the slurry consistencies plotted versus the insoluble solids content. The data for the baseline conditions slurry fall roughly along a line, but the NOC2 slurry has a higher consistency at about 12wt% and a much higher value at approximately 18wt%. The washed R1BC slurry consistency is similar to the unwashed slurry. The yield stresses of the slurries are plotted in Figure 5. The yield stresses are relatively linear with insoluble solids concentration up to about 16-18wt%, but then increase dramatically above 18wt%. The NOC2 slurry also had a much higher yield stress at approximately 18wt% than the baseline condition slurries did.

Table 17 R1BC Composition of Unwashed and Washed Slurry and Permeate

		Feed Slurry @ 1.45-1.60 wt% Insoluble Solids (Measured)	Unwashed Slurry @ 17.2 wt% Insoluble Solids (Calculated)	Washed Slurry @ 20.63 wt% Insoluble Solids (Measured)	Average Permeate from Unwashed Slurry (Measured)	Permeate from Washed Slurry (Measured)
ICPES (mg/L)	Al	7540	7790	4730	7530	3510
	B	<92	<100	<90	23.4	11.3
	Ba	198	2440	2440	<0.024	<0.02
	Ca	306	3090	3040	96.5	50.5
	Cd	36.1	61.6	45.3	36.1	18.8
	Co	<35	<34	<34	<0.1	<0.1
	Cr	162	311	251	146	69.6
	Cu	<35	174	174	3.8	0.4
	Fe	196	673	673	0.5	<0.1
	Li	<47	<45	<45	0.2	<0.2
	Mg	<41	235	234	<0.2	<0.2
	Mn	2320	37300	37300	0.3	0.7
	Mo	<69	<79	<67	28.6	13.9
	Na	135000	134000	76300	138000	66800
	Ni	270	1030	958	199	85.3
	P	768	1610	1290	568	372
	Pb	<302	1030	1030	39.4	10.3
	Si	<69	<74	<67	15.5	7.7
	Sn	<163	<157	<157	<0.7	<0.5
	Sr	5360	85500	85500	26.8	10.1
	Ti	<69	<67	<67	<0.3	<0.3
	V	<69	<67	<67	<0.3	<0.3
	Zn	<163	<158	<157	2.5	<0.7
	Zr	<23	143	143	0.5	0.2
	La	<324	<315	<314	<1.4	<1.4
	K	293	<957	<224	2100	843
	S		1240	NM	2830	1430
	Nd	<232	<225	<224	0.9	1.68
Ion Chrom. (mg/L)	NO_2^-	50900	37000	18200	44000	21700
	NO_3^-	134000	108000	57600	126000	57700
	Cl-	3030	2280	1134	2780	1320
	F-	1440	983	467	712	593
	PO_4^{-3}	2680	2280	1090	2370	1370
	SO_4^{-2}	7590	6970	3620	7010	3850
	formate	5770	5750	3220	4870	2900
	oxalate	1960	13200	10900	679	2610
Carbon (mg/L)						
	Total Organic Carbon	6220	5550	2980	NM	2960
	Total Inorganic Carbon	4950	5160	3340	NM	2090
	Total Carbon	11100	10700	6320	NM	5050
	Total Solids (wt%)	33.61	44.98	35.06	33.30	33.61
	Insoluble Solids (wt%)	1.60	17.17	20.63	NA	NA
	Estimated Specific Gravity	1.26	1.40	1.28	1.26	1.26

NM: not measured NA: not available <: measurement below detection limit

Table 18 Rheological Properties of Initial Precipitates, Concentrated Slurries, and Permeates

Batch	Sample	Insoluble Solids (wt%)	Mean Viscosity$^{\#\#}$ or Consistency$^{\#}$ Up (cp)	Mean Viscosity$^{\#\#}$ or Consistency$^{\#}$ Down (cp)	Mean Yield Stress Up (Pa)	Mean Yield Stress Down (Pa)	Fluid Behavior
R1BC	Precipitate Batch #5 (fresh precipitate)	1.45-1.60	3.84	3.55	none	none	Newtonian
R2BCL	Precipitate Batch (fresh precipitate)	1.30-1.60	4.22	4.22	none	none	Newtonian
R1BC	Precipitate Batch #1 (subjected to filtration)	1.45-1.60	4.08	3.72	none	none	Newtonian
R1BC	Concentrated Slurry*	13.3	11.7	11.8	4.94	3.60	non-Newtonian
	Concentrated Slurry*	17.2	14.6	16.8	15.3	10.3	non-Newtonian
	Concentrated Slurry**	19.3	15.2	20.7	23.2	14.5	non-Newtonian
	Washed Concentrated Slurry*	20.6	8.4	15.7	61.7	51.5	non-Newtonian
R2BCL	Concentrated Slurry*	14.8	11.6	12.1	4.14	2.72	non-Newtonian
	Concentrated Slurry*	18.8	16.6	18.2	12.83	9.82	non-Newtonian
R2NOC2	Concentrated Slurry*	12.3	16.3	20.9	11.89	4.78	non-Newtonian
	Concentrated Slurry*	18.5	43.3	64.2	65.2	33.5	non-Newtonian
R1BC	Permeate	none	3.10	NA	none	NA	Newtonian
	Washed Slurry Permeate	none	1.55	NA	none	NA	Newtonian

* Haake Z41 Double Concentric Geometry

** Haake 35mm, 2° Cone Geometry

\# Consistency for concentrated slurry samples

\#\# Viscosity for dilute precipitate samples

NA: not applicable

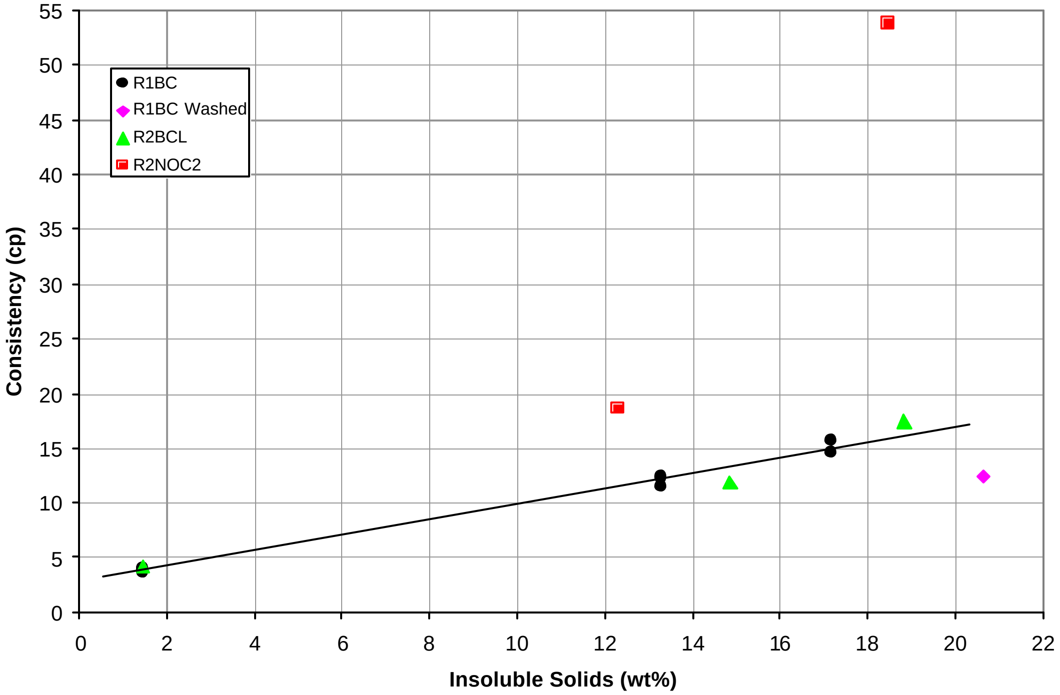


Figure 4 Slurry Consistencies

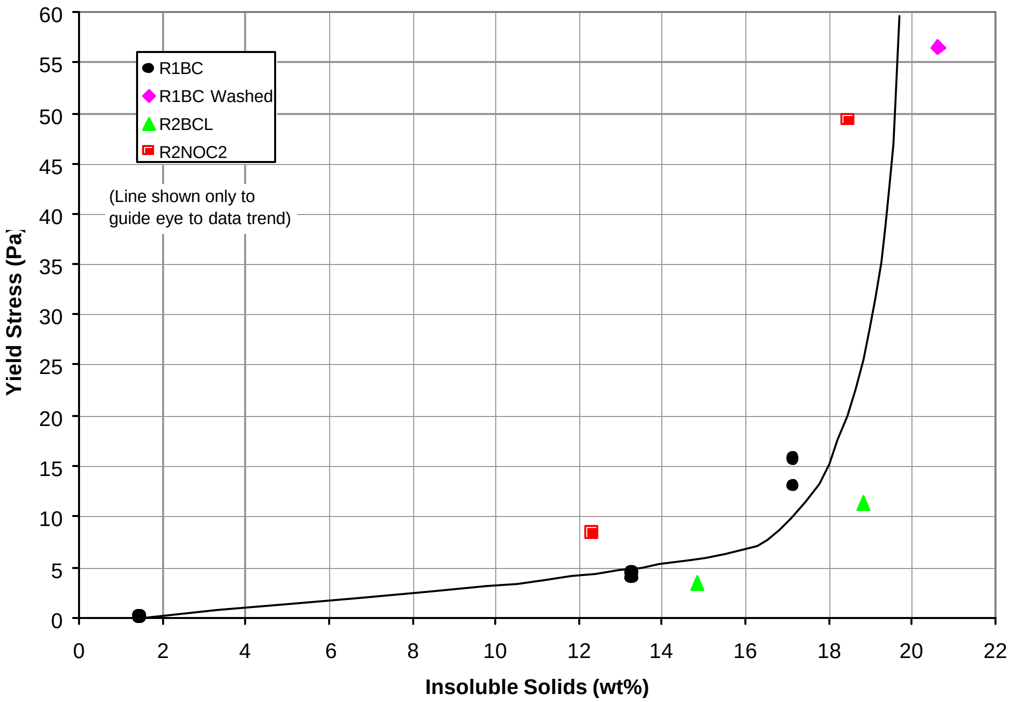


Figure 5 Slurry Yield Stresses

The high yield stresses for the more concentrated slurries made these difficult to mix, but still pumpable. The feed tank was agitated by a small turbine blade that was adequate at low concentrations, but provided poor mixing at high concentrations. The poor mixing at high solids concentrations resulted in short circuiting of the slurry flowing through the feed tank from the filter return to the pump inlet. Figure 6 shows a sketch of what appears to have probably been happening. The washed slurry, when concentrated to approximately 21 wt% insoluble solids, could not be mixed by the agitator. The yield stress of this material (51-62 Pa) would indicate that it would not pour well (and it did not pour well) and would require positive displacement pumps, possibly with screw suction, to effectively transport it. Visibly, this material was similar to gelatin and did not flow in a bottle when turned upside down. The R2NOC2 concentrated slurry had similar behavior. A photo of mixing of the concentrated slurry is shown in Figure 7. The report CD-ROM also contains a short movie of the poor mixing ("Feed-Mixing.mpg").

As the concentration of the insoluble solids increased, the slurry changed from Newtonian to non-Newtonian and the viscosity or consistency and yield stress increased. Because of these increases, the flow regime in the filter and associated piping moved from turbulent or transition flow to laminar. The Reynolds number for the R1BC slurry is plotted versus insoluble solids in Figure 8. The flow of the dilute slurry at 11 and 15 fps velocity is fully turbulent, while at 7 fps it is in the transition region. No data was taken on slurry properties in the region from 1.5-13 wt% insoluble solids, but the fluid up to at least about 4-5 wt% insoluble solids was probably still Newtonian. The transition Reynolds number plotted was calculated per Hansen.[10]

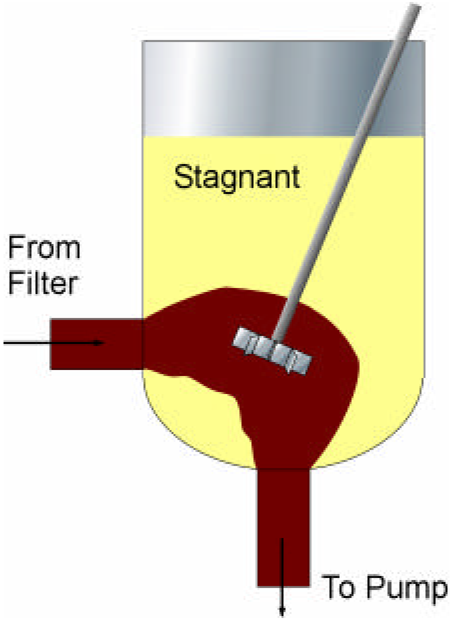


Figure 6 Feed Tank Sketch Showing Possible Stagnation

Figure 7 CUF Feed Slurry Mixing at Approximately 15-17 wt% Insoluble Solids

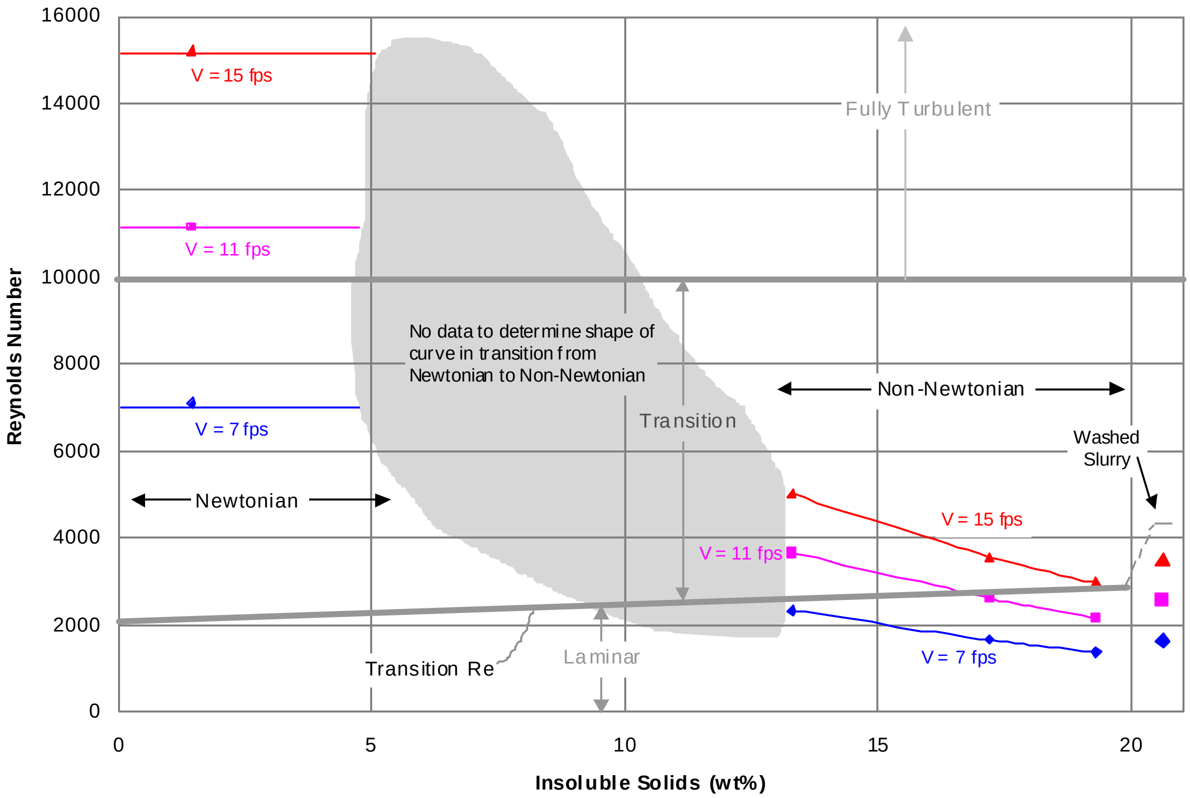


Figure 8 Reynolds Number in Filter Tube versus Insoluble Solids Content

3.2.5 Heat Transfer

At the high solids concentrations, the pump imparted significant thermal energy into the slurry, such that an auxiliary cooling mechanism had to be added to the CUF to keep the temperature in the required 20-30°C range. Heating of the slurry to 30°C occurred very quickly without the additional cooling. The heat load on the slurry cooler increased, indicating that the heat input into the slurry had increased. Given the increases in slurry consistency and yield stress, this energy increase is not surprising. During the first concentrations performed, the cooling system used was too small to maintain the temperature of the slurry below 30°C at the highest solids contents.

After the R1BC work, thermocouples were added to the inlet and outlet of both the slurry and cooling water sides of the cooler. Data were then taken periodically throughout the concentration of the R2NOC2 and R2BCL tests. Figure 9 shows the change in the heat load and the overall heat transfer coefficient while concentrating the R2NOC2 slurry. The overall heat transfer coefficients were calculated from the log-mean temperature differences across the heat exchanger and the cooling water flowrate. The values have a lot of scatter, partially due to variations in the cooling water flow that were not measured accurately; the cooling water flow was monitored periodically by measuring the volume collected over a time interval. The initial points on this figure (at <13 wt%) are plotted on the time axis, but these measurements were actually made during the several days preceding the >13.4 wt% data; these points are plotted versus time so the magnitude can be compared.

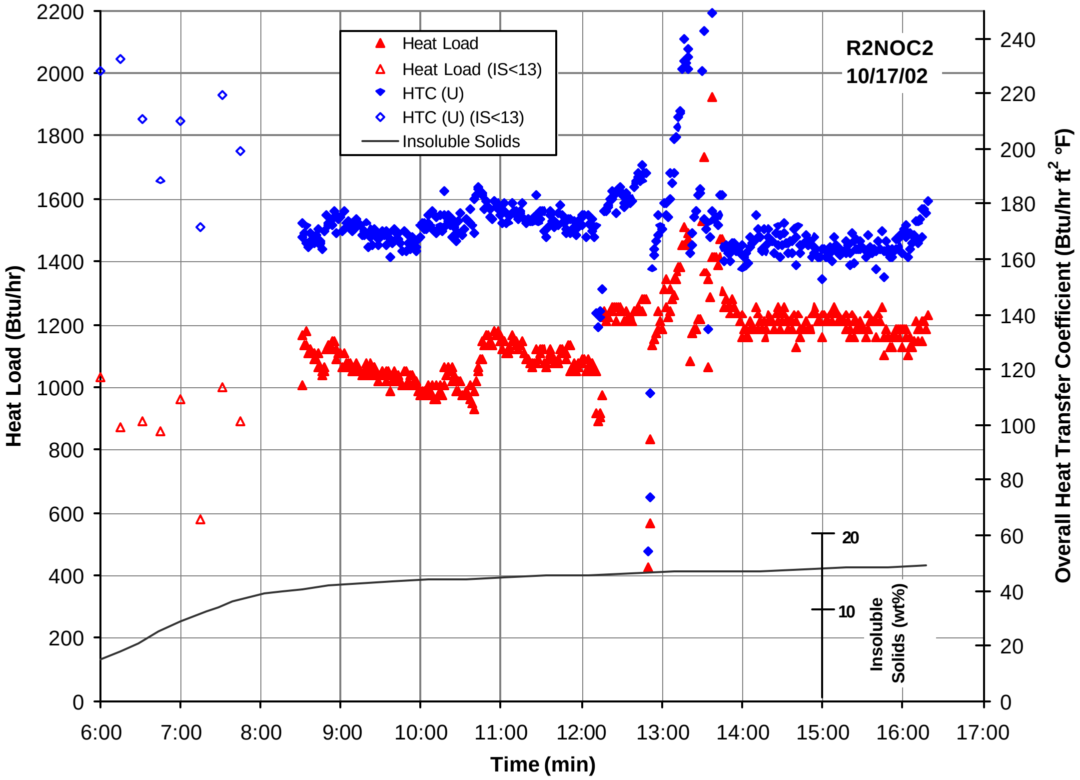


Figure 9 Heat Transfer During Concentration of R2NOC2 Slurry

3.2.6 Particle Size Distributions

The particle size distributions of samples from several of the runs were measured. Initially, the measurements were made by diluting the samples with deionized water. Later measurements were done by diluting with permeate from the R1BC run. The permeate should provide a better matrix match with samples and dissolution or precipitation due to pH or ionic strength changes should be minimized. The R1BC permeate was chosen because it was clear with no suspended solids (as determined by zeroing of the particle size instrument). The permeate from R2NOC2 was tried, but post-filtration precipitation was still occurring, which made this permeate unusable as a diluent. (The R1BC permeate had aged for several months.)

The particle size measurements were made using a Microtrac Particle Size Analyzer. This instrument has several measurement methods or modes, as shown in Table 19.

Table 19 Microtrac Particle Size Analyzer Methods

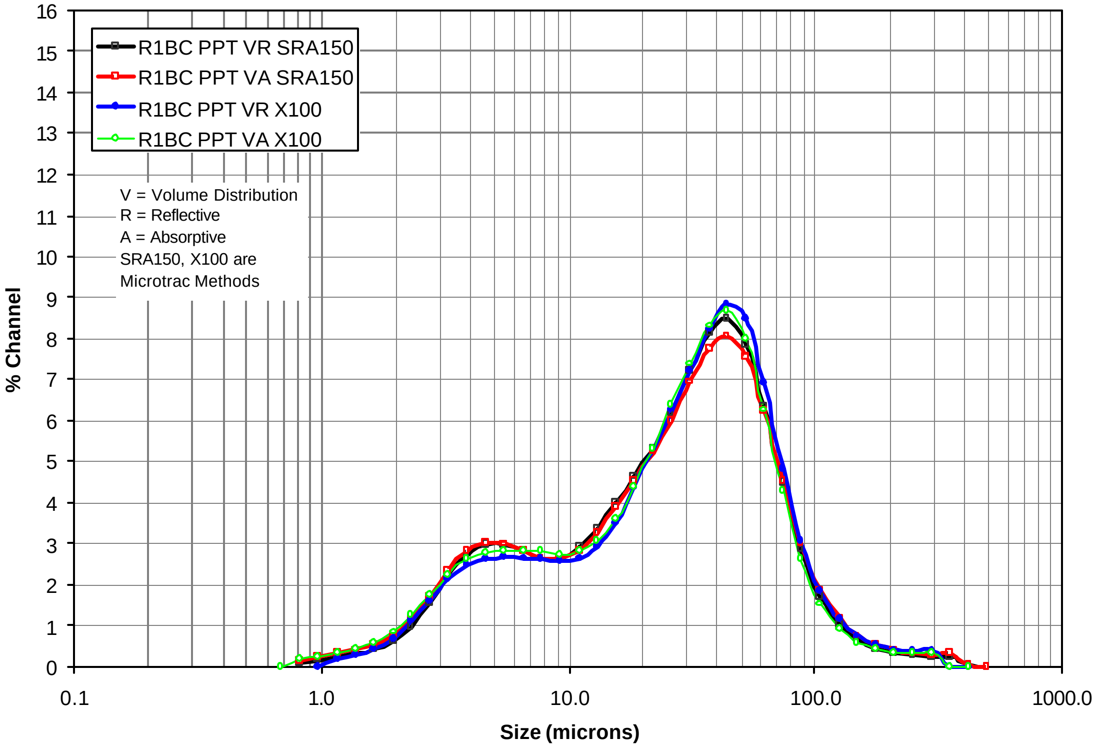
Method	Resolution	Range (μm)	Assumptions
SRA150, single laser diffraction analysis	Standard, 20 channels	0.7-700	None
	High, 40 channels	0.7-700	Particle transparency: • absorptive • reflective • assumed refractive index
X100, tri-laser diffraction analysis by "Unified Scatter Technique"	All, 40 channels	0.04-704	Particle transparency: • absorptive • reflective • assumed refractive index

The resolution of the standard SRA150 method, for which no assumptions need be made, was not sufficient to show significant differences in particle size distribution between samples. Therefore, the high-resolution SRA150 method and the X100 method were tried. For these methods, the transparency of the particles must be assumed. The choices are reflective (such as metals), absorptive (opaque materials), or a specific refractive index can be assumed. The correct choice for the Sr/TRU precipitates was not known, so both the reflective and absorptive methods were used. No runs with an assumed refractive index were made.

Figure 10-Figure 13 show volume distribution plots using both the SRA150 and X100 high-resolution modes. Each Figure shows a pair of samples: the initial unfiltered precipitate and the concentrated slurry. The SRA150 plots are generally sharper since particles smaller than 0.7μm are not quantified, whereas the X100 plots are broader since a larger range is measured.

In Figure 10a the volume distributions for the R1BC precipitate by the four methods are almost identical. Figure 10b shows the same material after filtration and concentration. The initial precipitate had a bimodal distribution with peaks at about 45 and 5 μm, whereas the filtered material was unimodal with a broad peak at 2-4μm. For the filtered slurry, the X100 method showed a much broader distribution with more particles smaller than 1μm than in the initial sample. The amount of material in the >10μm range decreased significantly.

Figure 11 compares the distributions for the R2NOC pilot-scale run. For this material, the initial bimodal distribution remained after filtration, with both the SRA150 and X100 methods showing increases in particles in the less than 4μm range. The R2NOC2 precipitate run in the CUF showed similar behavior, as shown in Figure 12. The R2BCL data shown in Figure 13 also shows similar trends, The bimodal distribution remains after filtration, but the amount of material >10μm decreases and that at around 2-3μm increases.



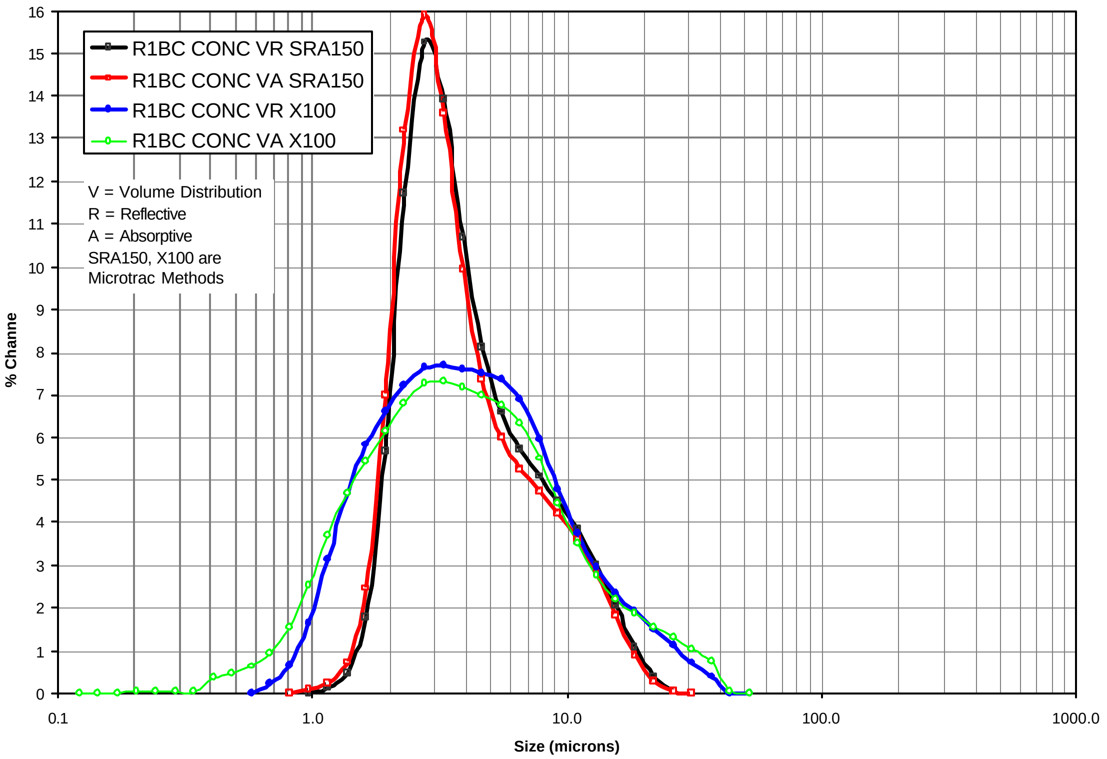
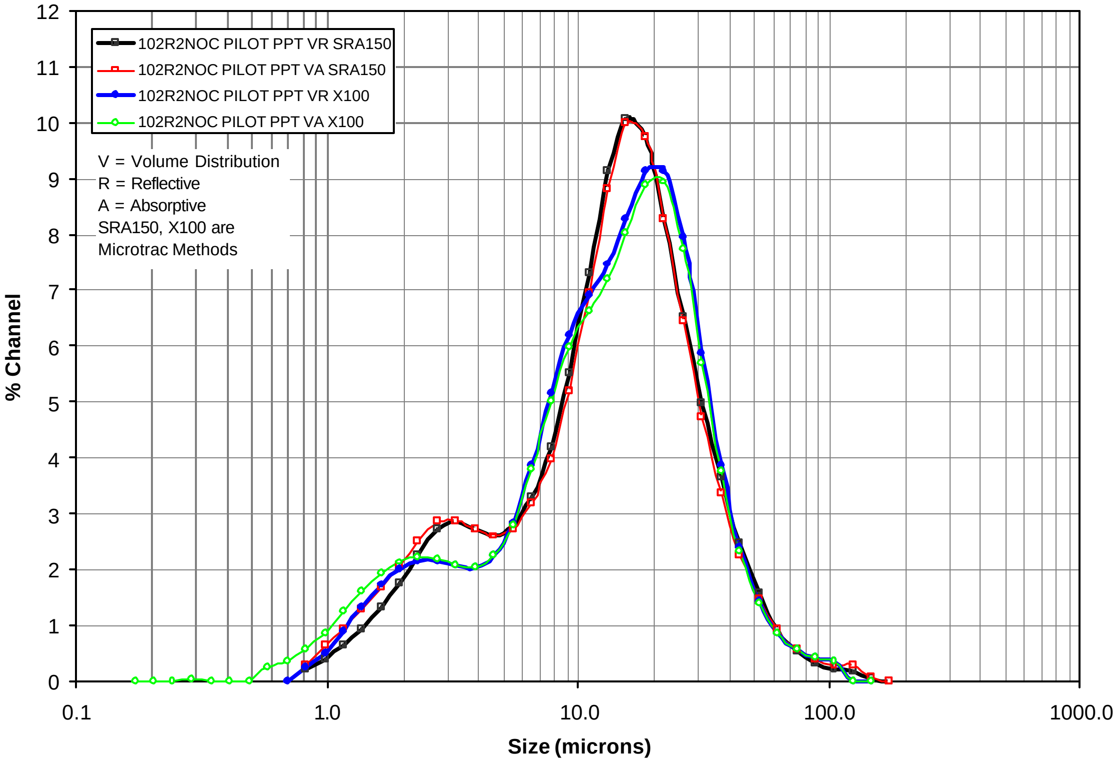


Figure 10 Particle Size Volume Distribution for R1BC Slurry



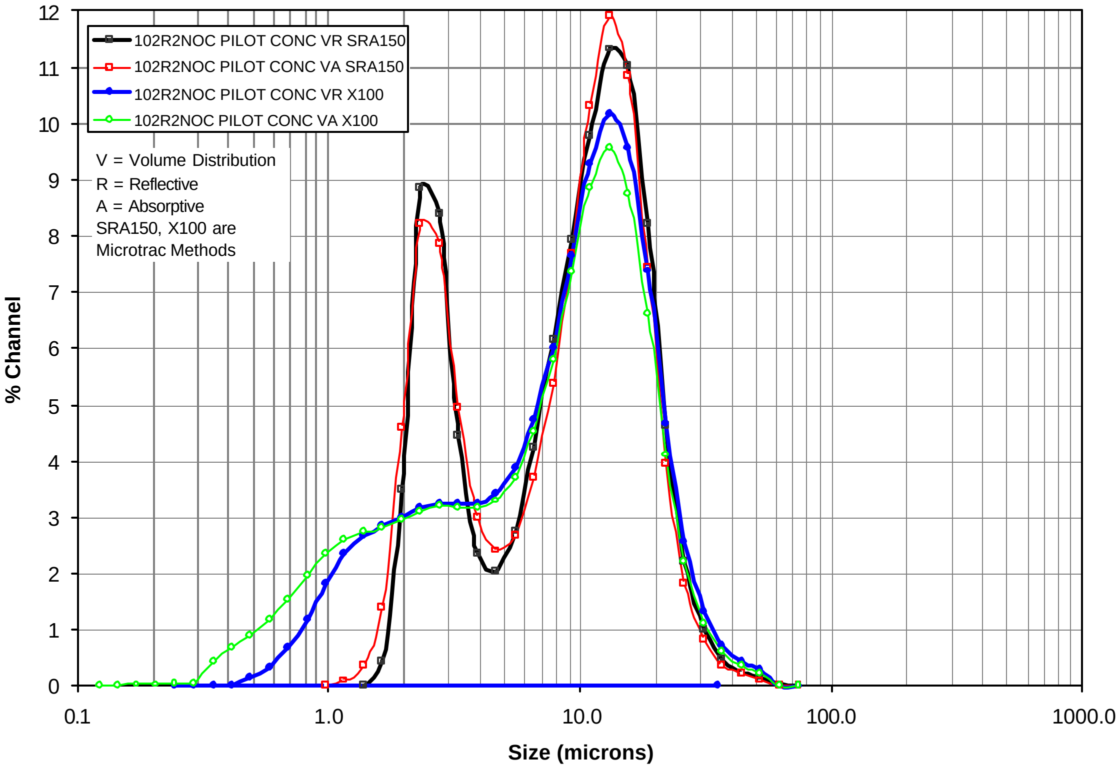


Figure 11 Particle Size Volume Distribution for R2NOC Pilot Scale Slurry

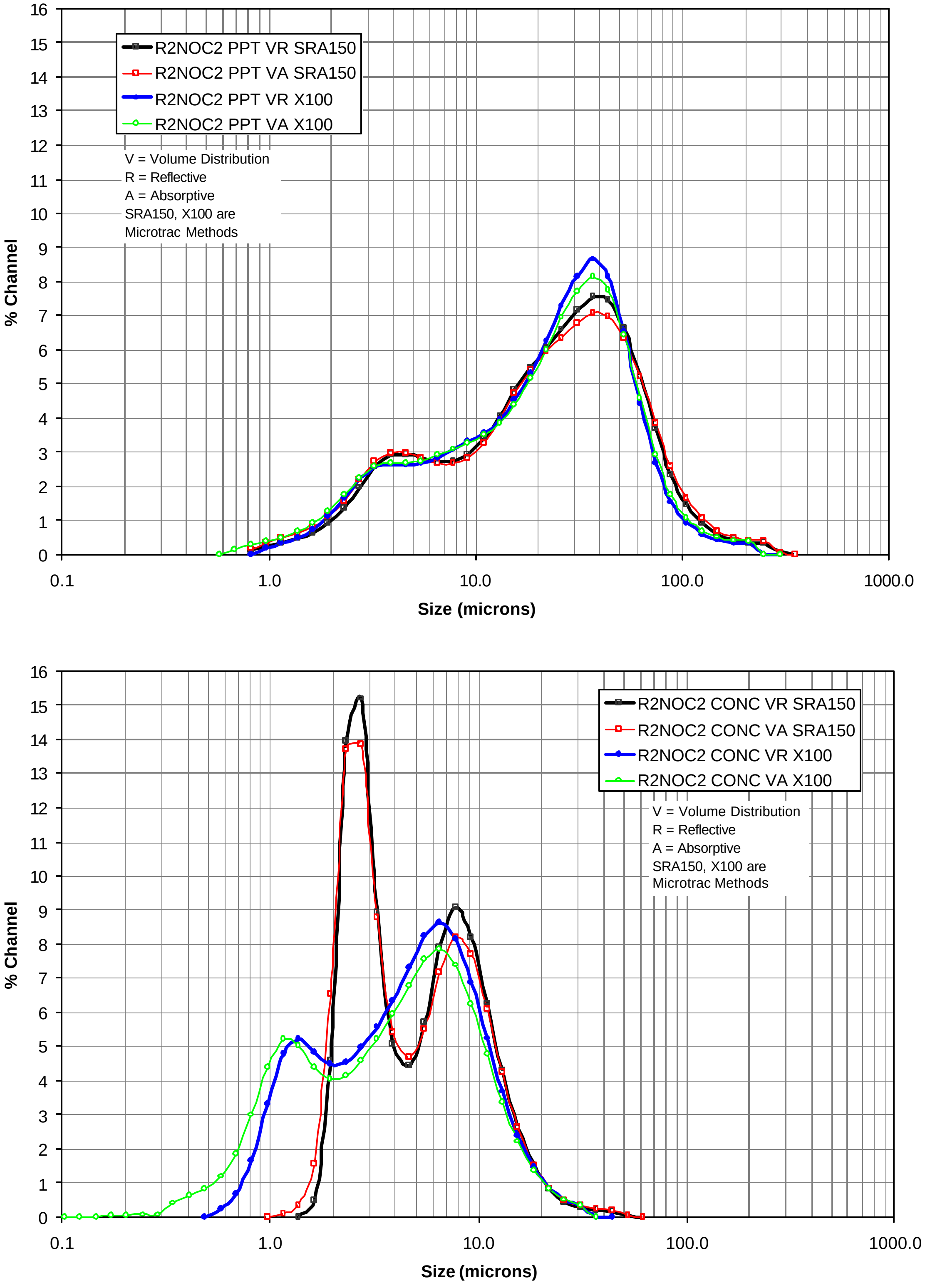


Figure 12 Particle Size Volume Distribution for R2NOC2 Slurry

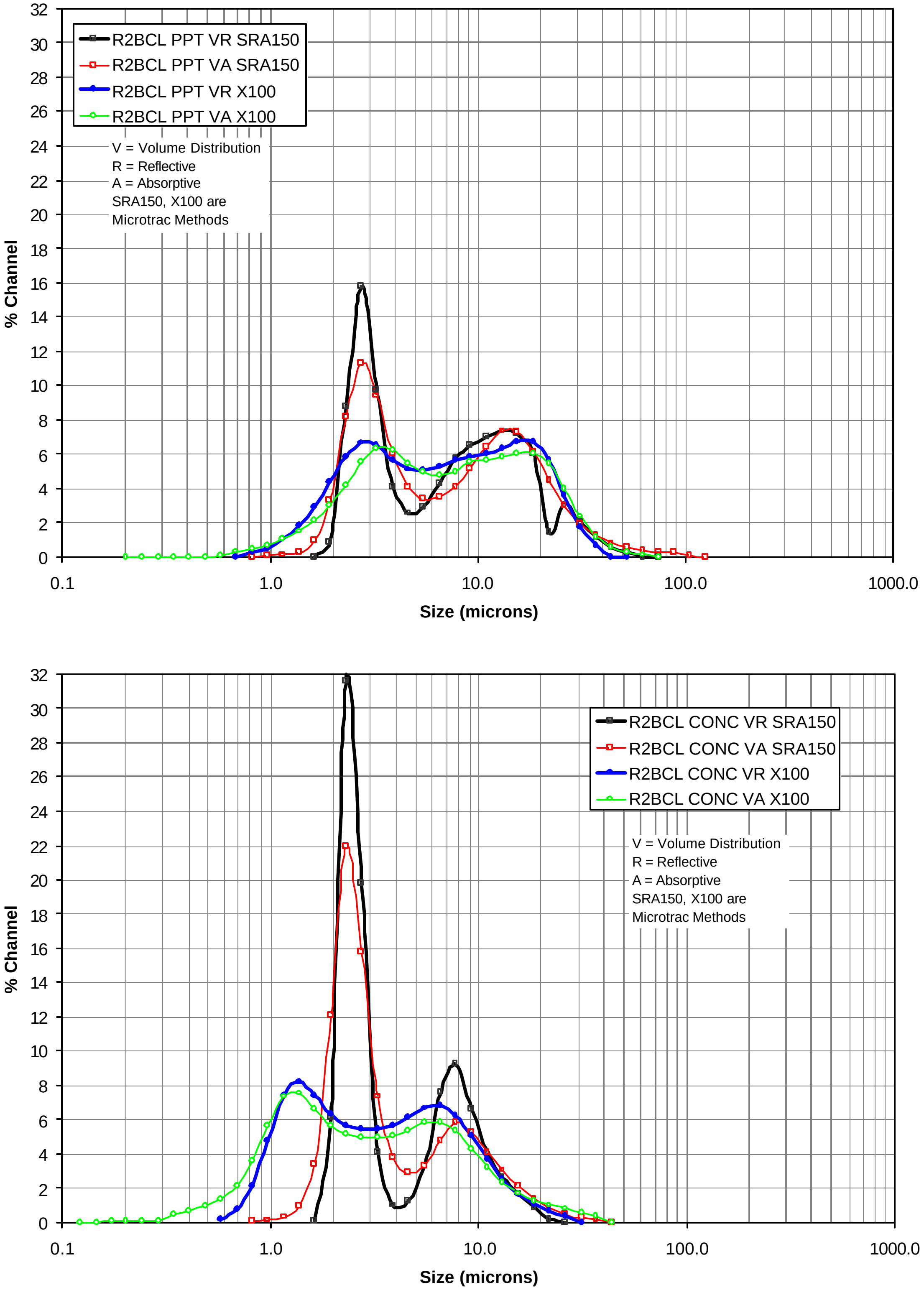


Figure 13 Particle Size Volume Distribution for R2BCL Slurry

Overall, the R2NOC and R2BCL samples have similar distribution shapes. The R1BC data stands out as being unimodal after filtering. One factor that could affect the R1BC data is aging. These samples were about four months old, whereas the others were less than a month old; agglomeration of the R1BC samples may have occurred.

Due to the significant differences in filterability of these precipitates, we proposed that the particle size distribution may be a factor. We expected that the samples that filtered poorly might have more small particles. Figure 14 shows the volume particle size distribution of the filtered slurries measured by the X100 method assuming absorptive particles. The distributions are numbered in order of filterability, with "1" being the best. It appears that there may be a correlation between the approximate shape of the distribution and filterability. The same is seen the X100 reflective data. Surprisingly, the most filterable slurries appear to have more smaller particles in the 1-4μm range and in general have a flatter distribution of particle sizes.

However, the SRA150 method data does not show as strong a correlation; data for the reflective assumption are shown in Figure 15. The best and worst stand out as in the X100 data, but the two intermediate sets do not. In the absorptive mode for the SRA150 method, there was no correlation at all; all of the distributions looked essentially the same. Since the SRA150 method does not quantify particles smaller than 0.7μm, any distributions plotted for materials with particles smaller than this would tend to be skewed towards larger particles, so the X100 data may be more applicable. Although filterability seems to correlate with the volumetric particle size distribution shape, there isn't really enough data to come to any firm conclusions about this relationship.

Figure 16 shows the X100 absorptive volume distribution for the initial precipitates. There is no correlation between filterability and the distribution, so prediction of filterability from initial particle size distribution does not appear to be possible. Additional volume distribution data plots are shown in the Appendix. Number distribution plots are also shown in the Appendix. For most of the samples, the number distribution data showed more smaller particles in the filtered slurries, as would be expected. There was not any apparent correlation between the number distribution data and filterability.

SEM photos of filtered samples from the R1BC and R2NOC1 runs are shown in Figure 17. Both photos were taken at approximately the same magnification. The samples were collected by dead-end filtration of the dilute precipitate slurries. The R2NOC1 slurry was about one week old, whereas the R1BC slurry was several months old. Both samples contain some sheet-like flat crystals, while R1BC contains numerous needle-like crystals (since R1BC sat for several months, crystallization may have occurred that would not be seen in the newer R2NOC1 material). There was no difference in appearance between the rest of the materials in either sample that would indicate why R1BC filtered so much better than R2NOC1.

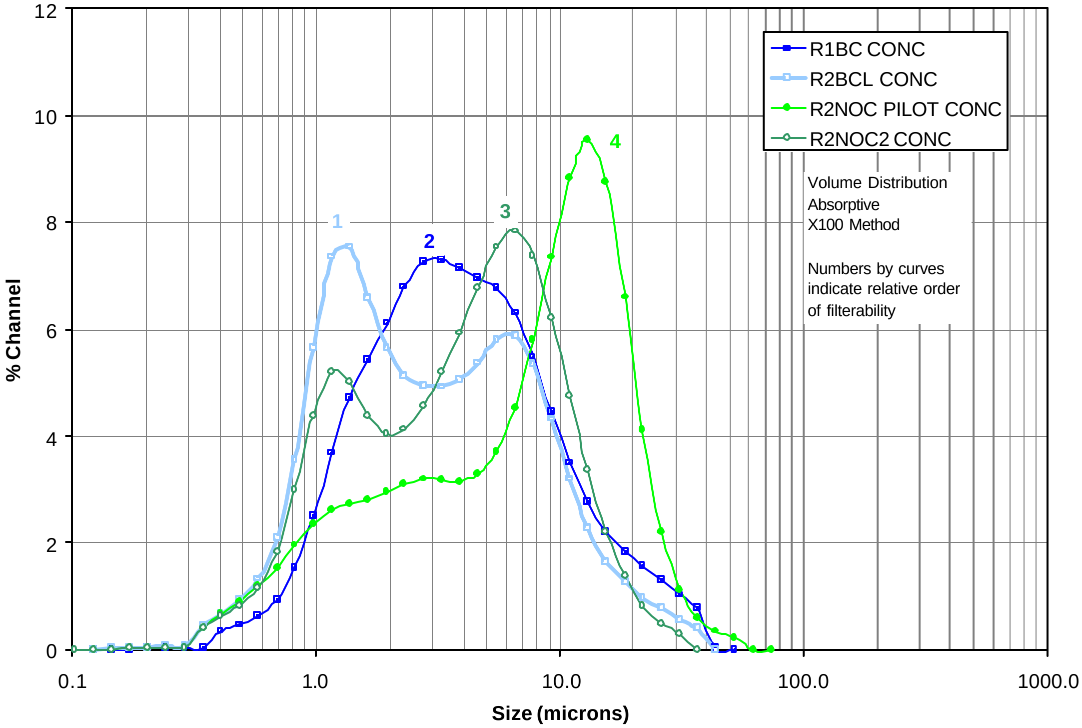


Figure 14 Volume Distributions of Concentrated Slurries by the X100 Absorptive Method

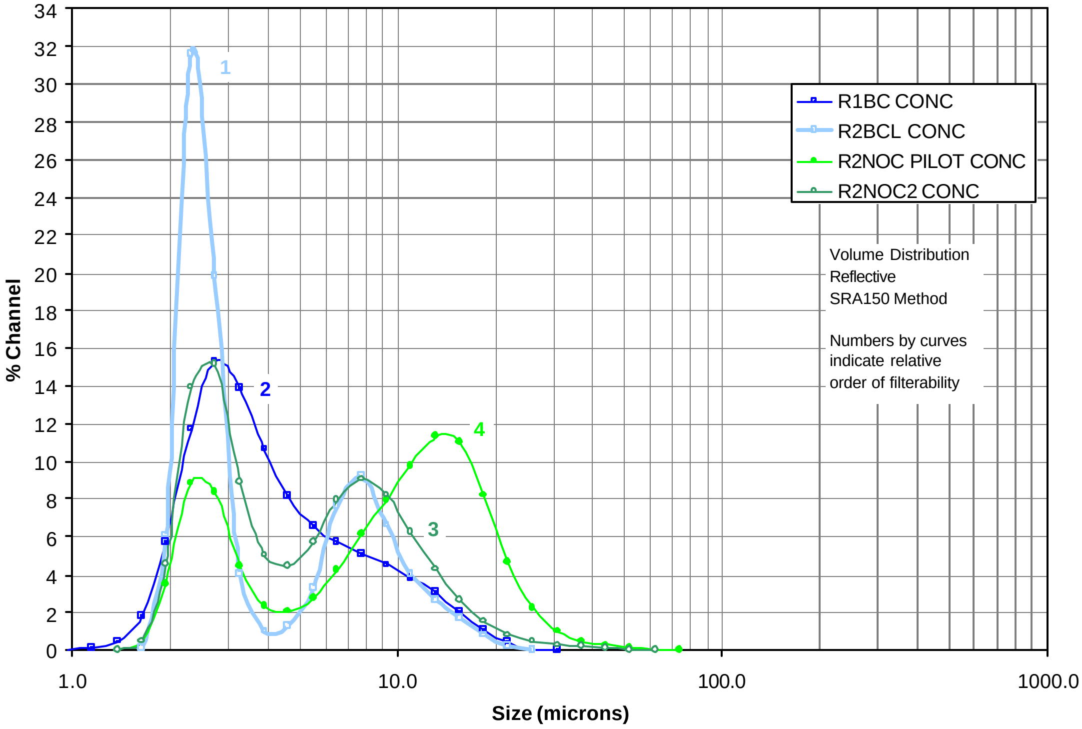
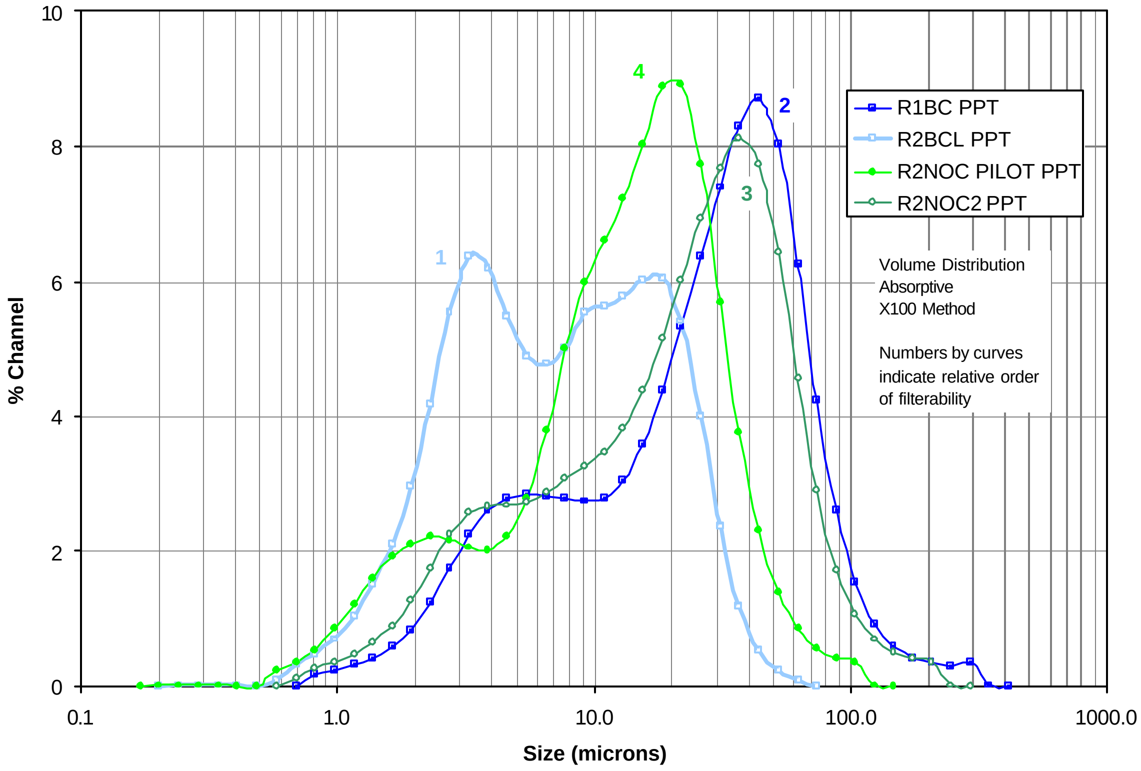


Figure 15 Volume Distributions of Concentrated Slurries by the SRA150 Reflective Method



3.2.7 Feed Decontamination – R1BC and R2NOC2 Data

For the R1BC data, the average concentration is compared to the calculated and measured precipitate compositions in Table 20. Results for the R2NOC2 run are also shown in this table. The ratio of the concentration in the permeate to the concentration in the precipitate feed is also shown in this table. This value can be called the “percent retained” and is related to the decontamination factor (DF) as shown in Figure 18. This “percent retained” is referred to as “feed based” since it uses the permeate and feed concentrations. A “slurry based” DF uses the concentrated slurry concentration rather than the feed concentration and so is more indicative of the overall separation ability of the process. A “batch based” DF can also be defined. This DF uses the total amounts of materials separated rather than concentrations, and is this based on the processing of an individual batch. This “batch based” DF is used later in this report when the removal of soluble species from the final concentrated slurry is described.

Figure 16 Volume Distributions of Initial Precipitates by the X100 Absorptive Method

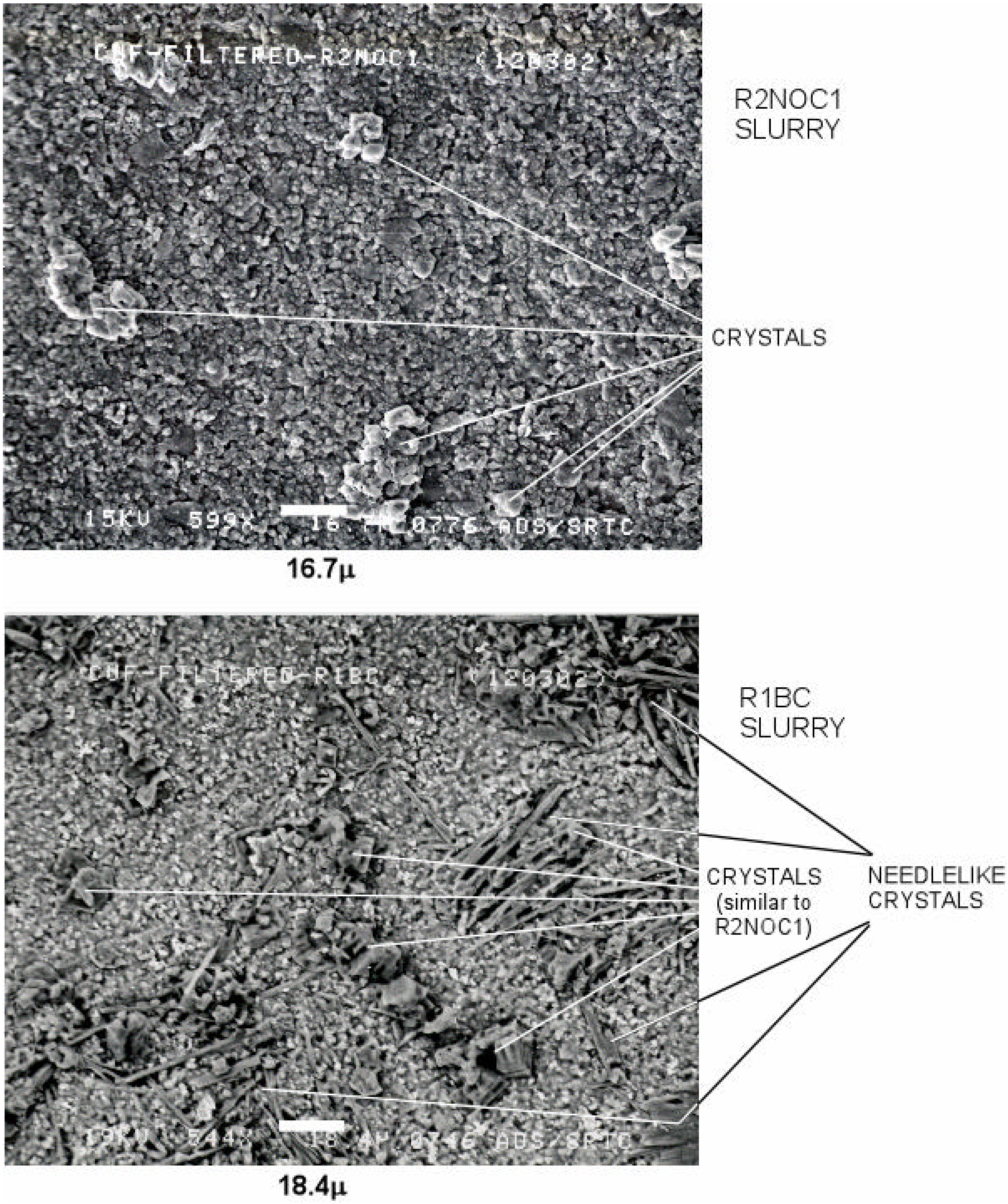


Figure 17 SEM Photo of Filtered R1BC & R2NOC1 Slurries

Table 20 Comparison of Permeate and Precipitate Compositions

	R1BC				R2NOC2		
	Average Permeate	Calculated Precipitate	Measured Precipitate	Percent Retained in Permeate (Feed Based)	Permeate	Measured Precipitate	Percent Retained in Permeate (Feed Based)
ICPES (mg/L)							
Al	7530	7670	7540	98.2-99.9	8980	8730	103
B	23.4	22.9	<92	approx. 100	**28.2**	**65.5**	**43.1**
Ba	<0.02	0.1	189	approx. 0	**<0.02**	**30.1**	**approx. 0**
Ca	**96.5**	**306**	**306**	**31.5-31.6**	**89.4**	**703**	**12.7**
Cd	36.07	37.9	36.1	95.1-99.8	4.1	45.5	89.0
Co	<0.088	2.1	<35	NA	<0.2	NA	NA
Cr	146	160	162	90.1-91.7	146	173	84.3
Cu	**3.8**	**15.1**	**<35**	**25.0**	**6.7**	**18.6**	**35.8**
Fe	**0.5**	**30.2**	**196**	**0.28-1.79**	**2.7**	**140**	**1.9**
Li	0.2	1.0	<47	24.0	<0.2	NA	NA
Mg	<0.2	0	<41	NA	**<0.2**	**27.9**	**<0.6**
Mn	**0.3**	**2770**	**2320**	**0.012-0.014**	**12.2**	**1870**	**0.7**
Mo	28.6	28.1	<69	102.0	34.5	37.9	91.2
Na	138000	135000	135000	102.0	136000	129000	106
Ni	**199**	**260**	**270**	**73.5-76.6**	275	324	84.9
P	**568**	**1140**	**768**	**49.7-74.0**	**775**	**1220**	**63.6**
Pb	**39.4**	**116**	**<302**	**34.0**	**47.4**	**274**	**17.3**
Si	15.5	6.5	<69	240.4	**27.1**	**88.0**	**30.8**
Sn	<0.7	0	<163	NA	**1.0**	**95.0**	**1.1**
Sr	**26.8**	**6570**	**5360**	**0.41-0.50**	**22.9**	**3210**	**0.7**
Ti	<0.3	0	<69	NA	<0.3	NM	NA
V	<0.3	0	<69	NA	<0.3	NM	NA
Zn	2.5	3.2	<163	79.3	**1.7**	**84.5**	**2.0**
Zr	**0.5**	**8.5**	**<23**	**5.78**	**3.2**	**10.0**	**31.5**
La	**1.4**	**9.9**	**<324**	**14.1**	**3.0**	**211**	**1.4**
K	2100	1270	293	NC	1840	1460	126
S	2830	2640	NM	107.1	3180	NM	NA
Nd	**0.9**	**19.9**	**<232**	**4.38**	**6.1**	**109**	**5.6**
Ce, Cs, Rb, W	NM	not shown	NM	NC			
Ion Chrom.							
NO_2^-	44000	48100	5900	86.4-91.4	50500	NA	NA
NO_3^-	126000	137000	134000	92.2-94.0	124000	NA	NA
Cl^-	2780	2980	3030	91.8-93.3	3150	NA	NA
F^-	**712**	**1080**	**1440**	**49.4-65.8**	1180	NA	NA
PO_4^{-3}	**2370**	**3500**	**2680**	**67.5-88.4**	3310	NA	NA
SO_4^{-2}	7010	7910	7590	88.6-92.3	8460	NA	NA
formate	4870	5240	5770	84.3-93.0	5860	NA	NA
oxalate	**679**	**397**	**1960**	**34.6-171.1**	1020	NA	NA

Bold indicates species with permeate concentrations significantly lower than in precipitate.

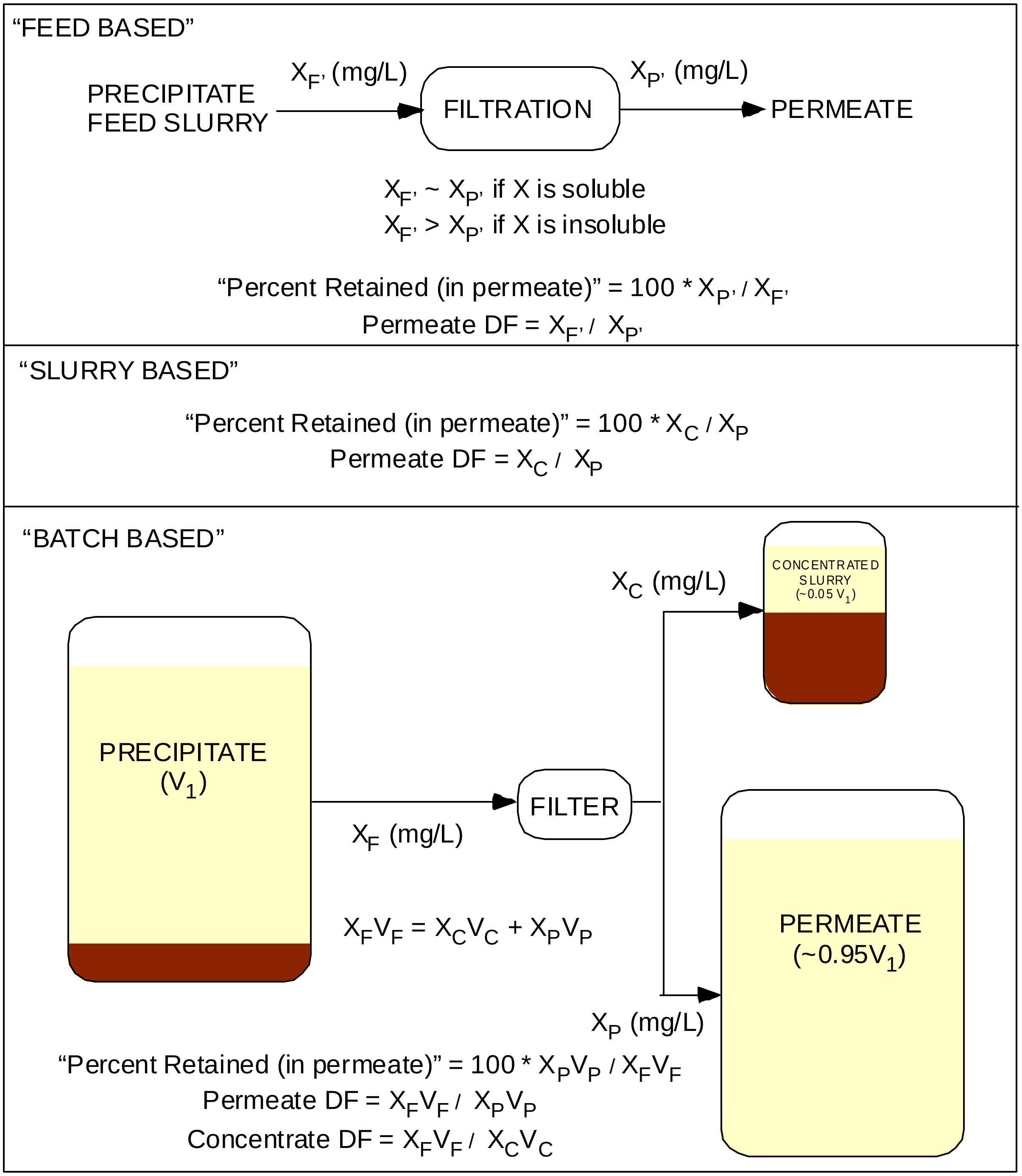


Figure 18 Definitions for Percent Retained and Decontamination Factor

The precipitate compositions in Table 20 for R1BC are the batch #1 calculated precipitate composition (adjusted for added Na and also for the dilution from the reagent additions) and the average measured composition; the R2NOC2 precipitate compositions shown are the measured values. Some species are precipitated along with Sr and Mn during the $Sr(NO_3)_2$ and $NaMnO_4$ additions; these have a lower concentration in the permeate and are shown in **bold** face. The percent retained in the permeate (feed based) is given for both data sets. For both R1BC and R2NOC2, the species that appear to have been precipitated (in addition to Sr and Mn) are Ca, Cu, Fe, P (PO_4^{-3}), Pb, Zr, La, Nd, Ni, F, and oxalate. Zinc was also removed to some extent. In R2NOC2, boron, Mg, Si, and Sn were also depleted in the permeate. The concentration of the remaining species are essentially the same in the permeate as in the precipitate. One exception is K, but the analyses for K by ICPES have been determined to be unreliable.

As previously stated, phosphorus (as PO_4^{-3}) and fluoride may have precipitated as sodium fluorophosphate. The drop in the fluoride concentration, on a molar basis, was greater than the drop in the phosphate, so this precipitate would not account for the entire amount of fluoride removed. Some of the Ca and most of the La, Nd, and Zn were added as the insoluble oxalates. Zr, Fe, and Ni were added as oxides and Pb as the sulfate. All of these are relatively insoluble, so these would be expected to be removed during filtration. Cu, which was removed, was added as the nitrate, but could have formed the insoluble oxalate.

Table 21 shows the amount retained in the permeate (expressed as the fraction retained) and the feed based DFs compared to the DFs calculated for data from the Large C run. Data shown as ">" for fraction retained means the feed concentration was below the detection limit, whereas values shown as "<" had permeate concentrations below detection limits. Since the DF is the inverse of the fraction retained, the sign on the corresponding DF is the opposite of the fraction retained value.

$$\begin{aligned}
\text{Fraction Retained} &= \frac{X}{<Y} = > \frac{X}{Y} \\
\text{or} &= \frac{<X}{Y} = < \frac{X}{Y} \\
\text{DF} &= \frac{<Y}{X} = < \frac{Y}{X} \\
\text{or} &= \frac{>Y}{X} = > \frac{Y}{X}
\end{aligned}$$

For the R1BC data, the permeate concentration used was the average of the concentrations measured and at 13.3 and 17.2 wt% insoluble solids. For the R2NOC2 data, the permeate composition was measured at approximately 10 wt% insoluble solids in the slurry. The DFs found in this work compare favorably with those found in the Large C work. In this work the DF for Cu was noticeably smaller, but the small amount present resulted in large uncertainty in the DF value. The DF for Fe was much higher here, and was similar to Zr and Nd. The DFs for nitrite, nitrate, and chloride should all be about unity since most of these should be very soluble. The measured values are all around 1.08. The higher DFs for fluoride and phosphate could possibly be due to formation of sodium fluorophosphate, which has been found in the simulant.[6]

Table 21 Decontamination Factors and Fraction Retained

	R1BC				Hot CUF	R2NOC2	
	Based on Calculated Feed Composition		Based on Measured Feed Composition			Based on Measured Feed Composition	
	Fraction Retained in Permeate	DF	Fraction Retained in Permeate	DF	DF	Fraction Retained in Permeate	DF
Al	0.98	1.02	1.00	1.00	0.9	1.03	0.97
B	1.02	0.98	>0.254	<3.9		0.43	2.32
Ca	0.32	3.17	0.32	3.17	2.4	0.13	7.86
Cd	0.95	1.05	1.00	1.00		0.89	1.12
Cr	0.92	1.09	0.90	1.11		0.84	1.19
Cu	0.25	4.00	>0.11	<9.3	>10	0.36	2.79
Fe	0.018	55.8	0.0028	363	5.3	0.02	52.8
La	<0.14	>7.1	>0.0043	<231		>0.014	<71
* Mn	0.013	74.7	NA	NA		NA	NA
** Mn	0.00012	8250	0.00015	6920		0.0065	153
Mo	1.02	0.98	>0.42	<2.4		0.91	1.10
Na	1.02	0.98	1.02	0.98		1.06	0.95
Ni	0.77	1.31	0.74	1.35	1.0	0.85	1.18
P	0.50	2.01	0.74	1.36	1.1	0.64	1.57
Pb	0.34	2.94	>0.13	<7.7		>0.17	<5.8
Si	2.40	0.42	>0.23	<4.4		0.31	3.24
* Sr	13.2	0.076	NA	NA		NA	NA
** Sr	0.0041	245	0.0050	200		0.0071	140
Zn	0.79	1.26	>0.016	<64	1.3	>0.020	<51
Zr	0.058	17.3	>0.021	<47		>0.32	<3.2
K	1.65	0.60	NC	NC		1.26	0.79
S	1.07	0.93	NC	NC	1.0	NC	NC
Nd	0.044	22.9	>0.0037	<267		0.056	18.0
NO_2^-	0.91	1.09	0.86	1.16		NA	NA
NO_3^-	0.92	1.08	0.94	1.06		NA	NA
Cl-	0.93	1.07	0.92	1.09		NA	NA
F-	0.66	1.52	0.49	2.03		NA	NA
PO_4^{-3}	0.68	1.48	0.88	1.13		NA	NA
SO_4^{-2}	0.89	1.13	0.92	1.08		NA	NA

* Mn and Sr values based on concentrations before additional of $Sr(NO_3)_2$ and $NaMnO_4$.
** Mn and Sr values based on concentrations after addition of $Sr(NO_3)_2$ and $NaMnO_4$.
NC = not calculated; NA = not available; see text for explanation of "<" and ">" values.

Whereas the goal of the precipitation is to remove Sr and TRU from the LAW (permeate) to minimize the volume of HLW produced, minimization of the amount of soluble non-radioactive species in the concentrated slurry is also required. The "permeate batch DF" or "batch percent retained" defined earlier is a good measure of the overall separation of the process. To calculate these measures, we can use either of the following equations, which should be equivalent:

$$\text{Permeate Batch DF} = \frac{x_F V_F}{x_P V_P} = \frac{x_F V_F}{x_F V_F - x_C V_C}$$

where $x_F = \text{concentration in feed slurry, mg/L}$

$x_P = \text{concentration in permeate, mg/L}$

$x_C = \text{concentration in concentrated slurry, mg/L}$

$V_F = \text{volume of feed slurry, L}$

$V_P = \text{volume of permeate, L}$

$V_C = \text{volume of concentrated slurry, L}$

However, due to measurement errors of both compositions and volumes, these are not equal. To judge the consistency of the data, the final washed slurry concentrations can be calculated from the initial slurry and permeate compositions and volumes:

$$x_C = \frac{x_F V_F - x_P V_P}{V_C}$$

The measured and calculated washed slurry compositions are shown in Table 22. The calculated washed slurry values for Al, Ca, Cd, Dr, Mn, Ni, and Sr match reasonably well. Other data shows more deviation due to the error in taking the differences between large numbers. Particularly, the calculated values for the anion data are all high. Other calculations were not reliable since below detection limit values had to be used.

Table 23 shows the batch basis DFs based on both the measured and calculated composition. The DFs for both permeate and concentrated slurry bases are given. The values calculated by differences (shown in italics) are less accurate. The DFs are not shown for species that required the use of data below instrumental detection limits to determine the DFs.

Table 22 Measured and Calculated Concentrated Washed Slurry Concentrations

	Measured (mg/L)	Calculated (mg/L)		Measured (mg/L)	Calculated (mg/L)		Measured (mg/L)	Calculated (mg/L)
Al	4730	4570	Mo	<67	*664*	La	<314	*5170*
B	<90	*1110*	Na	76300	34700	K	<224	NC
Ba	2440	3020	Ni	958	1280	Nd	<224	*3700*
Ca	3040	3410	P	1290	3390	NO_2^-	18200	135000
Cd	45.3	19.6	Pb	1030	*4240*	NO_3^-	57600	205000
Co	<34	*560*	Si	<67	*865*	Cl^-	1130	5620
Cr	251	343	Sn	<157	*2600*	F^-	467	11800
Cu	174	*505*	Sr	85500	85400	PO_4^{-3}	1090	6000
Fe	673	3140	Ti	<67	*1100*	SO_4^{-2}	3620	12800
Li	<45	*749*	V	<67	*1100*	formate	3220	16500
Mg	234	*654*	Zn	<157	*2570*	oxalate	10900	17800
Mn	37300	37200	Zr	143	*361*			

Values with "<" were below the detection limits. Values shown in *small blue italics* were calculated from one or more below detection limit (<) measurements.

Table 23 Permeate and Concentrate Decontamination Factors

	Permeate DF (from measured permeate and initial slurry)	*Permeate DF (from measured concentrate and initial slurry)*	*Concentrate DF (from measured permeate and initial slurry)*	Concentrate DF (from measured concentrate and initial slurry)
Al	1.04	*1.04*	*26.4*	25.5
Ca	3.28	*2.63*	*1.44*	1.61
Cd	1.04	*1.08*	*29.6*	12.8
Cr	1.15	*1.11*	*7.58*	10.4
Fe	387	*1.27*	*1.00*	4.68
Mn	6290	*NA*	*1.00*	1.00
Na	1.02	*1.04*	*62.3*	28.4
Ni	1.42	*1.28*	*3.39*	4.52
P	1.38	*1.12*	*3.63*	9.54
Sr	209	*221*	*1.00*	1.00
NO_2^-	1.20	*1.02*	*NC*	NC
NO_3^-	1.11	*1.03*	*NC*	NC
Cl^-	1.13	*1.02*	*NC*	NC
F^-	2.04	*1.02*	*NC*	NC
PO_4^{-3}	1.16	*1.03*	*NC*	NC
SO_4^{-2}	1.12	*1.03*	*NC*	NC
formate	1.22	*1.04*	*NC*	NC
oxalate	2.31	*1.53*	*NC*	NC

blue italics: calculated by differences, thus less accurate

3.2.8 Filtration – Remediation 1 Baseline Conditions

3.2.8.1 Clean Water and Strontium Carbonate Fluxes

"Clean water" and strontium carbonate fluxes were measured both before and after these experiments were started, per Table 24. The "clean water" fluxes were actually measured using deionized water filtered through a 0.1 μm filter followed by addition of reagent grade NaOH to a concentration of approximately 0.01M. Herein, this material will be referred to as "inhibited water" and the fluxes measured with it as "clean water" fluxes.

Prior to measuring the clean water and $SrCO_3$ fluxes, the CUF unit was cleaned by soaking in approximately 2M nitric acid. After soaking, the unit was flushed with several feed tank volumes of inhibited water to remove residual nitric acid. The pH of the water was checked during the flushing with inhibited water to assure that acid present was neutralized and flushed out. The clean water fluxes measured are shown in Figure 19. (The data reported was taken during the R1BC runs; data from the later runs with R2 simulant are similar, but are not reported here.)

Table 24 Factorial Design Test Conditions for R1BC

Test No. (Lab Book)	Task Plan Test No.	Trans-membrane Pressure (psi)	Crossflow Velocity (ft/s)
0.1µm Filtered Deionized Inhibited Water			
	1.0	10, 20, 30	11
5%wt Strontium Carbonate in 0.01M NaOH			
	1.1	10, 20, 30	11
0.1µm Filtered Deionized Inhibited Water			
	1.2	20	11
Feed			
1-a	1.3	40	11
1-b	1.15a	50	12
1-c	1.6	30	9
1-d	1.15b	30	15
1-e	1.15c	70	9
1-f	1.8	50	13
1-g	1.4	40	11
1-h	1.7	30	13
1-i	1.9	50	9
1-j	1.11	40	7
1-k	1.10	40	11
1-l	1.13	20	11
1-m	1.15	40	11
1-n	1.12	40	15
1-o	1.5	40	11
1-p	1.14	60	11
Dewatering to approx. 15 wt% Insoluble Solids			
	1.16	50	12
At approx. 15 wt% Insoluble Solids			
15-a	1.17	40	11
15-b	1.29a	50	12
15-c	1.20	30	9
15-d	1.29b	30	15
15-e	1.29c	70	9
15-f	1.22	50	13
15-g	1.18	40	11
15-h	1.21	30	13
15-i	1.23	50	9
15-j	1.25	40	7
15-k	1.24	40	11
15-l	1.27	20	11
15-m	1.29	40	11
15-n	1.26	40	15
15-o	1.19	40	11
15-p	1.28	60	11
Caustic Leaching/Washing			
Dewatering			
	1.30	50	12
Water Flush and 2M nitric acid cleaning			
	1.31	N/A	N/A
0.1µm Filtered Deionized Inhibited Water			
	1.32	20	11
5%wt Strontium Carbonate in 0.01M NaOH			
	1.33	10, 20, 30	11
0.1µm Filtered Deionized Inhibited Water			
	1.34	10, 20, 30	11

The system was initially cleaned by flushing with inhibited water, then soaked in approximately 2M nitric acid, and then flushed with inhibited water. The first clean water flux measured is shown as “Pre-Test #1”. The flux was approximately 1.3 gpm/ft^2 at about 18 psi TMP. Higher TMPs could not be achieved due to high backpressure in the permeate lines that was caused by the very high flowrate. After the clean water test, the first $SrCO_3$ test was performed. $SrCO_3$ results are shown below. The clean water flux after the $SrCO_3$ with only water flushing is shown as “Pre-Test #2”. This flux is significantly lower than the first water flux. Subsequent water fluxes performed immediately after $SrCO_3$ tests were consistently lower than after acid cleaning and fell within the blue band shown in Figure 19. The system was cleaned with nitric acid after $SrCO_3$ test #2 and the water flux after cleaning was very high (Pre-Test #3) at more than 2 gpm/ft^2 at 20 psi TMP. These data show that only water flushing after $SrCO_3$ to be inadequate to return the clean water flux to the original value; cleaning with nitric acid or acidification is required to eliminate all residual $SrCO_3$ slurry.

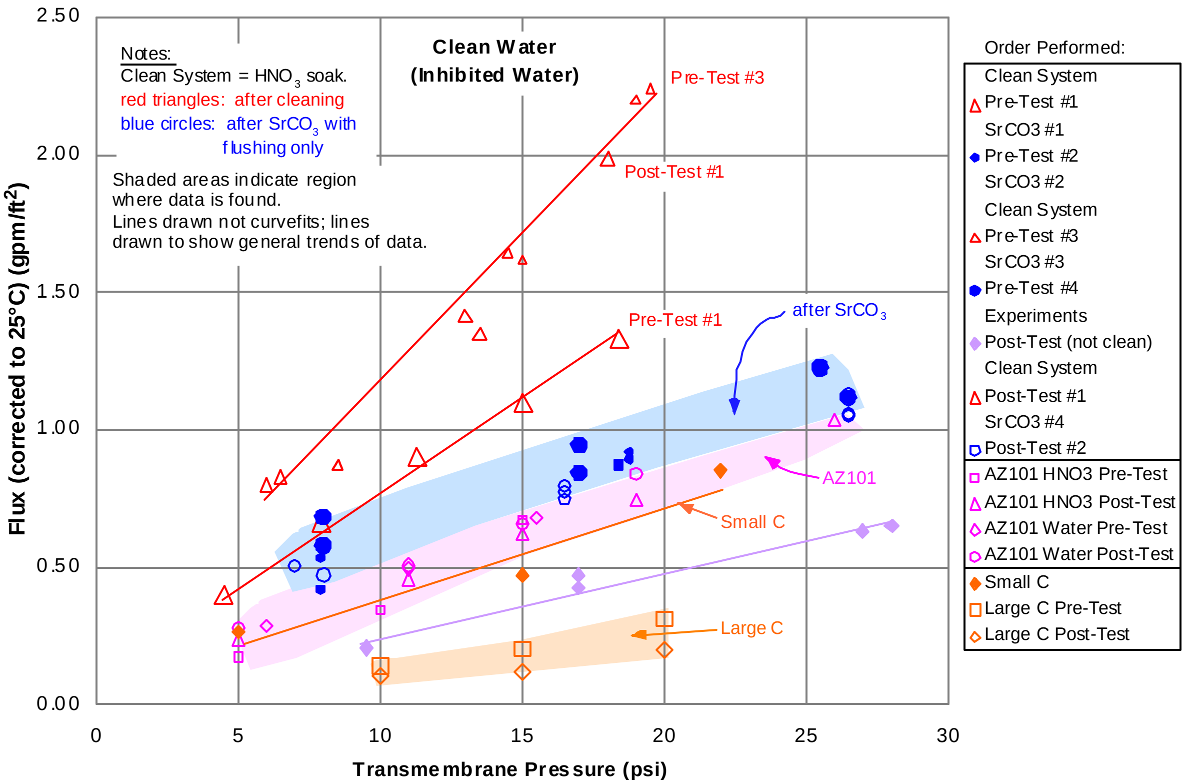


Figure 19 Clean Water Fluxes – R1BC Run

After completing all of the tests with the R1BC simulant slurry, including concentration and washing, the clean water fluxes were again measured. The first clean water test (Post-Test, not clean) gave results well below the previously determined values. These fluxes were measured after two flushes with inhibited water. The water in this test had visible solids, so the low result is not surprising. After cleaning, the clean water flux (Post-Test #1) returned to the same values as Pre-Test #3. For comparison, the clean water fluxes determined in the AZ101[11] simulant filtration, Small C,[12] and Large C[3] are also shown. The AZ101 and Small C values are essentially the same, while the Large C values are much lower than any of the other values. All of the values shown from previous work are much lower than those found in this work.

Clean water fluxes and $SrCO_3$ fluxes for this work are shown in Figure 20. The $SrCO_3$ fluxes (green band) were approximately one-third the fluxes determined for clean water (Pre-Test #1 and Post-Test #1). The “clean water” fluxes determined after only flushing out of the $SrCO_3$ (no dissolving with acid) were only about 30% higher (blue band). In general, the $SrCO_3$ fluxes were repeatable to within about ±0.2 gpm/ft^2.

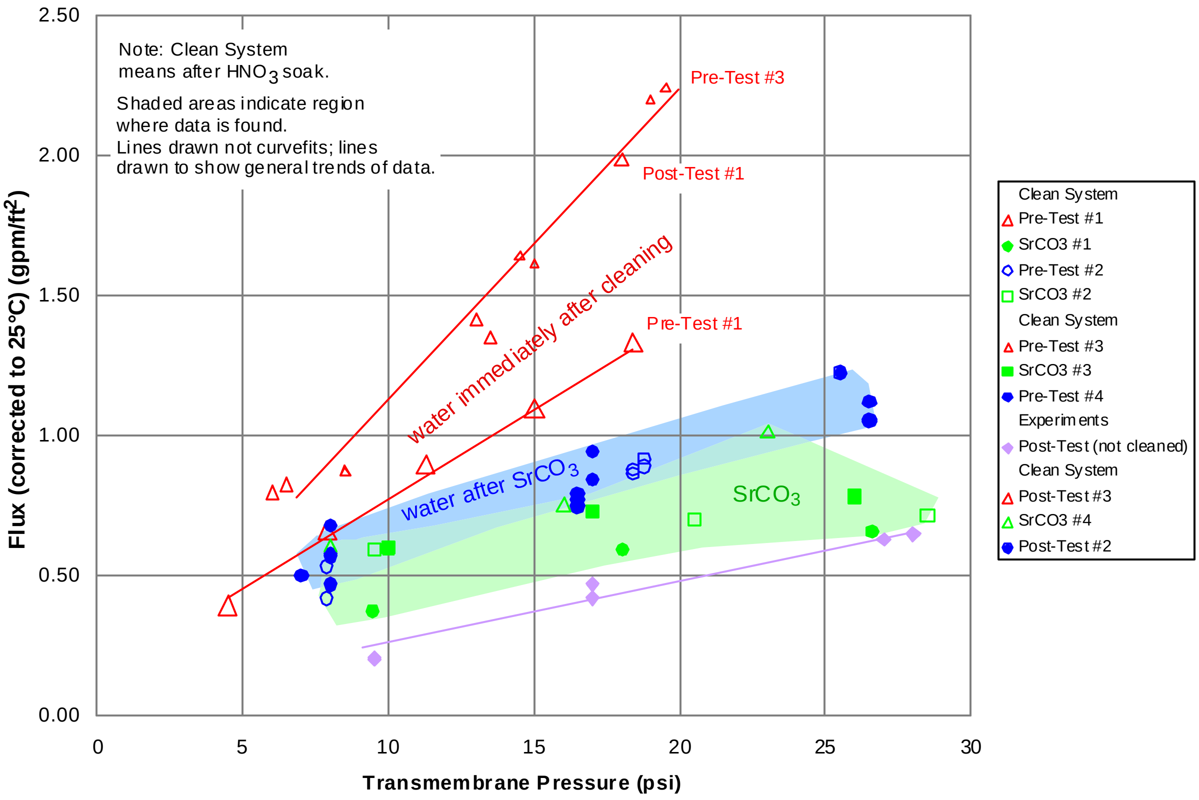


Figure 20 Clean Water and $SrCO_3$ Fluxes – R1BC Run

3.2.8.2 Factorial Design Tests – R1BC

The factorial design conditions for low solids concentration (slurry from precipitate batch #1), pre- and post-run flux tests, and design conditions for the runs with concentrated slurry (at 17.2 wt% insoluble solids) are shown in Table 24. The factorial design used at both low and high solids concentrations is also shown graphically in Figure 21. The factorial arrangement points are shown in solids symbols, while those added to match the Large C filtration work are shown as open symbols.

Each factorial design point was started with multiple backpulses at 45 psi. Filtration at the specified conditions was then started. Several plots of flux versus time are shown in Figure 22-Figure 24. In Figure 22, the fluxes in the first two runs of the set of factorials (1a, 1a’) are much higher than the subsequent values. Generally, it was seen that the first one or two runs immediately after cleaning are always much higher than later ones. This result is consistent with the observations of Nash et. al.[3] Figure 22 also shows that there was little effect of pressure on the flux and that the flux between runs decreases with time. The data in Figure 23 show that at 30 psi TMP, the flux increased with velocity. Overall, the flux was found to increase with velocity but to be for the most part independent of pressure. The effect of run order is shown in Figure 24 where the flux at 40 psi TMP and 11 fps velocity decreases with run order. A complete set of tabulated data and plots of the flux versus time for the low solids factorial and the factorial at 17.2 wt% are given in the Appendix.

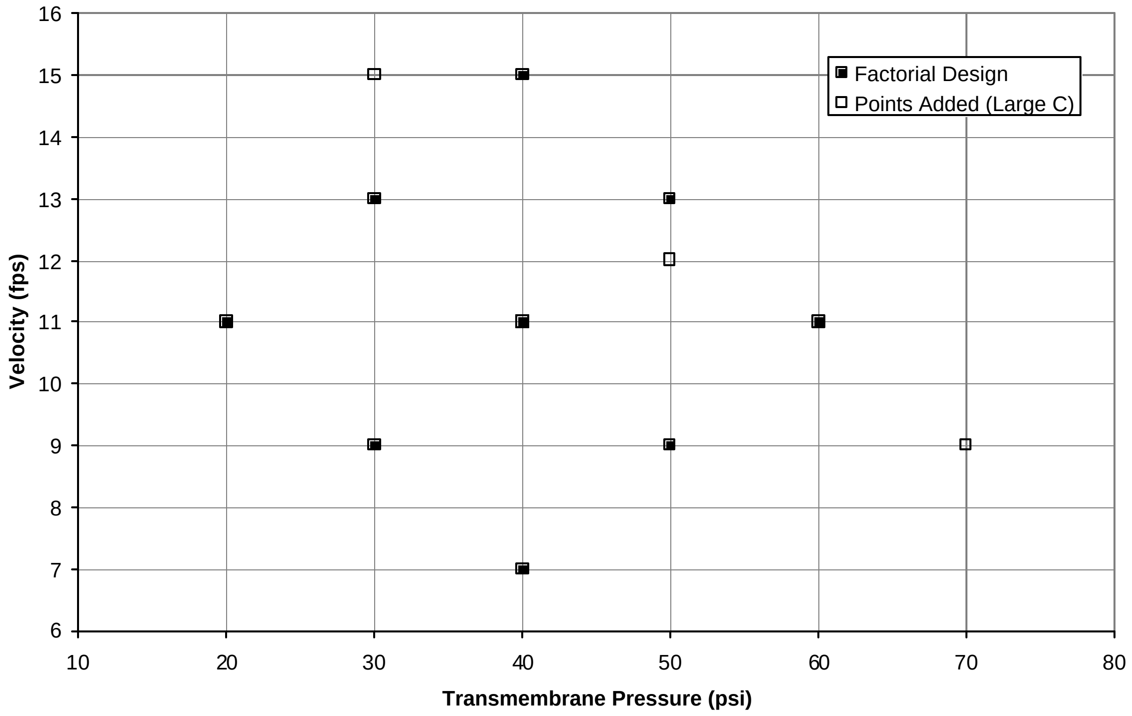
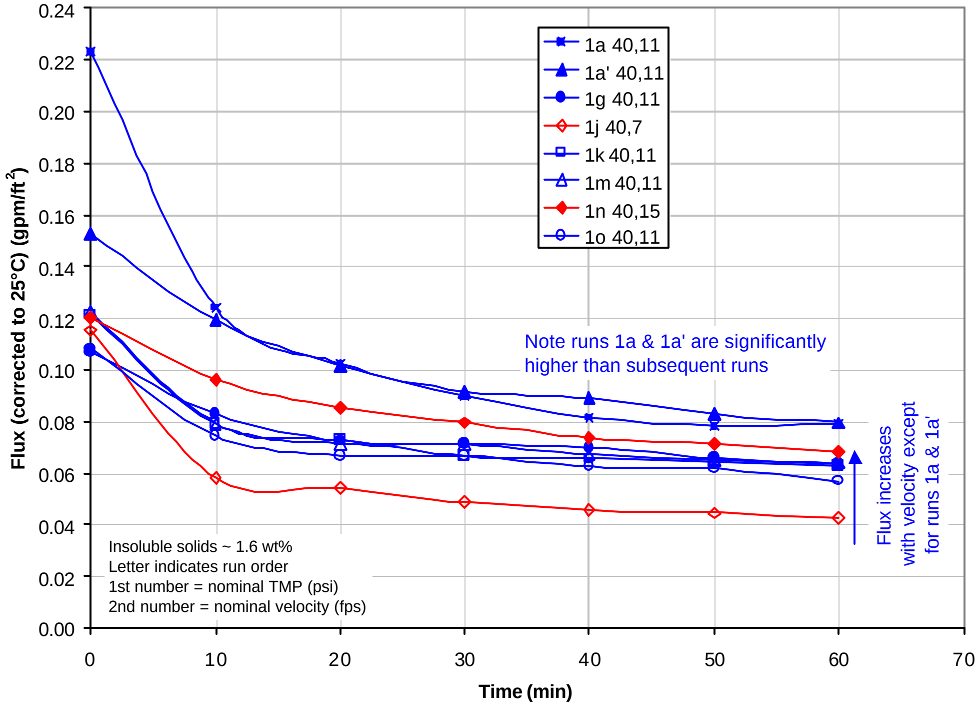


Figure 21 Factorial Design Arrangement: Low and High Solids Concentrations



Note runs 1a & 1a' are significantly higher than subsequent runs

Flux increases with velocity except for runs 1a & 1a'

Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

Figure 22 Low Solids Factorial: Flux versus Time at 11 fps Velocity.

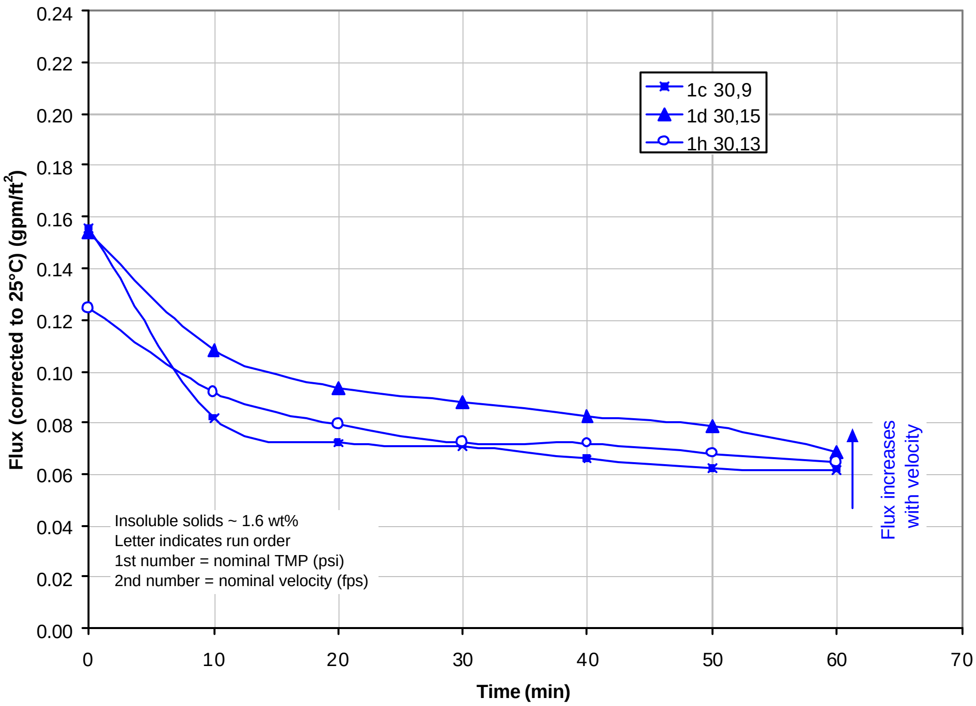


Figure 23 Low Solids Factorial: Flux versus Time at 30 psi TMP.

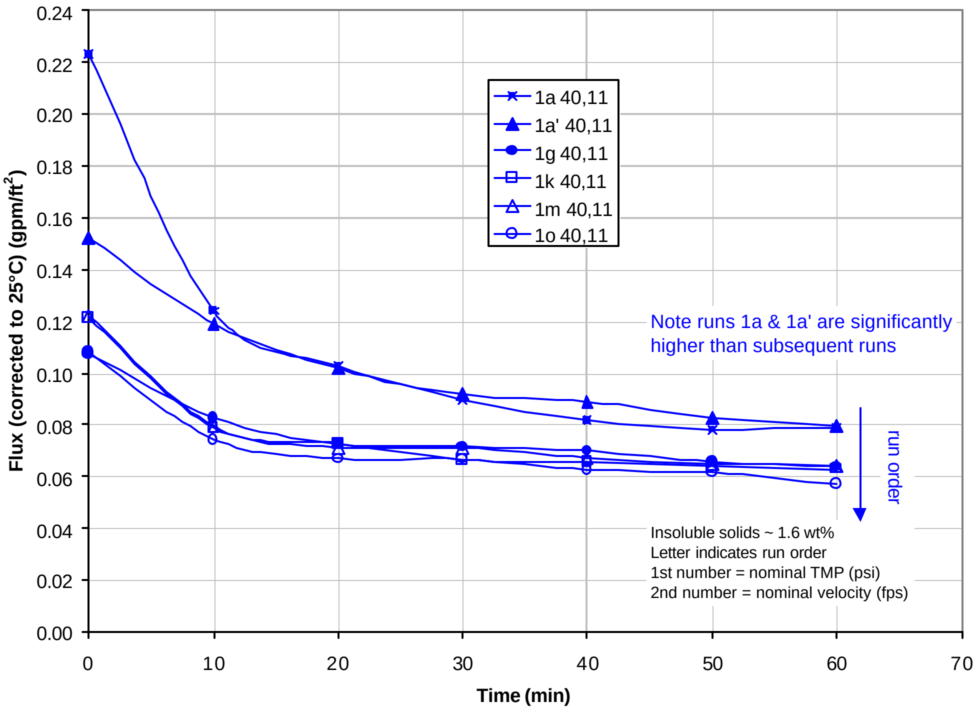


Figure 24 Low Solids Factorial: Flux versus Time at 11 fps Velocity, 40 psi TMP.

The data at the center point of the factorial design (40 psi TMP, 11 fps velocity) are plotted versus time along with data from the AN-102 Small C filtration[12] and the AN-107 pilot-scale simulant filtration[13] in Figure 25. The AN-107 data was fit to an exponential, while the Small

C data was fit to two bounding linear equations shown by the red region on the graph. The pilot AN-107 results are almost exactly the same as the results for this work.

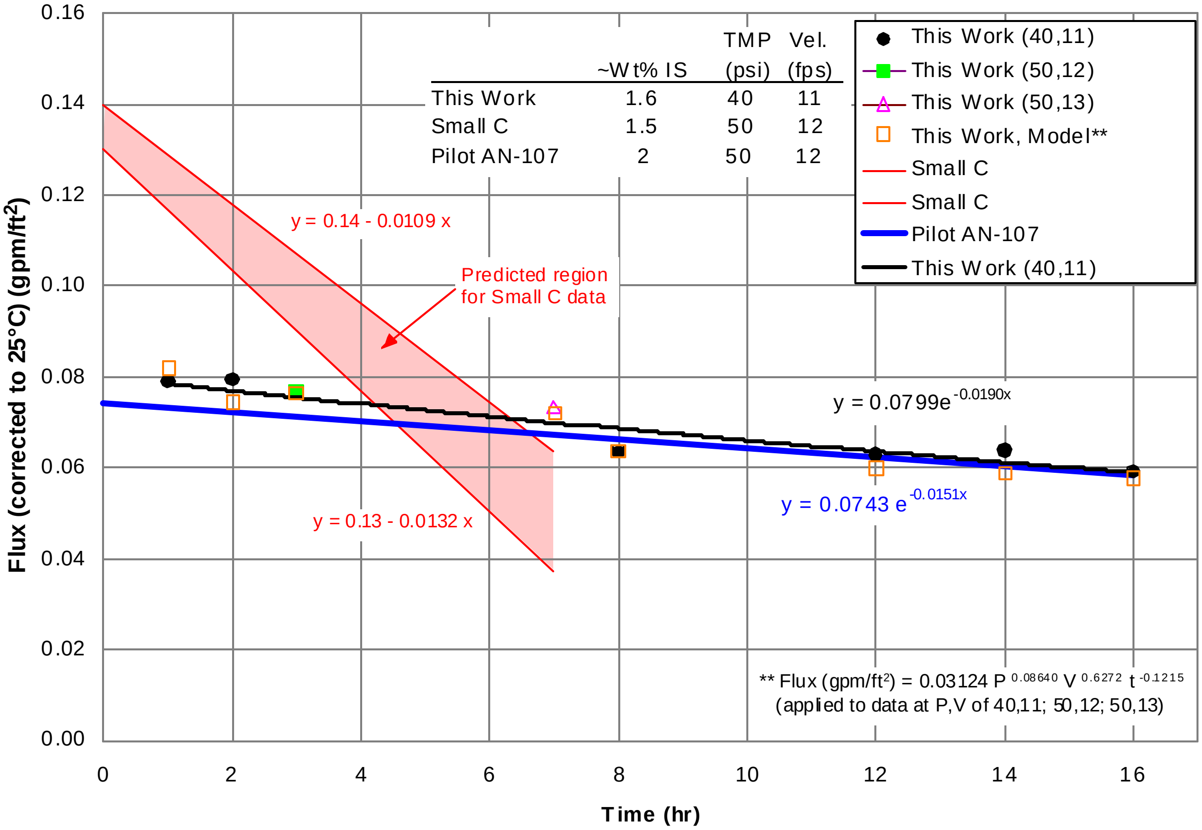


Figure 25 Comparison of Fluxes for this Work, AN-102 Small C, and AN-107 Pilot Simulant Filtration

The flux data at the low solids concentration and after concentration to 17.2 wt% were fit to models with TMP, velocity, and run time as variables. The best fit for the low concentration data was:

$$\text{Flux (gpm/ft}^2) = 0.01324\ P^{0.08640}\ V^{0.6272}\ t^{-0.1215} \qquad \text{Eq. 1}$$

where P = transmembrane pressure (psi)
V = velocity (fps)
t = elapsed filtration time since start of task (hr)

The effect of pressure was only marginally statistically insignificant at a 95% confidence, so it was left in the model. This model predicts that the maximum flux is achieved at the highest velocity and pressure and the lowest elapsed time. Of the conditions tested, the highest velocity was 15 fps, while the TMP was 40 psi, so this is the optimum set of conditions that were tested. The model predicts that even higher velocities and TMPs would give larger fluxes, but these conditions were not actually tested; these predictions are extrapolations beyond the range of the data.

Since the effect of pressure was marginally statistically insignificant, the data was also fit without the pressure dependence. The resulting prediction equation was:

$$\text{Flux (gpm/ft}^2) = 0.01990\ V^{0.5897}\ t^{-0.1227} \qquad \text{Eq. 1a}$$

The velocity and time exponents are both similar to the values found when the pressure effect was included.

At 17.2 wt% insoluble solids, the best fit of the flux data was:

$$\text{Flux (gpm/ft}^2) = 1.1519\ V^{0.5025}\ t^{-1.6473} \qquad \text{Eq. 2}$$

In this case, the effect of pressure was not significant. The highest flux is therefore predicted to occur at the highest velocity. Again, the highest velocity tested was 15 fps. Therefore, for both low and high solids concentrations, the flux is a function of velocity and elapsed filtration time.

The flux at 17.2 wt% insoluble solids was also fit to the same form of model, but with the time dependence found for the low insoluble solids slurry ($t^{-0.1215}$). The fit of the data with this model was not nearly as good as when the exponent on time was allowed to vary. However, the velocity dependence was closer to that seen at low insoluble solids concentrations:

$$\text{Flux (gpm/ft}^2) = 0.0008942\ V^{0.6265}\ t^{-0.1215} \qquad \text{Eq. 2a}$$

The 95% confidence intervals on the exponents on the velocity terms are:

Low Concentration:	0.6272 ± 0.1496	(from Eq. 1)
	0.5897 ± 0.1624	(from Eq. 1a)
High Concentration:	0.5025 ± 0.2330	(from Eq. 2)
	0.6265 ± 0.5275	(from Eq. 2a)

These confidence intervals overlap, so it cannot be concluded that the dependence on the velocity at the low and high insoluble solids concentrations is different. Therefore, this dependence may be the same in both cases. The exponents on the time (Eq. 1 or 1a vs. 2) are different because each set of factorial data (low and high concentration) was fit separately to the available data. Figure 26 shows a comparison of the time dependencies at high insoluble solids. The dependence $t^{-1.6473}$ is stronger than $t^{-0.1215}$, but as shown graphically, this difference in effects is in reality rather insignificant.

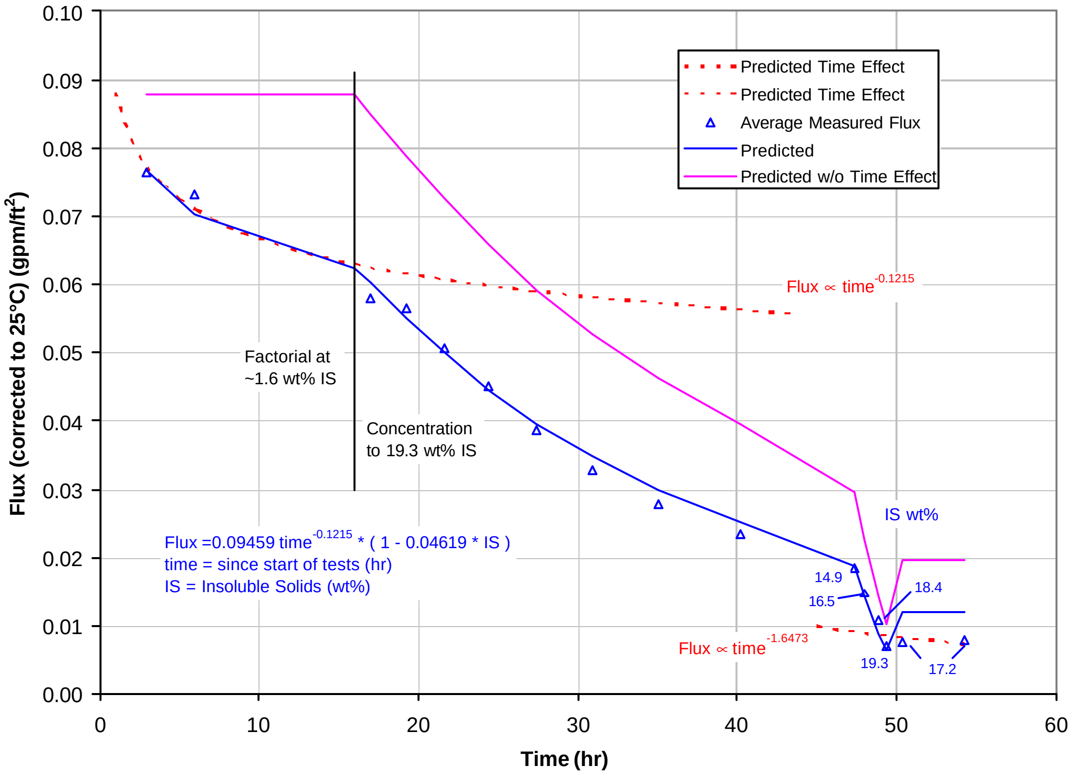


Figure 26 Predicted Flux versus Time for Concentration of Unwashed Slurry

3.2.8.3 Flux versus Insoluble Solids Concentration

After completing the factorial experiments on the first precipitate batch of the R1BC slurry, eight more batches were added and concentrated. The concentrating step of each batch was operated to maintain approximately a constant level in the filter feed tank by matching the permeate flowrate. Each addition of a precipitate batch was preceded by a backpulse. The flux versus time for all of the batches is shown in Figure 27. Note that the concentration steps were all conducted at nominally 50 psi TMP and 12 fps velocity. For each batch concentration, the flux started out higher and dropped to lower values with time, as expected. At the higher concentrations (starting at about 25 hr), this drop is not seen in the data because it took up to 20 minutes to get the first permeate flow reading after backpulsing (time was required to fill the permeate side of the filter and the associated tubing).

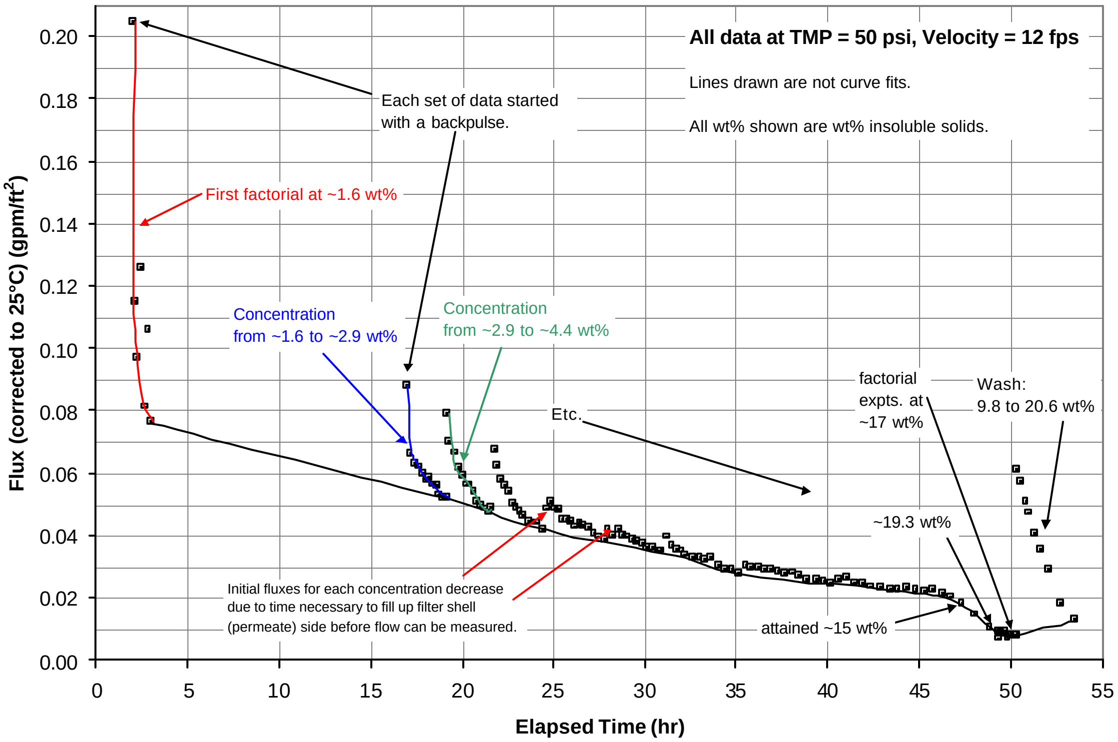


Figure 27 Flux During Concentration Showing Flux Decline

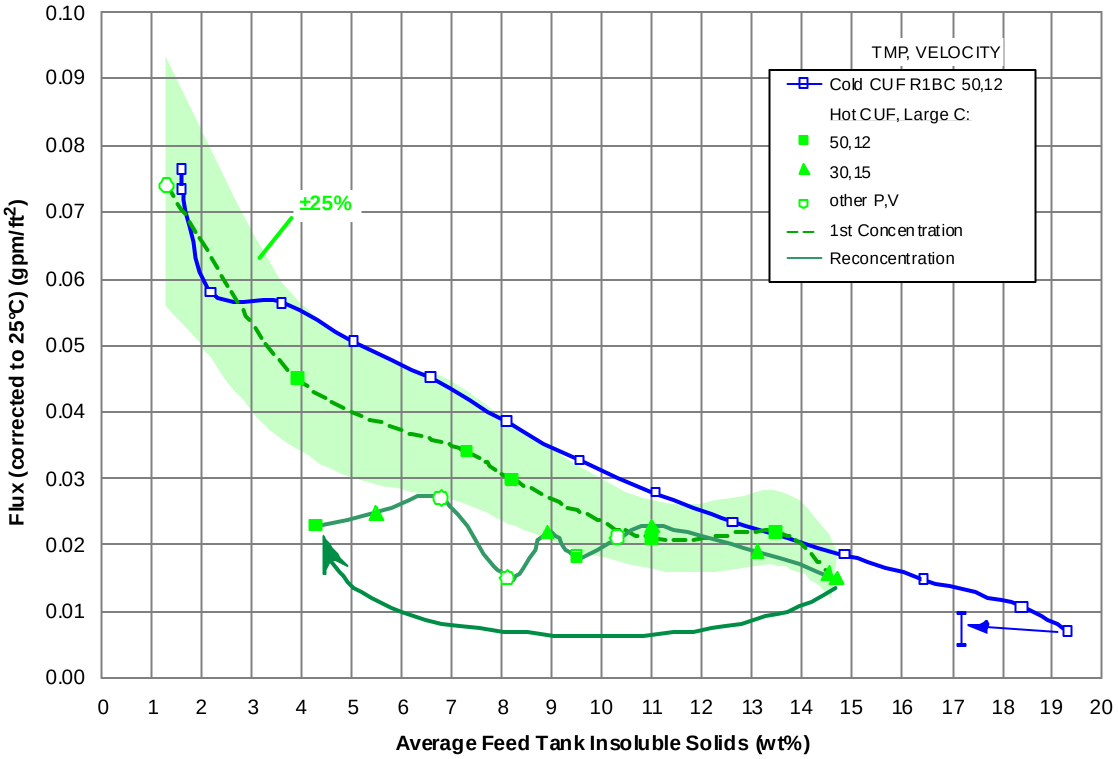


Figure 28 Comparison of Fluxes During R1BC Concentration

Figure 28 shows the R1BC data compared to fluxes measured during the AN-102 Large C work. The green band is a ±25% interval around the fluxes measured during the first concentration step in Large C; the other points for Large C were taken during re-concentration after the concentrated slurry was diluted. Eleven of fifteen of the R1BC data points are within this interval while the other four are within 32%. Four points for the Large C data at P=30, V=15 are also within the interval. Figure 28 also shows that the flux in the Large C work did not recover to the previous values at a given insoluble solids concentration; the R1BC runs show a similar occurrence when the slurry was diluted back to approximately 17.3 wt% insoluble solids for the factorial experiments. Figure 29 shows the R1BC data from Figure 28 along with the flux data taken during re-concentration after washing the concentrated slurry with inhibited water.

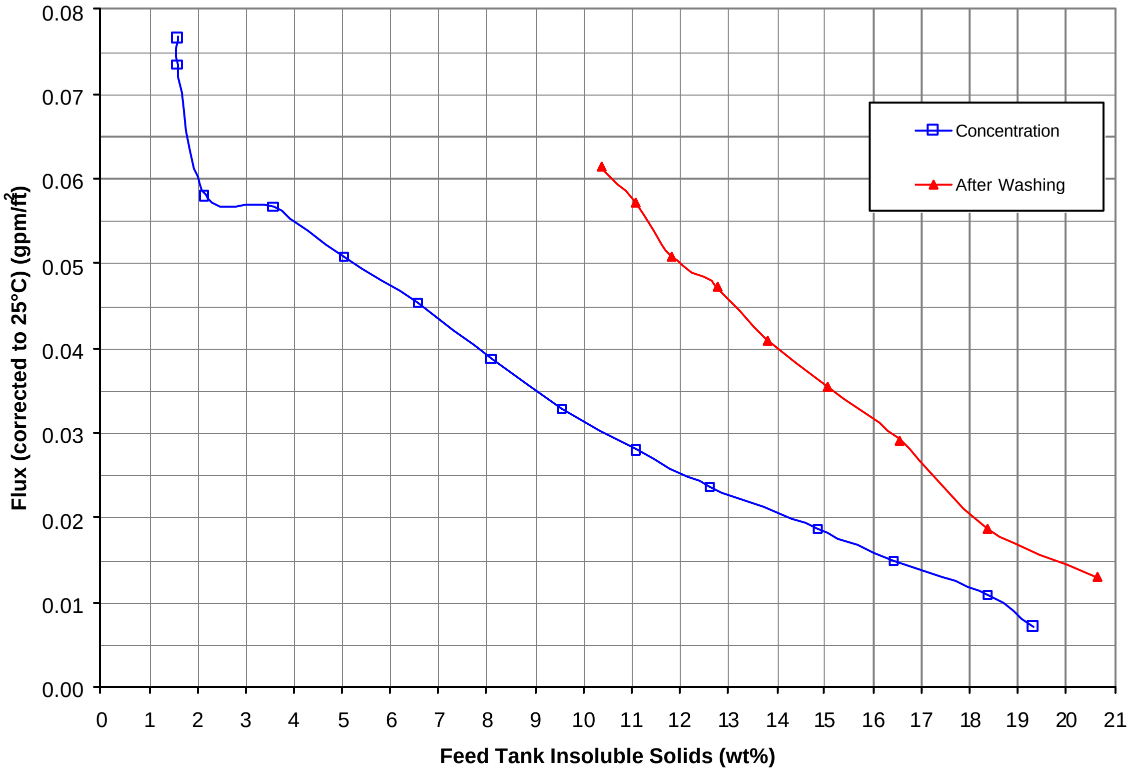


Figure 29 Fluxes for R1BC Concentration and Re-Concentration of Washed Slurry

The minimum average flux for the WTP for the AN-102 waste during dewatering is 0.02 gpm/ft^2.[1] All of the measured fluxes for R1BC for insoluble solids contents below 15 wt% were greater than this value. From 15 to approximately 19.3 wt%, the flux dropped below this value to about 0.007 gpm/ft^2. Even with these low values, the time averaged flux during dewatering, 0.036 gpm/ft^2, was much greater than 0.02 gpm/ft^2.

A simple model of the flux dependence on the insoluble solids content was developed for the R1BC data; this model used the flux decline with time predicted from the low solids factorial data. As shown in Figure 26, the power function drop in flux versus time ($\propto t^{-0.1215}$) was superimposed on the fluxes measured versus insoluble solids content. When the time effect was backed out, the was found to be a linear function of insoluble solids:

Flux (gpm/ft^2) = 0.09459 t$^{-0.1215}$ (1 – 0.04619 IS)

This function predicted all data points quite well, except for those taken during the high concentration factorial, which was performed after re-dilution. As stated previously, the flux after re-dilution was expected to rebound to the previous value at a similar solids content, but it did not. The predicted and measured fluxes are plotted versus insoluble solids content in Figure 30.

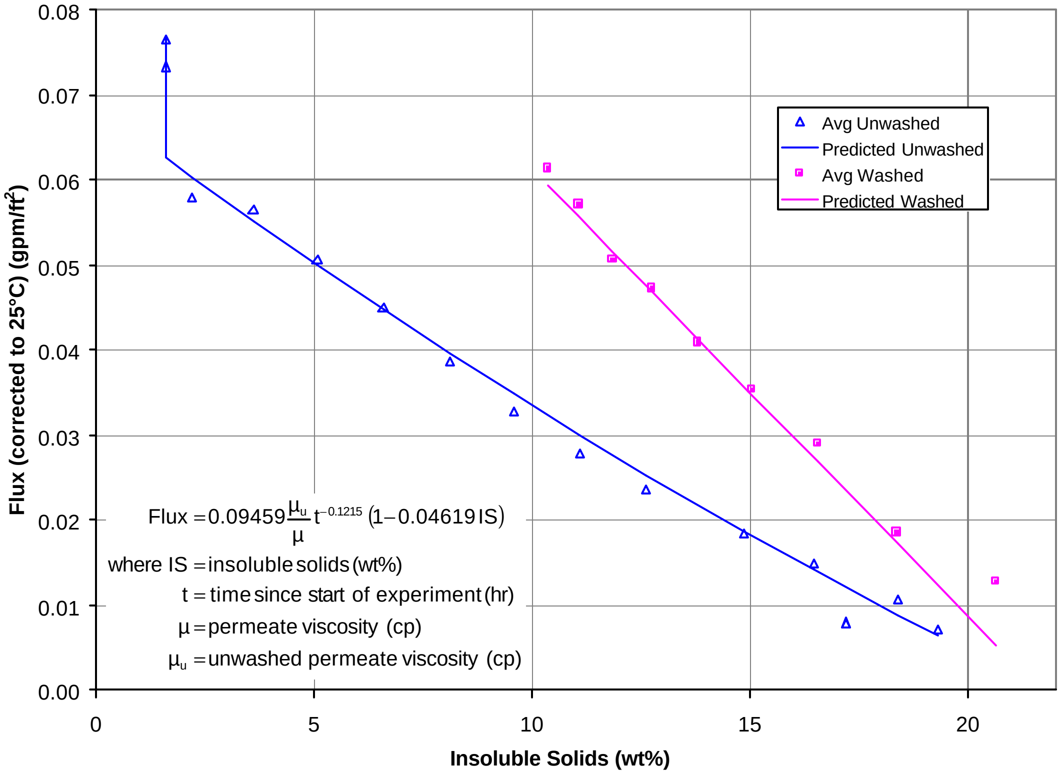


Figure 30 Predicted Flux versus Insoluble Solids Content

After dilution back to 17.2 wt%, the high concentration factorial design was performed, and then the slurry was washed with an equal volume of inhibited water. The flux versus insoluble solids for the concentration of the washed slurry was shown in Figure 28. The permeate viscosities measured were 3.10 and 1.55 cp before and after washing, respectively. Filter flux has been shown to be inversely proportional to permeate viscosity,[14] so a model with viscosity as a parameter was examined. A term for the effects of permeate viscosity was added to the model. The results are shown in Figure 30-Figure 31. The fit of the model is very good for both unwashed and washed slurry. Therefore, the overall model is:

$$\text{Flux} = 0.09459\frac{\mu_u}{\mu}t^{-0.1215}\left(1-0.04619\ \text{IS}\right)$$

where IS = insoluble solids (wt%)

t = time since start of experiment (hr)

μ = permeate viscosity (cp)

μ_u = unwashed permeate viscosity (cp)

The fit of this model is also shown by plotting the measured fluxes adjusted for the time factor and the permeate viscosity in Figure 32.

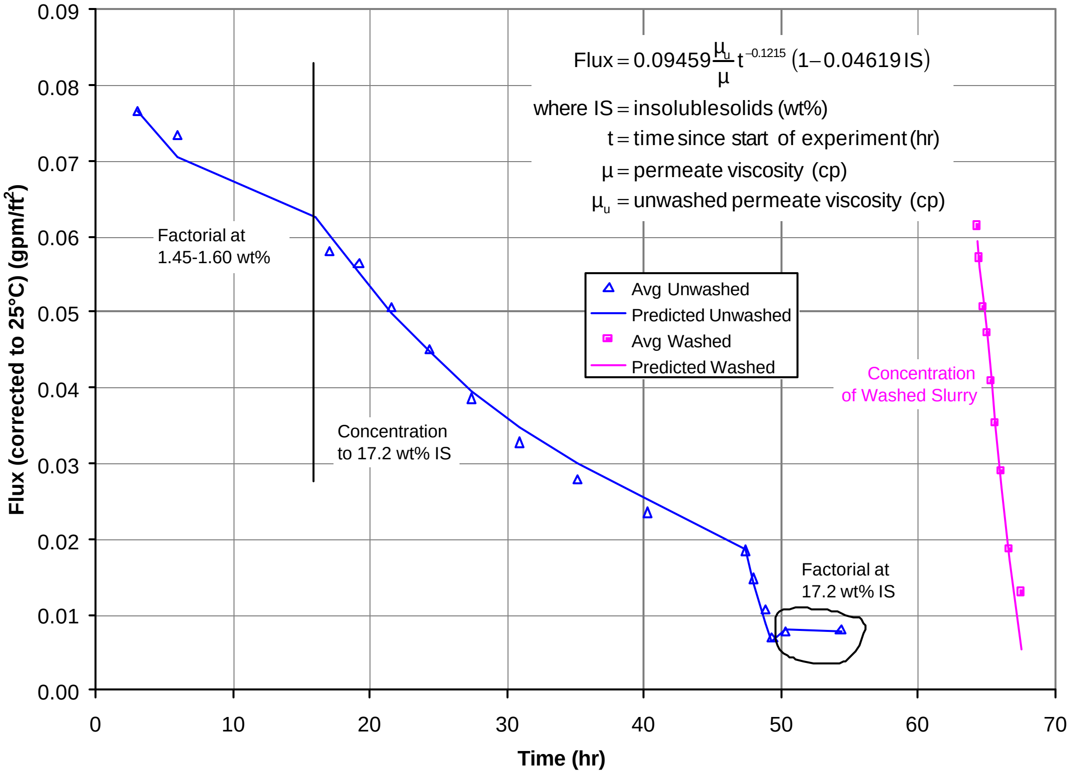


Figure 31 Predicted Flux versus Time for Concentration of Unwashed & Washed Slurry

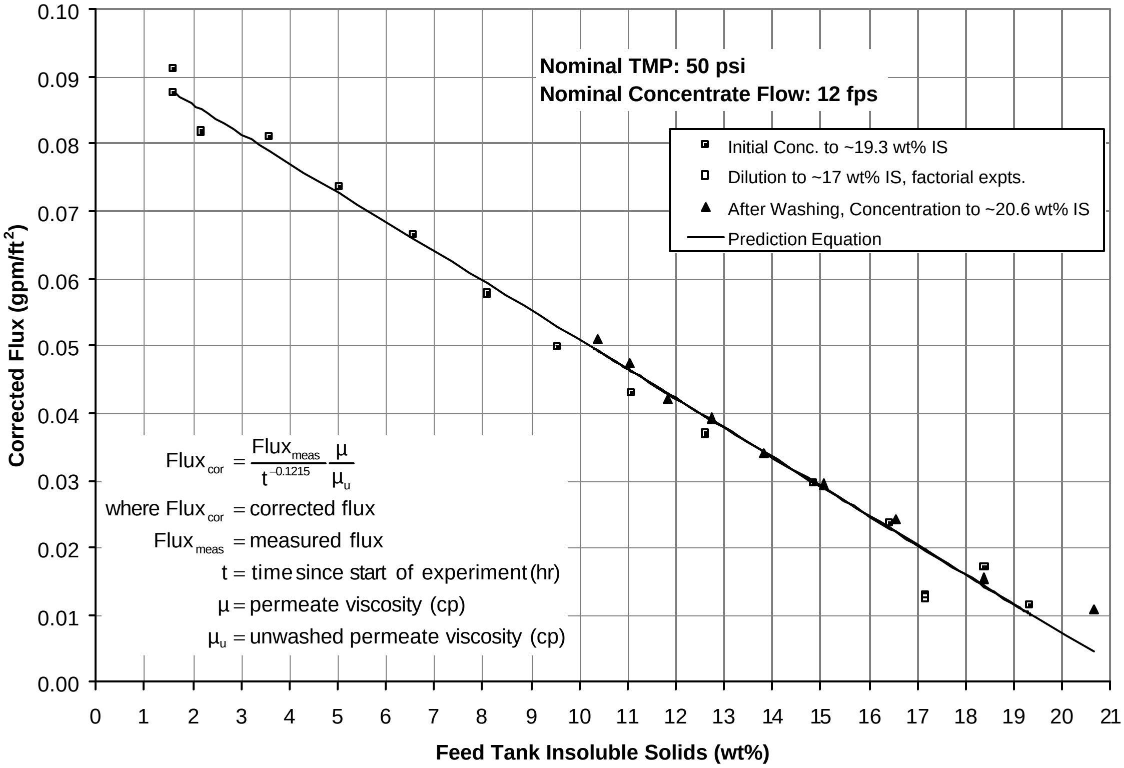


Figure 32 Flux Corrected for Time & Viscosity

3.2.9 Filtration – Remediation 2 Runs

The filtration fluxes versus insoluble solids content for the R2NOC runs (R2NOC1, R2NOC small, R2NOC2, pilot R2NOC) are shown in Figure 33. The initial R2NOC1 fluxes were less than the minimum 0.02 gpm/ft^2, so this run was immediately stopped. the small R2NOC run initial flux was about 0.067 gpm/ft^2, so the precipitation was repeated (R2NOC2). The initial flux was >0.119 gpm/ft^2 and it remained greater than 0.02 gpm/ft^2 until about 8 wt% insoluble solids was reached. After this, the flux dropped to approximately 0.007 gpm/ft^2 and the time-averaged flux was 0.013 gpm/ft^2. In Figure 33, the fluxes from a parallel run of the R2NOC conditions in the pilot unit are shown. (NOTE: The pilot unit fluxes given in this report are preliminary data and were not published at the time of this report. Refer to the pilot filtration program report (to be published) for the most accurate values. In the pilot unit, the flux dropped almost immediately below 0.02 gpm/ft^2, so the performance of the CUF and the pilot was not comparable with a similar feed. The CUF data, after about 5 wt% insoluble solids, was less than 50-65% of the Large C fluxes, while the pilot was only about 15-20% of the Large C results.

The R2 simulant with baseline conditions results are shown in Figure 34. The R2BC CUF and pilot runs were made using the same precipitate produced in the pilot precipitation unit. The CUF fluxes were typically twice both the Large C and the pilot unit results. The pilot results agreed very well with the Large C results. Figure 35 shows the CUF baseline conditions runs compared to the Large C results. One major difference between the R2BCL and the R1BC and Large C runs was that for R1BC and Large C, individual small batches of precipitate were made each day and then filtered so that little aging of the newly produced precipitate occurred. In contrast, the R2BCL run used material that was all produced at the same time, so the material used later in the concentration step was aged more than the earlier material.

For the same feed material, the cold CUF fluxes were always about a factor of two or higher than the pilot unit results. Possible reasons for this behavior can be given, but at this point are only hypotheses. The three possible reasons are:

1. The particle shear of the pilot centrifugal pump is greater than the CUF progressive cavity pump, resulting in smaller particles or a different particle size distribution.
2. The shear stress at the wall for the same axial velocity is higher in the CUF than in the pilot, which would be expected to result in higher fluxes.
3. The pilot unit tubes are longer than the CUF tubes. Therefore, a higher percentage of the length of the CUF tubes is subject to entrance effects, which would tend to give higher fluxes in the CUF.

All of these reasons result in higher fluxes in the CUF compared to the pilot unit.

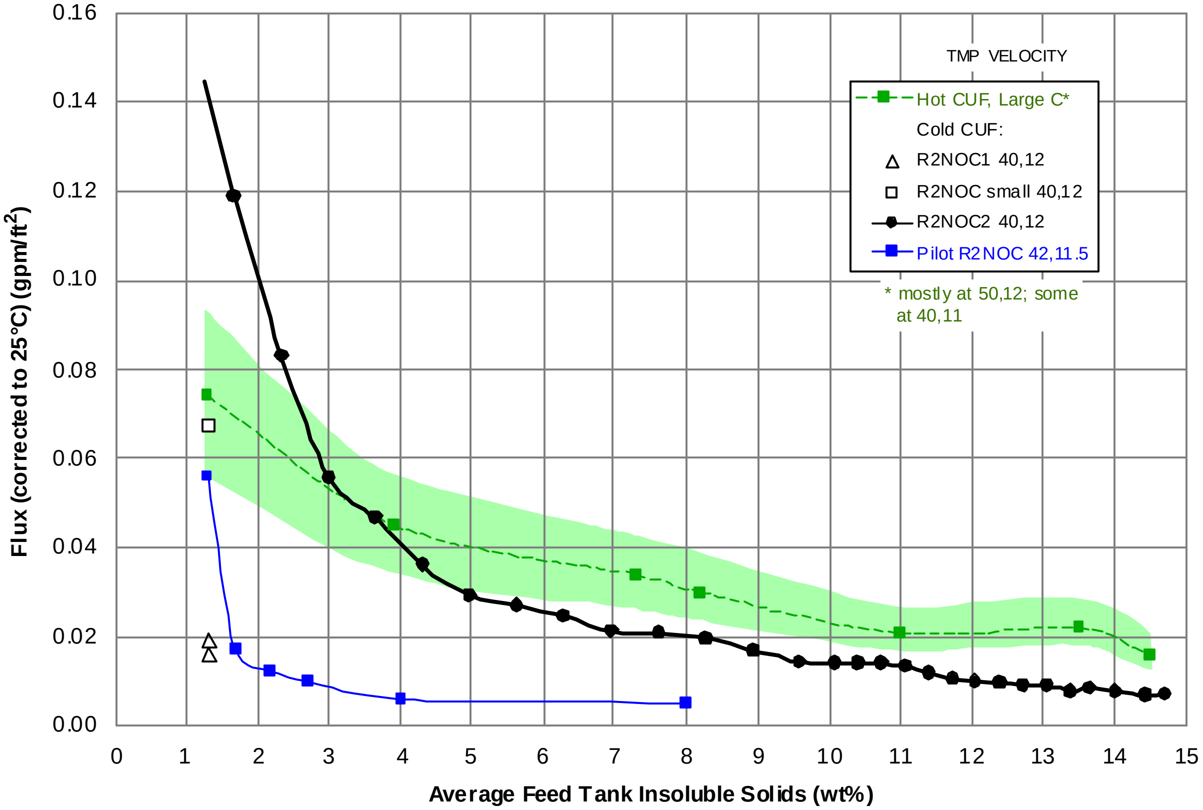


Figure 33 R2NOC Filtration Fluxes versus Insoluble Solids

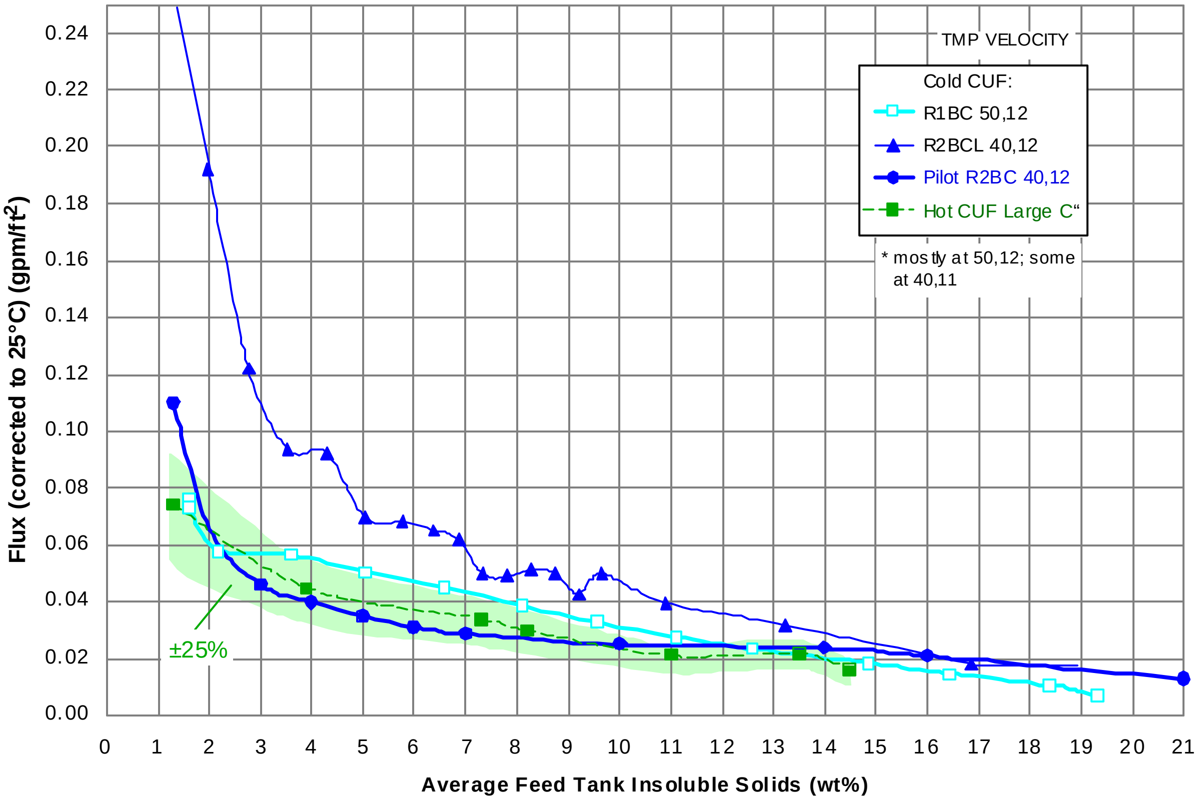


Figure 34 Baseline Conditions Fluxes versus Insoluble Solids

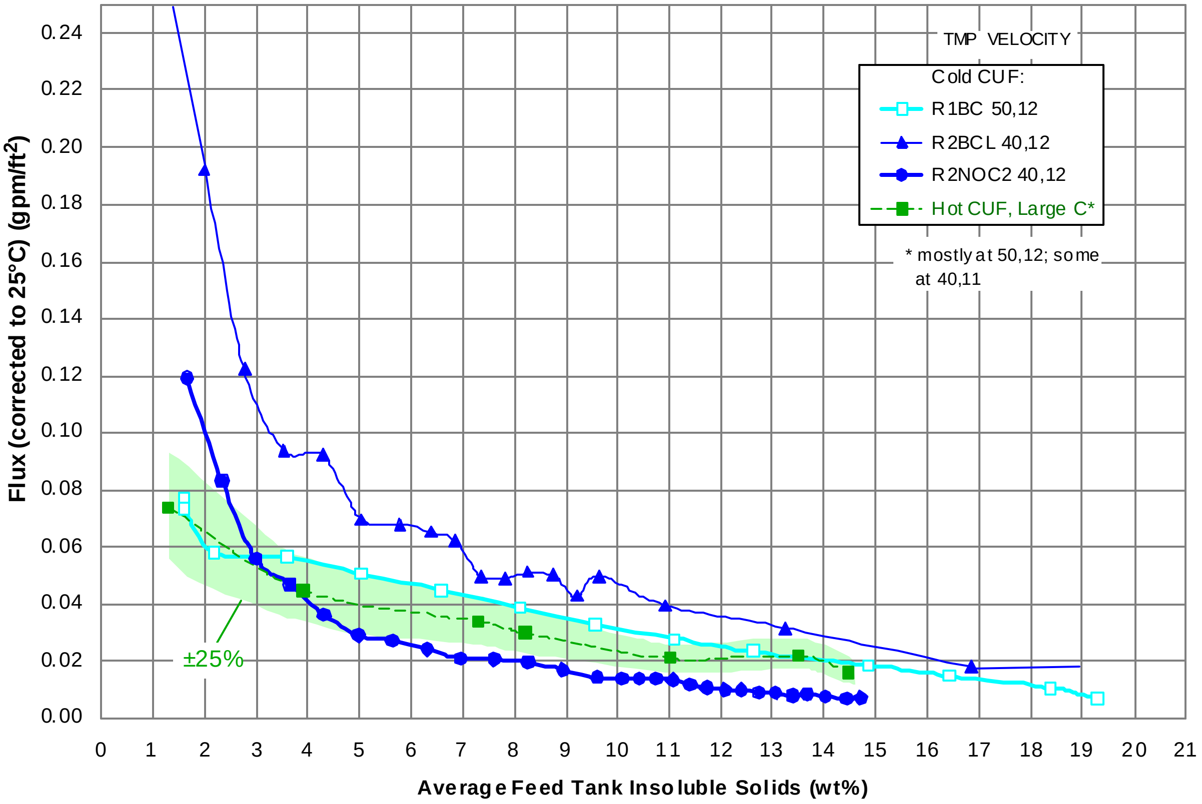


Figure 35 Flux Comparison of CUF Concentrations with Large C

4.0 FUTURE WORK

SRTC recommends the following further work:

1. Experimental work, probably on a large scale, should be performed to assure that the precipitation vessel will have adequate mixing so that the precipitate formed is filterable.
2. Related to item 1, the effect of mixing on the filterability of the precipitate should be investigated to determine if mixing has a significant influence on filtration.
3. Future optimization of the precipitation reactions should be coupled with crossflow filter tests to assure that the precipitate will be filterable. These tests should take the precipitate to at least 10 wt% insoluble solids, and preferably beyond.
4. The adequacy of mixing in the filter feed vessel should be tested with concentrated slurry (15 wt% or greater). The high consistency and especially the high yield stresses found for the concentrated slurry will make assuring adequate mixing difficult.

5.0 REFERENCES

[1] P. S. Townson, *AN-102 Simulant Sr/TRU Precipitation and Ultrafiltration Test Specification*, **24590-WTP-TSP-RT-01-019, Rev. 0**, River Protection Project – Waste Treatment Plant, Richland, WA (2001).

[2] M. R. Poirier, *Task Technical and Quality Assurance Plan for AN-102 Simulant Sr/TRU Precipitation and Ultrafiltration*, **SRT-RPP-2002-00007, Rev. 0, (WSRC-TR-2002-00038, Rev. 0)**, Savannah River Site, Aiken, SC (2002).

[3] C. A. Nash, H. H. Saito, W. R. Wilmarth, *Strontium-Transuranic Precipitation and Crossflow Filtration of 241-AN-102 Large C,* **WSRC-TR-2000-00506, Rev. 0 (SRT-RPP-2001-00006, Rev. 0)**, Savannah River Site, Aiken, SC (2000).

[4] M. R. Williams, *Task Technical and Quality Assurance Plan for RPP-WTP Pilot-Scale Precipitation Testing,* **SRT-RPP-2000-0050, Rev 2**, Savannah River Site, Aiken, SC (2002).

[5] P. S. Townson, *AN-102 Simulant Pilot Scale Ultrafiltration Test Specification,* **24590-WTP-TSP-RT-01-029, Rev. 0**, River Protection Project – Waste Treatment Plant, Richland, WA (2002).

[6] R. E. Eibling, *Development of a Supernate Simulant for Hanford Tank 241-AN-102 Waste*, **SRT-RPP-2002-00012. Rev. 0, DRAFT B (WSRC-TR-2002-00040, Rev. 0, DRAFT B)**, Savannah River Site, Aiken, SC (2002).

[7] C. J. Bannochie, *Standardized Baseline Precipitation Procedure for Hanford RPP Studies,* **WSRC-TR-2002-00138, Rev. 0 (SRT-RPP-2002-00073, Rev. 0)**, Savannah River Site, Aiken, SC (2002).

[8] P. S. Townson, electronic mail to J. R. Zamecnik, dated 4/22/02 (attached in Appendices).

[9] W. R. Wilmarth, V. H. Dukes, J. J. Mills, F. F. Fondeur, C. C. DiPrete and D. P. DiPrete, *Optimization Study for Strontium and Actinide Removal from 241-AN-107 Supernate*, **SRT-RPP-2002-00136. Rev. 0 (WSRC-TR-2002-00258, Rev. 0),** Savannah River Site, Aiken, SC (2002).

[10] E. K. Hansen, *Pipe Pressure Drop Calculation for Bingham Plastic, Power Law and Newtonian Fluids*, **SRT-RPP-2001-00226, Rev. 1**, Savannah River Site, Aiken, SC (2002).

[11] J. R. Zamecnik, M. A. Baich, *Evaluating The Effects Of Tri-Butyl Phosphate And Normal Paraffin Hydrocarbon In Simulated Low-Activity Waste Solution On Ultrafiltration*, **SRT-RPP-2002-00041, Rev. 0, WSRC-TR-2002-00108, Rev. 0**, Savannah River Site, Aiken, SC (2002).

[12] C. A. Nash, S. W. Rosencrance, W. R. Wilmarth, *Entrained Solids, Strontium-Transuranic Precipitation, and Crossflow Filtration of AN102 Small C*, **SRT-RPP-2000-0003, Rev. 0 (WSRC-TR-2000-00341, Rev. 0)**, Savannah River Site (2000).

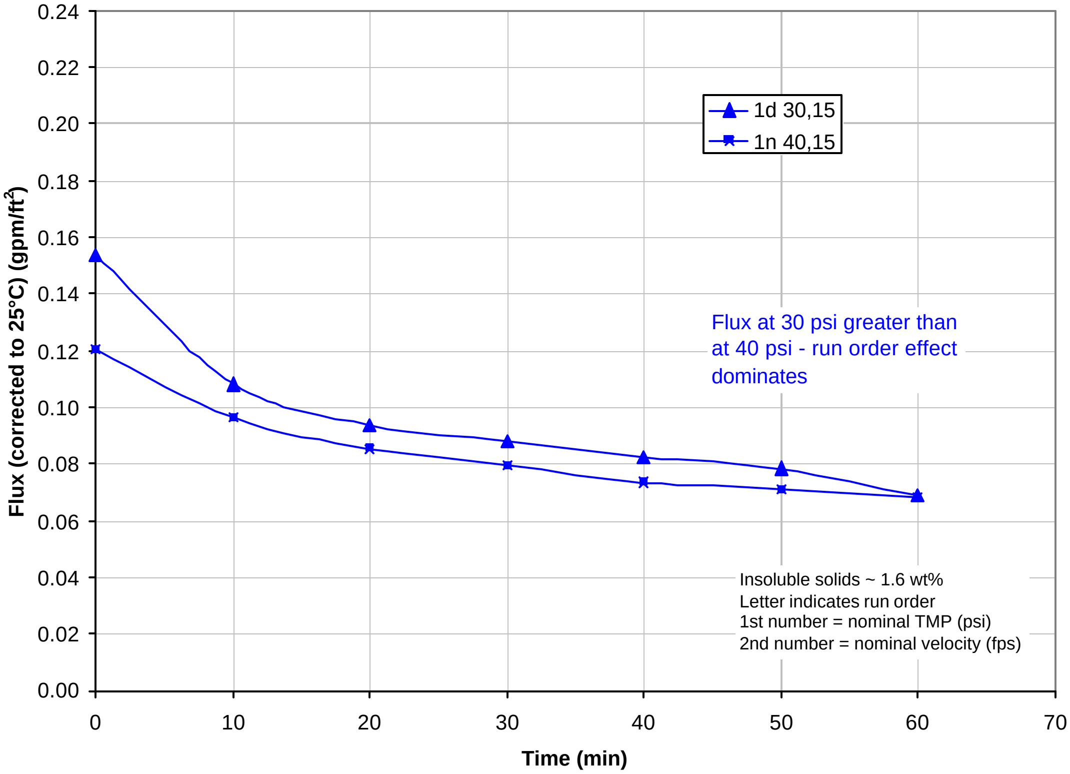
5.0 REFERENCES (CONTINUED)

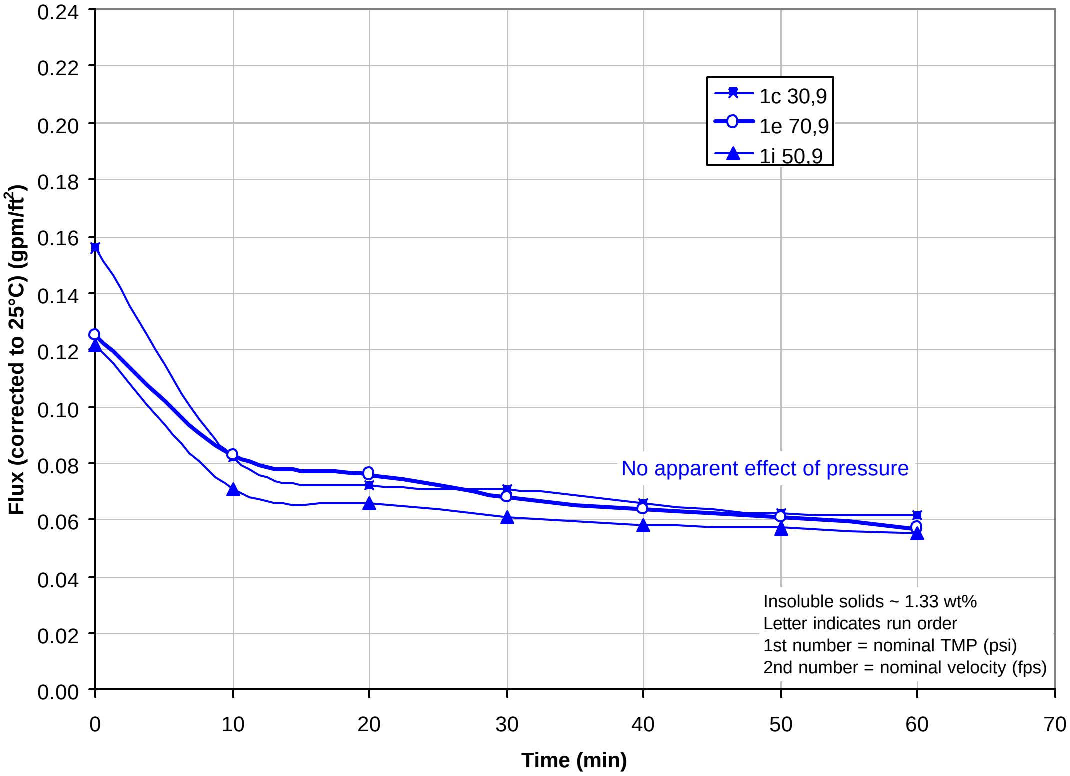
[13] M. R. Duignan, *Final Report: Pilot-scale Cross-flow Ultrafiltration Test Using a Hanford Site Tank 241-AN-107 Waste Simulant – Envelope C + Entrained Solids + Strontium-Transuranic Precipitation*, **BNF-003-98-0226, Rev. 0**, Savannah River Site (2000).

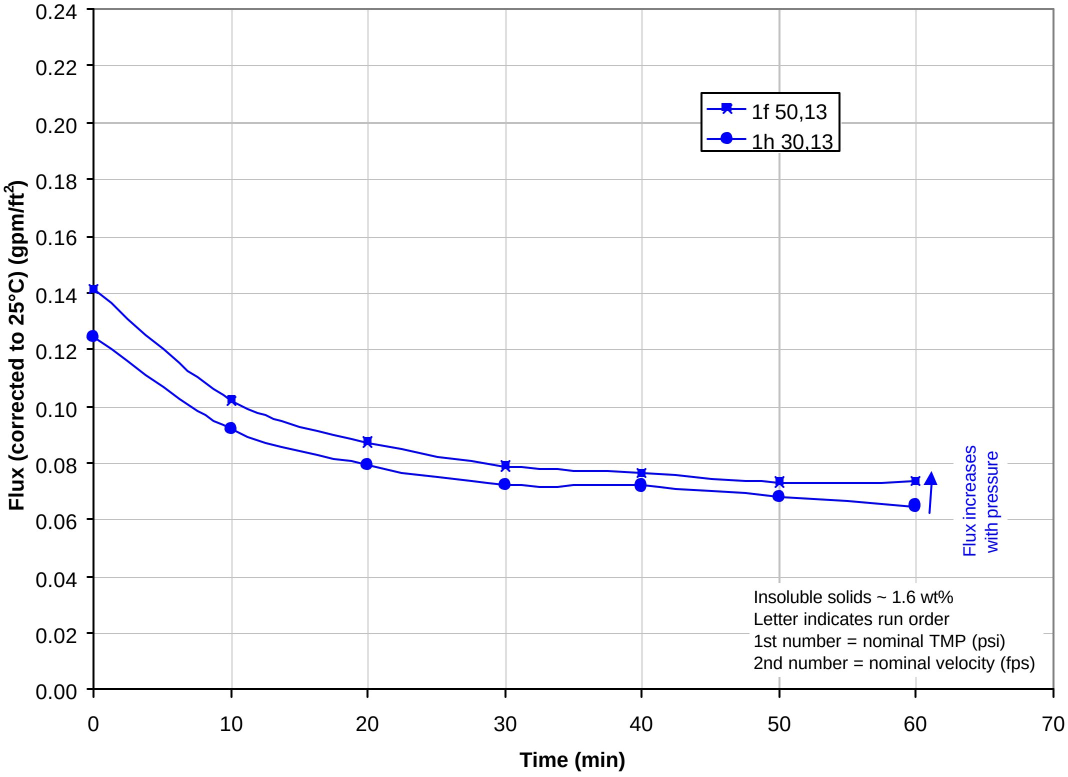
[14] M. C. Porter, *Handbook of Industrial Membrane Technology*, Noyes Publications, Park Ridge, NJ, p. 77 (1990).

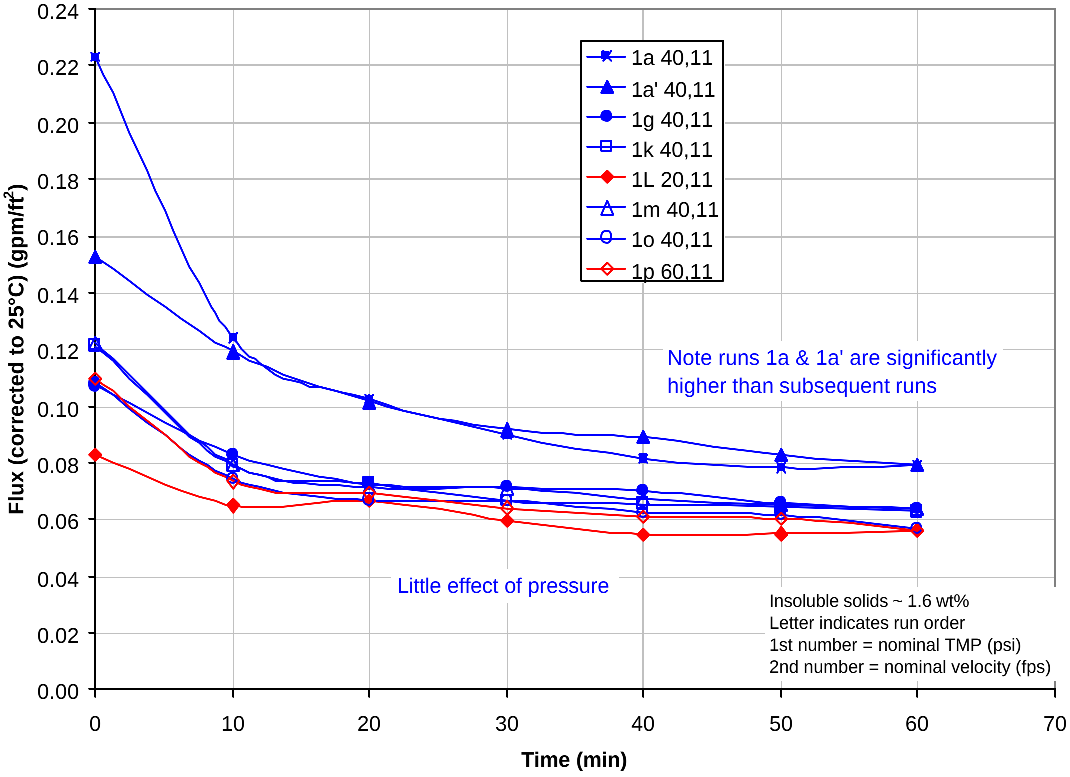
APPENDIX A1. GRAPHICAL FLUX DATA

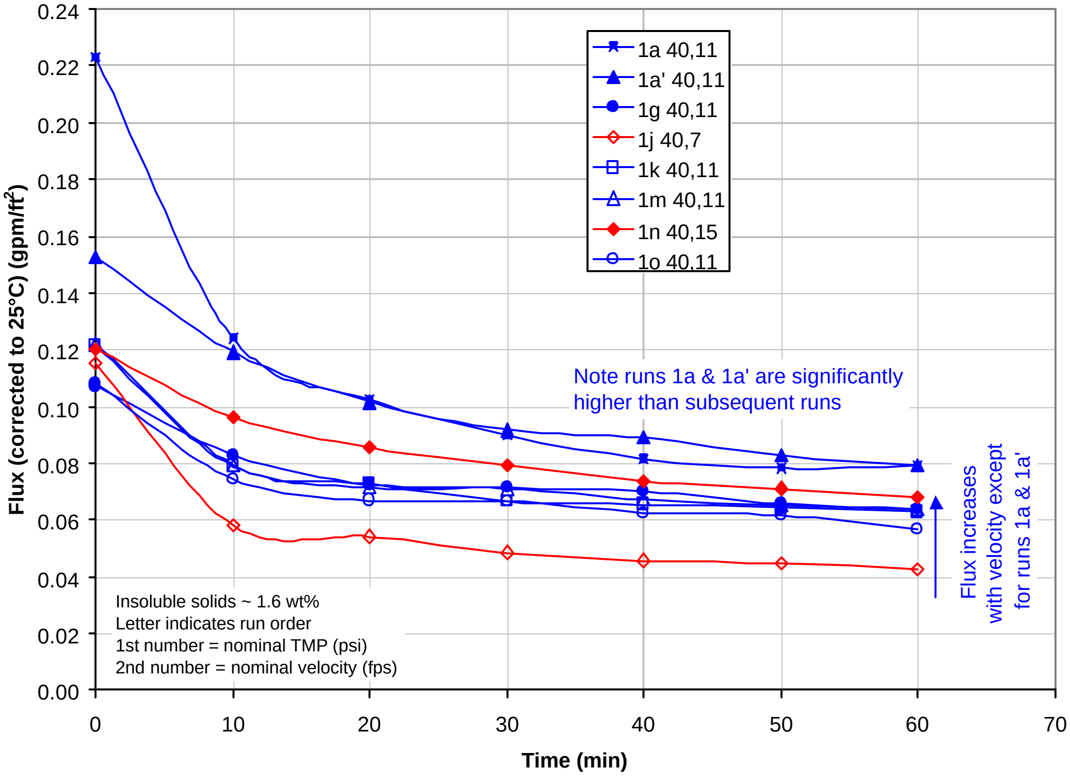
Graphs with data points numbered “1_” are for the low concentration slurry (approximately 1.45-1.60 wt% IS) factorial, whereas those numbered “15_” are for the factorial at about 17.2 wt% insoluble solids.

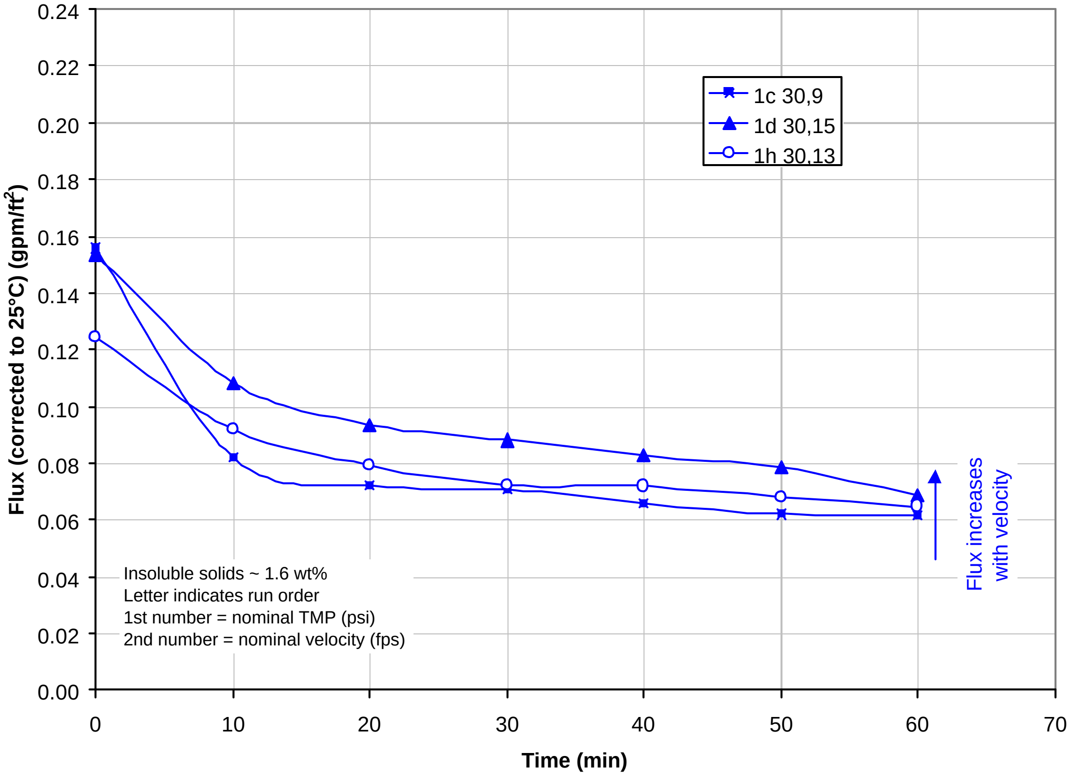

Flux (corrected to 25°C) (gpm/ft²)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1d 30,15
1n 40,15
Flux at 30 psi greater than at 40 psi - run order effect dominates
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

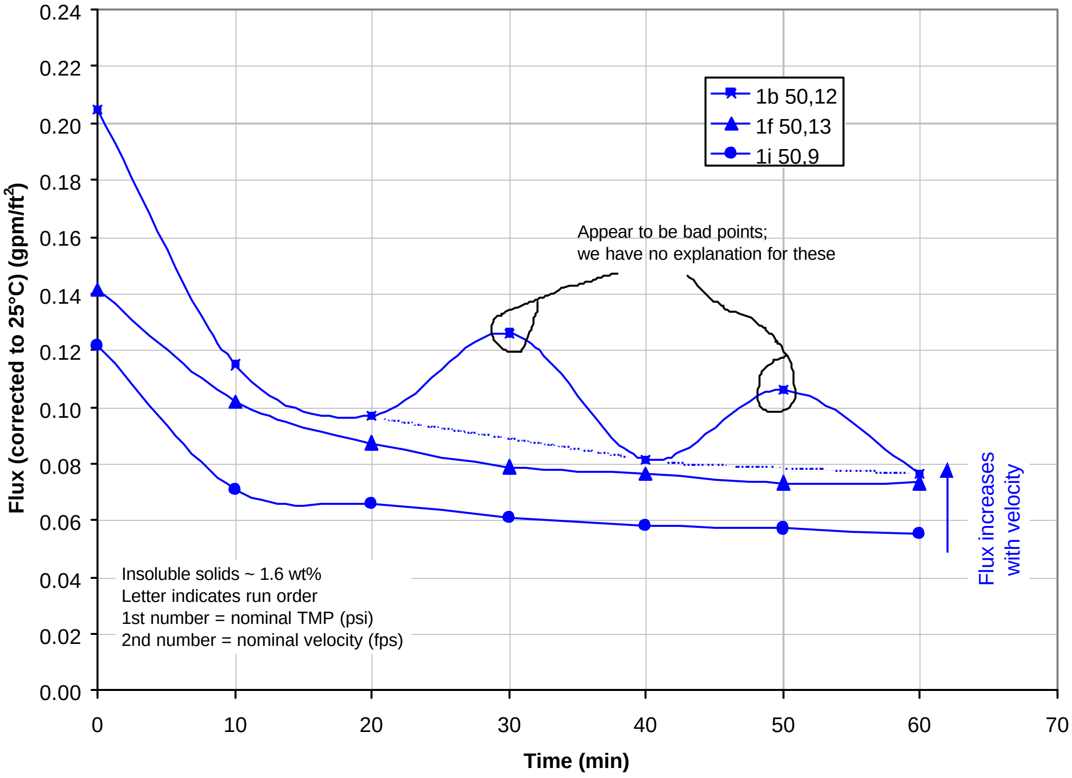

Flux (corrected to 25°C) (gpm/ft²)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1c 30,9
1e 70,9
1i 50,9
No apparent effect of pressure
Insoluble solids ~ 1.33 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

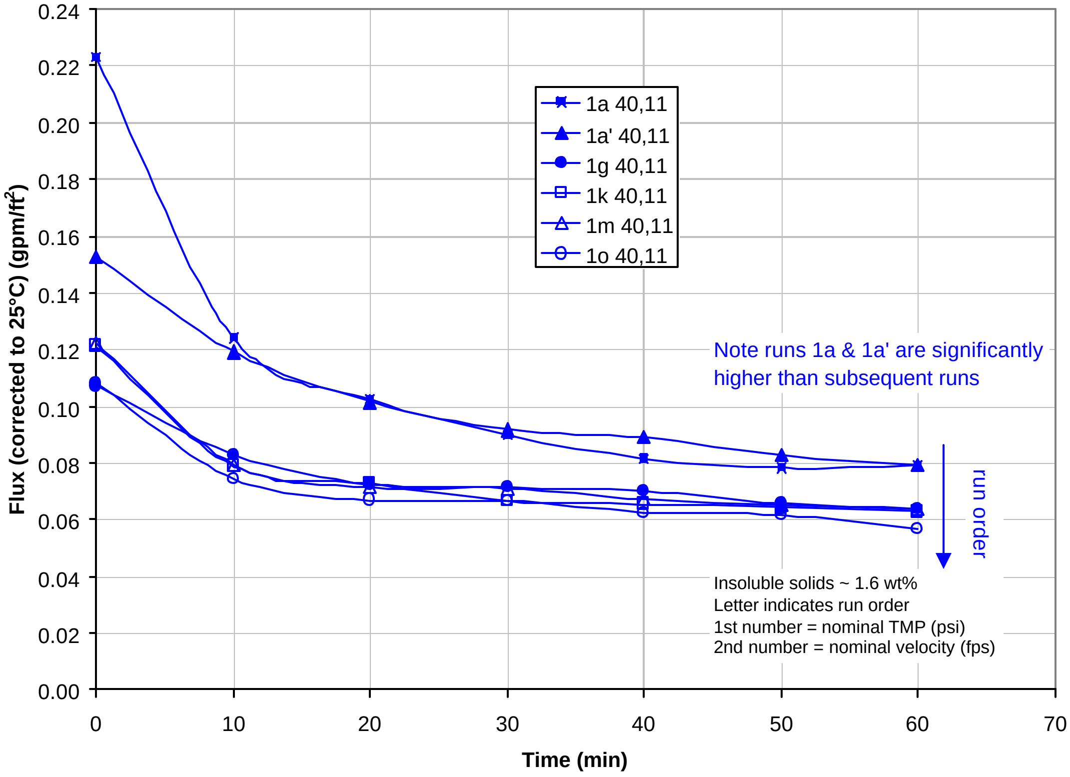

Flux (corrected to 25°C) (gpm/ft²)
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14
0.16
0.18
0.20
0.22
0.24
0
10
20
30
40
50
60
70
Time (min)
1f 50,13
1h 30,13
Flux increases with pressure
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

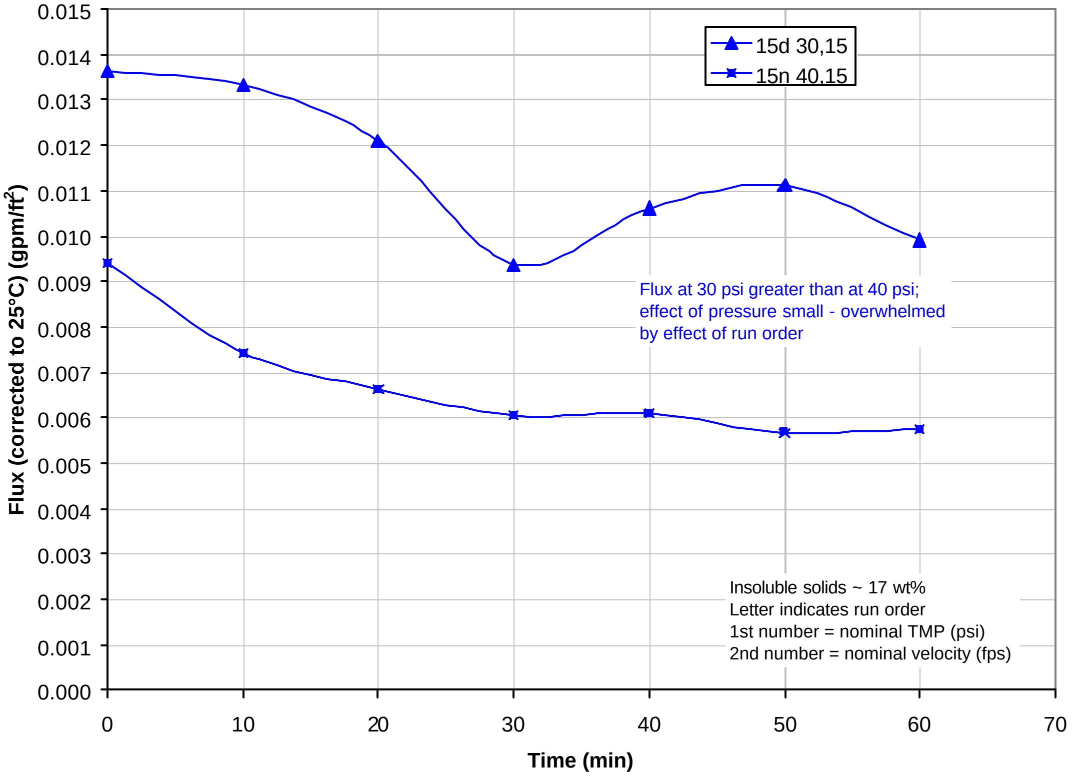

Flux (corrected to 25°C) (gpm/ft²)
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14
0.16
0.18
0.20
0.22
0.24
0
10
20
30
40
50
60
70
Time (min)
1a 40,11
1a' 40,11
1g 40,11
1k 40,11
1L 20,11
1m 40,11
1o 40,11
1p 60,11
Note runs 1a & 1a' are significantly higher than subsequent runs
Little effect of pressure
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

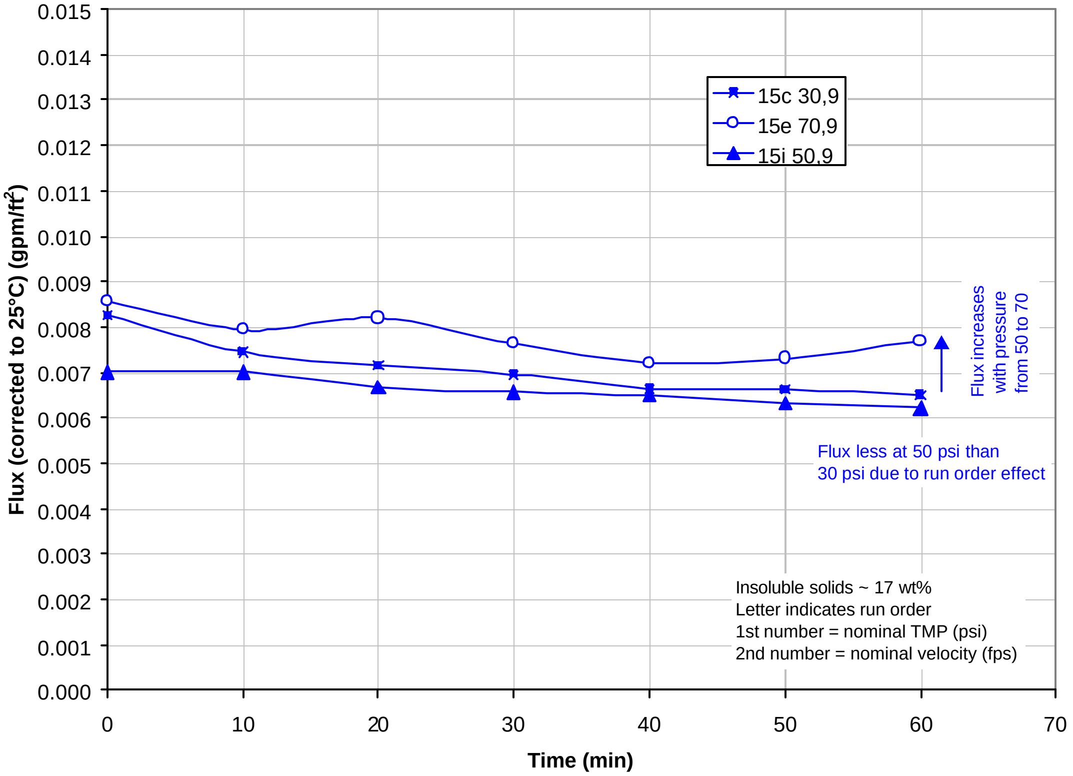

Flux (corrected to 25°C) (gpm/ft^2)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1a 40,11
1a' 40,11
1g 40,11
1j 40,7
1k 40,11
1m 40,11
1n 40,15
1o 40,11
Note runs 1a & 1a' are significantly higher than subsequent runs
Flux increases with velocity except for runs 1a & 1a'
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

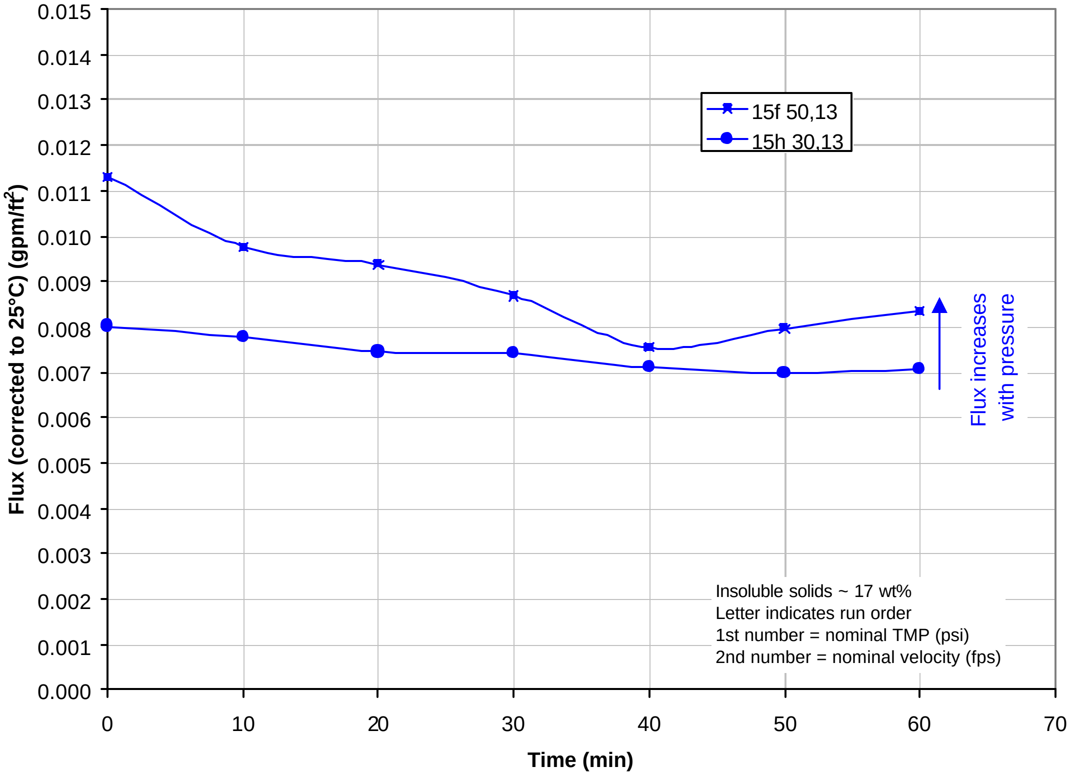

Flux (corrected to 25°C) (gpm/ft^2)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1c 30,9
1d 30,15
1h 30,13
Flux increases with velocity
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

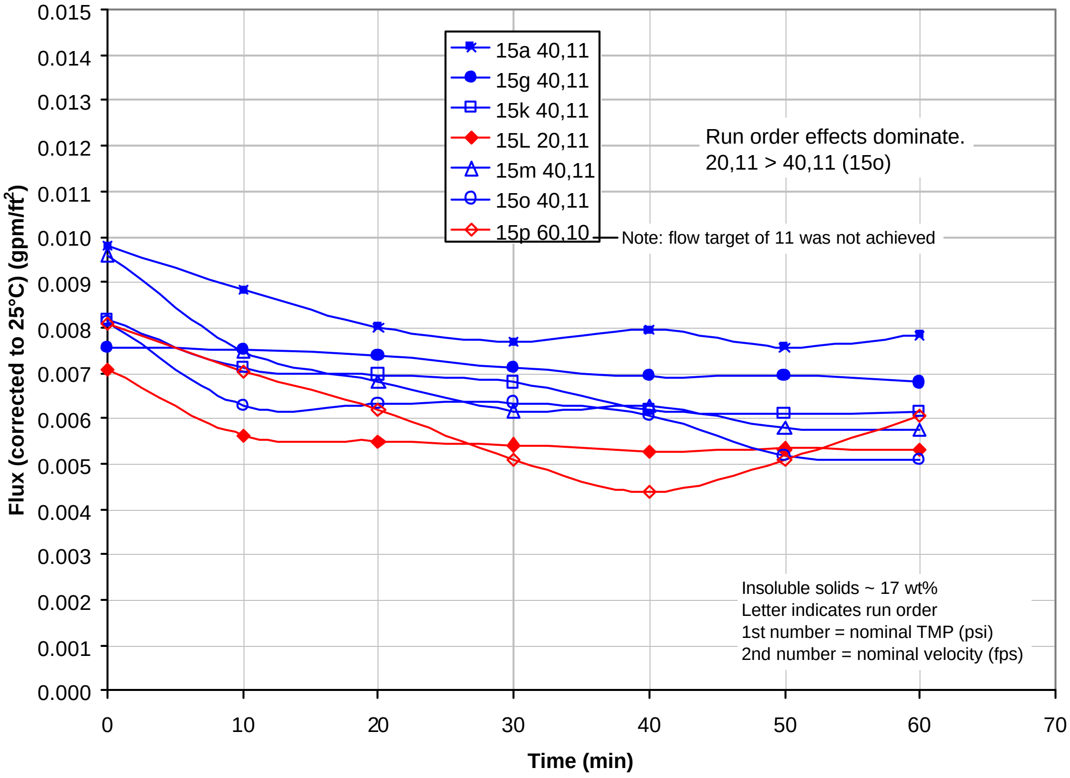

Flux (corrected to 25°C) (gpm/ft^2)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1b 50,12
1f 50,13
1i 50,9
Appear to be bad points;
we have no explanation for these
Flux increases
with velocity
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

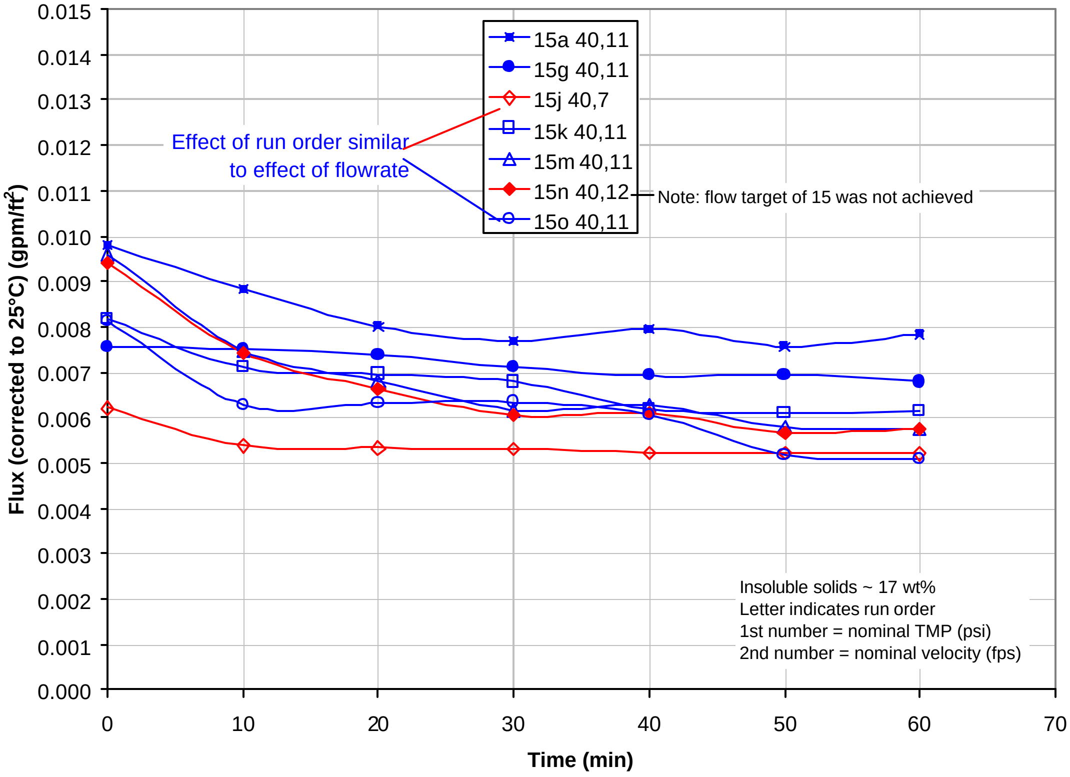

Flux (corrected to 25°C) (gpm/ft^2)
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
10
20
30
40
50
60
70
Time (min)
1a 40,11
1a' 40,11
1g 40,11
1k 40,11
1m 40,11
1o 40,11
Note runs 1a & 1a' are significantly
higher than subsequent runs
run order
Insoluble solids ~ 1.6 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

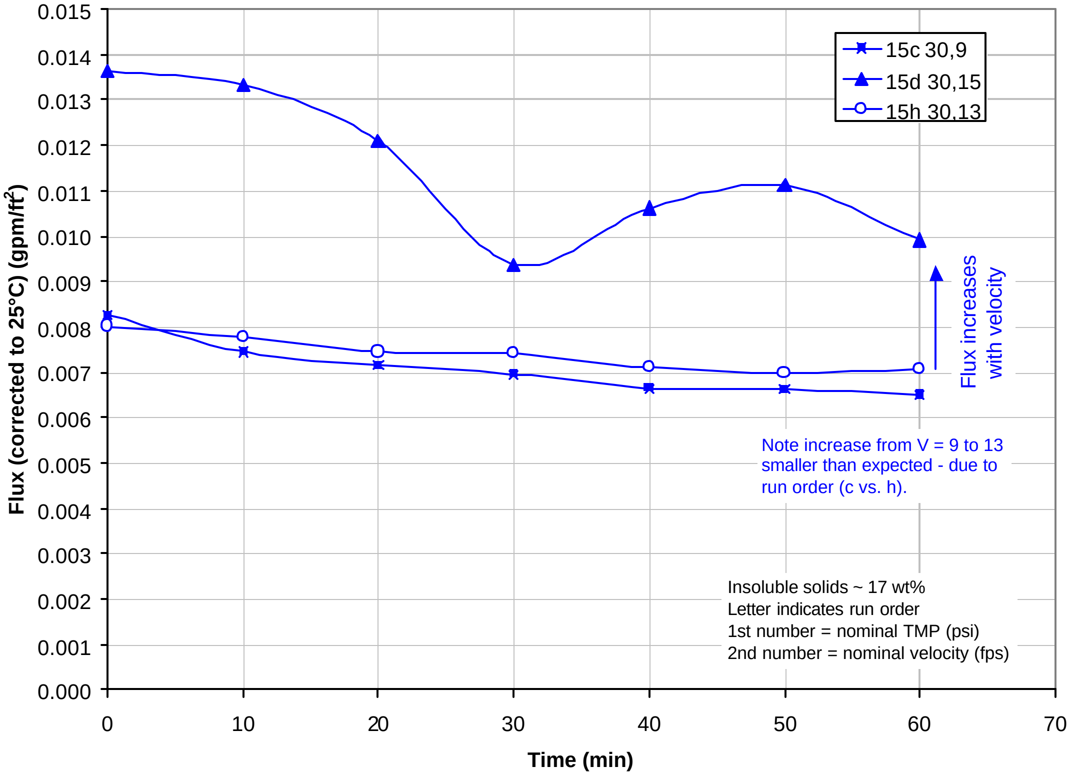

Flux (corrected to 25°C) (gpm/ft²)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15d 30,15
15n 40,15
Flux at 30 psi greater than at 40 psi;
effect of pressure small - overwhelmed
by effect of run order
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

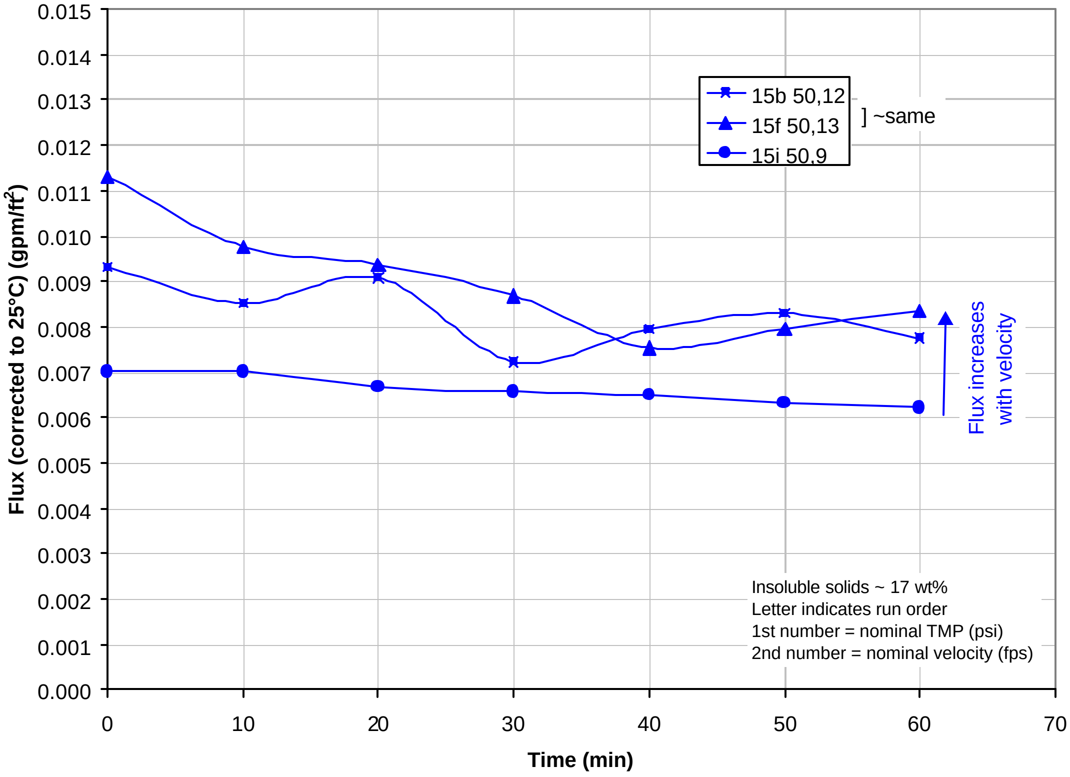

Flux (corrected to 25°C) (gpm/ft²)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15c 30,9
15e 70,9
15i 50,9
Flux increases
with pressure
from 50 to 70
Flux less at 50 psi than
30 psi due to run order effect
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

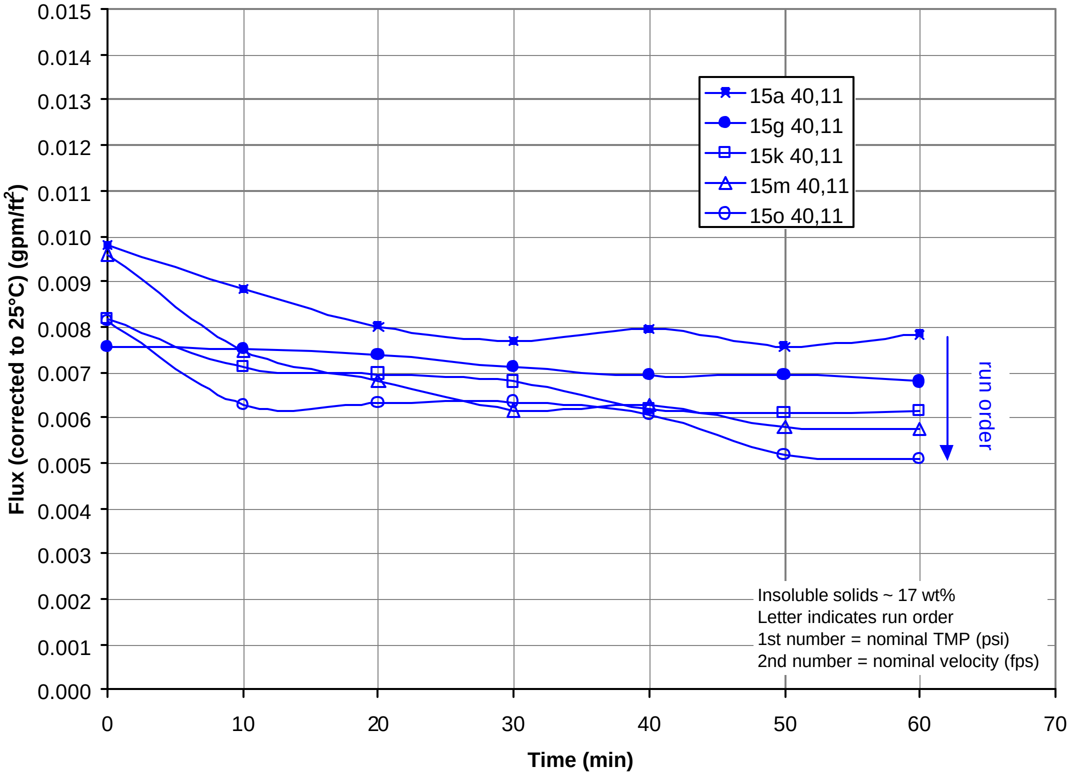

Flux (corrected to 25°C) (gpm/ft^2)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15f 50,13
15h 30,13
Flux increases with pressure
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)


Flux (corrected to 25°C) (gpm/ft^2)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15a 40,11
15g 40,11
15k 40,11
15L 20,11
15m 40,11
15o 40,11
15p 60,10
Run order effects dominate.
20,11 > 40,11 (15o)
Note: flow target of 11 was not achieved
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)


Flux (corrected to 25°C) (gpm/ft^2)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15a 40,11
15g 40,11
15j 40,7
15k 40,11
15m 40,11
15n 40,12
15o 40,11
Effect of run order similar to effect of flowrate
Note: flow target of 15 was not achieved
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)


Flux (corrected to 25°C) (gpm/ft^2)
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
10
20
30
40
50
60
70
Time (min)
15c 30,9
15d 30,15
15h 30,13
Flux increases with velocity
Note increase from V = 9 to 13 smaller than expected - due to run order (c vs. h).
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)


0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
Flux (corrected to 25°C) (gpm/ft2)
0
10
20
30
40
50
60
70
Time (min)
15b 50,12
15f 50,13
15i 50,9
] ~same
Flux increases with velocity
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)


0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
Flux (corrected to 25°C) (gpm/ft2)
0
10
20
30
40
50
60
70
Time (min)
15a 40,11
15g 40,11
15k 40,11
15m 40,11
15o 40,11
run order
Insoluble solids ~ 17 wt%
Letter indicates run order
1st number = nominal TMP (psi)
2nd number = nominal velocity (fps)

APPENDIX A2. RAW FLUX DATA

TTP #	Run	Target Velocity	Target	Time	Flow	Flow	Inlet P	Outlet P	Permeate	TMP	Permeate Volume	Permeate Time	Permeate Time	Total Permeate Time	Flux	Temp	Corr. Factor	Corrected Flux
		(fps)	(psi)	(min)	(gpm)	(fps)	(psig)	(psig)	(psig)	(psi)	(ml)	(min)	(sec)	(sec)	(gpm/ft^2)	(°C)		(gpm/ft^2)
1.17	15-a			0	3.85	11.18	44	37	2	39	40	5	39	339	0.0095	24	1.0286	0.00980
				10	3.72	10.81	44	37	2	39	40	5	56	356	0.0091	26	0.9723	0.00882
				20	3.70	10.75	44	38	3	38	40	6	43	403	0.0080	25	1.0000	0.00801
				30	3.74	10.86	45	39	2	40	40	7	0	420	0.0077	25	1.0000	0.00769
				40	3.86	11.21	43	38	2	39	40	6	46	406	0.0080	25	1.0000	0.00795
				50	3.88	11.27	44	38	2	39	40	6	55	415	0.0078	26	0.9723	0.00757
				60	3.74	10.86	44	38	2	39	40	6	52	412	0.0078	25	1.0000	0.00784
		40	11	Means:	3.78	10.99				38.79							SS FLUX:	0.00757
1.29a	15-b			0	4.06	11.79	51	46	2	47	40	5	28	328	0.0098	27	0.9456	0.00931
				10	4.18	12.14	53	45	2	47	40	6	30	390	0.0083	24	1.0286	0.00852
				20	4.08	11.85	52	45	2	47	40	6	6	366	0.0088	24	1.0286	0.00908
				30	4.10	11.91	50	45	2	46	40	7	15	435	0.0074	26	0.9723	0.00722
				40	4.09	11.88	53	46	2	48	40	6	25	385	0.0084	27	0.9456	0.00793
				50	4.09	11.88	54	45	2	48	40	6	29	389	0.0083	25	1.0000	0.00830
				60	4.08	11.85	54	47	2	49	40	6	45	405	0.0080	26	0.9723	0.00775
		50	12	Means:	4.10	11.90				47.00							SS FLUX:	0.00775
1.20	15-c			0	3.21	9.32	36	30	2	31	20	3	27	207	0.0078	23	1.0583	0.00826
				10	3.23	9.38	34	30	2	30	20	3	43	223	0.0072	24	1.0286	0.00745
				20	3.25	9.44	34	30	2	30	20	3	52	232	0.0070	24	1.0286	0.00716
				30	3.24	9.41	34	30	2	30	20	3	59	239	0.0068	24	1.0286	0.00695
				40	3.23	9.38	34	30	2	30	20	4	3	243	0.0066	25	1.0000	0.00664
				50	3.21	9.32	34	30	2	30	20	4	4	244	0.0066	25	1.0000	0.00662
				60	3.22	9.35	34	30	2	30	20	4	8	248	0.0065	25	1.0000	0.00651
		30	9	Means:	3.23	9.37				30.14							SS FLUX:	0.00651
1.29b	15-d			0	5.11	14.84	34	28	2	29	40	4	18	258	0.0125	22	1.0891	0.01363
				10	5.16	14.99	34	28	2	29	40	4	24	264	0.0122	22	1.0891	0.01332
				20	5.14	14.93	33	28	2	29	40	4	27	267	0.0121	25	1.0000	0.01209
				30	5.11	14.84	34	28	2	29	40	5	35	335	0.0096	26	0.9723	0.00937
				40	5.07	14.73	33	27	2	28	40	5	22	322	0.0100	23	1.0583	0.01061
				50	5.08	14.76	33	27	2	28	40	4	59	299	0.0108	24	1.0286	0.01111
				60	5.06	14.70	33	27	2	28	40	5	8	308	0.0105	27	0.9456	0.00991
		30	15	Means:	5.10	14.83				28.50							SS FLUX:	0.00991

TTP #	Run	Target Velocity	Target	Time	Flow	Flow	Inlet P	Outlet P	Permeate	TMP	Permeate Volume	Permeate Time	Permeate Time	Total Permeate Time	Flux	Temp	Corr. Factor	Corrected Flux
		(fps)	(psi)	(min)	(gpm)	(fps)	(psig)	(psig)	(psig)	(psi)	(ml)	(min)	(sec)	(sec)	(gpm/ft^2)	(°C)		(gpm/ft^2)
1.29c	15-e			0	3.08	8.95	73	68	2	69	40	5	56	356	0.0091	27	0.9456	0.00858
				10	3.06	8.89	74	68	2	69	40	6	25	385	0.0084	27	0.9456	0.00793
				20	3.08	8.95	73	67	2	68	40	6	23	383	0.0084	26	0.9723	0.00820
				30	3.09	8.98	74	68	2	69	40	6	40	400	0.0081	27	0.9456	0.00763
				40	3.12	9.06	73	67	3	67	40	7	4	424	0.0076	27	0.9456	0.00720
				50	3.11	9.03	73	67	3	67	40	7	10	430	0.0075	26	0.9723	0.00730
				60	3.10	9.01	74	67	3	68	40	6	48	408	0.0079	26	0.9723	0.00770
		70	9	Means:	3.09	8.98				68.00							SS FLUX:	0.00730
1.22	15-f			0	4.45	12.93	53	48	2	49	40	4	38	278	0.0116	26	0.9723	0.01129
				10	4.48	13.01	54	49	2	50	40	4	56	296	0.0109	29	0.8948	0.00976
				20	4.45	12.93	53	50	2	50	40	5	0	300	0.0108	30	0.8707	0.00937
				30	4.48	13.01	54	49	2	50	40	5	24	324	0.0100	30	0.8707	0.00868
				40	4.43	12.87	54	48	2	49	40	6	45	405	0.0080	27	0.9456	0.00754
				50	4.41	12.81	53	46	3	47	40	6	57	417	0.0077	24	1.0286	0.00797
				60	4.41	12.81	52	46	3	46	40	6	6	366	0.0088	27	0.9456	0.00834
		50	13	Means:	4.44	12.91				48.36							SS FLUX:	0.00795
1.18	15-g			0	3.80	11.04	44	39	2	40	20	3	34	214	0.0075	25	1.0000	0.00755
				10	3.83	11.13	44	39	2	40	20	3	35	215	0.0075	25	1.0000	0.00751
				20	3.87	11.24	44	39	2	40	20	3	33	213	0.0076	26	0.9723	0.00737
				30	3.84	11.15	45	40	2	41	20	3	47	227	0.0071	25	1.0000	0.00711
				40	3.74	10.86	44	38	2	39	20	4	0	240	0.0067	24	1.0286	0.00692
				50	3.78	10.98	45	38	2	40	20	3	46	226	0.0071	26	0.9723	0.00695
				60	3.78	10.98	45	40	2	41	20	3	58	238	0.0068	25	1.0000	0.00678
		40	11	Means:	3.81	11.06				39.71							SS FLUX:	0.00678
1.21	15-h			0	4.47	12.98	35	27	3	28	20	3	16	196	0.0082	26	0.9723	0.00801
				10	4.45	12.93	34	27	2	29	20	3	28	208	0.0078	25	1.0000	0.00776
				20	4.45	12.93	35	27	2	29	20	3	37	217	0.0074	25	1.0000	0.00744
				30	4.40	12.78	34	27	2	29	20	3	38	218	0.0074	25	1.0000	0.00741
				40	4.40	12.78	34	27	2	29	20	3	41	221	0.0073	26	0.9723	0.00710
				50	4.46	12.96	34	27	2	29	20	3	45	225	0.0072	26	0.9723	0.00698
				60	4.50	13.07	34	27	2	29	20	3	36	216	0.0075	27	0.9456	0.00707
		30	13	Means:	4.45	12.92				28.50							SS FLUX:	0.00707
1.23	15-i			0	3.19	9.27	53	47	3	47	20	3	57	237	0.0068	24	1.0286	0.00701
				10	3.11	9.03	55	51	3	50	20	3	57	237	0.0068	24	1.0286	0.00701
				20	3.14	9.12	54	50	3	49	20	4	9	249	0.0065	24	1.0286	0.00667
				30	3.15	9.15	54	50	3	49	20	4	6	246	0.0066	25	1.0000	0.00656
				40	3.13	9.09	54	50	3	49	20	4	9	249	0.0065	25	1.0000	0.00648
				50	3.14	9.12	54	50	3	49	20	4	15	255	0.0063	25	1.0000	0.00633
				60	3.09	8.98	54	50	3	49	20	4	13	253	0.0064	26	0.9723	0.00621
		50	9	Means:	3.14	9.11				48.86							SS FLUX:	0.00621

TTP #	Run	Target Velocity	Target	Time	Flow	Flow	Inlet P	Outlet P	Permeate	TMP	Permeate Volume	Permeate Time	Permeate Time	Total Permeate Time	Flux	Temp	Corr. Factor	Corrected Flux
		(fps)	(psi)	(min)	(gpm)	(fps)	(psig)	(psig)	(psig)	(psi)	(ml)	(min)	(sec)	(sec)	(gpm/ft^2)	(°C)		(gpm/ft^2)
1.25	15-j			0	2.39	6.94	43	39	3	38	20	4	13	253	0.0064	26	0.9723	0.00621
				10	2.38	6.91	43	39	3	38	20	4	52	292	0.0055	26	0.9723	0.00538
				20	2.39	6.94	43	39	3	38	20	5	3	303	0.0053	25	1.0000	0.00533
				30	2.38	6.91	44	40	3	39	20	5	13	313	0.0052	24	1.0286	0.00531
				40	2.34	6.80	44	40	3	39	20	5	18	318	0.0051	24	1.0286	0.00522
				50	2.43	7.06	44	40	3	39	20	5	28	328	0.0049	23	1.0583	0.00521
				60	2.41	7.00	44	40	3	39	20	5	29	329	0.0049	23	1.0583	0.00519
		40	7	Means:	2.39	6.94				38.57							SS FLUX:	0.00519
1.24	15-k			0	3.83	11.13	43	38	3	38	20	3	18	198	0.0082	25	1.0000	0.00815
				10	3.72	10.81	43	38	3	38	20	3	54	234	0.0069	24	1.0286	0.00710
				20	3.74	10.86	44	39	3	39	20	3	59	239	0.0068	24	1.0286	0.00695
				30	3.79	11.01	44	39	3	39	20	3	58	238	0.0068	25	1.0000	0.00678
				40	3.77	10.95	44	39	3	39	20	4	14	254	0.0064	26	0.9723	0.00618
				50	3.76	10.92	44	38	3	38	20	4	17	257	0.0063	26	0.9723	0.00611
				60	3.75	10.89	44	38	3	38	20	4	16	256	0.0063	26	0.9723	0.00613
		40	11	Means:	3.77	10.94				38.07							SS FLUX:	0.00613
1.27	15-L			0	3.75	10.89	23	19	3	18	20	3	36	216	0.0075	27	0.9456	0.00707
				10	3.73	10.84	23	18	3	18	20	4	39	279	0.0058	26	0.9723	0.00563
				20	3.68	10.69	23	19	3	18	20	4	54	294	0.0055	25	1.0000	0.00549
				30	3.74	10.86	23	18	3	18	20	5	7	307	0.0053	24	1.0286	0.00541
				40	3.69	10.72	23	18	3	18	20	5	15	315	0.0051	24	1.0286	0.00527
				50	3.81	11.07	23	19	3	18	20	5	12	312	0.0052	24	1.0286	0.00532
				60	3.73	10.84	23	19	3	18	20	5	13	313	0.0052	24	1.0286	0.00531
		20	11	Means:	3.73	10.84				17.79							SS FLUX:	0.00531
1.29	15-m			0	3.71	10.78	42	38	3	37	20	2	53	173	0.0093	24	1.0286	0.00960
				10	3.73	10.84	44	39	3	39	20	3	42	222	0.0073	24	1.0286	0.00748
				20	3.72	10.81	43	38	3	38	20	3	57	237	0.0068	25	1.0000	0.00681
				30	3.70	10.75	44	39	3	39	20	4	22	262	0.0062	25	1.0000	0.00616
				40	3.68	10.69	44	39	3	39	20	4	25	265	0.0061	24	1.0286	0.00627
				50	3.74	10.86	43	38	3	38	20	4	39	279	0.0058	25	1.0000	0.00579
				60	3.71	10.78	43	38	3	38	20	4	49	289	0.0056	24	1.0286	0.00575
		40	11	Means:	3.71	10.79				37.86							SS FLUX:	0.00575
1.26	15-n			0	4.24	12.32	44	38	3	38	20	2	47	167	0.0097	26	0.9723	0.00940
				10	4.18	12.14	44	39	3	39	20	3	20	200	0.0081	28	0.9198	0.00743
				20	4.20	12.20	43	37	3	37	20	3	38	218	0.0074	29	0.8948	0.00663
				30	4.17	12.11	44	38	3	38	20	3	59	239	0.0068	29	0.8948	0.00605
				40	4.13	12.00	44	37	3	38	20	3	57	237	0.0068	29	0.8948	0.00610
				50	4.24	12.32	43	37	3	37	20	4	22	262	0.0062	28	0.9198	0.00567
				60	4.17	12.11	43	37	3	37	20	4	18	258	0.0063	28	0.9198	0.00576
		40	15	Means:	4.19	12.17				37.57							SS FLUX:	0.00576

TTP #	Run	Target Velocity	Target	Time	Flow	Flow	Inlet P	Outlet P	Permeate	TMP	Permeate Volume	Permeate Time	Permeate Time	Total Permeate Time	Flux	Temp	Corr. Factor	Corrected Flux
		(fps)	(psi)	(min)	(gpm)	(fps)	(psig)	(psig)	(psig)	(psi)	(ml)	(min)	(sec)	(sec)	(gpm/ft^2)	(°C)		(gpm/ft^2)
1.19	15-o			0	3.78	10.98	43	39	3	38	20	3	8	188	0.0086	27	0.9456	0.00812
				10	3.70	10.75	43	38	3	38	20	4	3	243	0.0066	27	0.9456	0.00628
				20	3.72	10.81	42	37	3	37	20	4	2	242	0.0067	27	0.9456	0.00631
				30	3.76	10.92	43	37	3	37	20	4	1	241	0.0067	27	0.9456	0.00634
				40	3.76	10.92	43	38	3	38	20	4	5	245	0.0066	28	0.9198	0.00606
				50	3.70	10.75	43	37	3	37	20	4	39	279	0.0058	29	0.8948	0.00518
				60	3.70	10.75	43	37	3	37	20	4	52	292	0.0055	28	0.9198	0.00509
		40	11	Means:	3.73	10.84				37.21							SS FLUX:	0.00509
1.28	15-p			0	3.60	10.46	63	57	3	57	20	3	14	194	0.0083	26	0.9723	0.00809
				10	3.51	10.20	63	58	3	58	20	3	37	217	0.0074	27	0.9456	0.00704
				20	3.56	10.34	62	56	3	56	20	3	53	233	0.0069	29	0.8948	0.00620
				30	3.57	10.37	63	56	3	57	20	4	37	277	0.0058	30	0.8707	0.00508
				40	3.47	10.08	62	56	3	56	20	5	30	330	0.0049	29	0.8948	0.00438
				50	3.41	9.91	62	56	3	56	20	5	0	300	0.0054	27	0.9456	0.00509
				60	3.36	9.76	63	56	3	57	20	4	19	259	0.0062	26	0.9723	0.00606
		60	11	Means:	3.50	10.16				56.50							SS FLUX:	0.00518

APPENDIX A3. ANALYTICAL DATA

ADS #	USER ID	Description	Analyses	Value	Units
300180741	CCUF-AN102-SL-SIMUL2	supernate simulant	aluminate	0.497	M
			free OH	0.235	M
			carbonate	0.456	M
			sp gr	1.32	
			total base	1.186	M
			total solids	37.22	wt%
300184313	CCUF-AN102-SIMUL2	supernate simulant	suspended solids	0.31	wt%
			total solids	37.20	wt%
300178724	CCUF-AN102-PE-BAT1	permeate	F	733	mg/L
			formate	4880	mg/L
			Cl	2770	mg/L
			NO2-	44700	mg/L
			NO3-	126000	mg/L
			PO4-3	2360	mg/L
			SO4=	7080	mg/L
			oxalate	702	mg/L
300178725	CCUF-AN102-PE-10%	permeate	F	46100	mg/L
			formate	129000	mg/L
			Cl	2790	mg/L
			NO2-	732	mg/L
			NO3-	2240	mg/L
			PO4-3	7400	mg/L
			SO4=	4820	mg/L
			oxalate	712	mg/L
			total solids	33.30	wt%
300178726	CCUF-AN102-PE-15%	permate	F	41100	mg/L
			formate	124000	mg/L
			Cl	2770	mg/L
			NO2-	672	mg/L
			NO3-	2500	mg/L
			PO4-3	6540	mg/L
			SO4=	4900	mg/L
			oxalate	623	mg/L
			total solids	33.61	wt%
300178727	CCUF-AN102-PE-WASH	permeate from washed slurry	F	21700	mg/L
			formate	57700	mg/L
			Cl	1320	mg/L
			NO2-	593	mg/L
			NO3-	1370	mg/L
			PO4-3	3850	mg/L
			SO4=	2900	mg/L
			oxalate	2610	mg/L
300178715	CCUF-AN102-SL-BAT1-WET	batch 1 precipitate	F	36300	mg/L
			formate	95900	mg/L
			Cl	2160	mg/L
			NO2-	1030	mg/L
			NO3-	1910	mg/L
			PO4-3	5420	mg/L
			SO4=	4120	mg/L
			oxalate	1400	mg/L
			total carbon	10000	mg/L
			inorganic carbon	4440	mg/L
			organic carbon	5580	mg/L
			total solids	33.59	wt%
			total solids	33.10	wt%
			suspended solids	1.88	wt%

ADS #	USER ID	Description	Analyses	Value	Units
300178716	CCUF-AN102-SL-WASH-WET	washed slurry	F	14200	mg/kg slurry
			formate	45000	mg/kg slurry
			Cl	886	mg/kg slurry
			NO2-	365	mg/kg slurry
			NO3-	855	mg/kg slurry
			PO4-3	2830	mg/kg slurry
			SO4=	2520	mg/kg slurry
			oxalate	8530	mg/kg slurry
			total carbon	6320	mg/L
			inorganic carbon	3340	mg/L
			organic carbon	2980	mg/L
			total solids	35.06	wt%
			suspended solids	20.63	wt%
300178729	CCUF-AN102-SL-10%	slurry at ~10wt% IS	total solids	42.14	wt%
			suspended solids	13.33	wt%
300178728	CCUF-AN102-SL-BAT5	batch 5 percipitate	total solids	33.56	wt%
			suspended solids	1.60	wt%
300178730	CCUF-AN102-SL-15%	slurry at ~15wt% IS	total solids	44.98	wt%
			suspended solids	17.17	wt%

Notebook WSRC-NB-2002-93 p. 45				
File = Zamecnik Report 8-6-2002.xls				
Concentration in original sample in ug/g (ppm)				
USER ID	CCUF-AN102-SL-BAT1-AQREG	CCUF-AN102-SL-WASH-AQREG	CCUF-AN102-SL-BAT1-NA2O2	CCUF-AN102-SL-WASH-NA2O2
	slurry batch 1	washed slurry	slurry batch 1	washed slurry
	aqua regia	aqua regia	sodium peroxide	sodium peroxide
ADS 300-	178720	178721	178717	178718
Al	16200	10300	16300	10800
B	<200	<200	<200	<200
Ba	395	5260	419	5610
Ca	661	6630	2030	6930
Cd	79	102	77	100
Co	<75	<75	<75	<75
Cr	346	468	355	650
Cu	<75	376	<75	398
Fe	280	1440	567	1560
Li	<100	<100	<100	<100
Mg	<90	539	<90	506
Mn	5290	80800	4710	85500
Mo	<150	<150	<150	<150
Na	291000	170000	NA	NA
Ni	601	2120	565	2150
P	1880	3080	1430	2670
Pb	<650	2180	<650	2390
Si	<150	<150	<150	<150
Sn	<350	<350	<350	<350
Sr	13000	191000	10100	190000
Ti	<150	<150	<150	<150
V	<150	<150	<150	<150
Zn	<350	<350	<350	<350
Zr	<50	318	NA	NA
La	<700	<700	<700	<700
K	632	<500	NA	NA
Nd	<500	<500	<500	<500

Use sp gr and total solids content to convert to mg/L.

Notebook WSRC-NB-2002-93 p. 35			
File = Zamecnik Report 7-18-2002.xls			
Concentration in original sample in mg/L (ppm)			
USER ID	CCUF-AN102-PE-BAT1	CCUF-AN102-PE-10%	CCUF-AN102-PE-15%
Permeate	batch 1	@13 wt%	@17 wt%
ADS 300-	178724	178725	178726
Al	7350	7540	7700
B	22.4	23.5	24.2
Ba	<0.024	<0.024	<0.024
Ca	91.7	95.8	102
Cd	34.5	35.6	38.1
Co	<0.088	<0.088	<0.088
Cr	149	153	137
Cu	3.49	3.67	4.15
Fe	0.53	0.46	0.63
Li	0.24	0.26	0.22
Mg	<0.168	<0.168	<0.168
Mn	0.17	0.34	0.50
Mo	28.4	28.5	29.0
Na	133000	138000	142000
Ni	195	200	201
P	573	577	553
Pb	36.2	39.9	42.1
Si	15.3	15.2	16.2
Sn	<0.7	<0.7	<0.7
Sr	28.0	26.1	26.4
Ti	<0.28	<0.28	<0.28
V	<0.26	<0.26	<0.26
Zn	3.03	2.24	2.35
Zr	0.47	0.44	0.55
La	<1.4	<1.4	<1.4
K	1770	2150	2380
S	2750	2820	2910
Nd	0.64	1.21	0.76

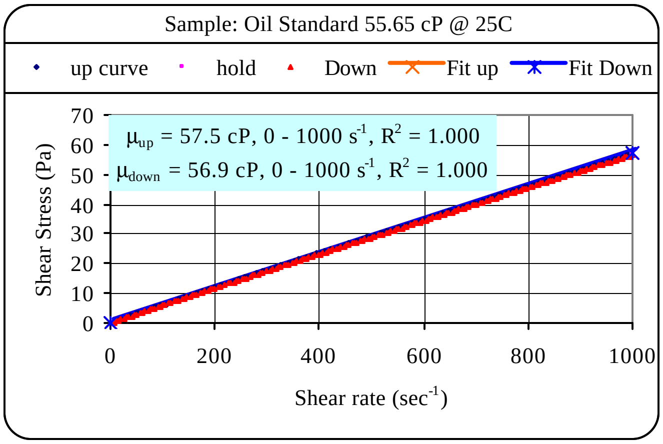
Notebook WSRC-NB-2002-93 p. 42			
File = Zamecnik Report 8-12-2002.xls			
Concentration in original sample in mg/L (ppm)			
USER ID	CCUF-AN102-PE-WASH		
	permeate from washed slurry		
ADS 300-	178727		
Al	3510		
B	11.3		
Ba	<0.024		
Ca	50.5		
Cd	18.8		
Co	<0.088		
Cr	69.6		
Cu	0.44		
Fe	<0.088		
Li	<0.2		
Mg	<0.168		
Mn	0.69		
Mo	13.9		
Na	66800		
Ni	85.3		
P	372		
Pb	10.3		
Si	7.69		
Sn	<0.52		
Sr	10.10		
Ti	<0.28		
V	<0.26		
Zn	<0.74		
Zr	0.22		
La	<1.4		
K	843		
Re	<0.1		
S	1430		
Nd	1.68		
Ag	<0.6		

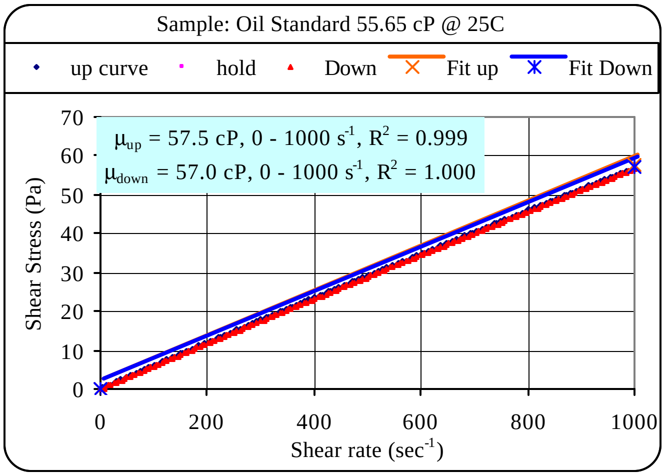
DESCRIPTION	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1	102R2 NOC1
	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT	R2NOC1 PPT SOLIDS	R2NOC1 PERMEATE
	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-X	102R2-SL-INIT-SOL	102R2-P-INIT-X
ADS #300- or MOBILE LAB	185115, 185181, 185114		185115, 185179, 185114		MOBILE LAB	MOBILE LAB	MOBILE LAB	188650	185181	185182
ICPES mg/L										
Units	mg/kg slurry	mg/L	mg/kg slurry	mg/L					mg/kg	mg/L
Dissolution	NA2O2	NA2O2	AQUA REGIA	AQUA REGIA						
Al	6830	8875	6780	8810					30300	8050
B	51.3	66.7	57.1	74.2					235	24.4
Ba	<5.0	<6	<5.0	<6					138	<0.024
Ca	391	508	289	376					14000	79.1
Cd	32.6	42.4	33.4	43.4					192	32.8
Co	<10	<13	<10	<13					114	<0.30
Cr	237	308	143	186					881	159
Cu	17.0	22.2	16.2	21.0					580	5.75
Fe	409	531	38.3	49.7					3030	2.28
Li	<20	<26	<20	<26					<100	0.270
Mg	13.8	17.9	15.3	19.9					944	<0.168
Mn	1290	1676	1330	1728					80600	14.0
Mo	32.8	42.6	22.5	29.2					<100	30.4
Na	NA		103000	133845					NA	125000
Na AA										
Ni	301	391	234	305					2340	221
P	2150	2794	1730	2248					13300	870
Pb	<150	<195	<150	<195					4000	41.5
Si	37.7	48.9	<20	<26					1220	12.7
Sn	<50	<65	<50	<65					<250	<0.60
Sr	1760	2287	2100	2729					104000	50.2
Ti	<20	<26	<20	<26					<20	<0.28
V	<20	<26	<20	<26					<20	<0.26
Zn	<50	<65	<50	<65					<50	1.18
Zr	NA		<15						NA	2.27
La	<100	<130	<100	<130					<650	<1.4
K	NA		1260						NA	1680
K AA										
S										2930
Nd	<50	<65	<50	<65					975	0.947
Ce										<1.54
Cs										
Rb										
W										
Ion Chrom (mg/L or mg/kg)										
Units	mg/kg	mg/L	mg/kg	mg/L						
NO2-	41900	54448	48700	63284						
NO3-	106000	137743	123000	159834						
Cl	2730	3548	3340	4340						
F	910	1183	603	784						
PO4[-3]	3200	4158	3760	4886						
SO4=	6590	8563	8810	11448						
COOH-	4840	6289	4720	6133						
C2O4=	1230	1598	1410	1832						
Carbon (mg/L)										
TOC	10500									9890
TIC	7370									7710
TC	17900									17600
Wet Chem										
CO3= (M)	0.550									
CO3= mg/L	33005									
FREE OH- (M)	0.150									
FREE OH- (mg/L)										
TOTAL BASE (M)	1.50									
TOTAL SOLIDS (wt%)				MOBILE LAB ->	36.6	35.6	33.4	33.0		
TOTAL SOLIDS (wt%)				MOBILE LAB ->	37.2	35.9	33.4			
TOTAL SOLIDS (wt%)										
TOTAL SOLIDS (wt%)										
SUSPENDED SOLIDS (wt%)				MOBILE LAB ->	6.00	4.56	1.19	0.740		
SUSPENDED SOLIDS (wt%)				MOBILE LAB ->	5.20	4.96	1.33			
SUSPENDED SOLIDS (wt%)										
SUSPENDED SOLIDS (wt%)				filtration, still wet ->	4.04					
SPECIFIC GRAVITY				lab measurement ->	1.27			1.27		1.19

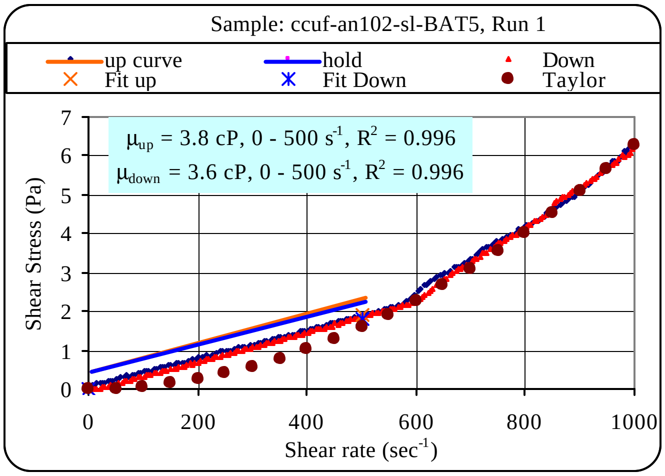
DESCRIPTION	102R2 NOC2	102R2 NOC2	102R2 NOC2	102R2 NOC2	102R2 NOC2	102R2 NOC2	102R2 NOC2			102R2 NOC2
	R2NOC2 PPT		R2NOC2 PPT		R2NOC2 SLURRY ALL FED	R2NOC2 SLURRY ~20%	PERMEATE AT 2/3 FED			PERMEATE AT ~20%
	102R2NOC2-SL-INIT-X	102R2NOC2-SL-INIT-X	102R2NOC2-SL-INIT-X	102R2NOC2-SL-INIT-X	102R2NOC2-SL-15X	102R2NOC2-SL-20X	102R2NOC2-P-2/3B	102R2NOC2-SL-INIT-X	102R2NOC2-SL-INIT-X	102R2NOC2-P-20
ADS #300- or MOBILE LAB	186089, 186090, 186091, 198092				186094	186095	186093			187817
ICPES mg/L										
Units	mg/kg	mg/L	mg/kg	mg/L				mg/L	mg/L	
Dissolution										
Al	20900	8848	6830	8606			8980	8848	8606	
B	<220	<93	<30	<38			28.2	93.0	38.0	
Ba	95.2	40.3	15.8	19.9			<0.024	40.3	19.9	
Ca	2620	1109	235	296			89.4	1109	296	
Cd	110	46.6	35.2	44.4			40.5	46.6	44.4	
Co	<50	<21	<10	<13			<0.20			
Cr	460	195	120	152			146	195	152	
Cu	<50	<21	12.8	16.2			6.65	21.0	16.2	
Fe	478	202	61.7	77.7			2.65	202	77.7	
Li	<100	<42	<20	<25			<0.2			
Mg	<85	<36	15.7	19.8			<0.168	36.0	19.8	
Mn	4560	1931	1430	1802			12.2	1931	1802	
Mo	97.2	41.2	27.5	34.6			34.5	41.2	34.6	
Na	NA		102000	128520			136000		128520	
Na AA										
Ni	806	341	244	307			275	341	307	
P	3290	1393	829	1044			775	1393	1044	
Pb	<700	<296	<200	<252			47.4	296	252	
Si	352	149	21.4	27.0			27.1	149	27.0	
Sn	<300	<127	<50	<63			1.03	127	63.0	
Sr	8850	3747	2120	2671			22.9	3747	2671	
Ti	<100	<42	<20	<25			<0.28			
V	186	78.7	<20	<25			<0.26			
Zn	<250	<106	<50	<63			1.67	106	63.0	
Zr	NA		<8.0	<10			3.15		10.0	
La	<700	<296	<100	<126			2.97	296	126	
K	3640	1541	1090	1373			1840	1541	1373	
K AA										
S							3180			
Nd	324	137	64.9	81.8			6.07	137	81.8	
Ce							8.95			
Cs										
Rb										
W										
Ion Chrom (mg/L or mg/kg)										
Units	mg/kg	mg/L								
NO2-	38500	48510					50500			
NO3-	92100	116046					124000			
Cl	2490	3137					3150			
F	971	1223					1180			
PO4[-3]	2900	3654					3310			
SO4=	6590	8303					8460			
COOH-	4510	5683					5860			
C2O4=	1260	1588					1020			
Carbon (mg/L)										
TOC	10100									
TIC	9610									
TC	19700									
Wet Chem										
CO3= (M)										
CO3= mg/L										
FREE OH- (M)										
FREE OH- (mg/L)										
TOTAL BASE (M)										
TOTAL SOLIDS (wt%)	33.6			ADS	43.1	47.4				35.4
TOTAL SOLIDS (wt%)				MOBILE LAB->	43.6					
TOTAL SOLIDS (wt%)										
TOTAL SOLIDS (wt%)				ADS	42.5					
SUSPENDED SOLIDS (wt%)	1.32			ADS	12.3	18.5	CALC			
SUSPENDED SOLIDS (wt%)				MOBILE LAB->	13.9					
SUSPENDED SOLIDS (wt%)										
SUSPENDED SOLIDS (wt%)										
SPECIFIC GRAVITY	1.26				1.37					

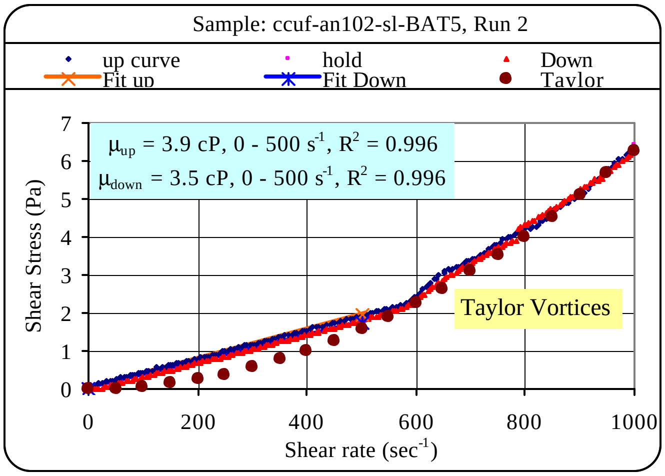
APPENDIX A4. RHEOLOGICAL DATA

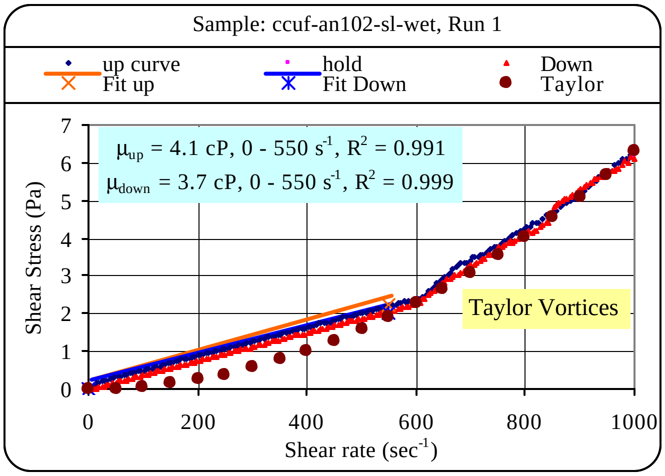
R1BC Slurries:

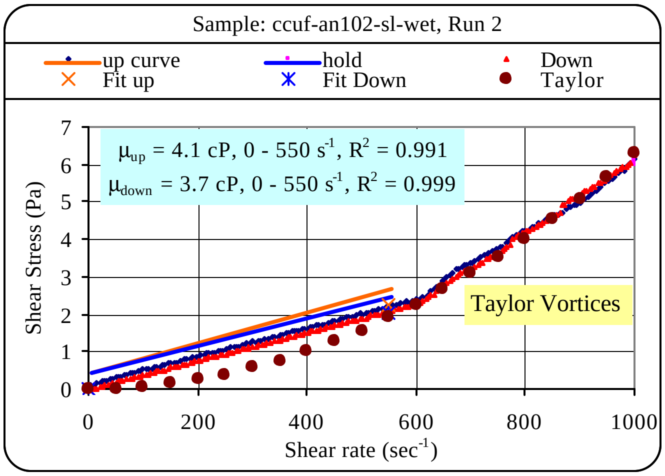


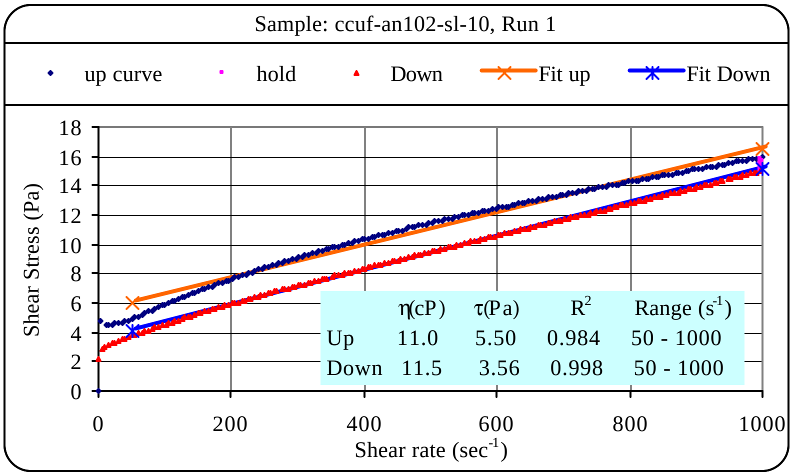





Sample: ccuf-an102-sl-BAT5, Run 2

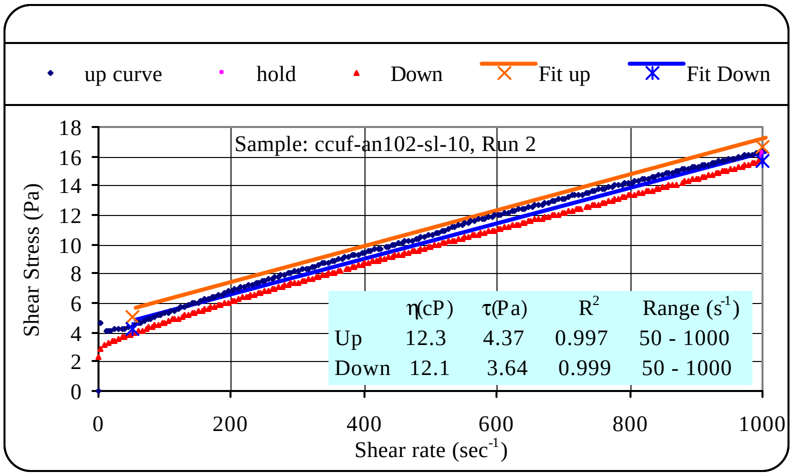

Sample: ccuf-an102-sl-wet, Run 1


Sample: ccuf-an102-sl-wet, Run 2



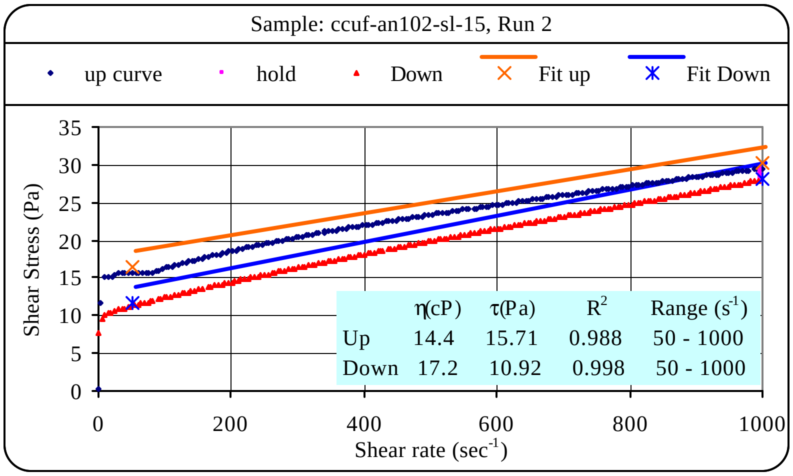
Sample: ccuf-an102-sl-10, Run 1

	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	11.0	5.50	0.984	50 - 1000
Down	11.5	3.56	0.998	50 - 1000



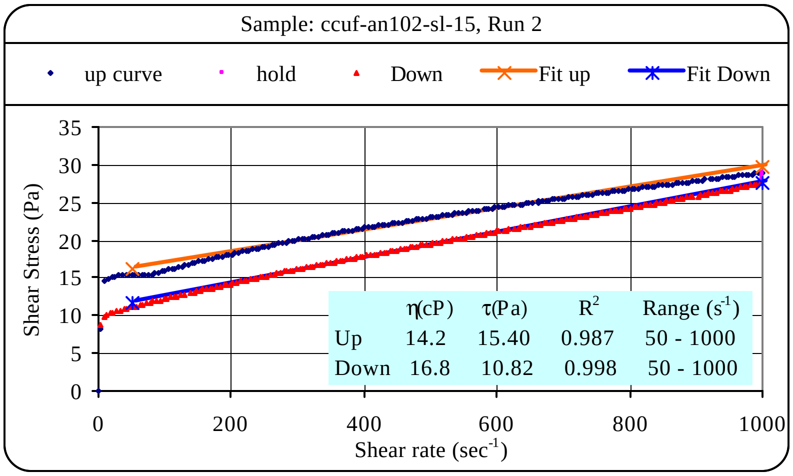
Sample: ccuf-an102-sl-10, Run 2

	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	12.3	4.37	0.997	50 - 1000
Down	12.1	3.64	0.999	50 - 1000

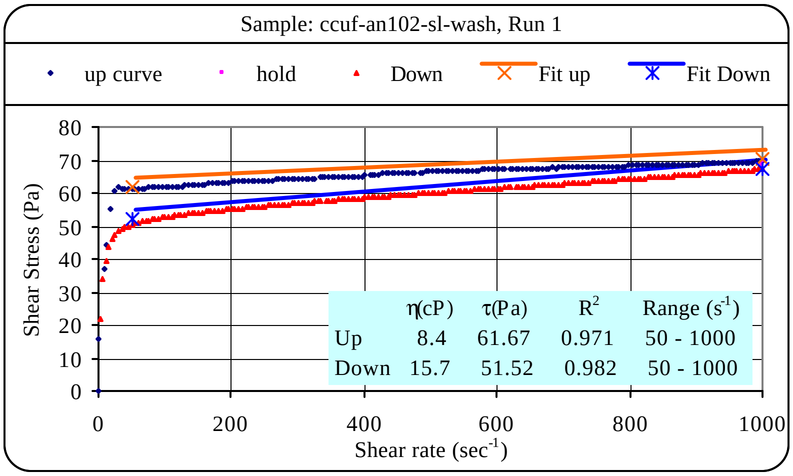


Sample: ccuf-an102-sl-15, Run 2

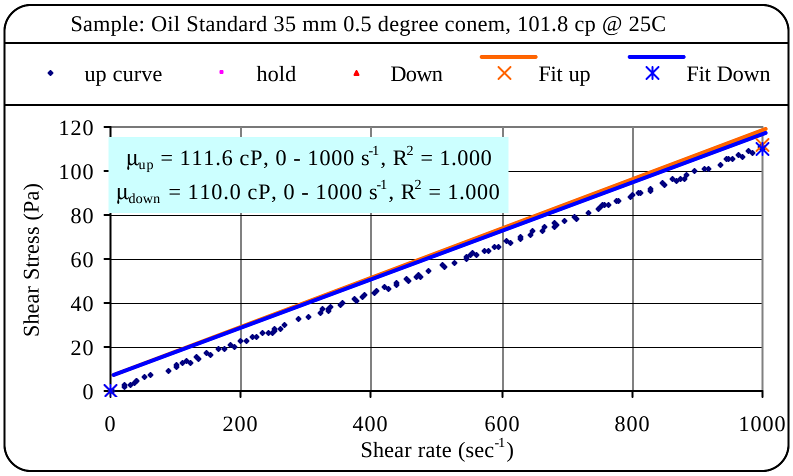
	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	14.4	15.71	0.988	50 - 1000
Down	17.2	10.92	0.998	50 - 1000



	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	14.2	15.40	0.987	50 - 1000
Down	16.8	10.82	0.998	50 - 1000

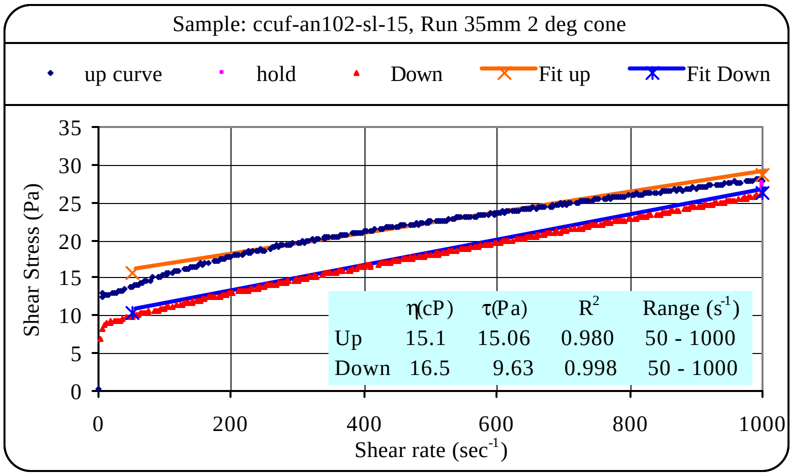


	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	8.4	61.67	0.971	50 - 1000
Down	15.7	51.52	0.982	50 - 1000

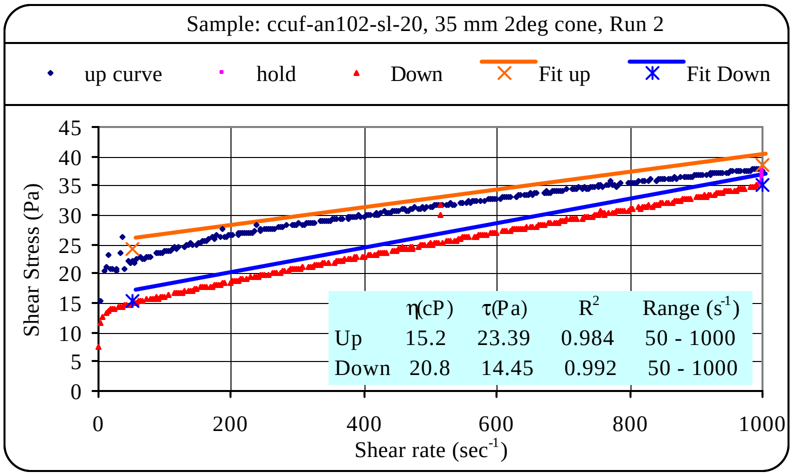


μ_{up} = 111.6 cP, 0 - 1000 s^{-1}, R^2 = 1.000
μ_{down} = 110.0 cP, 0 - 1000 s^{-1}, R^2 = 1.000

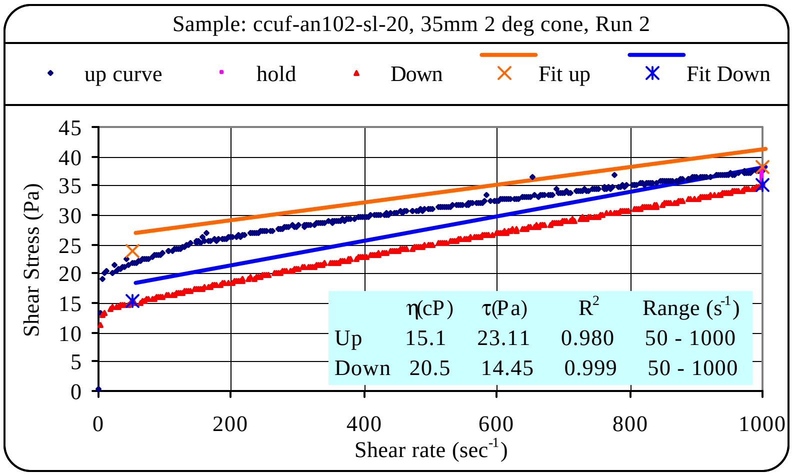
Outlier points in the graphs following are due to an instrument communication problem. The number of outlier points is insignificant to the fitted viscosity or consistency and yield stress values.


Sample: ccuf-an102-sl-15, Run 35mm 2 deg cone

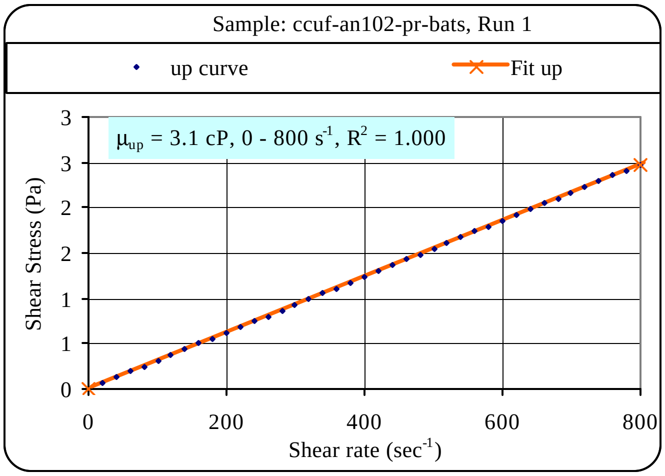
	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	15.1	15.06	0.980	50 - 1000
Down	16.5	9.63	0.998	50 - 1000

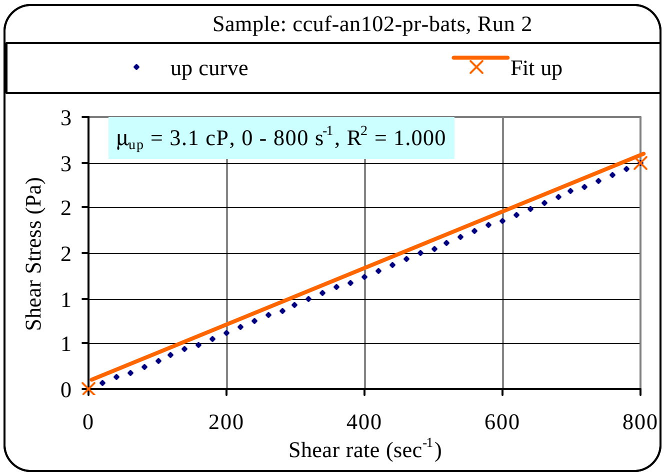

Sample: ccuf-an102-sl-20, 35 mm 2deg cone, Run 2

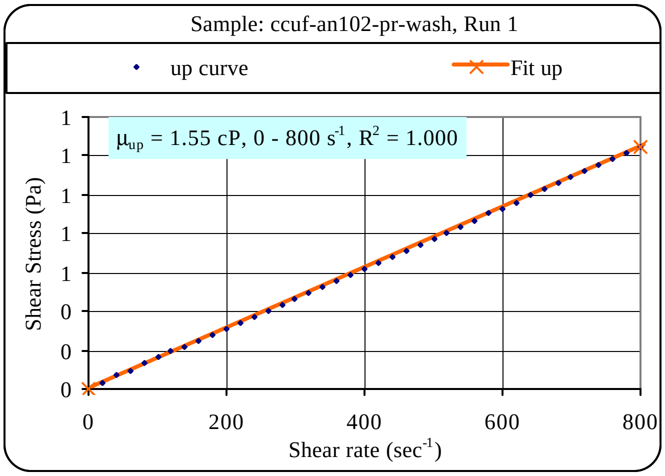
	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	15.2	23.39	0.984	50 - 1000
Down	20.8	14.45	0.992	50 - 1000

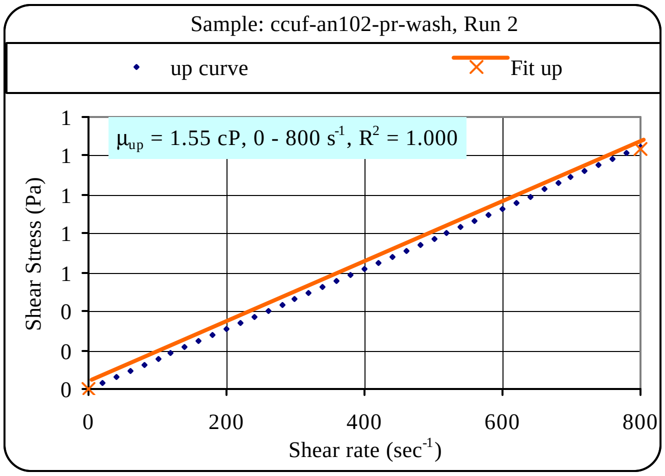

Sample: ccuf-an102-sl-20, 35mm 2 deg cone, Run 2

	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	15.1	23.11	0.980	50 - 1000
Down	20.5	14.45	0.999	50 - 1000

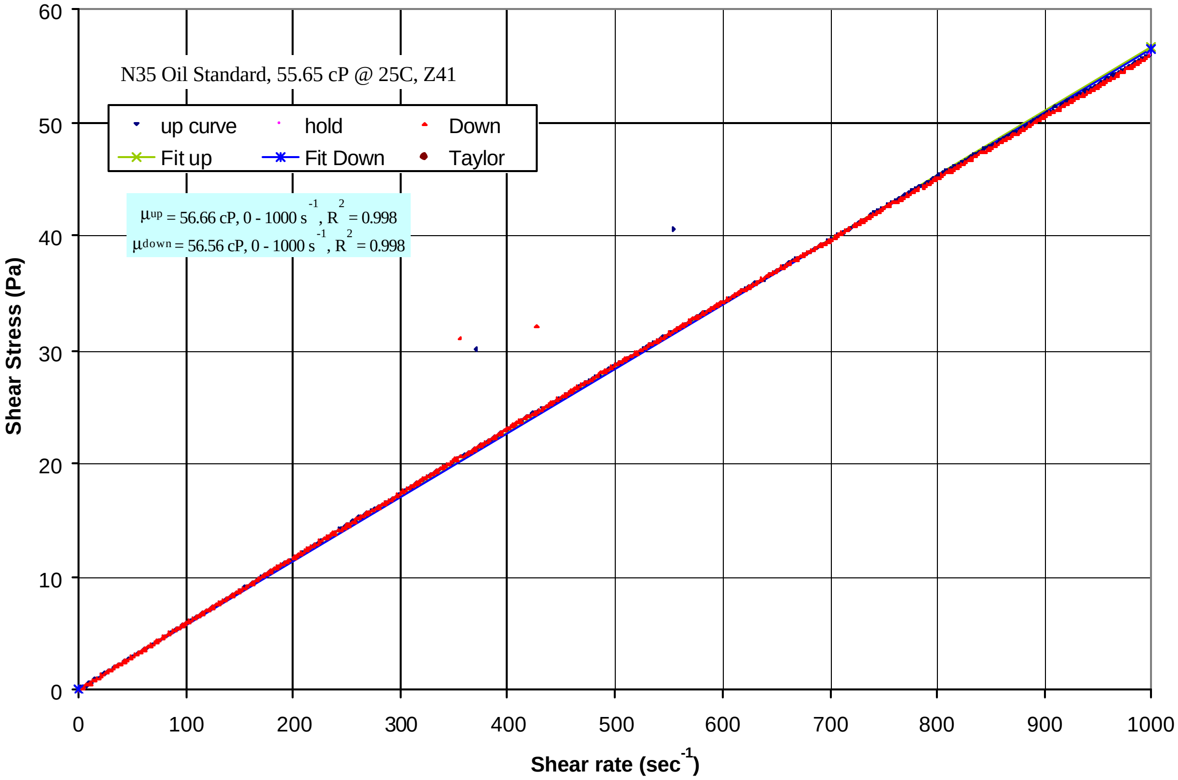

Sample: ccuf-an102-pr-bats, Run 1
up curve
Fit up
μup = 3.1 cP, 0 - 800 s-1, R2 = 1.000
Shear Stress (Pa)
0
1
1
2
2
3
3
0
200
400
600
800
Shear rate (sec-1)

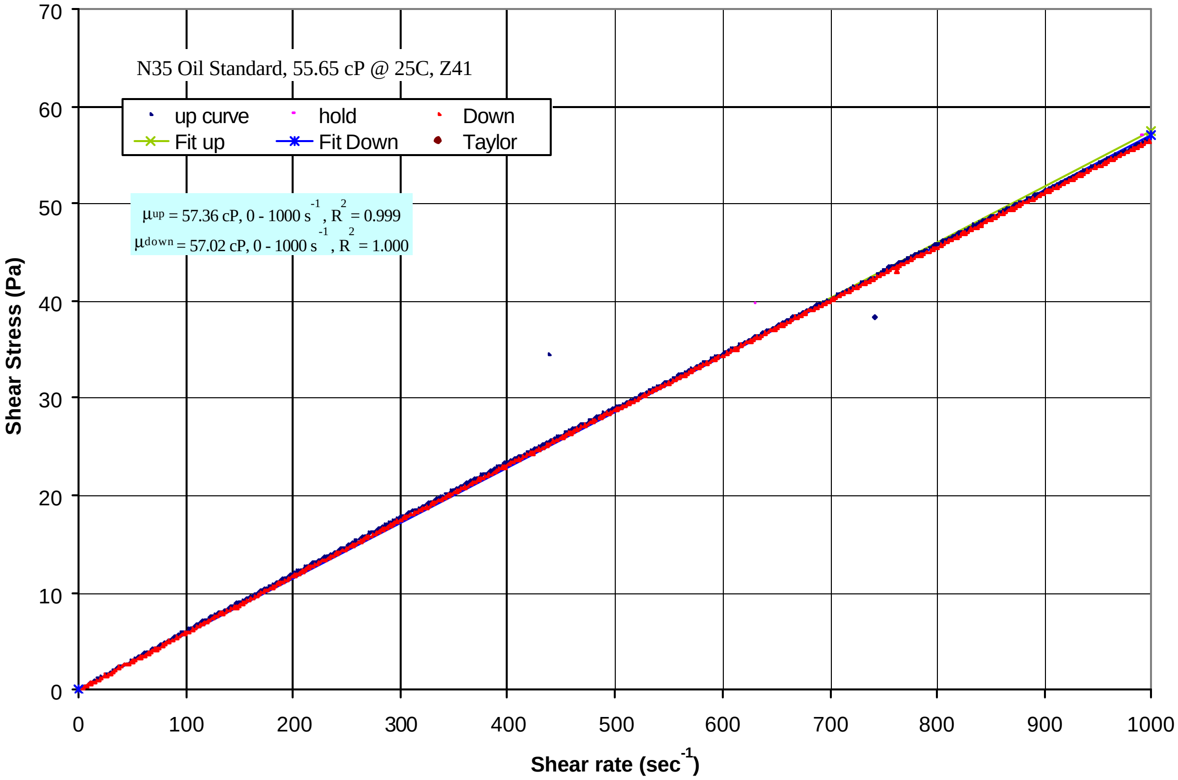

Sample: ccuf-an102-pr-bats, Run 2
up curve
Fit up
μup = 3.1 cP, 0 - 800 s-1, R2 = 1.000
Shear Stress (Pa)
0
1
1
2
2
3
3
0
200
400
600
800
Shear rate (sec-1)

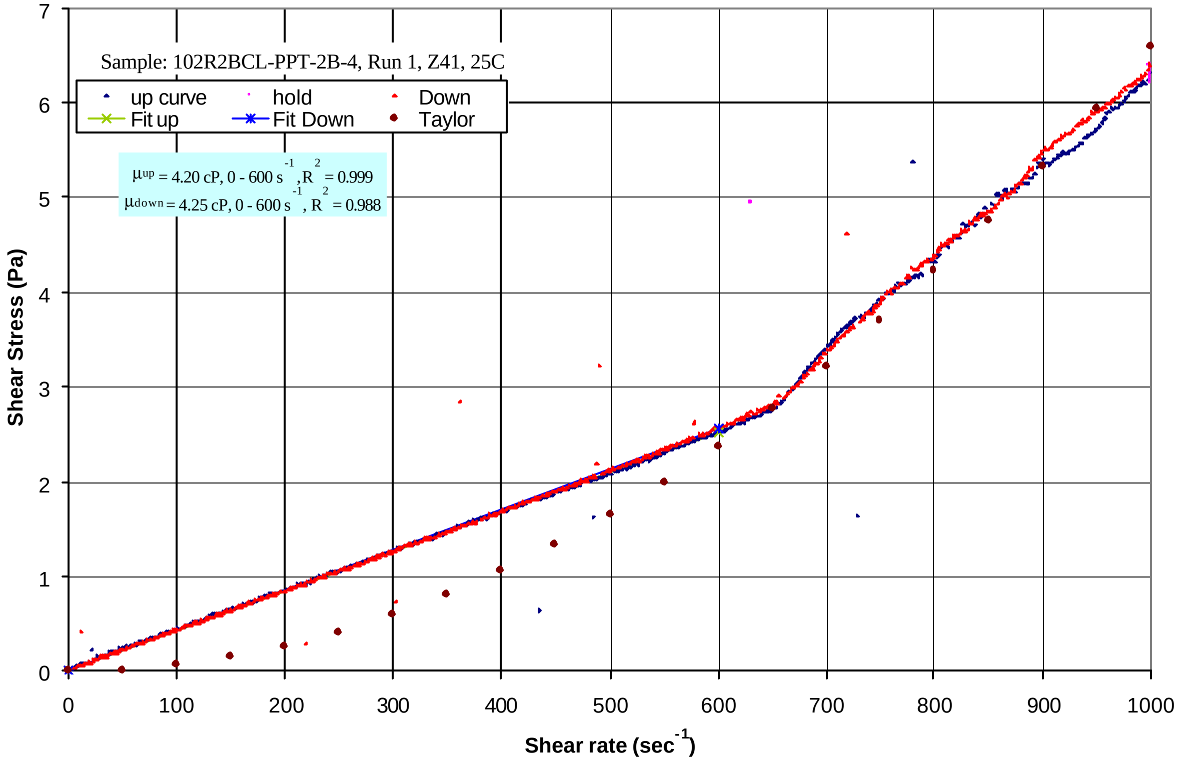

Sample: ccuf-an102-pr-wash, Run 1
up curve
Fit up
μ_{up} = 1.55 cP, 0 - 800 s^{-1}, R^2 = 1.000
Shear Stress (Pa)
1
1
1
1
1
0
0
0
0
200
400
600
800
Shear rate (sec^{-1})

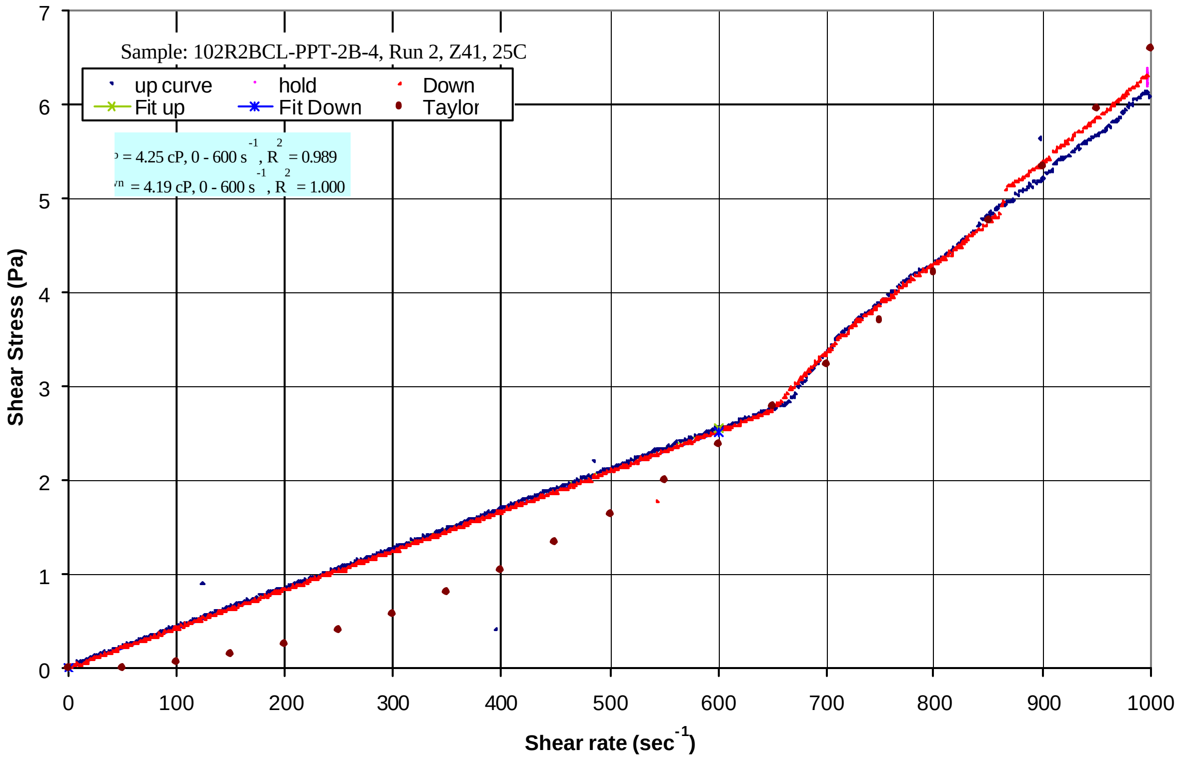

Sample: ccuf-an102-pr-wash, Run 2
up curve
Fit up
μ_{up} = 1.55 cP, 0 - 800 s^{-1}, R^2 = 1.000
Shear Stress (Pa)
1
1
1
1
1
0
0
0
0
200
400
600
800
Shear rate (sec^{-1})

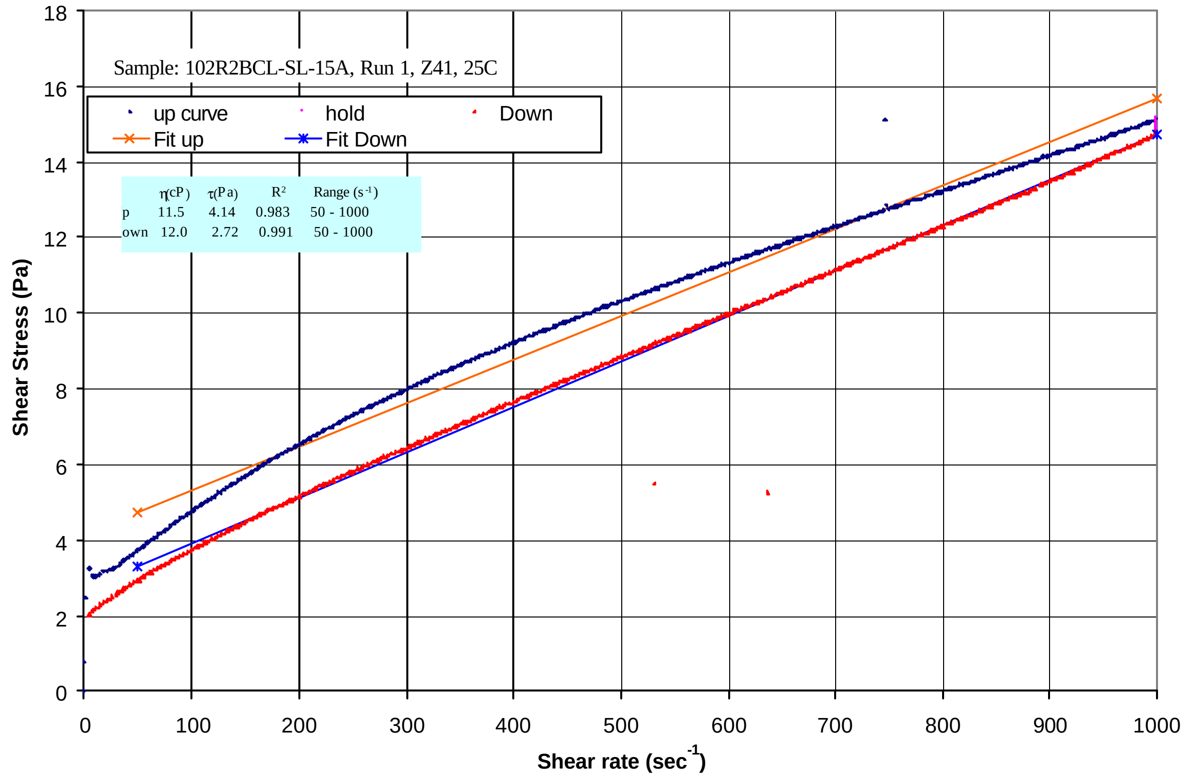
R2BCL and R2NOC2 Slurries:

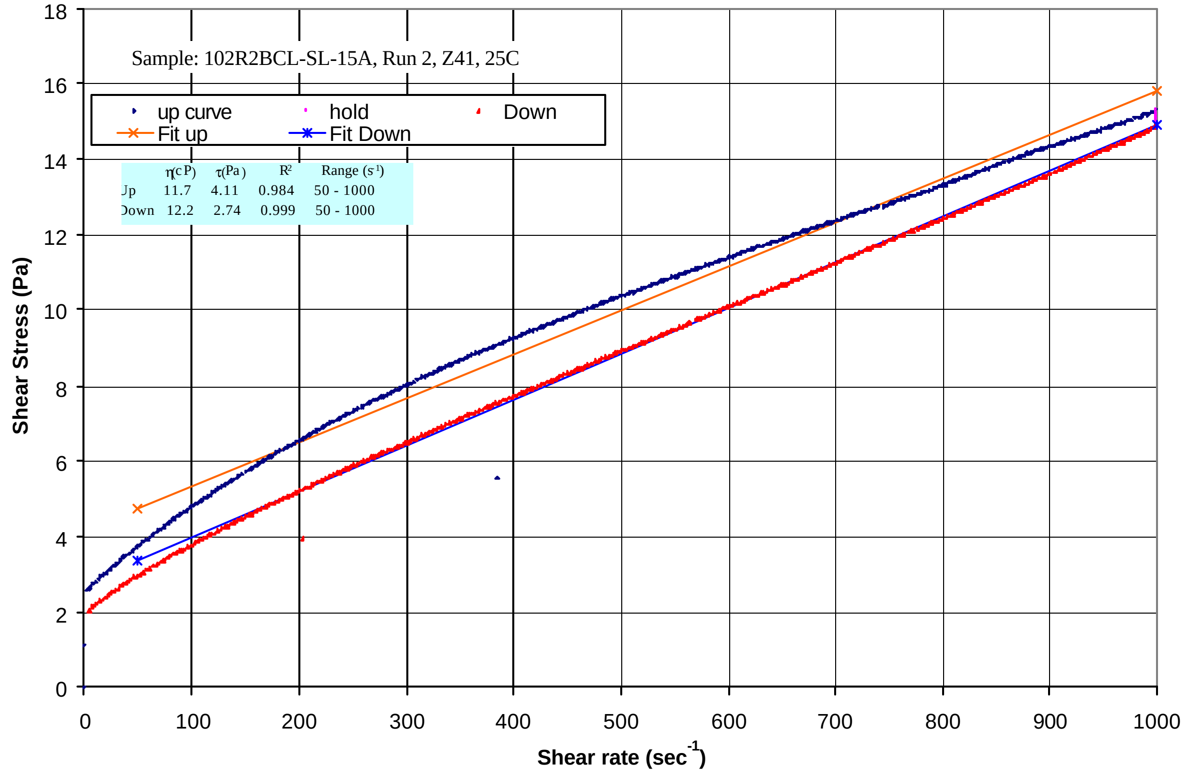


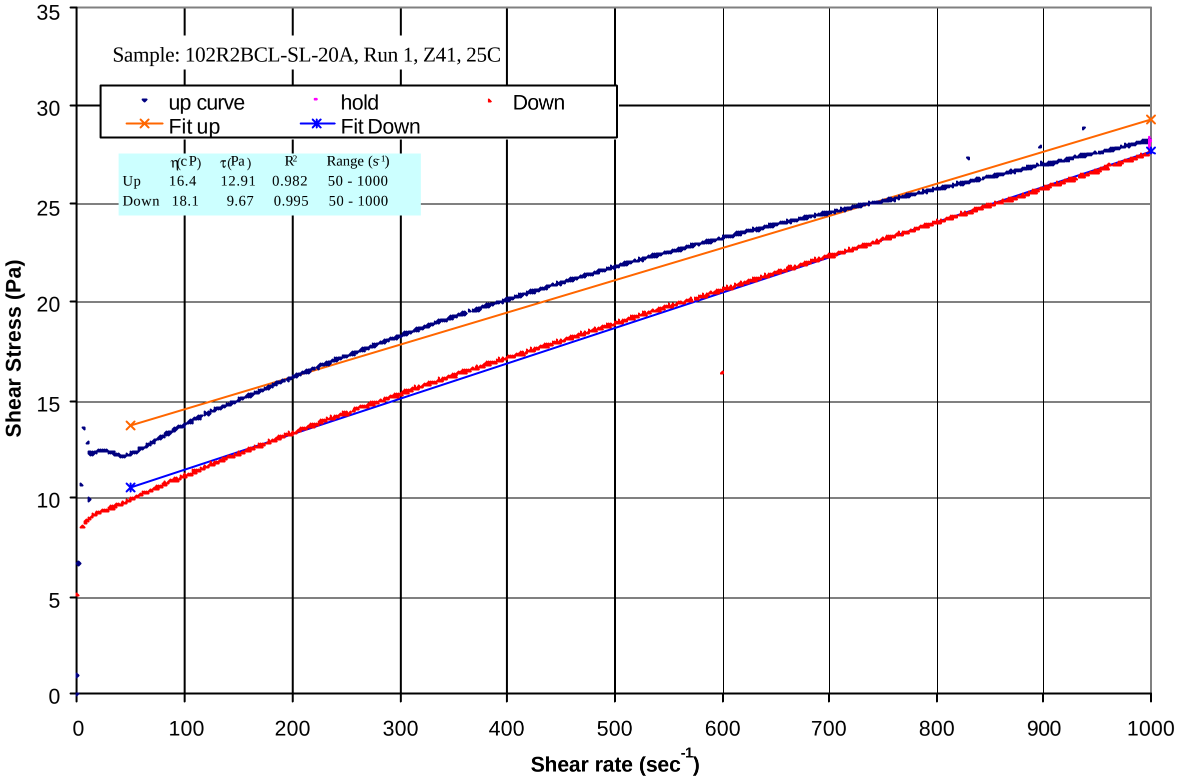


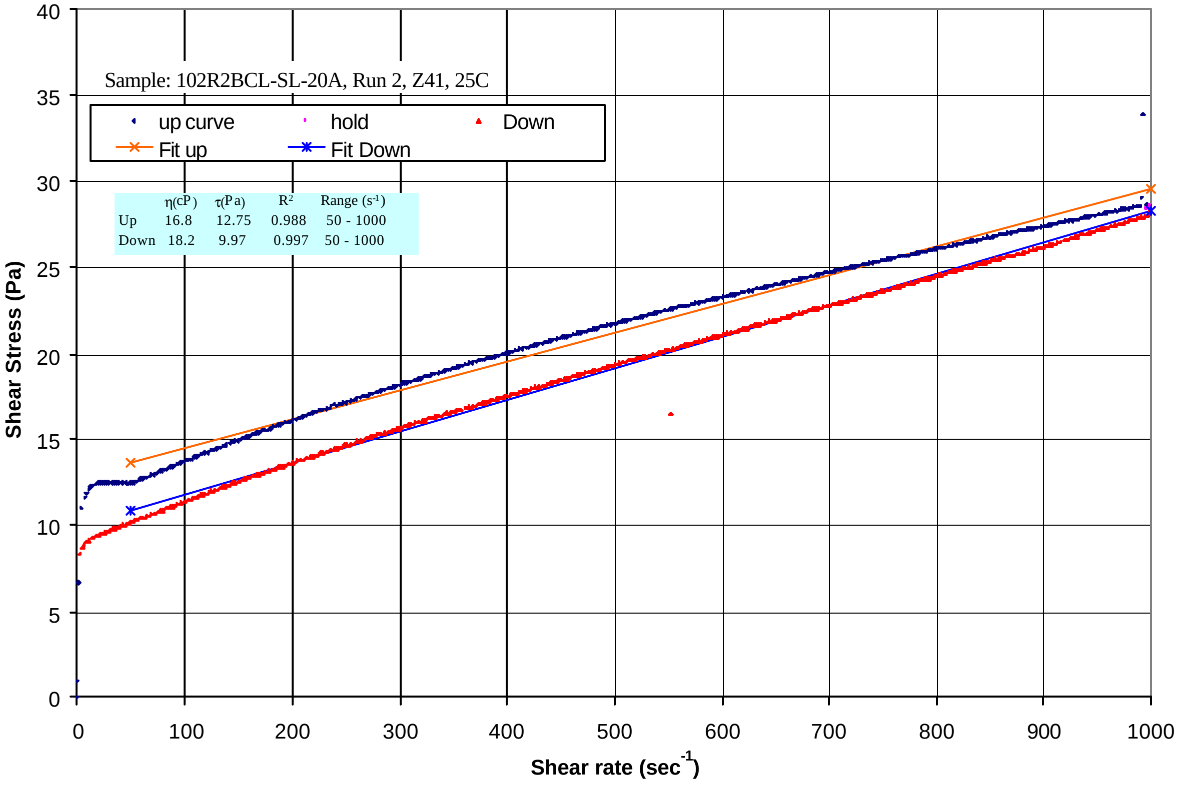

Sample: 102R2BCL-PPT-2B-4, Run 1, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
Taylor
μ_{up} = 4.20 cP, 0 - 600 s^{-1}, R^2 = 0.999
μ_{down} = 4.25 cP, 0 - 600 s^{-1}, R^2 = 0.988
Shear Stress (Pa)
0
1
2
3
4
5
6
7
Shear rate (sec^{-1})
0
100
200
300
400
500
600
700
800
900
1000

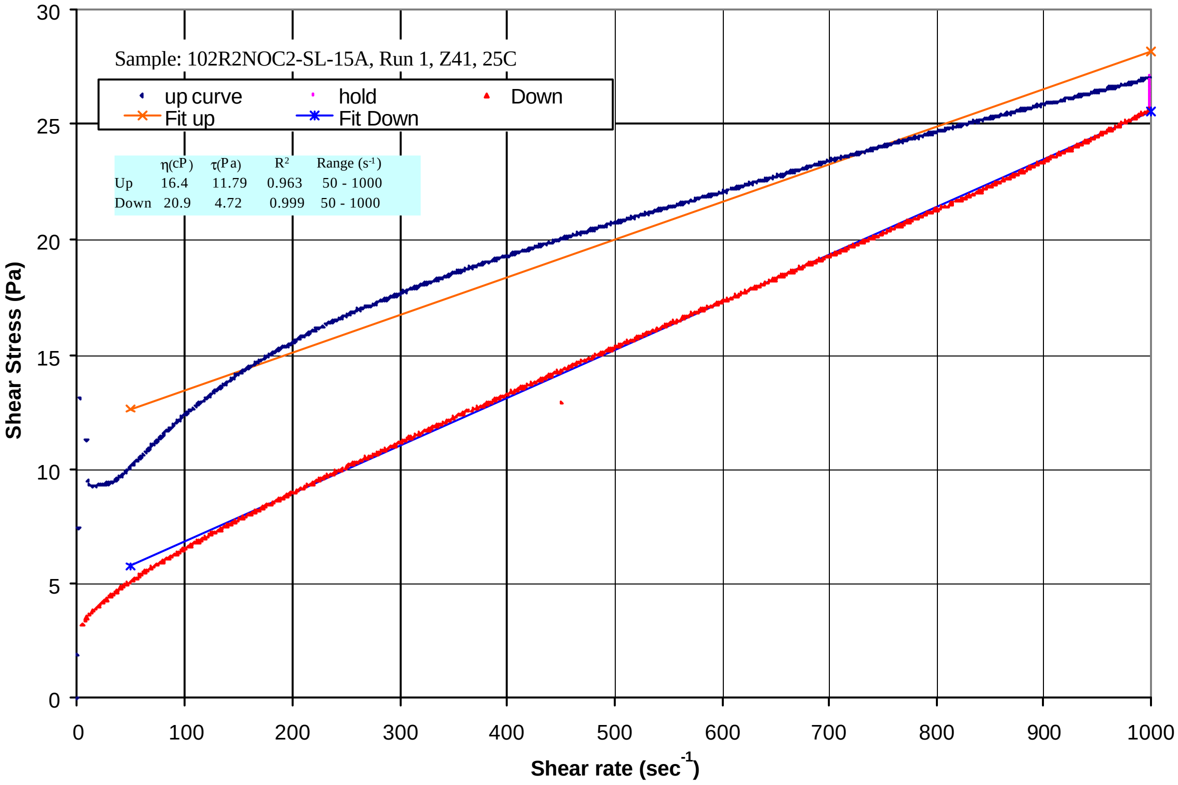

Sample: 102R2BCL-PPT-2B-4, Run 2, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
Taylor
= 4.25 cP, 0 - 600 s^{-1}, R^2 = 0.989
= 4.19 cP, 0 - 600 s^{-1}, R^2 = 1.000
Shear Stress (Pa)
0
1
2
3
4
5
6
7
Shear rate (sec^{-1})
0
100
200
300
400
500
600
700
800
900
1000


Sample: 102R2BCL-SL-15A, Run 1, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
p 11.5 4.14 0.983 50 - 1000
own 12.0 2.72 0.991 50 - 1000
Shear Stress (Pa)
0
2
4
6
8
10
12
14
16
18
0
100
200
300
400
500
600
700
800
900
1000
Shear rate (sec-1)

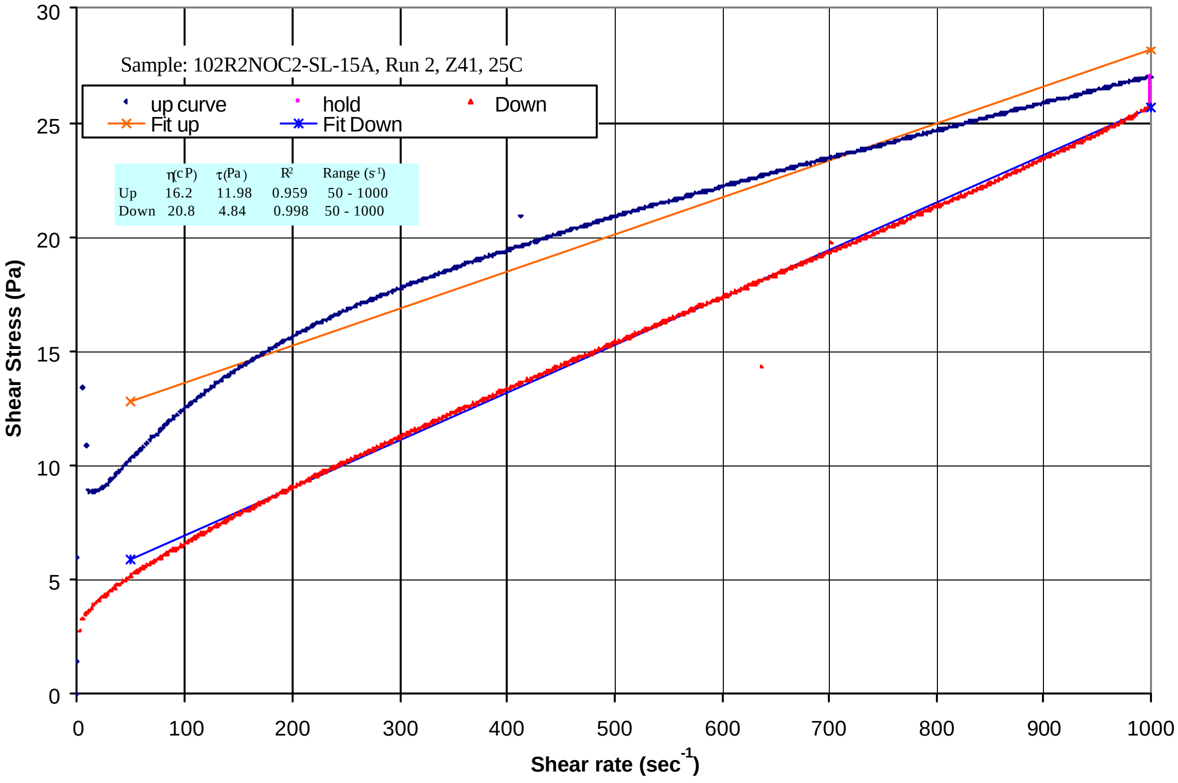

Sample: 102R2BCL-SL-15A, Run 2, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
Up 11.7 4.11 0.984 50 - 1000
Down 12.2 2.74 0.999 50 - 1000
Shear Stress (Pa)
0
2
4
6
8
10
12
14
16
18
0
100
200
300
400
500
600
700
800
900
1000
Shear rate (sec-1)


Sample: 102R2BCL-SL-20A, Run 1, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
Up 16.4 12.91 0.982 50 - 1000
Down 18.1 9.67 0.995 50 - 1000
Shear Stress (Pa)
0
5
10
15
20
25
30
35
Shear rate (sec⁻¹)
0
100
200
300
400
500
600
700
800
900
1000

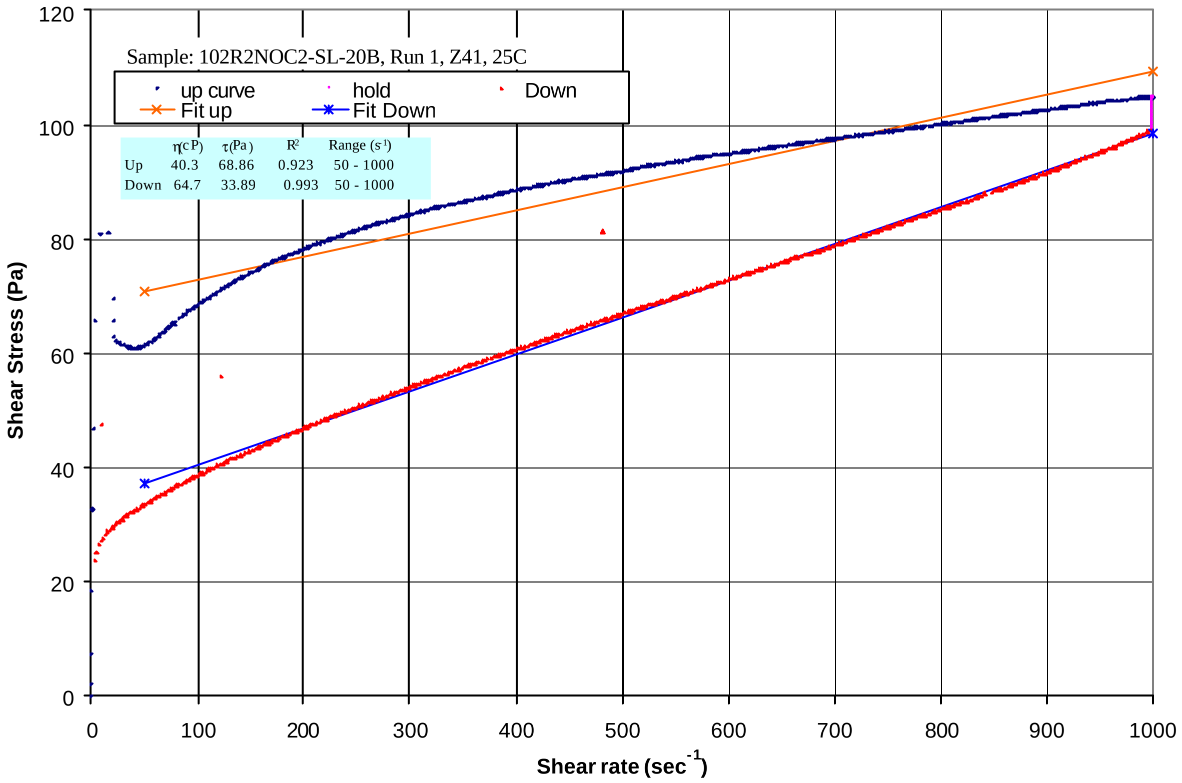

Sample: 102R2BCL-SL-20A, Run 2, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
Up 16.8 12.75 0.988 50 - 1000
Down 18.2 9.97 0.997 50 - 1000
Shear Stress (Pa)
0
5
10
15
20
25
30
35
40
Shear rate (sec⁻¹)
0
100
200
300
400
500
600
700
800
900
1000

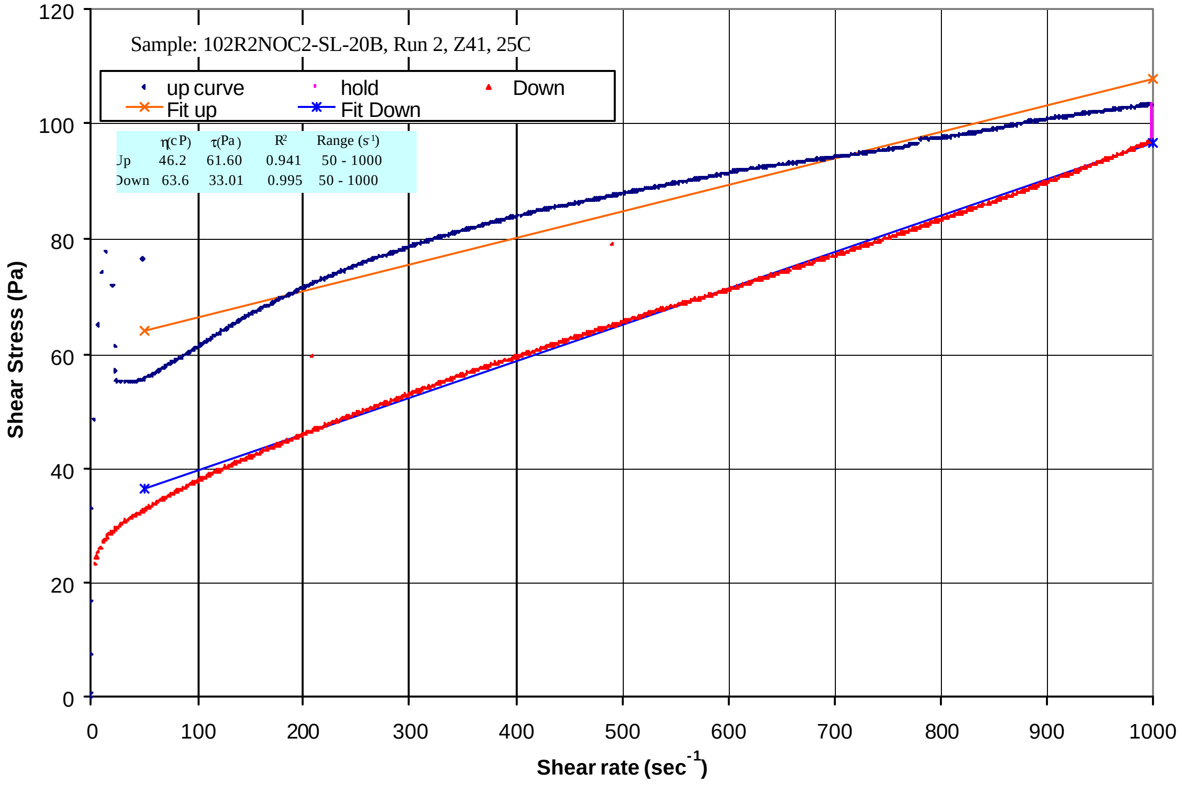


	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	16.4	11.79	0.963	50 - 1000
Down	20.9	4.72	0.999	50 - 1000



	η(cP)	τ(Pa)	R^2	Range (s^{-1})
Up	16.2	11.98	0.959	50 - 1000
Down	20.8	4.84	0.998	50 - 1000


Sample: 102R2NOC2-SL-20B, Run 1, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
Up 40.3 68.86 0.923 50 - 1000
Down 64.7 33.89 0.993 50 - 1000
Shear Stress (Pa)
0
20
40
60
80
100
120
0
100
200
300
400
500
600
700
800
900
1000
Shear rate (sec⁻¹)


Sample: 102R2NOC2-SL-20B, Run 2, Z41, 25C
up curve
hold
Down
Fit up
Fit Down
η(cP) τ(Pa) R² Range (s⁻¹)
Jp 46.2 61.60 0.941 50 - 1000
own 63.6 33.01 0.995 50 - 1000
Shear Stress (Pa)
0
20
40
60
80
100
120
0
100
200
300
400
500
600
700
800
900
1000
Shear rate (sec⁻¹)

APPENDIX A5. JMP CURVE FITTING DATA

AN102R1BC DATA
LOW WT% SOLIDS FACTORIAL

$\ln F = a + v \ln V + p \ln P + u \ln t$
or
$F = e^a V^v P^p t^u$

F = flux (gpm/ft^2)
V = velocity (fps)
P = TMP (psi)
t = time since start of tests (hr)
a, v, p, u = parameters

Summary of Fit

RSquare	0.918762
RSquare Adj	0.900015
Root Mean Square Error	0.049574
Mean of Response	-2.75008
Observations (or Sum Wgts)	17

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.36132469	0.120442	49.0078
Error	13	0.03194882	0.002458	Prob > F
C. Total	16	0.39327351		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-4.324277	0.250122	-17.29	<.0001
ln time	-0.121504	0.015588	-7.79	<.0001
ln TMP	0.0863982	0.040071	2.16	0.0504
ln V	0.6272042	0.069238	9.06	<.0001

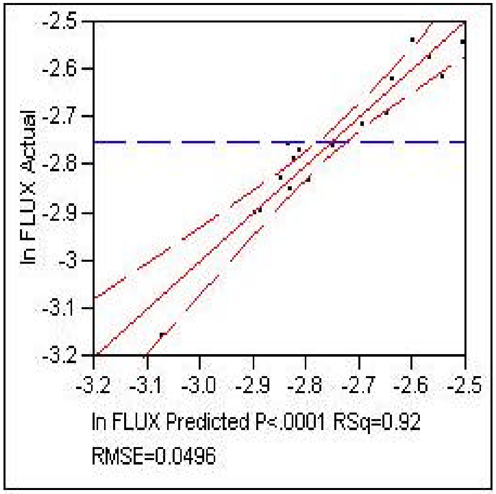
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ln time	1	1	0.14932253	60.7595	<.0001
ln TMP	1	1	0.01142540	4.6490	0.0504
ln V	1	1	0.20166984	82.0596	<.0001

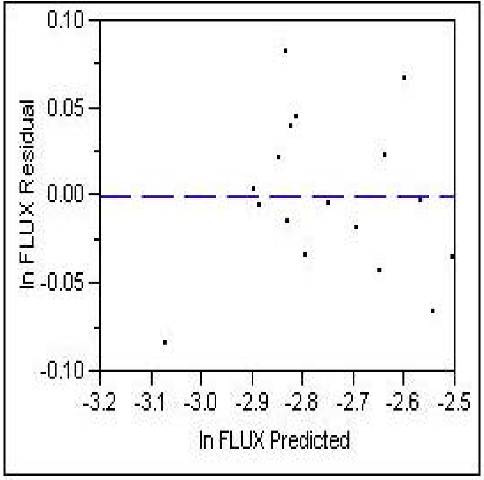
Correlation of Estimates

Corr	Intercept	ln time	ln TMP	ln V
Intercept	1.0000	-0.163	-0.755	-0.812
ln time	-0.163	1.0000	0.0356	0.0288
ln TMP	-0.755	0.0356	1.0000	0.2515
ln V	-0.812	0.0288	0.2515	1.0000

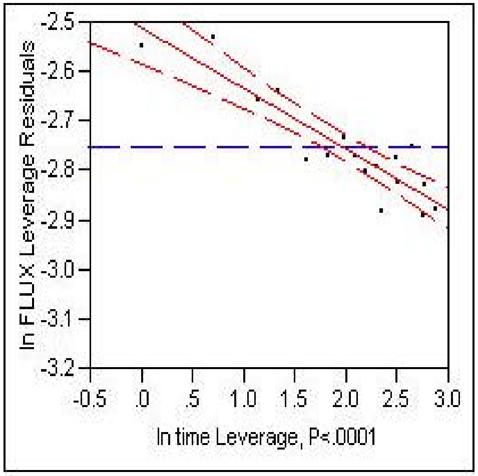
Actual by Predicted Plot



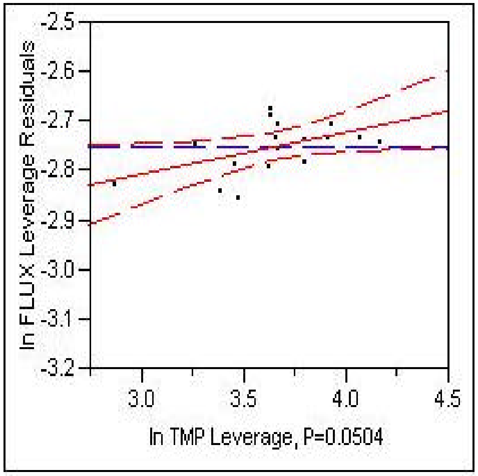
Residual by Predicted Plot



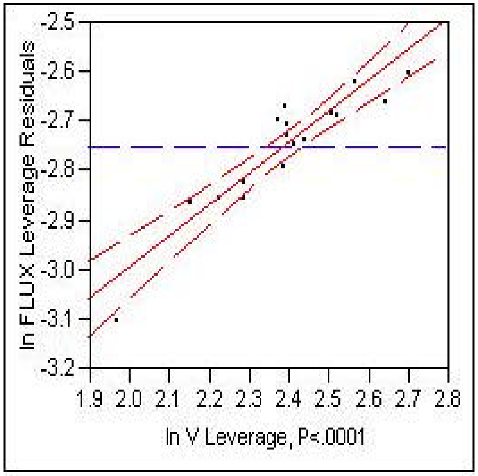
ln time
Leverage Plot



ln TMP
Leverage Plot



ln V
Leverage Plot



AN102R1BC DATA
HIGH WT% (approximately 17.2) SOLIDS FACTORIAL

$\ln F = a + v \ln V + u \ln t$
or
$F = e^a V^v t^u$

F = flux (gpm/ft^2)
V = velocity (fps)
P = TMP (psi)
t = time since start of tests (hr)
a, v, p, u = parameters

Note: pressure was not statistically significant

Summary of Fit

RSquare	0.870774
RSquare Adj	0.850893
Root Mean Square Error	0.072618
Mean of Response	-5.04014
Observations (or Sum Wgts)	16

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.46194402	0.230972	43.7995
Error	13	0.06855413	0.005273	Prob > F
C. Total	15	0.53049814		<.0001

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4159563	0.980182	0.42	0.6782
ln time	-1.647299	0.225335	-7.31	<.0001
ln V	0.50251	0.106916	4.70	0.0004

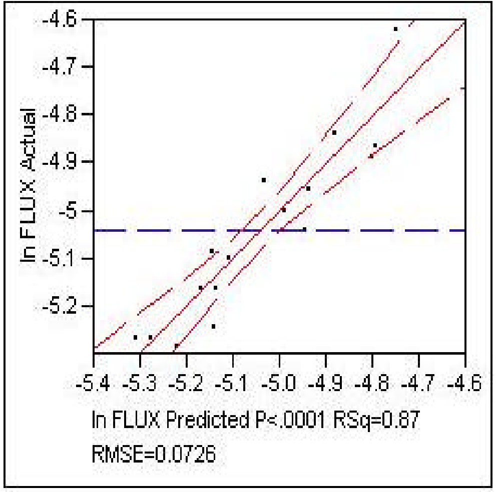
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ln time	1	1	0.28182351	53.4425	<.0001
ln V	1	1	0.11649086	22.0903	0.0004

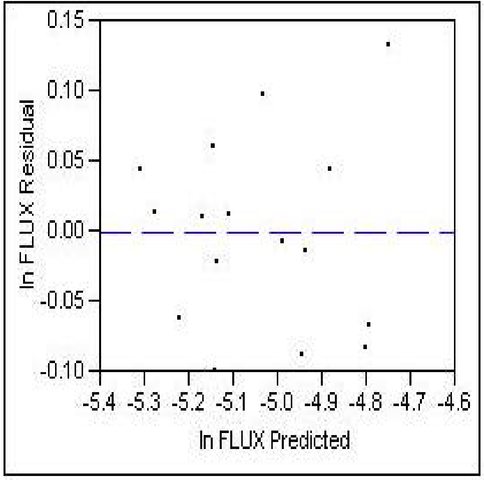
Correlation of Estimates

Corr	Intercept	ln time	ln V
Intercept	1.0000	-0.966	-0.396
ln time	-0.966	1.0000	0.1478
ln V	-0.396	0.1478	1.0000

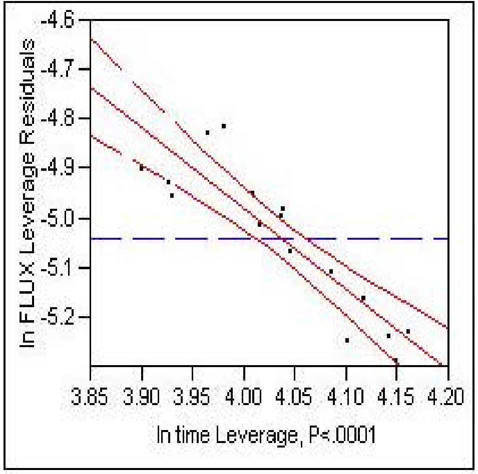
Actual by Predicted Plot



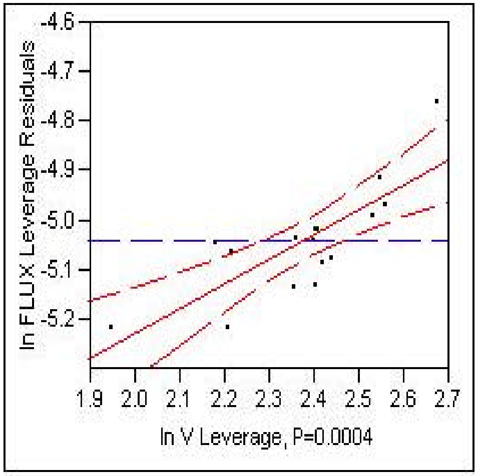
Residual by Predicted Plot



ln time
Leverage Plot



ln V
Leverage Plot



Flux versus Time for Concentration of Slurry

$F = a\,(1 - b\,S - c\,R)\,t^{-0.1215}$

F = flux (gpm/ft^2)
t = time since start of tests (hr)
S = insoluble solids (wt%)
R = 0 if before redilution back to 17.2 wt%; = 1 if after (2 data points)
a, b, c = parameters

R factor used to account for flux after redilution that did not return to previous value at same solids content.

Nonlinear Fit

```
Converged in the Gradient

Criterion                Current        Stop Limit
Iteration                      2                60
Shortening                     0                15
Obj Change          0.0000864071         0.0000001
Prm Change          0.0022076205         0.0000001
Gradient             8.6407135e-9          0.000001
Parameter          Current Value
a                   0.0945850528
b                    0.046194566
c                   0.0716954074

SSE = 0.0000325408
N = 16
Alpha = 0.050
Convergence Criterion = 0.00001
Goal SSE for CL = 0.0000442235
```

Solution

```
                SSE        DFE           MSE          RMSE
       0.0000325408         13     0.0000025     0.0015821
Parameter               Estimate    ApproxStdErr      Lower CL       Upper CL
a                   0.0945850528        0.001049    0.09231882     0.09685128
b                    0.046194566      0.00072735    0.04460366     0.04774819
c                   0.0716954074      0.02249778    0.02332142     0.12055594
```

Correlation of Estimates

```
                               a              b              c
a                         1.0000         0.4945        -0.2090
b                         0.4945         1.0000        -0.5232
c                        -0.2090        -0.5232         1.0000
```

APPENDIX A6. SIMULANT RECIPES

AN102 REMEDIATION 1 (R1)

Final SRS AN102 at 6.5 Molar Sodium recipe

Volume of Feed		1000	mL

0.5% Accuracy on masses and volumes is sufficient

Tare Weight		grams

	grams	Actual Wt, grams
Water	200	

Transition Metals and Complexing agents

Compounds	Formula	Mass Needed	Actual Wt, grams
Cadmium Nitrate	Cd(NO3)2.4H2O	0.14	
Calcium Nitrate	Ca(NO3)2.4H2O	2.36	
Cerium Nitrate	Ce(NO3)3.6H2O	0.00	■
Cesium Nitrate	CsNO3	0.019	
Cobalt Nitrate	Co(NO3)2.6H2O	0.01	
Copper Nitrate	Cu(NO3)2.2.5H2	0.07	
Ferric Nitrate	Fe(NO3)3.9H2O	0.25	
Lanthanum Nitrate	La(NO3)3.6H2O	0.04	
Lead nitrate	Pb(NO3)2	0.24	
Magnesium Nitrate	Mg(NO3)2.6H2O	0.00	■
Manganous Chloride	MnCl2.4H2O	0.09	
Neodymium Nitrate	Nd(NO3)3.6H2O	0.08	
Nickel Nitrate	Ni(NO3)2.6H2O	1.68	
Potassium Nitrate	KNO3	4.11	
Rubidium Nitrate	RbNO3	0.01	
Strontium Nitrate	Sr(NO3)2	0.005	
Zinc Nitrate	Zn(NO3)2.6H2O	0.02	
Zirconyl Nitrate	ZrO(NO3)2.H2O	0.03	
Disodium Ethylenediaminetetra	Na2C10H14N2O8.2H2O	2.92	
n-(2-Hydroxyethyl)ethylenediaminetriacetic acid	C10H18N2O7	0.30	
Sodium Gluconate	HOCH2(CHOH)4COONa	1.34	
Citric Acid	C6H8O7.H2O	4.22	
Nitrilotriacetic Acid	C6H9NO6	0.21	
Iminodiacetic Acid	C4H7NO4	3.71	
Succinic Acid	C4H6O4	0.03	
Glutaric Acid	C5H8O4	0.05	
Adipic Acid	C6H10O4	0.20	
Azelaic Acid	C9H16O4	0.85	

Suberic Acid	C8H14O4	1.49	
Ammonium Acetate	NH4CH3COO	0.51	
Boric acid	H3BO3	0.17	
Sodium Chloride	NaCl	6.38	
Sodium Fluoride	NaF	3.09	
Sodium Sulfate	Na2SO4	15.22	
Potassium Molybdate	K2MoO4	0.09	

In separate container mix the following

Add	Formula	Mass Needed	Actual Wt, grams
Sodium Hydroxide	NaOH	79.31	
Aluminum Nitrate	Al(NO3)3.9H2O	139.03	
Sodium Phosphate	Na3PO4.12H2O	18.01	
Sodium Tungstate	Na2WO4.2H2O	0.25	
Sodium Metasilicate	Na2SiO3.9H2O	0.08	
Sodium formate	NaHCOO	10.36	
Sodium Glycolate	HOCH2COONa	11.14	
Sodium Acetate	NaCH3COO.3H2	0.55	
Sodium Oxalate	Na2C2O4	0.57	

Add	grams	Actual Wt, grams
Water	200	

Mix thoroughly. Then add this solution to the Vessel

Add	Formula	Mass Needed	Actual Wt, grams
Sodium Chromate	Na2CrO4	0.64	
Sodium Carbonate	Na2CO3	48.45	

Mix thoroughly.

Mix	Formula	Mass Needed	Actual Wt, grams
Sodium Nitrate	NaNO3	119.84	
Sodium Nitrite	NaNO2	94.49	
Water		100	

Add and Mix thoroughly.

Add	Formula	Mass Needed	Actual Wt, grams
Water	H2O	236.53	

Record Final Weight		grams

Note: the Final water addition is based on 1.309 g/mL density.

Measure the Density ______ g/mL

For INFO ONLY
The final addition of water would be 236.53 grams based upon
a density of 1.30855 g/mL.

Solution Labeling Final AN-102 Simulant at 6.5 M Na

AN102 REMEDIATION 2 (R2)

Final SRS AN102R2 at 6.5 Molar Sodium recipe

Volume of Feed		1000	mL

0.5% Accuracy on masses and volumes is sufficient

Tare Weight		grams

	grams	Actual Wt, grams
Water	200	

Transition Metals and Complexing agents

Compounds	Formula	Mass Needed	Actual Wt, grams
Cadmium Nitrate	Cd(NO3)2.4H2O	0.14	
Calcium Nitrate	Ca(NO3)2.4H2O	2.36	
Cerium Nitrate	Ce(NO3)3.6H2O	0.00	
Cesium Nitrate	CsNO3	0.019	
Cobalt Nitrate	Co(NO3)2.6H2O	0.01	
Copper Nitrate	Cu(NO3)2.2.5H2O	0.07	
Ferric Nitrate	Fe(NO3)3.9H2O	0.25	
Lanthanum Nitrate	La(NO3)3.6H2O	0.04	
Lead nitrate	Pb(NO3)2	0.24	
Magnesium Nitrate	Mg(NO3)2.6H2O	0.00	
Manganous Chloride	MnCl2.4H2O	0.09	
Neodymium Nitrate	Nd(NO3)3.6H2O	0.08	
Nickel Nitrate	Ni(NO3)2.6H2O	1.68	
Potassium Nitrate	KNO3	4.11	
Rubidium Nitrate	RbNO3	0.01	
Strontium Nitrate	Sr(NO3)2	0.005	
Zinc Nitrate	Zn(NO3)2.6H2O	0.02	
Zirconyl Nitrate	ZrO(NO3)2.H2O	0.03	
Disodium Ethylenediaminetetraacetate	Na2C10H14N2O8	2.92	
n-(2-Hydroxyethyl)ethylenediaminetriacetic acid	C10H18N2O7	0.30	
Sodium Gluconate	HOCH2(CHOH)4C	1.34	
Citric Acid	C6H8O7.H2O	4.22	
Nitrilotriacetic Acid	C6H9NO6	0.21	
Iminodiacetic Acid	C4H7NO4	3.71	
Succinic Acid	C4H6O4	0.03	
Glutaric Acid	C5H8O4	0.05	
Adipic Acid	C6H10O4	0.20	
Azelaic Acid	C9H16O4	0.85	
Suberic Acid	C8H14O4	1.49	
Ammonium Acetate	NH4CH3COO	0.51	
Boric acid	H3BO3	0.17	
Sodium Chloride	NaCl	6.38	
Sodium Fluoride	NaF	3.09	
Sodium Sulfate	Na2SO4	15.22	
Potassium Molybdate	K2MoO4	0.09	

In separate container mix the following

Add	Formula	Mass Needed	Actual Wt, grams
Sodium Hydroxide	NaOH	79.31	
Aluminum Nitrate	Al(NO3)3.9H2O	139.03	
Sodium Phosphate	Na3PO4.12H2O	18.01	
Sodium Tungstate	Na2WO4.2H2O	0.25	
Sodium Metasilicate	Na2SiO3.9H2O	0.08	
Sodium formate	NaHCOO	10.36	
Sodium Glycolate	HOCH2COONa	11.14	
Sodium Acetate	NaCH3COO.3H2	0.55	
Sodium Oxalate	Na2C2O4	0.57	

Add	grams	Actual Wt, grams
Water	200	

Mix thoroughly. Then add this solution to the Vessel

Add	Formula	Mass Needed	Actual Wt, grams
Sodium Chromate	Na2CrO4	0.64	
Sodium Carbonate	Na2CO3	78.98	

Mix thoroughly.

Mix	Formula	Mass Needed	Actual Wt, grams
Sodium Nitrate	NaNO3	87.85	
Sodium Nitrite	NaNO2	80.71	
Water		100	

Add and Mix thoroughly.

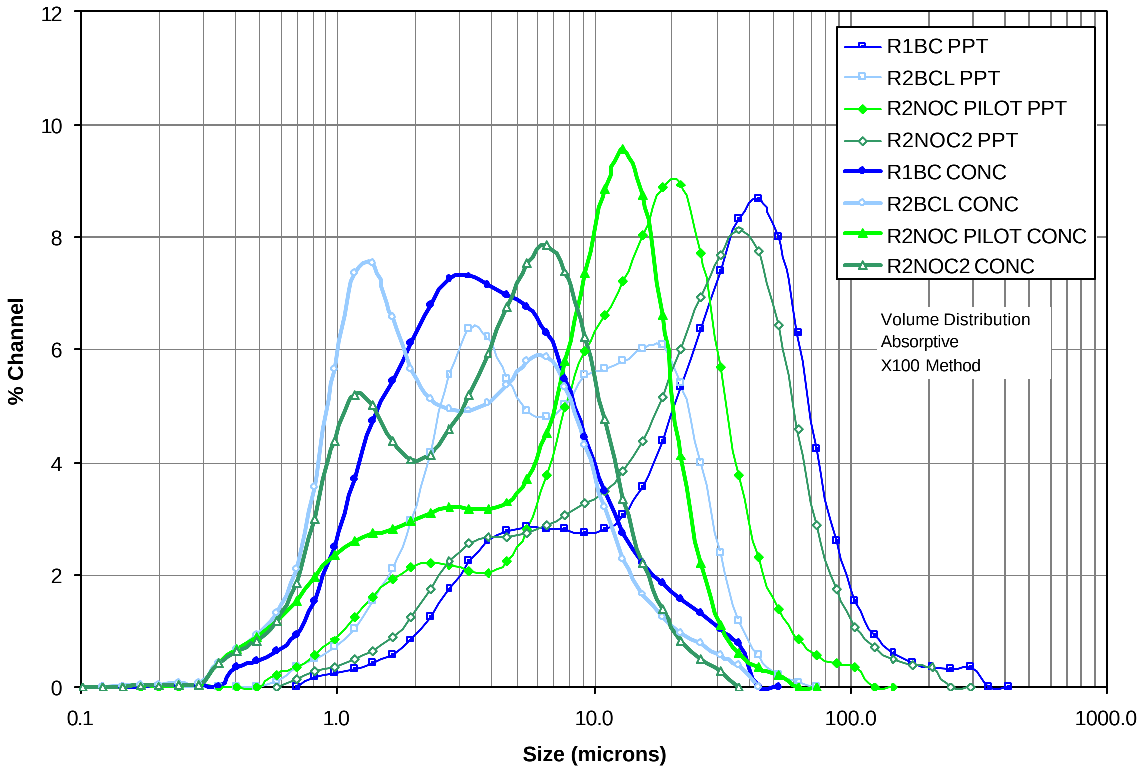
Add	Formula	Mass Needed	Actual Wt, grams
Water	H2O	241.54	

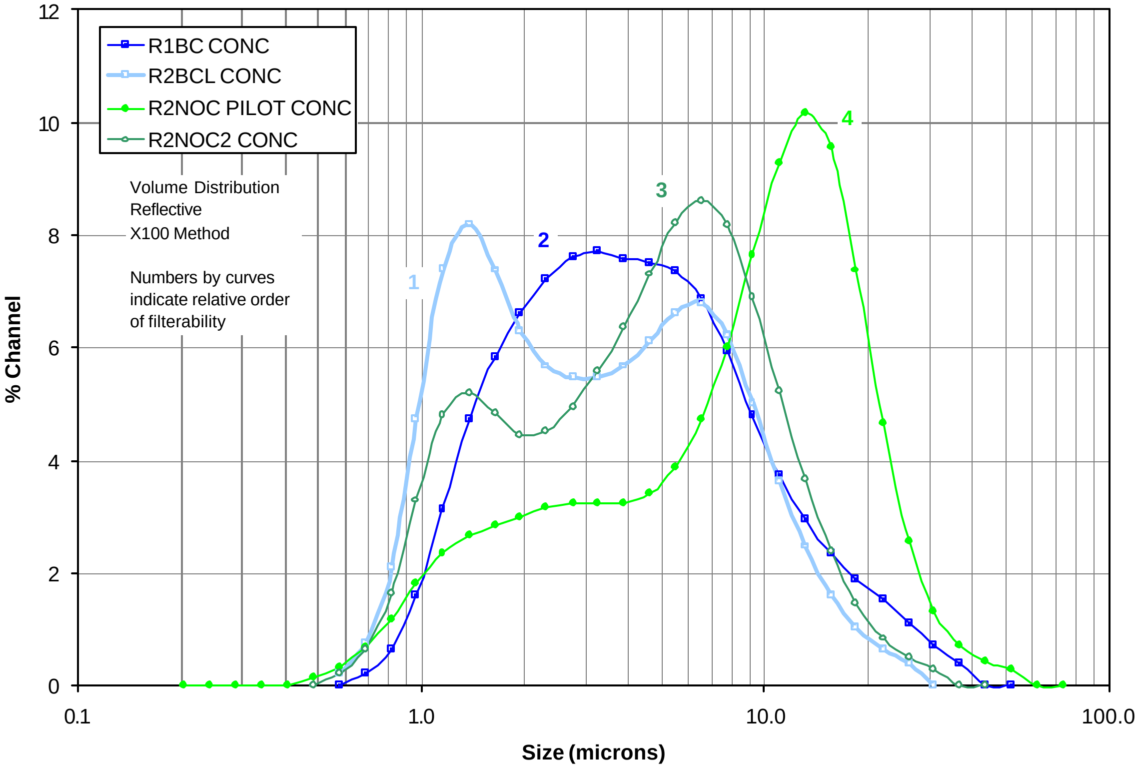
Record Final Weight ______ grams

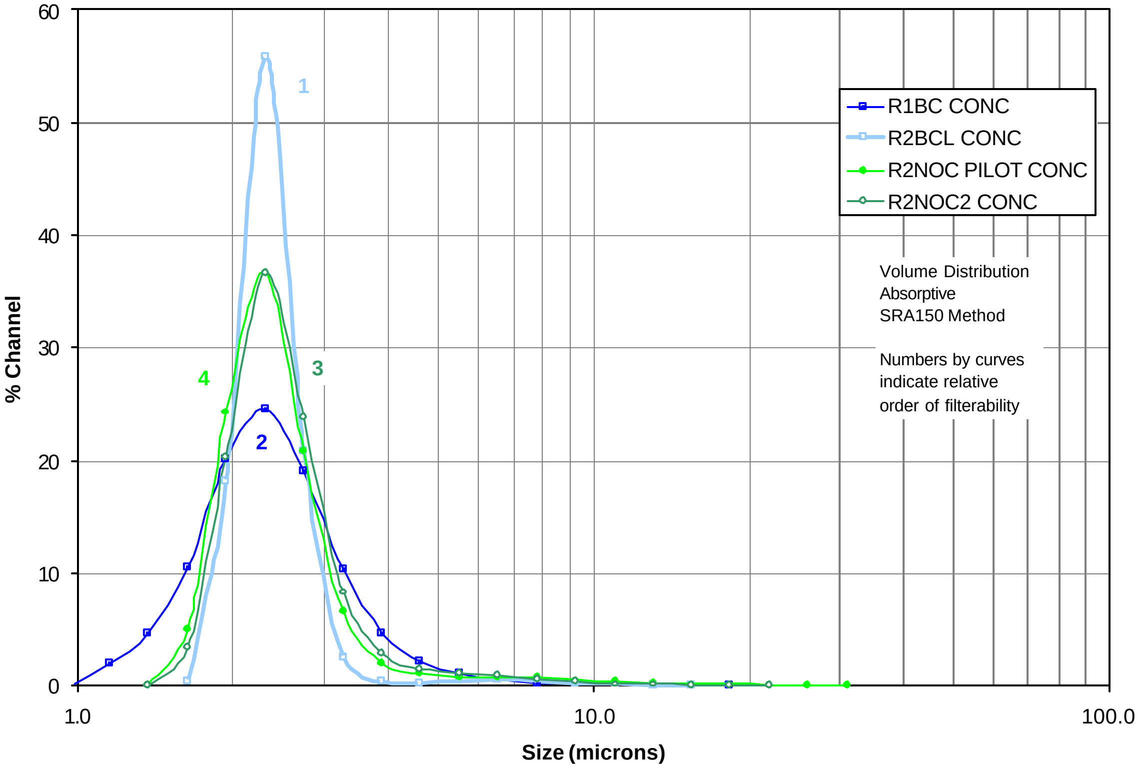
Note: the Final water addition is based on 1.298 g/mL density.

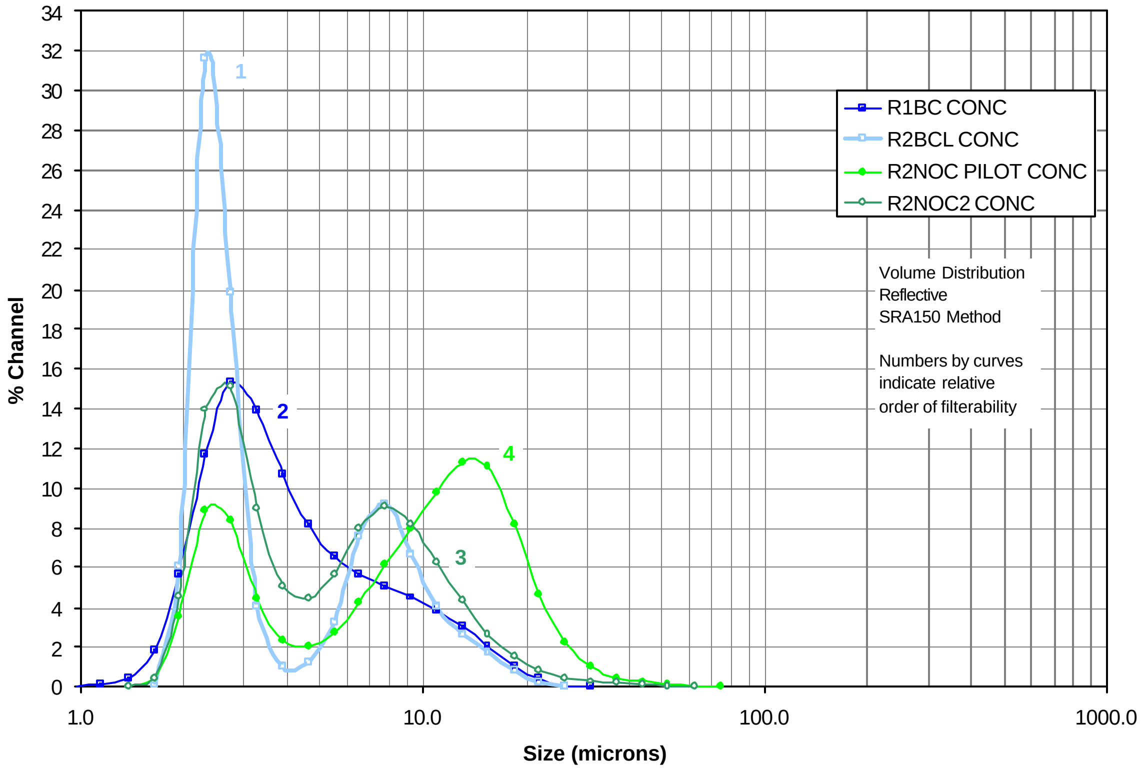
APPENDIX A7. PARTICLE SIZE DISTRIBUTION DATA

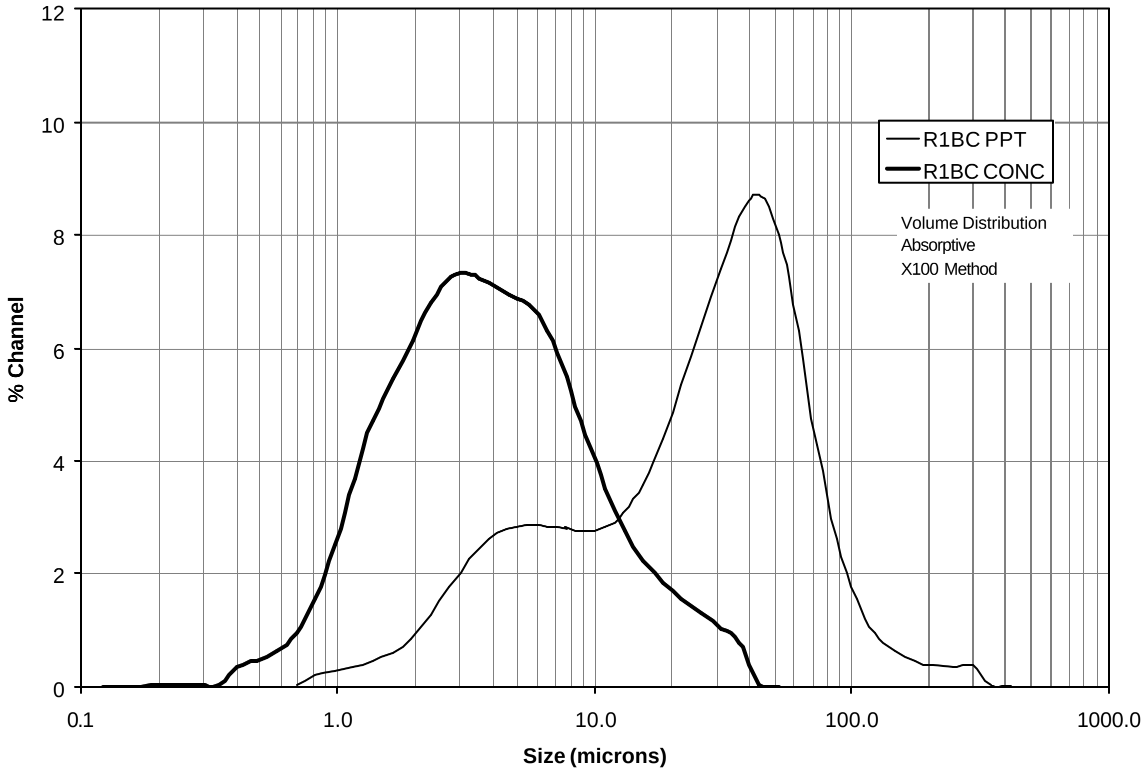
Graphical Data - Volume Distributions

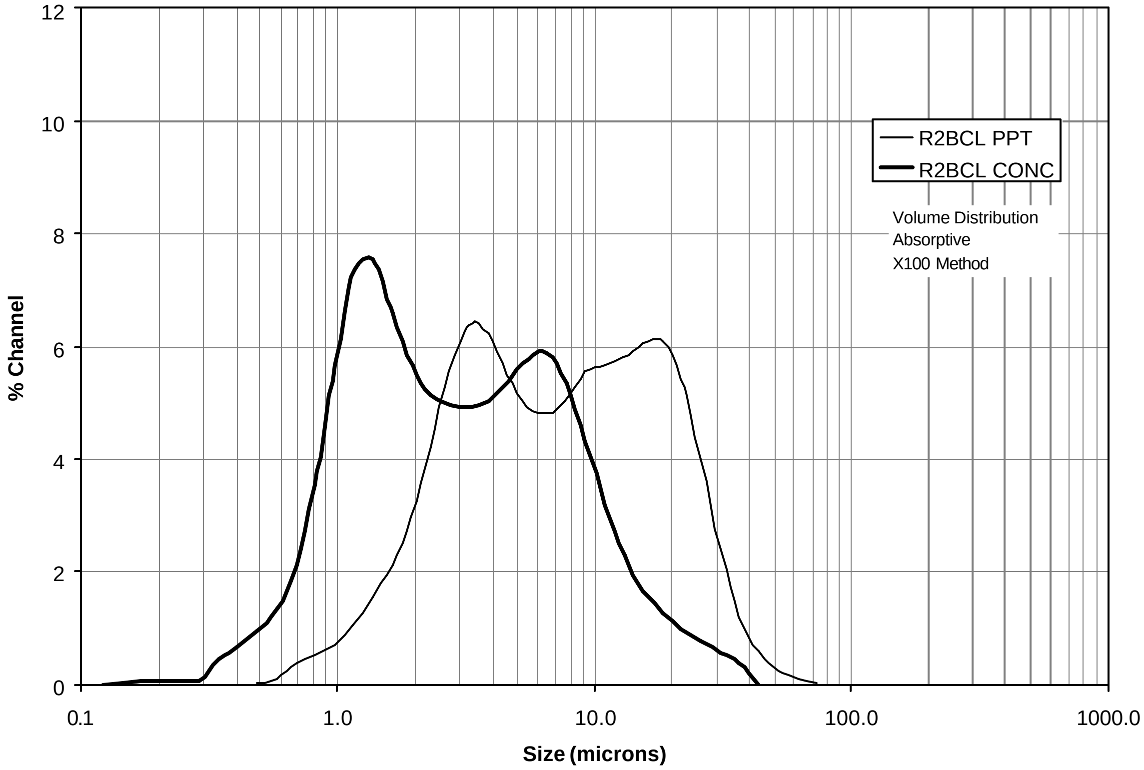


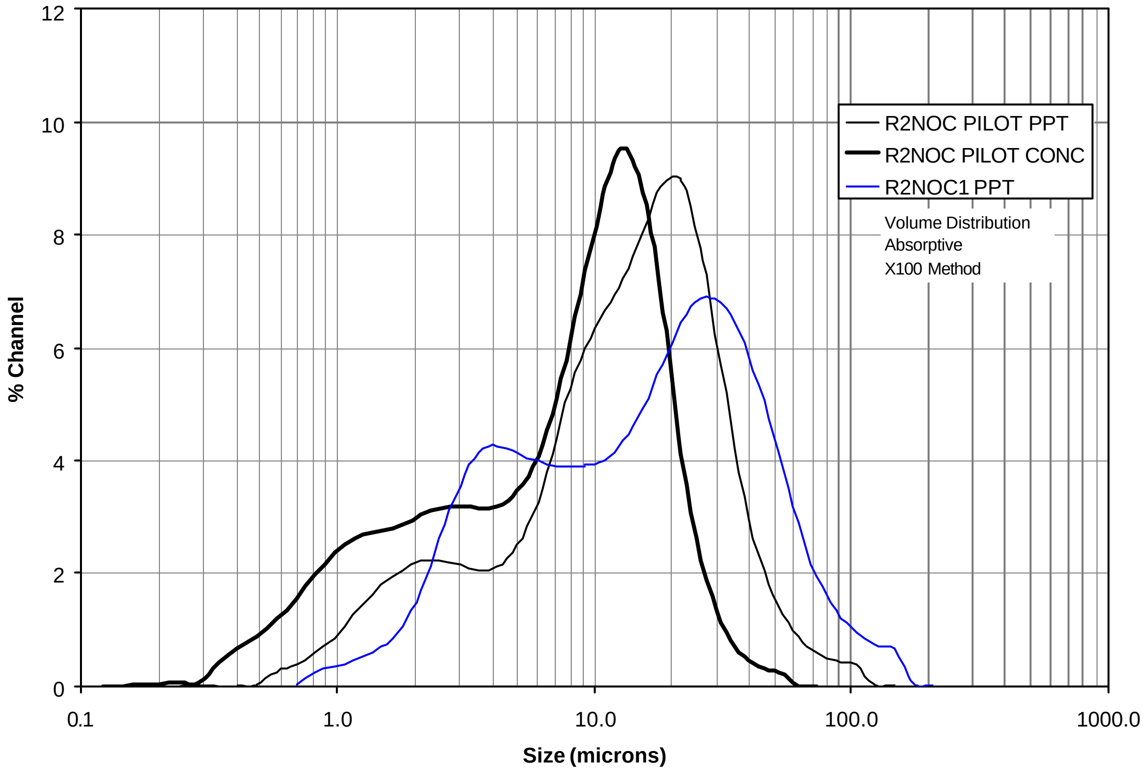

% Channel
Size (microns)
0
2
4
6
8
10
12
0.1
1.0
10.0
100.0
R1BC CONC
R2BCL CONC
R2NOC PILOT CONC
R2NOC2 CONC
Volume Distribution
Reflective
X100 Method
Numbers by curves
indicate relative order
of filterability
1
2
3
4

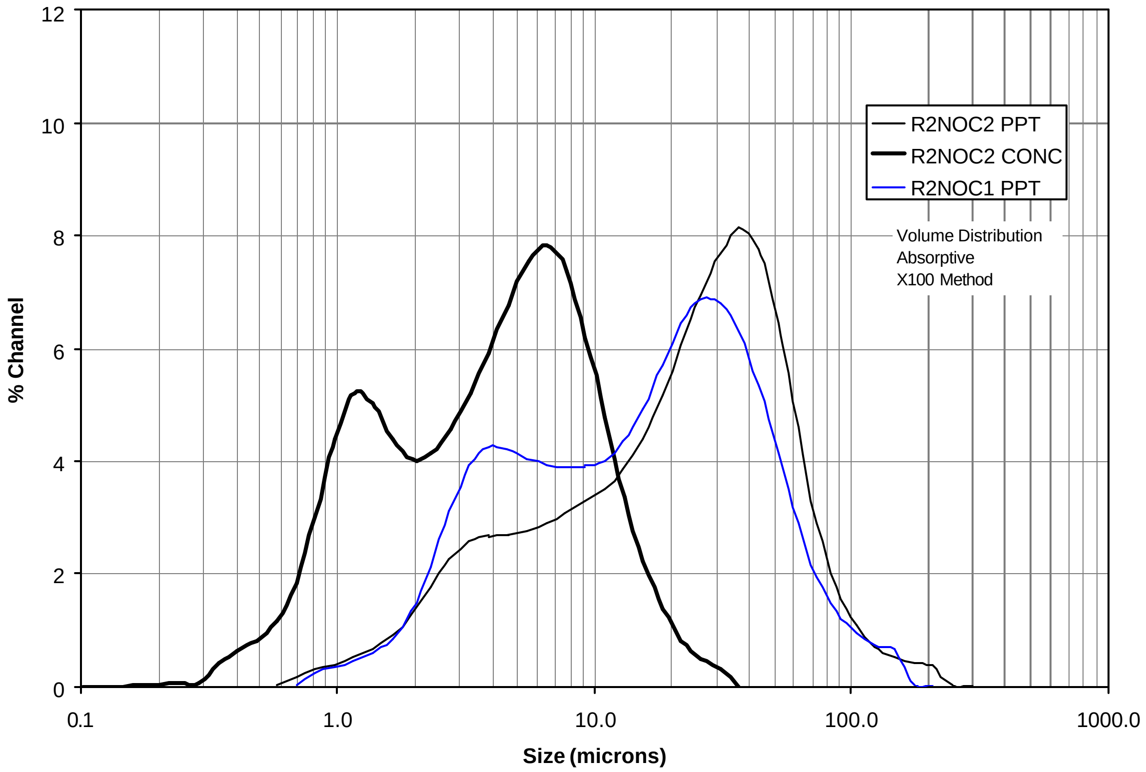

% Channel
Size (microns)
0
10
20
30
40
50
60
1.0
10.0
100.0
R1BC CONC
R2BCL CONC
R2NOC PILOT CONC
R2NOC2 CONC
Volume Distribution
Absorptive
SRA150 Method
Numbers by curves
indicate relative
order of filterability
1
2
3
4


34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
% Channel
1.0
10.0
100.0
1000.0
Size (microns)
1
2
3
4
R1BC CONC
R2BCL CONC
R2NOC PILOT CONC
R2NOC2 CONC
Volume Distribution
Reflective
SRA150 Method
Numbers by curves
indicate relative
order of filterability

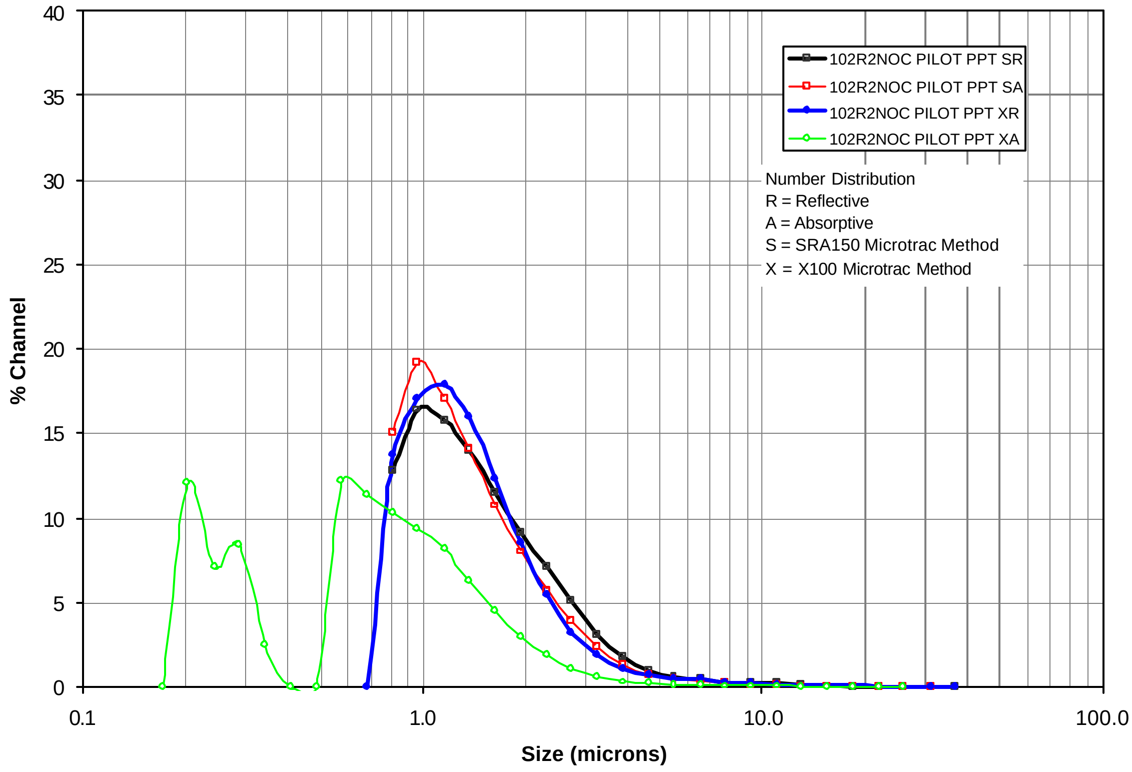

12
10
8
6
4
2
0
% Channel
0.1
1.0
10.0
100.0
1000.0
Size (microns)
R1BC PPT
R1BC CONC
Volume Distribution
Absorptive
X100 Method

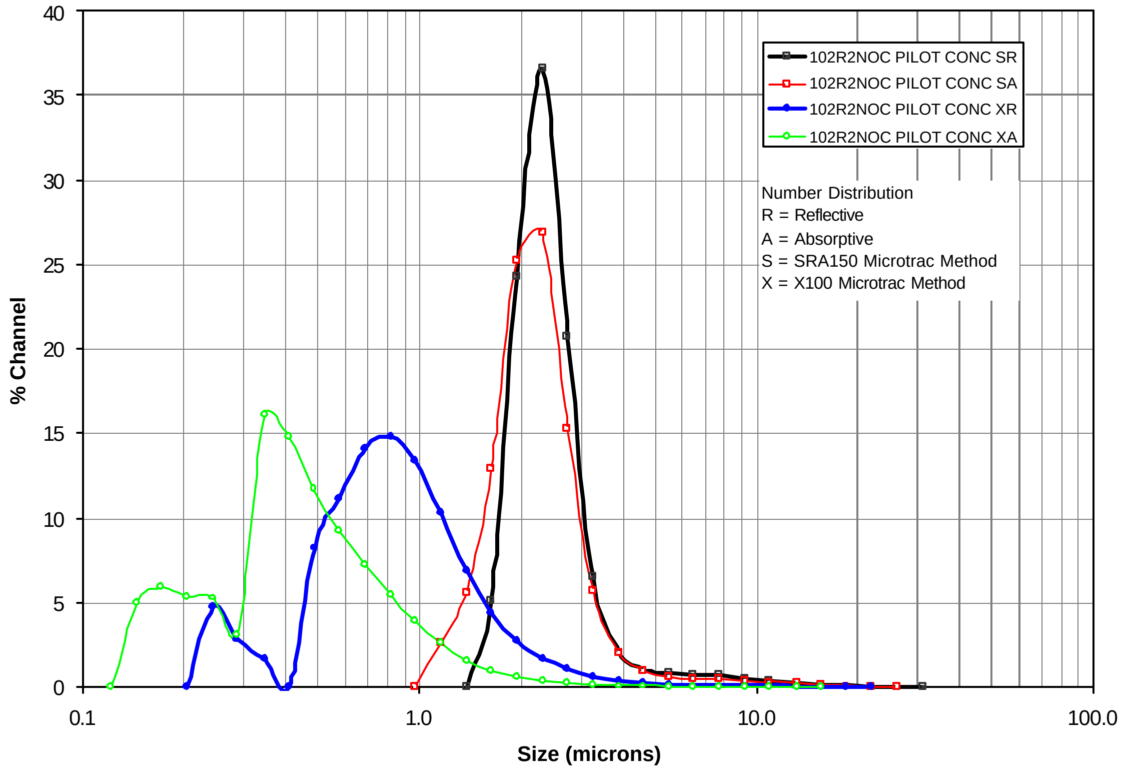

12
10
8
6
4
2
0
% Channel
0.1
1.0
10.0
100.0
1000.0
Size (microns)
R2BCL PPT
R2BCL CONC
Volume Distribution
Absorptive
X100 Method

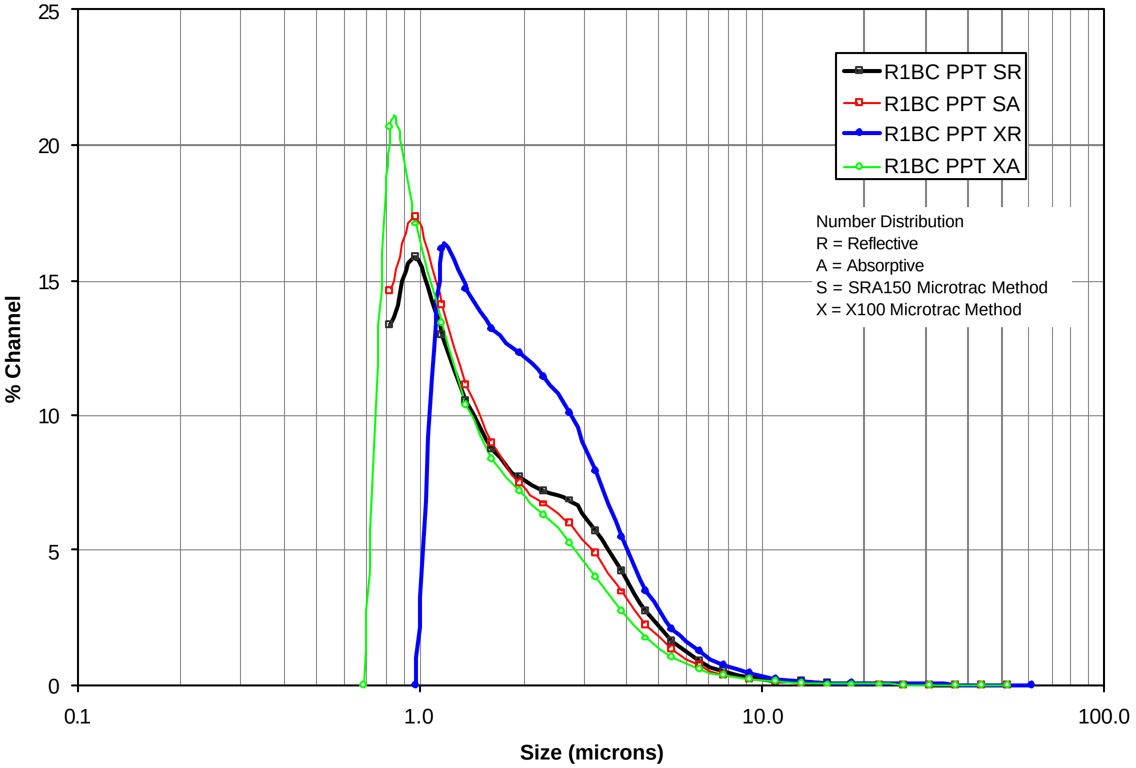

12
10
8
6
4
2
0
% Channel
0.1
1.0
10.0
100.0
1000.0
Size (microns)
R2NOC PILOT PPT
R2NOC PILOT CONC
R2NOC1 PPT
Volume Distribution
Absorptive
X100 Method

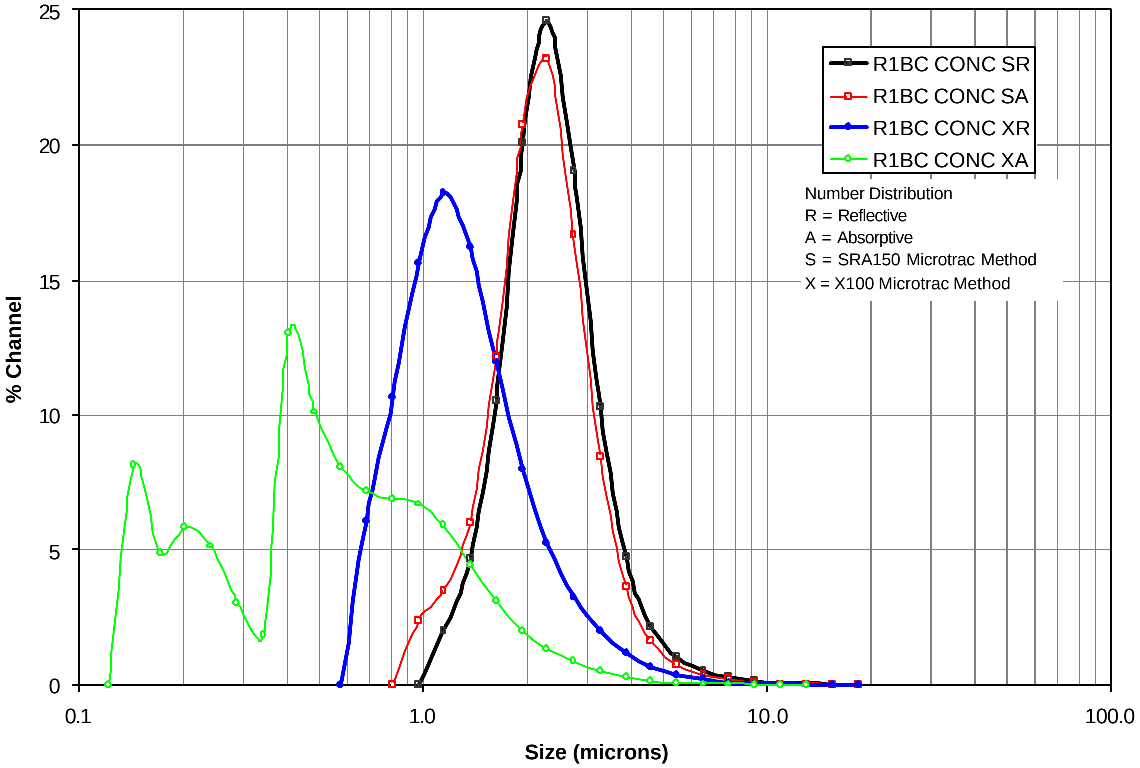

12
10
8
6
4
2
0
% Channel
0.1
1.0
10.0
100.0
1000.0
Size (microns)
R2NOC2 PPT
R2NOC2 CONC
R2NOC1 PPT
Volume Distribution
Absorptive
X100 Method

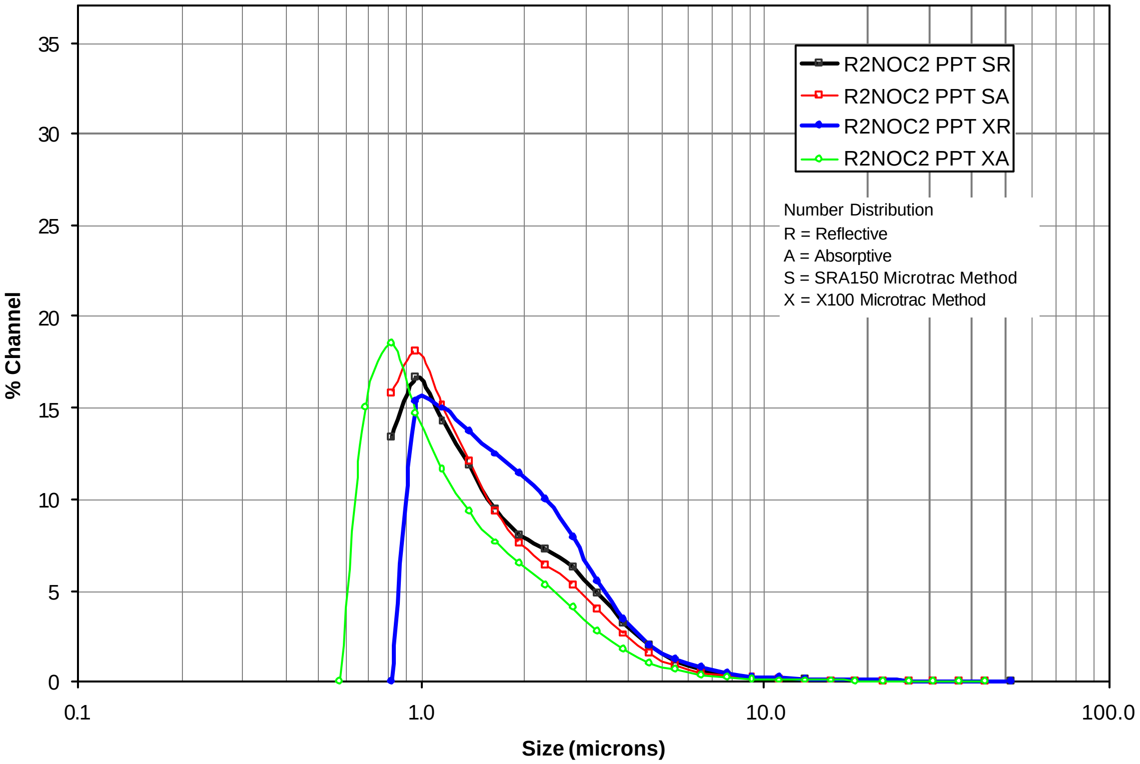
Number Distributions

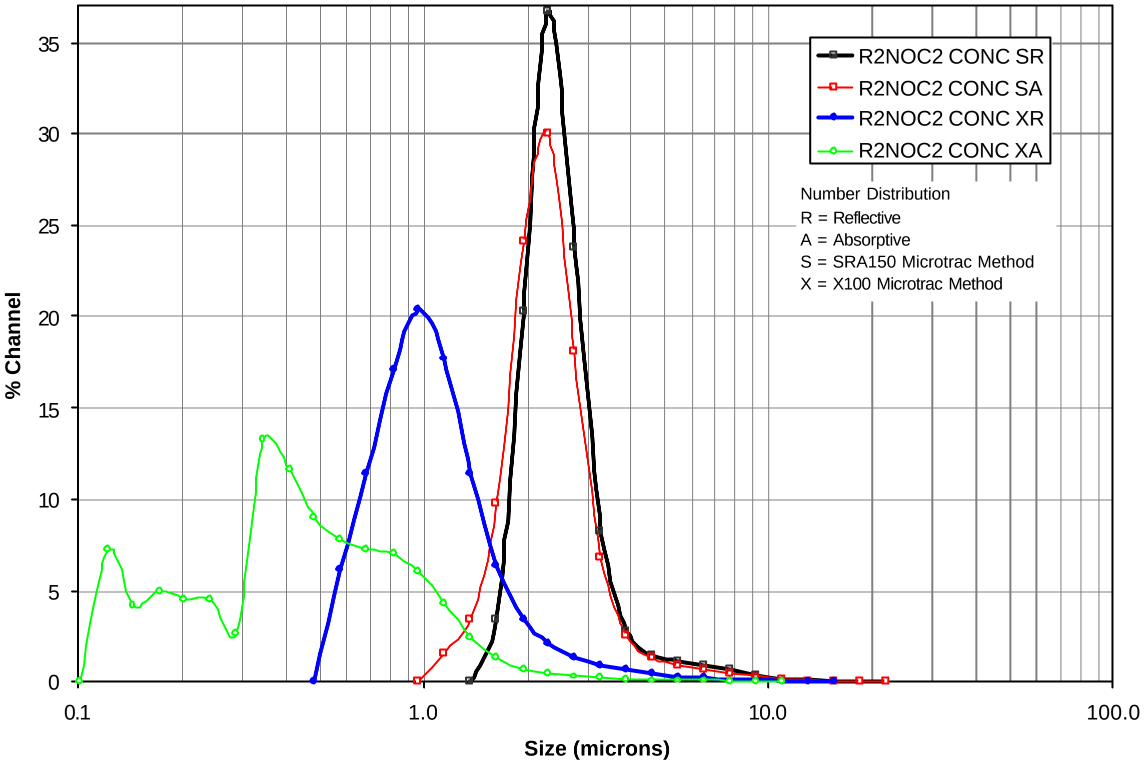


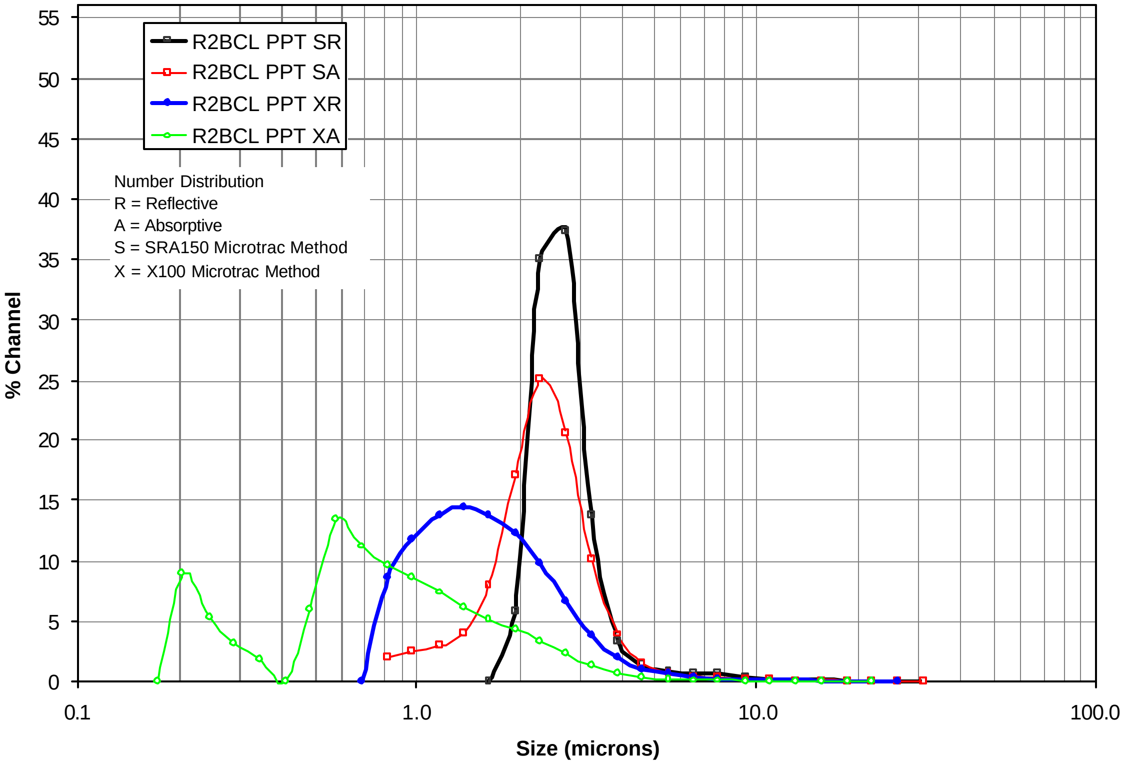


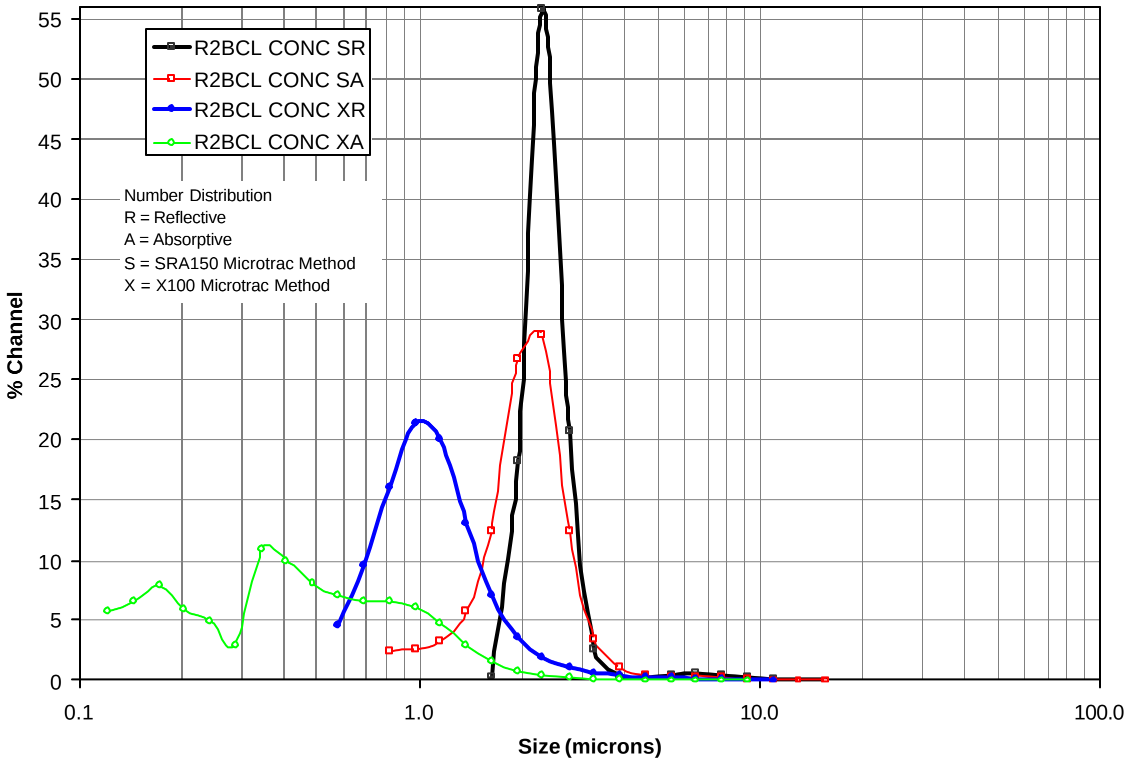

25
20
15
10
5
0
% Channel
0.1
1.0
10.0
100.0
Size (microns)
R1BC PPT SR
R1BC PPT SA
R1BC PPT XR
R1BC PPT XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method

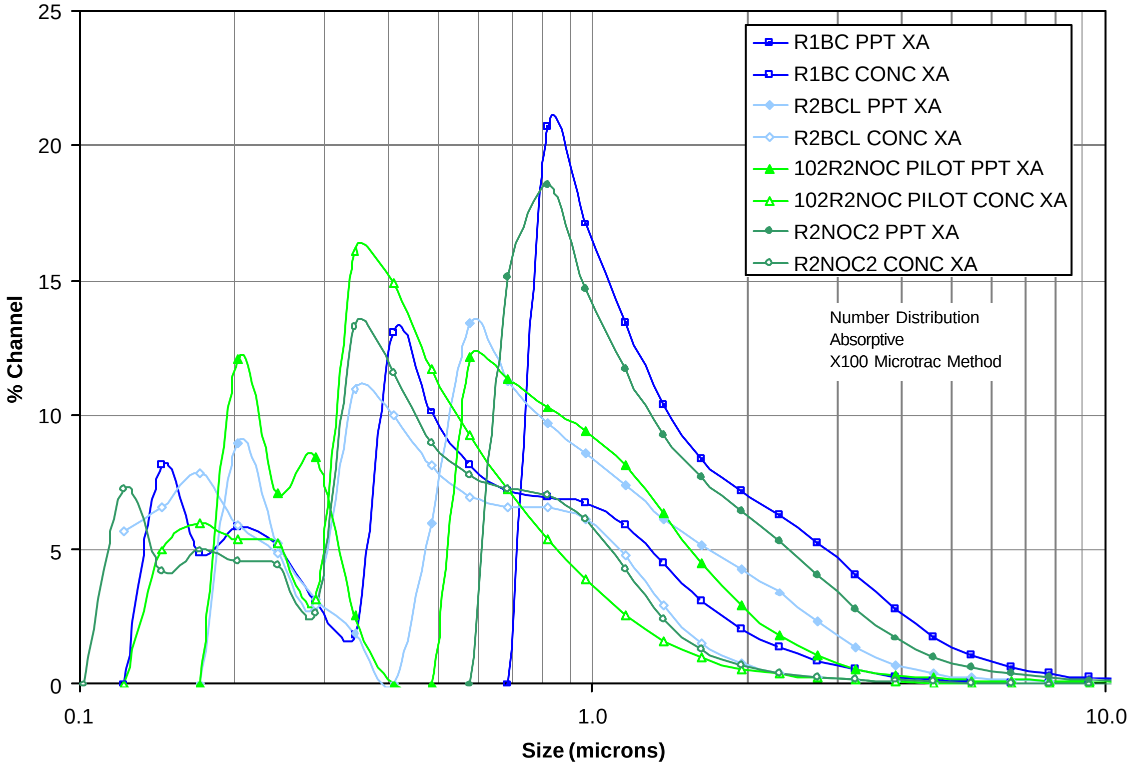

25
20
15
10
5
0
% Channel
0.1
1.0
10.0
100.0
Size (microns)
R1BC CONC SR
R1BC CONC SA
R1BC CONC XR
R1BC CONC XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method

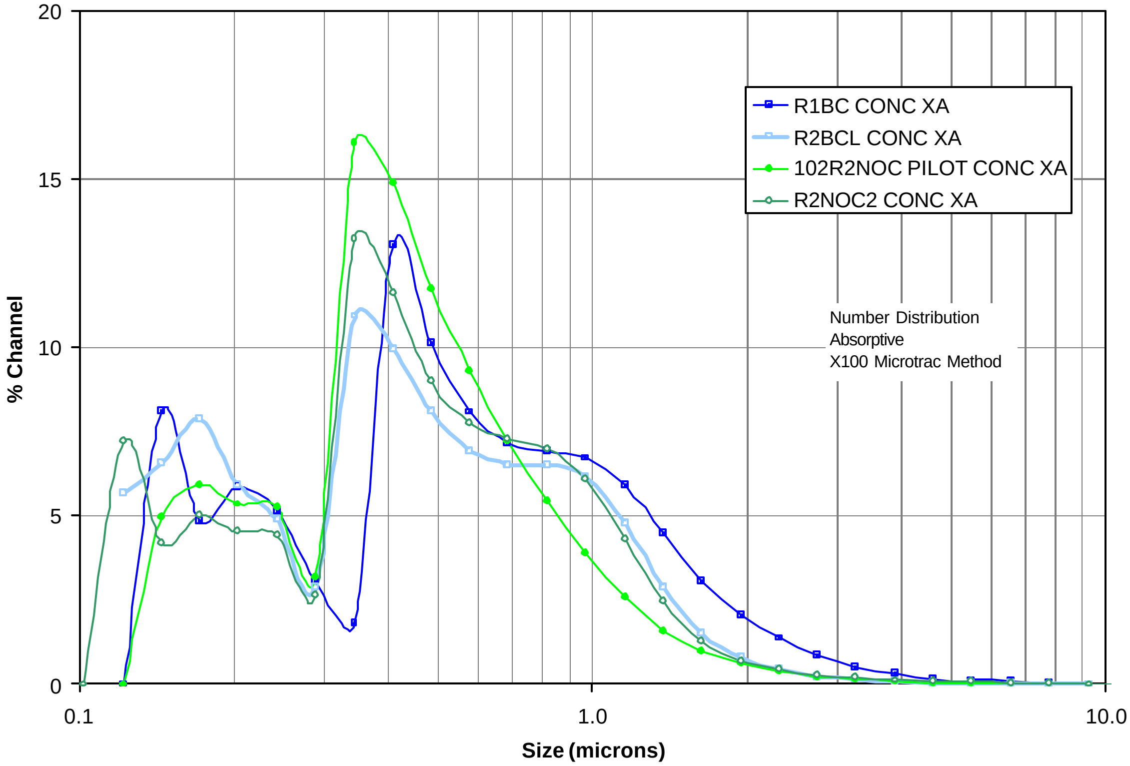

R2NOC2 PPT SR
R2NOC2 PPT SA
R2NOC2 PPT XR
R2NOC2 PPT XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method
% Channel
0
5
10
15
20
25
30
35
0.1
1.0
10.0
100.0
Size (microns)


R2NOC2 CONC SR
R2NOC2 CONC SA
R2NOC2 CONC XR
R2NOC2 CONC XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method
% Channel
0
5
10
15
20
25
30
35
0.1
1.0
10.0
100.0
Size (microns)


R2BCL PPT SR
R2BCL PPT SA
R2BCL PPT XR
R2BCL PPT XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method
% Channel
0
5
10
15
20
25
30
35
40
45
50
55
0.1
1.0
10.0
100.0
Size (microns)


R2BCL CONC SR
R2BCL CONC SA
R2BCL CONC XR
R2BCL CONC XA
Number Distribution
R = Reflective
A = Absorptive
S = SRA150 Microtrac Method
X = X100 Microtrac Method
% Channel
0
5
10
15
20
25
30
35
40
45
50
55
0.1
1.0
10.0
100.0
Size (microns)


25
20
15
10
5
0
% Channel
0.1
1.0
10.0
Size (microns)
R1BC PPT XA
R1BC CONC XA
R2BCL PPT XA
R2BCL CONC XA
102R2NOC PILOT PPT XA
102R2NOC PILOT CONC XA
R2NOC2 PPT XA
R2NOC2 CONC XA
Number Distribution
Absorptive
X100 Microtrac Method


20
15
10
5
0
% Channel
0.1
1.0
10.0
Size (microns)
R1BC CONC XA
R2BCL CONC XA
102R2NOC PILOT CONC XA
R2NOC2 CONC XA
Number Distribution
Absorptive
X100 Microtrac Method

APPENDIX A8. EMAIL FROM TOWNSON TO ZAMECNIK

"Townson, Paul S."
<pstownso@bechtel.com>

To: "'jack.zamecnik@srs.gov'" <jack.zamecnik@srs .gov>
cc:
Subject: RE: Questions about Sr/TRU precipitation recipe in AN102 Cold

04/22/2002 11:05 AM

Jack,

Looks like an old set of numbers slipped through the net! Well spotted! I have an excel spreadsheet which does all the calcs for me and it takes into account all dilutions to end up with the correct concentrations. The output from it is shown below: The first five lines show the input data, the next four the targets, the next four the actual concentrations achieved and the last four the amount of each reagent to add.

	Sample:	241-AN-102	
	Starting Na: (M)	7	
DATA INPUT	Starting OH: (M)	0	
	NaMnO4 strength	1	
	Strontium strength	1	
	Sodium hydroxide strength (M)	19	
	Target Na after water addition (M)	6.0	
SOLVER	Target final OH (M)	0.9	Can only be changed in the macro itself
TARGETS	Target final Sr (M)	0.075	
	Target final MnO4(M)	0.05	
	Final Na (M)	5.864	
ACTUAL	Final OH (M)	0.900	
ACHIEVED	Final Sr (M)	0.075	
	Final MnO4 (M)	0.050	
	Water	0.167	l per litre original sample
DATA OUTPUT	NaOH	0.067	l per litre original sample
	Sr(NO3)2 addn	0.106	l per litre original sample
	NaMnO4 addn	0.071	l per litre original sample

-----Original Message-----
From: Jack Zamecnik
Sent: Monday, April 22, 2002 7:37 AM
To: Townson, Paul S.
Cc: Michael Poirier
Subject: Questions about Sr/TRU precipitation recipe in AN102 Cold CUF Test Spec

Paul:

The attached document shows my analysis of the recipe for the Sr/TRU precipitation.

It appears that the water added to bring the Na molarity to 6M was not accounted for in the subsequent calculations.

Could you please look this over and let me know if my analysis is correct? Either way, please let me know how to proceed.

Thanks,

Jack Zamecnik, Ph. D.
Westinghouse Savannah River Co.
Aiken, SC 29808