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Comparison Testing of Multiple Spherical Resorcinol-Formaldehyde Resins for the River Protection Project—Waste Treatment Plant

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for W. Tamosaitis
ACCEPTED FOR
WTP PROJECT USE

November 2006

Test Specification: 24590-PTF-TSP-RT-04-0001, Rev. 0
Test Plan: TP-RPP-WTP-368, Rev. 0
Test Exceptions: 24590-PTF-TEF-RT-04-00001
24590-PTF-TEF-RT-04-00031
24590-PTF-TEF-RT-05-00005
24590-PTF-TEF-RT-05-00007
24590-PTF-TEF-RT-06-00001
R&T Focus Area: Pretreatment
Test Scoping Statement(s): A-225

Battelle—Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-04-0001, Rev. 0 and Test Plan TP-RPP-WTP-368, Rev. 0 and Test Exceptions 24590-PTF-TEF-RT-04-00001, 24590-PTF-TEF-RT-04-00031, 24590-PTF-TEF-RT-05-00005, 24590-PTF-TEF-RT-05-00007, and 24590-PTF-TEF-RT-06-00001. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

Gordon H. Beeman, Manager
WTP R&T Support Project

11/2/06
Date

History Sheet

Rev	Date	Reason for revision	Revised by
0	September 2006	New Document	SK Fiskum
1	October 2006	Revision 1 corrects transcriptional errors. Figure 5.19 was updated to correct for offset in the plotted data. Table 5.18 was updated to show the residual Cs concentration on the eluted SL-644 resin following water rinse on the H-form mass basis instead of the Na-form mass basis. Only 2 data entries were affected: Cycle 1 residual Cs changed from 0.749 to 0.959 μg/g; Cycle 2 residual Cs changed from 0.530 to 0.679 μg/g. Table 5.19 residual Cs concentration on SL-644 (AP-101 processing) was updated with change from 0.75 μg Cs / g Na-form resin to 0.96 μg Cs / g H-form resin.	SK Fiskum

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Terms and Abbreviations

AP-101	AP-101 tank waste simulant diluted to 5 M Na
APEL	Advanced Process Engineering Laboratory
ASO	Analytical Support Operations
ASR	analytical services request
ASTM	American Society for Testing and Materials
AV	apparatus volume
AZ-102	AZ-102 tank waste simulant concentrated to 5 M Na
BNI	Bechtel National, Inc.
BS	blank spike
BSC	Boulder Scientific Corporation
BT	breakthrough
BV	bed volume
DF	decontamination factor
DI	deionized (water)
DO	dissolved oxygen
DOE	U.S. Department of Energy
EDI	water rinse following elution
EQL	estimated quantitation limit
F	furnace (method)
FD	feed displacement
FDI	water rinse following feed displacement
FMI	Fluid Metering, Inc., Syosset, NY
GEA	gamma energy analysis
HLW	high-level waste
HS	headspace (fluid)
IBC	IBC Advanced Technologies, Inc., American Fork, Utah
IC	ion chromatography
ICN	interim change notice
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
IDL	instrument detection limit
L/D	length-to-diameter ratio

LAW	low-activity waste
MB	Microbeads
LCS	laboratory control sample
MDL	method detection limit
MS	matrix spike
NA	not applicable
ND	not detected
NIST	National Institute of Standards and Technology
NM	not measured
NR	not required
ORP	Office of River Protection (DOE)
PB	preparation blank
PNWD	Battelle—Pacific Northwest Division
PSD	particle-size distribution
QA	quality assurance
QC	quality control
QAPjP	Quality Assurance Project Plan
RF	resorcinol-formaldehyde
RPD	relative percent difference
RPP-WTP	River Protection Project-Waste Treatment and Immobilization Plant
RPL	Radiochemical Processing Laboratory (PNWD facility)
R&T	research and technology
RTD	resistance temperature detector
RV	resin volume
SRNL	Savannah River National Laboratory
SRTC	Savannah River Technology Center
TBC	Tensile Bolt Cloth
TIC	total inorganic carbon
TOC	total organic carbon
TSS	technical scoping statement
WTPSP	Waste Treatment Plant Support Project

Terms of Measurement

μCi	microcuries
μg	micrograms
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed, dimensionless
F-factor	ratio of dry resin mass to wet resin mass, dimensionless
g	gram
h	hour
meq	milliequivalents
mL	milliliter
psi	pounds per square inch

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Testing Summary

The Department of Energy (DOE) has directed Bechtel National, Inc. (BNI) to evaluate the alternate Cs ion exchanger, spherical resorcinol-formaldehyde (RF) for use in the River Protection Project-Waste Treatment Plant (RPP-WTP).[(a)] Preliminary testing with spherical RF under technical scoping statement (TSS) A-222[(b)] indicated it had adequate capacity, selectivity, and kinetics to perform in the plant according to the flowsheet needs. It appeared to have superior elutability and hydraulic performance compared to the existing alternatives: ground gel RF and SuperLig® 644 (SL-644).[(c)] Additional testing was needed to study the effects of spherical RF formulation and curing on Cs ion exchange performance. Furthermore, because the spherical RF had historically been produced in bench-scale quantities; testing was also needed to evaluate impacts associated with large-scale production.

Battelle—Pacific Northwest Division (PNWD) was contracted to evaluate different formulations of spherical RF resins and provide data supporting WTP's selection of one spherical RF formulation. Work was conducted under contract number 24590-101-TSA-W000-00004 satisfying the needs defined in TSS A-225[(d)] for the evaluation of the impact of manufacturer variation on RF resin performance.

Objectives

The primary test objective was to provide data supporting WTP's selection of one formulation of spherical RF resin for scale-up production and plant application. This objective was implemented through sub-objectives, including:

- determine Cs loading and elution behavior as a function of spherical resin formulation
- compare RF resin performance data with SL-644 resin performance data
- obtain performance data to support refinement of purchase specifications for RF resin
- provide performance data using RF resin from the scale-up vendor, and scale-up production

These test objectives are further discussed in Table S.1.

(a) R Peterson, H Babad, L Bray, J Carlson, F Dunn, A Pajunen, I Papp, and J Watson. 2002. *WTP Pretreatment Alternative Resin Selection,* 24590-PTF-RPT-RT-02-001, Rev. 0, Bechtel National, Inc., Richland, WA.

(b) S Barnes, R Roosa, and R Peterson. 2003. *Research and Technology Plan,* 24590-WTP-PL-RT-01-002, Rev. 2, Bechtel National, Inc., Richland, WA.

(c) SL-644 is solely available through IBC Advanced Technologies, Inc., American Fork, UT.

(d) M. Thorson. 2002. *Alternative Ion Exchange Resin Supplemental Research and Technology Plan – Case 20,* 24590-PTF-PL-RT-02-002, Rev. 0, Bechtel National, Inc., Richland, WA.

Table S.1. Test Objectives

Test Objective	Objective Met?	Discussion
Provide data supporting WTP's selection of one formulation of spherical RF resin for scale-up production and plant application	Yes	This test objective was met by fulfilling the next five sub-objectives. PNWD tested 26 spherical RF resins provided by the developmental vendor and the scale-up vendor.
Determine Cs load and elution behavior as a function of spherical resin formulation as well as a variety of physical properties	Yes	Fifty single-cycle and/or multi-cycle load and elution tests were conducted on 26 different RF resins. Shrink-swell and bed density data were determined, and in selected cases, particle morphology and size were determined. Test results are summarized in Sections 5.0 through 14.0.
Compare RF resin performance data with SL-644 resin performance data	Yes	Resin comparison included column loading and elution performance as well as oxygen consumption and post-oxygenated effects on loading and elution profiles. The comparative column-performance results are presented in Figure S.3 and Sections 5.0, 6.0, 14.0, and 16.0.
Obtain performance data to support refinement of purchase specifications for RF resin	Yes	Purchase specification performance data were derived from the column loading and elution performance results, physical property results (skeletal density, morphology, and particle size distribution).
Provide performance data using RF resin from the scale-up vendor, and scale-up production	Yes	Physical-property and load and elution test results were obtained from scale-up resin batches tested under Wave 3, 3a, 4a, and 4b. Wave 3 tested resins prepared in 50-gal and 75-gal batches and Waves 4a and 4b tested resins prepared in ~70-gal production batches. (A 70-gal production batch is equivalent to ~100 gal Na-form pretreated resin.) Cesium loading data are summarized in Figure S.2. Additional resin properties are provided in Sections 11.0 to 14.0.

Test Exceptions

Specific test details were modified in Test Exceptions 24590-PTF-TEF-RT-04-00001, 24590-PTF-TEF-RT-04-00031, 24590-PTF-TEF-RT-05-00005, 24590-PTF-TEF-RT-05-00007, 24590-PTF-TEF-RT-06-00001. Table S.2 summarizes the test exceptions to the test plan and provides a discussion of the impacts on the tests.

Table S.2. Test Exceptions

Test Exception ID	Test Exceptions	Discussion
24590-PTF-TEF-RT-04-00001	Include an extra single-cycle test (Wave 1a) on two new resins of smaller particle diameter than the Wave 1 resins. Report the load cycle profile using AP-101 simulant and the post-eluted column Cs content.	The additional testing was implemented.
	Pretreat resins (subsequent to Wave 1 test resins where testing is already completed) according to the standard protocol P1-RF. Complete resin pretreatment before physical property measurements and column performance testing.	The more extended pretreatment implemented in compliance with Protocol P1-RF resulted in a relaxed H-form bead that was 20 to 30% larger than the as-received form. Comparisons between Wave 1 physical property testing and first cycle load and elute testing needs to take the different pretreatment operations into consideration.
	Limit formaldehyde testing to the Wave 1 test resins only.	No further information on the formaldehyde concentrations in solution was required by the project.
	Change the interval for determining constant resin mass during F-factor measures from 2 h to 7 h.	This practice was standardized between Savannah River National Laboratory (SRNL) and PNWD testing and was consistent with Protocol P1-RF.
	Allow the alternative use of a pH meter and electrode or pH paper to determine effluent solution pH during regeneration test and optimization.	Use of the pH electrode allowed for higher accuracy, as appropriate for this test.
24590-PTF-TEF-RT-04-00031	Allow the option of conducting the analysis for ^{137}Cs in the effluent and eluate using liquid scintillation counting instead of gamma energy analysis (GEA).	The added flexibility in counting options allowed for rapid data analysis while the GEA detector systems were being repaired.
	Allow flexibility in process parameters for the oxygenated regeneration test through the SL-644 media. Continue follow-on processing as indicated in the test plan provided the system does not pressurize; if pressure continues to be problematic, discuss the path forward with the research and technology (R&T) lead.	Oxygen-saturated regeneration solution was to be processed for 510 BVs through the resin beds. The test on SL-644 had to stop early because of excessive (>10 psi) pressure drop through the resin bed. Processing with fresh regeneration solution continued the high pressure drop. Changing the feed to AP-101 simulant slowly resolved the problem, and processing continued per the test plan.
24590-PTF-TEF-RT-05-00005	Add additional ion exchange resin testing Waves 2a, 2b, and 3a.	Additional ion exchange loading and elution testing was conduced as indicated. The changes allowed more extensive evaluation of Cs loading effects based on permutations in resin manufacturing conditions.
	Delete repeat cycle testing from Wave 1 activity and add two new resins from the developmental vendor for testing.	
	Add additional ion exchange system for concurrent testing.	
	Eliminate most of the AZ-102 simulant testing and replace testing with AP-101 simulant.	More testing was conducted with AP-101 simulant because it was thought to provide a more challenging test matrix than the AZ-102 simulant.

Table S.2 (Contd)

Test Exception ID	Test Exceptions	Discussion
24590-PTF-TEF-RT-05-00007	Expand the allowed regeneration sodium hydroxide molarity from 0.5 M to a range of 0.25 M to 1.25M. Expand process volume from 5 to 10 BVs to a range of 1 to 10 BVs. Specific direction on molarity and volume to be used for any given test will be provided by the R&T lead.	Changes were applied to Wave 3 and on because other testing was already completed. Regeneration solution was maintained at 0.5 M molarity and 6 BVs for A-225 testing per concurrence of the R&T lead.
	Change eluant nitric acid molarity from 0.5 M to a range of 0.1 to 0.6 M. Expand volume range from 12 to 40 BVs to a range of 12 to 105 BVs for elution. Specific direction on molarity and volume to be used for any given test was to be provided by the R&T lead.	Changes were applied to Wave 3 and on because other testing was already completed. Wave 3 incorporated the use of 0.25 M and 0.4 M nitric acid concentrations for elution. Wave 3a, 4a, and 4b used 11 to 29 BVs of 0.5 M nitric acid.
	Conduct Wave 3a testing in conjunction with Wave 4 testing. (Note, Wave 3a tested the spent resin from the A-215 24-inch column test at SRNL.)	Wave 3a testing was conducted concurrently with Wave 4a testing and per the R&T lead's direction. Wave 3a test resin was processed in two resin beds; one incorporated a stop-flow condition during load step and the other bed incorporated upflow elution.
	Conduct a resin speed of neutralization test to support selection of a flowrate for upflow regeneration in the hydraulic testing in A-224. Delineated steps may be changed with concurrence of the R&T lead.	The resin neutralization test was conducted as written through the titration phase. The remainder of the test was conducted under the direction of the R&T lead, which was different than written in the test exception. After testing, it was decided that the test design was not especially helpful toward the end goal.
24590-PTF-TEF-RT-06-00001	Expand the resin pretreatment options per the direction of the R&T lead.	The WTP is looking to reduce Na consumption and waste generation in the plant. Selected pretreatment options were evaluated to minimize the Na usage.
	Split test Wave 4 into two different test waves, Wave 4a and Wave 4b. Conduct Wave 4b testing at a later date (from Wave 4a) and allow up to six load/elute tests.	Wave 4a was conducted concurrent with Wave 3a testing, 1/06. The split of Wave 4 (into 4a and 4b) allowed for additional scale-up resin batches to be tested with results generated in a timely manner to the project. Wave 4b was conducted 5/06 with two test resins. One resin tested AP-101 and AZ-102; the other resin tested AP-101 and alternate pretreatment processing.
	Include additional test scope in Wave 4b: assessment of minimal pretreatment conditions and high (45°C) temperature load conditions.	The change allowed assessment in a side-by-side test of minimal pretreatment options and their corresponding effects on Cs loading performance. The temperature effect could not be assessed because the jacketed columns received from the vendor were defective.

Results and Performance Against Success Criteria

The test success criteria are listed in Table S.3.

Table S.3. Test Success Criteria

Success Criteria	Explanation
Data supplied supporting selection of a RF formulation most likely to meet plant requirements	Load profiles were obtained for resin formulations submitted for testing. Additionally, most ion exchange test resins were evaluated for Cs elution profile and residual Cs loading, post-elution (consistent with the test plan). Shrink-swell characteristics were obtained during pretreatment and ion exchange processing. Particle size distribution, particle density, bed density, and micrographs were obtained for a selected (as determined by the R&T lead) subset of the resins. The relative Cs load performances of the resins allowed the R&T lead to select appropriate variations in processing conditions of spherical RF. Final scale-up production conditions were determined from the various scoping tests.
Data supplied supporting confirmation that the spherical RF resin can be successfully prepared at scale-up proportions (100-gallon production batches).	RF resin production was scaled up to 50-gal to 75-gal production lots (a 64-gal production lot of H-form resin expands to 100-gal Na-form resin). Subsamples were tested in a congruent manner to the previous tests to determine efficacy of the production scale-up. Collected data included Cs loading and elution profiles, residual Cs concentration, shrink-swell characteristics, particle density, particle-size distribution (PSD), bed density, and micrographs. The resin production was shown to be successfully scaled up to the 100-gal size (Na-form resin) lot production.

Micrographs showed the RF resins were uniformly spherical with few broken pieces. A typical sample is shown in Figure S.1. Micrographs of resin bead cross-sections revealed a visible core structure in the first test wave. Subsequent test resins, prepared from different processing conditions by the manufacturer, appeared uniform in structure throughout the bead cross-section.

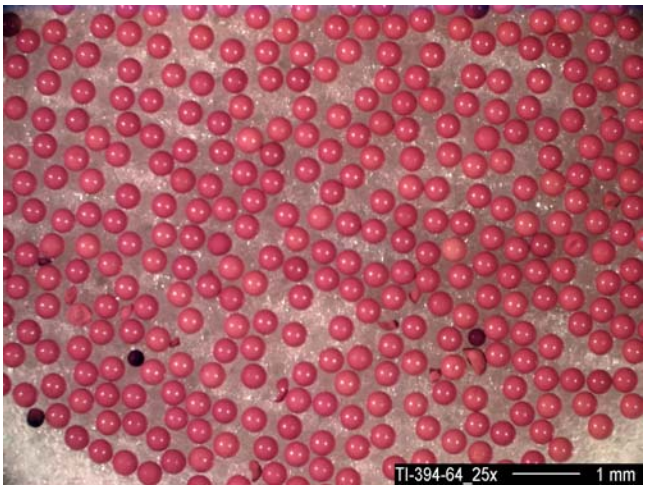

Figure S.1. Micrograph of Spherical RF, Test Wave 4a, MB 5J-370/686 (72-gal Production Batch)

PSDs were generally very tight, consistent with observations in the micrographs. Particle densities did not vary much from 1.62 g/mL for Na-form resin and 1.48 g/mL for H-form resin. Shrink-swell characteristics did not vary significantly. Generally, the resins contracted 20% on conversion from the Na-form to the H-form. The Microbeads (MB) resin bed densities were slightly lower than the Boulder Scientific Corp. (BSC) product resin bed densities, 0.366 vs. 0.416 g H-form resin per mL settled H-form resin volume, respectively.

Cesium loading profile results from the four scale-up resin productions are summarized in Figure S.2. Comparison of the AP-101 simulant (5.9 mg/L Cs) load profiles shows the scale-up product was consistent with respect to the Cs load characteristics. The AZ-102 (52 mg/L Cs) loading profile is also shown. The high-K waste (AP-101 at 0.7 M K) bounded the worst-case load performance for RF resin. The minimum WTP process design flowsheet[(a)] volumes for Envelope A and C wastes are 72-BVs processed at 2.2 BV/h by three columns in series. In the AP-101 simulant tests, the Cs effluent contract limit was reached after processing ~50 to 60 BVs through one ion exchange column at 1.5 BV/h; ~135 to 140 BVs were processed before reaching 50% breakthrough. The WTP process design flowsheet volume

(a) JW Olson, *System Description for Cesium Removal Using Ion Exchange—System CXP*, 24590-PTF-3YD-CXP-00001, 12/2001.

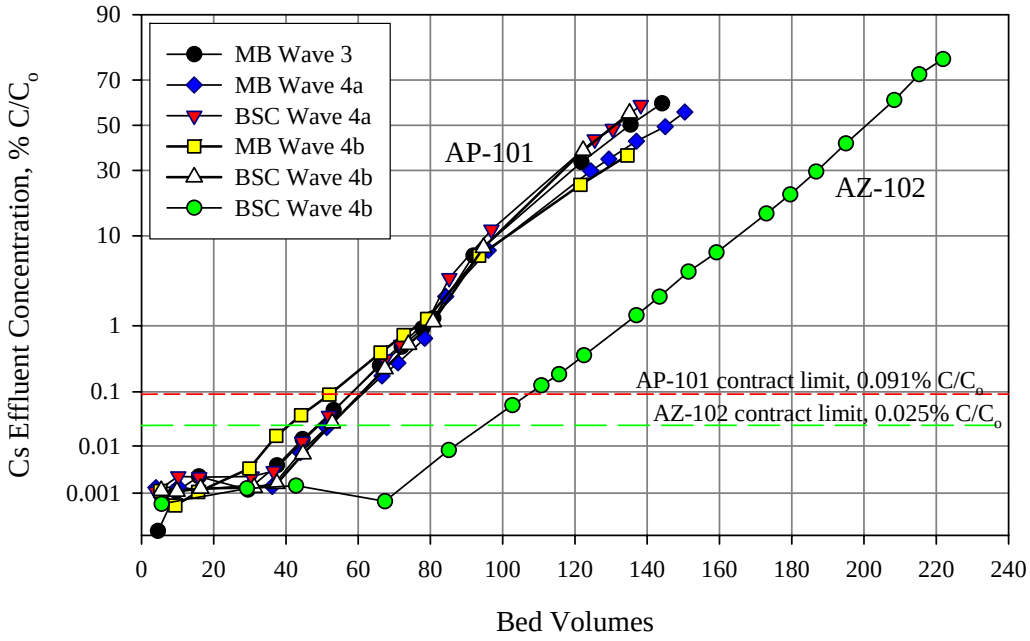

Figure S.2. Cs Ion Exchange Loading Performance with Multiple Large-Scale Production Lots in AP-101 and AZ-102 Simulant Feeds

Conditions	**AP-101**	**AZ-102**
Flowrate	1.5 BV/h (0 to 80 BVs), 3 BV/h (>80 BVs)	1.5 BV/h
Process temperature	19 to 24°C	19 to 24°C
Na concentration	4.89 M	5.18 M
K concentration	0.679 M	0.145 M
Cs concentration	5.86 mg/L	52.2 mg/L

for Envelope B waste is 18 to 43 BVs. The AZ-102 simulant test demonstrated the Cs contract limit was reached after processing 105 BVs, and the 50% breakthrough was reached after processing 200 BVs. The RF resin was found to meet plant processing flowsheet requirements for both types of simulant tank waste feeds.

Figure S.3 shows the direct comparison of the SL-644 ion exchange performance results with the spherical RF (scale-up production batches). Processing the high Cs (AZ-102simulant) waste resulted in similar performance. The Cs in the AP-101 matrix broke through the RF resin sooner than the SL-644.

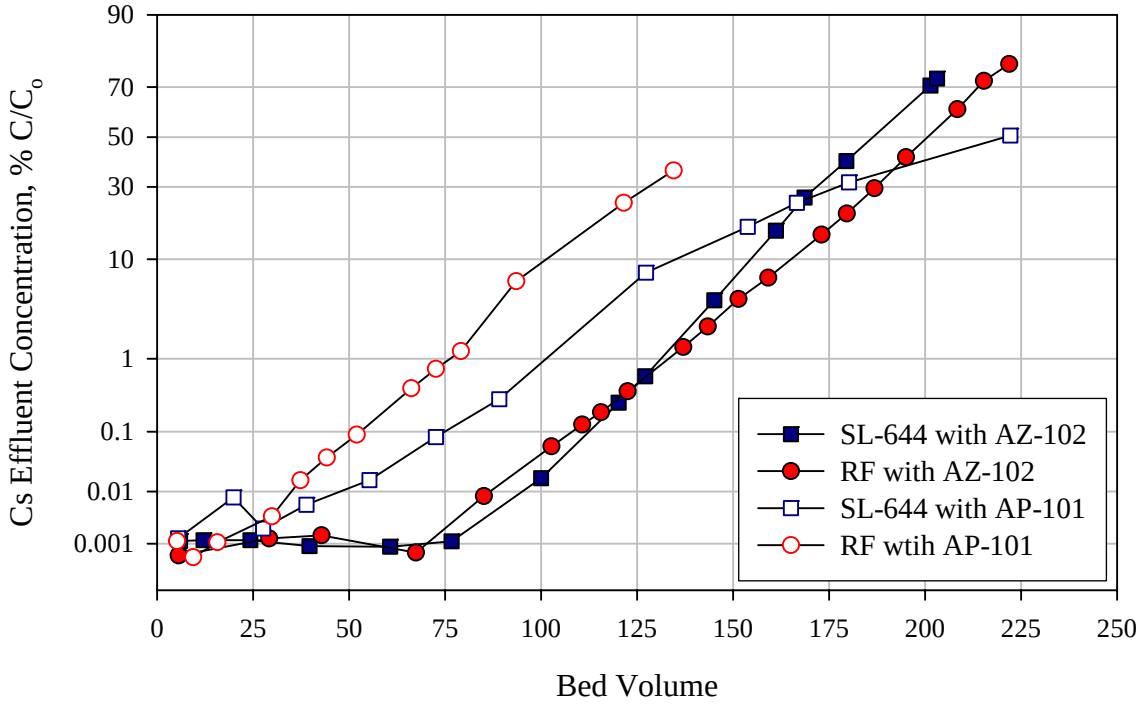

Figure S.3. Comparison of SL-644 (Lot C-01-11-05-02-35-60, 18 to 40 mesh Na-form) and Spherical RF (Lot 3380-3-0201) Cs Loading Performance Results

Quality Requirements

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization and to the approved test plan, TP-RPP-WTP-368, Rev. 0. This work was performed to the quality requirements of NQA-1-1989 Part I, Basic and Supplementary Requirements, NQA-2a-1990, Part 2.7 and DOE/RW-0333P, Rev 13, Quality Assurance Requirements and Descriptions (QARD). These quality requirements were implemented through PNWD's *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements were implemented through WTPSP's Statement of Work (WTPSP-SOW-005) with the Radiochemical Processing Laboratory (RPL) Analytical Service Operations (ASO).

PNWD addressed internal verification and validation activities by conducting an Independent Technical Review of the final data report in accordance with PNWD's procedure QA-RPP-WTP-604. This review verifies that the reported results were traceable, inferences and conclusions were soundly based, and the reported work satisfied the test plan objectives. This review procedure is part of PNWD's WTPSP Quality Assurance Requirements and Description Manual.

R&T Test Conditions

Table S.4 summarizes the various R&T test conditions and a brief discussion how the test conditions were followed.

Table S.4. R&T Test Conditions

R&T Test Condition	Were Test Condition(s) Followed?
Sub-sampling	Yes, resins were sampled using the coring technique in suitable volumes for follow-on testing. Resins were stored in the original vendor-supplied container. Wet resins were stored wet, dry resins stored dry. The water (as applicable) and head spaces were flushed with nitrogen after sampling.
Resin Pretreatment	Yes, Wave 1 pretreatment consisted of water washing the resin before loading in the column and conducting physical property testing. Wave 1b and subsequent testing utilized pretreatment steps defined in the protocol agreed to by PNWD, SRTC, and BNI (one shrink-swell cycle out of column, one shrink-swell cycle inside the column), P1-RF. Wave 4b, in part, evaluated R&T lead-directed minimal pretreatment steps.
Formaldehyde Testing	Yes, Wave 1 pretreatment process solutions were tested using formaldehyde test strips. Follow-on test waves were exempt from this testing.
Bulk Resin Characterization	Yes, resin physical properties were tested on resins selected by the R&T lead. The morphology was evaluated using optical microscopy on dry H-form resin. The shrink-swell behavior was measured as part of the column testing by noting resin bed height in all matrices: deionized water, 0.5 M HNO_3, 0.5 M NaOH, and AP-101 simulant. The bulk wet density was determined as part of column testing with reference mass based on the dry H-form resin. The H-form and Na-form PSDs were measured using a Micro TRAC S3000 Particle Size Analyzer. Particle density was determined using water pycnometry.
Simulant Preparation and Analysis	Yes, the AP-101 and AZ-102 simulants were prepared per instruction by Noah Technologies, San Antonio, TX. The AZ-102 hydroxide was 36% high. The AP-101 Cl was 20% low in the first preparation and 20% high in the second preparation. The R&T lead indicated that these variations would be acceptable for testing.
Regeneration Optimization	Yes, all processing was conducted as indicated in the test plan; the individual steps are too numerous to recount here (see Section 4.0).
RF Resin Mass Determination	Yes and No. A duplicate sample was measured for dry mass determination as indicated. The entire quantity of duplicate sample was taken to dryness instead of duplicate 0.25 to 0.50-g sub-samples. Constant mass was determined when the resin mass change was <0.5% over a 7-h period at 50°C.
RF Column Preconditioning	Yes, all steps were followed as indicated in the test plan; see Section 3.0 and 5.0 through 14.0.
SL-644 Resin Preparation	Yes, SL-644 was taken from the preconditioned stock available from SRTC, in the Na-form, in water. No pretreatment was needed (see Section 5.0).

Table S.4 (Contd)

R&T Test Condition	Were Test Condition(s) Followed?
Load and Elution Performance	Yes, the loading and elution testing was conducted according to the Process Conditions defined in the test plan, Table 3. Cesium ion exchange load and elution characteristics were tested downflow in a single-column format. Resin bed volumes (BVs) were nominally 20-mL in a 2-cm ID glass column with a nominal length-to-diameter ratio of 3 when the resin was in the Na-form. Most testing was conducted with the AP-101 simulant; additional testing was conducted with AZ-102 simulant. All feeds were spiked with ^{137}Cs tracer to allow for rapid determination of Cs concentration by GEA. Load and elution processing was conducted according to nominal plant design and throughput. The AZ-102 and AP-101 simulants were loaded at 1.5 BV/h; after processing ~80 BVs, the flow rate for AP-101 was increased to 3.0 BV/h. Elution was conducted at 1.4 BV/h with one test elution conducted in upflow. After elution and water rinse, the column assemblies (resin bed plus glassware) were counted directly by GEA to evaluate residual Cs content on the resin. The individual steps are too numerous to describe in this section; see Sections 3.0 and 5.0 through 14.0.
Oxygen Exposure	Yes, all testing was conducted according to the test plan. The SL-644 oxygen exposure had to be stopped early because of the catastrophic failure of the resin (see Section 6.0).
Eluate Metals Content	Yes, the R&T lead selected one eluate composite for analysis: Wave 3 resin 5E-370/641 eluted with 0.4 M HNO_3. See Section 11.0.
Spent Resin Cs Content Extrapolation	Yes, the spent resin Cs content was extrapolated for each elution sample taken, where Cs was actually determined at the end of an ion exchange process test, in accordance with direction from the R&T lead. Therefore, Cs content on the resin bed as a function of elution volume was reconstructed. See Sections 5.0 through 14.0.
Resin Neutralization	Yes, this test was conducted as defined in the test plan and as modified with concurrence of the R&T lead (who was present during most of the test).

Simulant Use

Because of inherent problems with obtaining and handling radioactive tank wastes, tank waste simulants were used for column testing. A simulant solution of Envelope A waste Tank AP-101 supernatant was previously developed (Russell et al. 2003) and was used for the testing described here. Additionally, the AZ-102 simulant formulation used previously for TSS A-222 (Fiskum et al. 2004), was tested to compare the results with those from prior efforts.

The use of simulants for this testing scope provided an adequate basis for resin performance comparisons. The use of actual tank wastes was not necessary to evaluate incremental improvements in resin performance.

Discrepancies and Follow-on Tests

None.

1.0 Introduction

Forty years of plutonium production at the U.S. Department of Energy (DOE) Hanford Site in southeastern Washington State has left a legacy of liquid waste generated as a byproduct of reprocessing operations. The wastes are a complex mixture composed mostly of sodium nitrate, nitrite, hydroxide, and sulfate, along with a broad spectrum of minor and trace metals, organics, and radionuclides stored in underground storage tanks. The sources and compositions of the tank wastes have been previously described (Agnew et al. 1997). The tank waste is designated as high-level waste (HLW) and requires burial in a geologic repository after solidification. Reduction in HLW volume can be realized by removing ^{137}Cs and transuranics from the large volume fraction of the non-radioactive constituents and thus allow significant savings in disposal costs.

The DOE Office of River Protection (ORP) has contracted Bechtel National Incorporated (BNI) to build a processing plant, the River Protection Project-Waste Treatment and Immobilization Plant (RPP-WTP).[(a)] The RPP-WTP will chemically separate the highly radioactive components (specifically ^{137}Cs and, in some cases, ^{90}Sr and transuranics) of the tank waste from the bulk (non-radioactive) constituents and immobilize the fractionated wastes by vitrification. An overview of the RPP-WTP unit operations has been previously described (Nash et al. 2004). The plant will produce two waste streams: a high-volume low-activity waste (LAW), that is, waste depleted of ^{137}Cs, ^{90}Sr, and transuranics, and a low-volume HLW (the ^{137}Cs, ^{90}Sr, and transuranic-rich fraction).

The RPP-WTP contract statement of work specifies cesium ion exchange for removing ^{137}Cs from tank waste supernatant to achieve a ^{137}Cs loading of 0.3 Ci/m^3 or less in the immobilized LAW product.[(b)] Further, the contract specifies that cesium ion exchange will use the elutable SuperLig® 644 (SL-644) resin (registered trademark of IBC Advanced Technologies, Inc., American Fork, UT) or a DOE-approved equivalent. SL-644 is a proprietary material that is solely available through IBC Advanced Technologies. To provide an alternative to this sole-source resin supply, DOE-ORP directed BNI to initiate a three-stage process for selecting and potentially implementing an alternative ion exchange resin for cesium removal in the RPP-WTP.[(c)]

BNI completed the first step of this process with the recommendation that resorcinol-formaldehyde (RF) resin be pursued as a potential alternative to SL-644.[(d)] The RF resin is an organic-based resin developed at Westinghouse Savannah River Company in the late 1980s. It was selected as an alternative cesium ion exchange technology for the Initial Pretreatment Module project (Swanson et al. 1994), and extensive testing was performed to support that project during the late 1980s to early 1990s (Bray et al. 1996; Brown et al. 1995b and 1996; Kurath et al. 1994). Both batch- and column-testing of the ground-gel RF resin was conducted at Pacific Northwest (National) Laboratory (PNL/PNNL)[(e)] and the Savannah

(a) The RPP-WTP is currently under construction.
(b) DOE Contract No. DE-AC27-01RV14136 (DOE 2000) Section C.7.d.1.iii.
(c) CCN 030290, Letter from CB Reid, ORP, to RF Naventi, BNI, dated March 13, 2002.
(d) R Peterson, H Babad, L Bray, J Carlson, F Dunn, A Pajunen, I Papp, and J Watson. 2002. *WTP Pretreatment Alternative Resin Selection* 24590-PTF-RPT-RT-02-001, Rev. 0, Bechtel National, Inc., Richland, WA.
(e) Before October 1995, the laboratory was called Pacific Northwest Laboratory. After October 1995, the name was changed to Pacific Northwest National Laboratory.

River Laboratory. The resin was found to have a high loading and selectivity for cesium from Hanford Site tank wastes, and the cesium could be eluted from the resin under acidic conditions.

BNI completed the second step of this process by developing an implementation plan.[(a)] As part of the first stage of RF testing, RF resin performance for Cs-removal was assessed using batch contact and column testing under the technical scoping statement (TSS) A-222 (Fiskum et al. 2004). Side by side testing of spherical RF,[(b)] ground gel RF, and SL-644 using AZ-102 simulant (a high Cs concentration waste form) showed that the spherical resin had adequate capacity and kinetics, better elution performance, and lower pressure drop during column operations than the ground-gel RF and SL-644. BNI completed the first stage of the implementation plan by selecting the spherical RF resin formulation, which provided the best combination of characteristics required for WTP operations.

DOE-ORP directed BNI to initiate second-stage testing designed to evaluate spherical RF resin for cold commissioning in the WTP.[(c)] One part of the second stage testing, provided in Appendix C of the *Research and Technology Plan*[(d)] under TSS A-225, defines the need to determine the impact of manufacturer variation on RF resin performance. Minor changes to the spherical RF resin formulation and curing could affect resin capacity, kinetics, and possibly cesium selectivity.

Spherical RF had only been produced in small laboratory-scale quantities; large-scale preparation of spherical RF posed engineering challenges, and the success of the scale-up processing needed to be tested. Scale-up production was conducted by both Microbeads and Boulder Scientific Corporation (BSC, Mead, CO) under sub-contract to Microbeads. Product testing was required during the various steps of the technology transfer and scale-up process to assess resin performance and product quality. The TSS A-225 was expanded to include the effect of manufacturer scale-up on resin performance as well as batch-to-batch variability in the scale up process.

Battelle—Pacific Northwest Division (PNWD) was contracted to provide data to evaluate the various spherical RF resin formulations according to TSS A-225 under contract number 24590-101-TSA-W000-00004. The objectives of this work were to provide data to BNI for their use in:

- selecting one RF resin formulation for scale-up production by determining
  - bulk properties, including particle-size distribution (PSD), morphology, shrink-swell characteristics, and bed density
  - Cs loading and elution characteristics as a function of spherical RF formulation
- comparing RF loading and elution performance data to SL-644 performance data
- evaluating batch-to-batch variability in scale-up production

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(a) M. Thorson. 2002. *Alternative Ion Exchange Resin Supplemental Research and Technology Plan – Case 20,* 24590-PTF-PL-RT-02-002, Rev. 0, Bechtel National, Inc., Richland, WA.

(b) Spherical RF production is a patented process by Microbeads (Skedsmorkeset, Norway).

(c) Schepens, 2004. CCN 083069, Letter from R. J. Schepens, ORP, to J. P. Henschel, BNI, "Direction to Perform Required Cesium Ion Exchange Alternative Resin Testing", 03-WEC-006, effective date February 25, 2004.

(d) S Barnes, R Roosa, and R Peterson. April 2003. Research and Technology Plan. 24590-WTP-PL-RT-02-002, Rev. 0, Bechtel National, Inc., Richland, WA.

- refining a RF resin purchase specification.

All work was conducted according to test specification 24590-PTF-TSP-RT-04-0001, Rev. 0,[(a)] test plan TP-RPP-WTP-368, Rev. 0,[(b)] and test exceptions 24590-WTP-TEF-RT-04-00001, 24590-PTF-TEF-RT-04-00031, 24590-WTP-TEF-RT-05-00005, 24590-WTP-TEF-RT-05-00007, and 24590-PTF-TEF-RT-06-00001.

This report presents the results obtained over 2 years supporting the TSS A-225 study and satisfying the task objectives. To this end, 59 spherical RF resins were received, 28 of which were selected for specific performance testing. Resin morphology, physical characteristics, and loading and elution performances are presented.[(c)]

(a) MR Thorson. May 2004. *Column and Chemical Testing of Variations of Spherical Resorcinol Formaldehyde*. River Protection Project-Waste Treatment Plant, Bechtel National Inc., Richland, WA.

(b) SK Fiskum. June 2004. *Column Performance Testing of Variations Spherical Resorcinol Formaldehyde Resins, Stage 2*. Rev. 0. Battelle—Pacific Northwest Division. Richland, WA.

(c) The supporting electronic media were delivered to MR Thorson, R&T lead for the alternative cesium ion exchange resin activities, under letter RPP-WTP-06-673 from GH Beeman to MR Thorson September 20, 2006.

2.0 Report Layout

The results defined in this report were generated from extensive spherical RF resin testing over the course of 2 years, meeting the needs defined under TSS A-225.

The original test scope under TSS A-225 included RF resin regeneration testing and performance testing of ~11 resins (33 load and elute tests) in four test waves associated with resin preparation by selected vendors. The work scope increased as new resin formulations were developed for testing and as additional resin evaluation procedures were requested. Over the 2-year study interval, resin pretreatment was standardized and the need for formaldehyde testing was eliminated. As test needs and conditions evolved and the scope increased, the PNWD test plan (TP-RPP-WTP-368) was modified accordingly, through the Waste Treatment Plant Support Project (WTPSP) document change process. The final scope of this report presents test results of

- 28 resins
- 50 loading and elution tests
- physical properties
- regeneration optimization
- oxygen attack
- pretreatment minimization
- SL-644 comparison testing.

To best present the large breadth of experimental design, testing, results, and interpretations, this report was broken out into sections relevant to the specific sub-test objectives. The experimental section is presented in a broad-brush perspective. It includes overarching experimental design, such as ion exchange column design and general processing, as well as unchanging procedures such as those implemented for skeletal density measurement and particle size distribution. Specific process details and variations, such as process ion exchange volumes and flowrates, are presented in the sections with the experimental data.

The report chapters were prepared based on the resin test wave[(a)] to present the specific physical property and ion exchange performance data. Special tests, such as resin regeneration, resin oxidation, and resin neutralization testing, were broken out into separate chapters. Thus, with the overarching experimental section, each chapter can generally stand scrutiny and evaluation separate from the other sections. The report format is as follows:

- Section 3.0: Overarching experimental details are provided that generally apply to all sections.
- Section 4.0: An initial test was conducted on reserve resin stocks from TSS A-222 testing to determine the optimum regeneration reagent and volume. The selected regeneration solution was used for follow-on testing.

(a) A test wave is defined as a group of resins tested concurrently.

- Section 5.0: Wave 1 testing incorporated four resins prepared by SINTEF (Trondheim, Norway). Embedded with the Wave 1 testing was resin exposure to an oxygen-rich regeneration solution and its effect on resin ion exchange characteristics.
- Section 6.0: Specifics of the oxygen-rich exposure on the resin and follow-on (Cycle 3 Wave 1) resin ion exchange performance are presented.
- Sections 7.0 through 10.0: ion exchange performances and physical properties are presented from a variety of small-scale resin lots prepared under differing manufacturing conditions (Waves 1b through 2b).
- Section 11.0 through 14.0: Wave 3 through Wave 4b ion exchange performances and physical properties are presented on the scale-up resin lots.
- Section 15.0: Resin neutralization testing results are presented.
- Section 16.0: Generalized comparative summary of resin testing is presented.
- Section 17.0: Summary of results and conclusions are presented.

The authors hope this format creates a user-friendly report by providing the relevant data in concise, well-defined sections.

3.0 Experimental

This section describes the general experimental approach and relevant details used for spherical RF resin handling, testing, data reduction, and reporting. Specific process details, as they pertain to individual test waves, are discussed with the test wave results. All raw data generated in support of this testing are maintained in the project files at PNWD under Project 42365 records inventory and disposition system.

3.1 Spherical RF Test Resins

Many RF resins were received over the course of 2 years. The resins were tested in several series designated as numbered waves (1-4) and sub-waves. In all cases, Microbeads (Skedsmokorset, Norway) created the seed material used for the spherical RF production. SINTEF[(a)] (Trondheim, Norway) and Microbeads prepared numerous resin batches under various processing conditions for testing. Microbeads sub-contracted Boulder Scientific Corporation (BSC, Mead, CO) to prepare spherical RF resin using the Microbeads patent under the supervision of Microbeads personnel.

Different resin lots were prepared under slightly different conditions in an effort to optimize Cs ion exchange capacity and selectivity. Varied production parameters included relative resorcinol-to-formaldehyde mole ratio, curing time, curing temperature, and excess monomer (RF) added beyond the theoretical capacity of the resin sphere. However, resins BRF-14, TI394-11 (BSC 3380-2P-0100), and TI394-15 (Microbeads 5C-370/522) were synthesized under similar conditions; resins TI394-16 (Microbeads 5C-370/523) and TI394-61 (Microbeads 5E-370/639) were synthesized under similar conditions. Actual production parameters were confidential and were provided directly to the Research and Technology (R&T) lead (MR Thorson) from Microbeads.

Test Waves 3 and on evaluated resin quality and reproducibility from the scaled-up production process. Resin TI394-62 (Microbeads 5E-370/641) from Wave 3, and resins from Wave 4a and Wave 4b were manufactured under similar process conditions.

Table 3.1 summarizes the PNWD-assigned internal resin IDs, resin manufacturer, lot number, lot size, and preparation and receipt dates. The resins (and associated information) are subdivided according to test wave. Each RF resin was received in the H-form under water.

3.2 Resin Sub-Sampling

Each resin was sampled consistent with American Society for Testing and Materials (ASTM) Method 2687, "Standard Practices for Sampling Particulate Ion-Exchange Materials." After sub-sampling, most of the remaining container void space was filled with deionized (DI) water. Residual gas head-space was flushed with N_2 cover gas to minimize resin contact with oxygen.

(a) SINTEF prepared the first test wave of resins. Microbeads resumed the manufacturing process for all subsequent test waves.

Table 3.1. Test Resin Summary

Resin Internal ID	Manufacturer	Lot Number	Lot Size[a]	Manufacture Date	Receipt Date
Wave 1					
BRF-14	SINTEF	BRF-14	2 L	6/04	7/19/04
BRF-15	SINTEF	BRF-15	2 L	6/04	7/19/04
BRF-16	SINTEF	BRF-16A-B-C	2 L	6/04	7/19/04
BRF-17	SINTEF	BRF-17	2 L	6/04	7/19/04
BRF-18	SINTEF	BRF-18	2 L	6/04	7/19/04
SL-644	IBC Advanced Technologies	C-01-11-05-02-35-60[b]	250 gal	11/02	7/03
Wave 1b					
420	Microbeads	PS-420	< 2 L	[c]	10/18/04
424	Microbeads	PS-424	< 2 L	10/04	10/18/04
Wave 2					
TI394-11	BSC	BSC-3380-2P-0100	0.5 gal	3/05	3/18/05
TI394-12	BSC	BSC-3380-2P-0101	0.5 gal	3/05	3/18/05
TI394-13	BSC	BSC-3380-2P-0102	0.5 gal	3/05	3/18/05
TI394-14	BSC	BSC-3380-2P-0103	0.5 gal	3/05	3/18/05
TI394-5	Microbeads	PS-493	0.15 L	3/05	3/14/05
TI394-8	Microbeads	PS-501	0.15 L	3/05	3/14/05
Wave 2a					
TI394-15	Microbeads	5C-370/522	2 gal	4/05	4/12/05
TI394-16	Microbeads	5C-370/523	2 gal	4/05	4/12/05
Wave 2b					
TI394-17	Microbeads	PS-513	0.15 L	4/05	4/20/05
TI394-18	Microbeads	PS-514	0.15 L	4/05	4/20/05
TI394-19	Microbeads	PS-515	0.15 L	4/05	4/20/05
TI394-20	Microbeads	PS-517	0.15 L	4/05	4/20/05
TI394-21	Microbeads	PS-518	0.15 L	4/05	4/20/05
TI394-9	Microbeads	PS-502	0.15 L	4/05	3/14/05
Wave 3					
TI394-61	Microbeads	5E-370/639	50 gal	5/05	6/6/05
TI394-62	Microbeads	5E-370/641	75 gal	5/05	6/6/05
Wave 3a					
TI-394-63[d]	Microbeads	5E-370/641	75 gal	5/05	11/3/05
Wave 4a					
TI394-64	Microbeads	5J-370/686	72 gal	9/05	10/21/05
TI394-65	BSC	3380-3-0200	66 gal	11/05	12/19/05
Wave 4b					
TI394-72	Microbeads	6C-370/745	74 gal	4/06	4/25/06
TI394-73	BSC	3380-3-0201	66 gal	5/06	5/12/06

(a) The spherical RF lot size represents the as-produced H-form resin. Typically, the resin expands 50 to 60% upon first conversion to the Na-form.
(b) The SL-644 sample was wet-screened in the Na-form to 18 to 40 mesh at SRNL.
(c) The parameter was not provided.
(d) Sub-sample of resin that was processed through 16-cycles at Savannah River National Laboratory (SRNL).

3.3 Resin Pretreatment

Resin pretreatment evolved as a result of experience gained during the testing campaigns. Wave 1 resin pretreatment incorporated a simple water wash before processing. The Wave 1 pretreatment is detailed in Section 5.0. For Wave 1b and subsequent test waves, resin pretreatment utilized a full resin expansion and contraction cycle in an open beaker format, consistent with the protocol, P1-RF, *Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column,*[(a)] which was approved after Wave 1 testing was complete. The P1-RF pretreatment steps were further delineated in test instruction TI-RPP-WTP-394.[(b)]

The pretreatment (P1-RF protocol) steps are summarized in Figure 3.1. The nominal resin volumes represent settled resin in graduated cylinders. The resin was settled in the graduated cylinder by tapping with a bung. The volumes were recorded once a constant volume was achieved.

An aliquot of as-received resin was taken and the volume (RV) was determined. The aliquot was transferred to a beaker and soaked for 30 min in 5 RVs DI water; the slurry was agitated every 10 min. After allowing the resin to settle, the water was removed, and 5 RVs of 1 M NaOH was added. The slurry was agitated every 10-min during the first hour and then the mixture was soaked overnight. The contact solution was verified to be basic after the first 30 min and again after soaking overnight. The solution was removed, and the resin was washed three successive times with 3 RVs water; each rinse contacted the resin for 30 min with agitation every 10 min. The pH of the final water-rinse supernatant was generally between 12.5 and 13.0. The resin was converted back to the H-form by removing the final water-rinse supernatant and soaking the washed resin in 10 RVs 0.5 M HNO_3 for 2 h with agitation every 10 min. This was followed by three successive rinses with 3 RVs of DI water, each rinse contacted the resin for 30 min with agitation every 10 min. The pretreated resin volume was measured, and then the resin was sub-divided into the appropriate processing fractions as shown in Figure 3.1 for column testing, PSD, microscopy, and dry mass determination.

The balance of the resin was converted back to the Na-form by repeating the 1 M NaOH soaking steps and water rinse steps. At least three water rinses were applied, bringing the final solution pH to ~12.7 to 12.9. Additional water rinses did not appreciably change the equilibrated solution pH. Efforts to reach an equilibrium pH of 12.5 were abandoned after subjecting the resin to a total of five water rinses.

(a) WTP doc. no. 097893, CA Nash and CE Duffey, August 17, 2004, *Hanford RPP-WTP Alternate Resin Program - Protocol P1-RF: Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column*.

(b) SK Fiskum, TI-RPP-WTP-394, *Resin Pretreatment and Physical Properties Evaluation of Spherical RF Resins*, 2004.

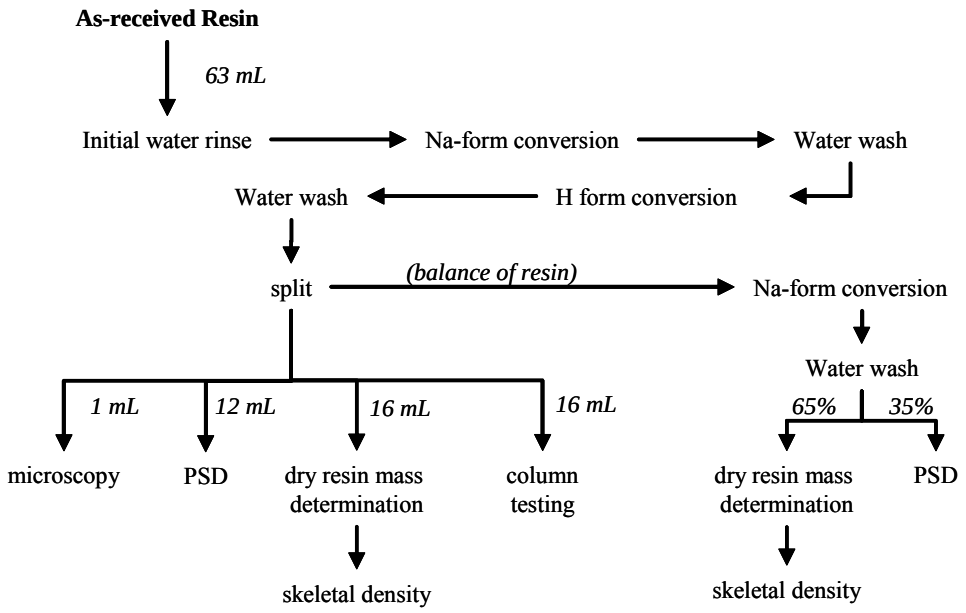


Figure 3.1. Typical Sample Pretreatment and Splitting for Physical Property and Column Performance Testing, Waves 1b through 4b

3.4 Optical Microscopy

Wet resin aliquots (~1 mL) were dried at room temperature under vacuum or nitrogen flush to a free-flowing state, which generally took ~4 h. Constant dry mass was neither required nor obtained.

Optical micrographs of the dried resins were taken at 10×, 25×, and 70× magnification to characterize particle shape and morphology. Resin sub-samples were prepared by covering the vial container opening with tape and inverting the container. Resin spheres were randomly distributed across the tape without preference to defect orientation. Several observations were made of the resin before the micrographs were taken to help confirm that the photographed regions were representative of the overall resin.

A fraction of resin particles was randomly selected for cross-sectioning. Several resin beads from each lot were individually sectioned using a scalpel. Micrographs were taken of the cross-sections at 70× magnification.

3.5 Particle Size Distribution

The PSD was measured on each of the resins in both the H-form and the Na-form. This analysis was performed according to procedure[(a)] using a MicroTRAC-S3000 Particle Size Analyzer. Standards traceable to the National Institute of Standards and Technology (NIST) (nominal 230-µm, 480-µm, and 750-µm diameter) were measured before and after the resin sample measurements to verify the accuracy of the results. The dispersion liquid for the H-form resins was DI water; the Na-form resins were dispersed in 0.01 M NaOH. Each sample was analyzed in duplicate. The particle-size analyzer was only

(a) W Buchmiller, TPR-RPP-WTP-222, Rev. 1, *S3000 Microtrac Particle Size Analyzer.*

capable of measuring particle sizes up to a maximum of 1410 μm. This constraint did not appear to bias the data because the sample peak results were generally well below this maximum.

The Microtrac S3000 reported particle diameters on a volume basis (m_v), number basis (m_n), and a surface area basis (m_a). The m_v, m_n, m_a, and standard deviation (sd) values were calculated according to the following equations:

$$m_v = \frac{\sum V_i d_i}{\sum V_i} \quad (3.1); \qquad m_n = \frac{\sum (V_i / d_i^{\,2})}{\sum (V_i / d_i^{\,3})} \quad (3.2);$$

$$m_a = \frac{\sum V_i}{\sum V_i / d_i} \quad (3.3); \qquad sd = \frac{(84\% - 16\%)}{2} \quad (3.4)$$

where V_i is the particle volume, and d_i is the particle diameter. For populations of uniform diameter, m_v, m_n, and m_a will be in close agreement.

3.6 Shrink-Swell Characteristics

The resin shrink-swell characteristics were measured during column processing (constrained) and during select pretreatment steps (unconstrained). In-column shrink-swell characteristics were measured simply with a millimeter scale to determine resin bed height in each process reagent; the resin bed volume was calculated based on the column diameter of 2 cm. The pretreatment shrink-swell characteristics were based on resin volumes measured in graduated cylinders after open-beaker equilibration.

3.7 Dry-Bed Density

The dry-bed density was determined for the Na-form resin and the H-form resin. The H-form resin dry-bed density was evaluated from two different sources of volume measurements: in-column process volume and out-of column graduated cylinder volume. One mass source applied to both cases. The Na-form bed density was only determined from out-of column processing.

The dry H-form resin mass loaded into the ion exchange columns was determined indirectly. Two duplicate volumes of settled H-form resin slurries were measured in a 25-mL graduated cylinder. One sample was used for column testing. Excess water was removed from the second sample, which was then taken to dryness under vacuum at 50°C. Dryness was defined as a resin mass change of <0.5% over a 7-h period.[(a)] The dry H-form resin mass in the ion exchange column was calculated relative to the volumes taken and the dry mass of the second sample according to Equation 3.5.

$$M_c = \frac{V_c * M_d}{V_d} \quad (3.5)$$

(a) The dried resin was subsequently used as the starting material for H-form skeletal density determination (see Section 3.8).

where M_c = mass of dry H-form resin loaded in the column
V_c = volume of wet H-form resin transferred to the column
V_d = volume of wet H-form duplicate resin used for dry mass determination
M_d = mass of dry H-form duplicate resin used for dry mass determination.

The in-column processing dry-bed density (δ_{DRB}) was calculated according to Equation 3.6.

$$\delta_{DRB} = \frac{M_c}{V} \tag{3.6}$$

The resin volume (V) in the column varied according to process feed. The mass basis (M_c) was restricted to the dry H-form resin mass. The dry-bed densities from pretreatment processing were similarly calculated substituting M_d and V_d for M_c and V in Equation 3.6.

The measured quantity of Na-form resin slurry was phase-separated and dried similarly to the H-form resin, establishing constant mass. The dry bed density for the Na-form resin was estimated by substituting the dry Na-form mass and wet Na-form volume for M_c and V in Equation 3.6.

The calculated Na-form resin density may be biased high because water could potentially still be incorporated in the "dried" resin. Barnett and Landman (1993) showed that the energy required for hydrated water removal from the Na ion markedly increased as the hydration number decreased below 4. Arm and Blanchard (2004) indicated that drying ground gel RF under vacuum at 50°C may not be sufficient to remove all waters of hydration. This was based on the discrepancy between the derived Na capacity from the I-factor calculation and Na consumption during column testing. Furthermore, the amount of water washing may further change the amount of Na associated with the resin. The equilibrated solution pH did not change appreciably with subsequent water washes, indicating that the resin was functioning like a buffer, releasing Na ions with each water wash. (See Section 15.0 for further discussion.)

3.8 Skeletal Density

Skeletal densities were measured in a calibrated 25-mL pycnometer consistent with ASTM Method D 854-02, *Specific Gravity of Soil Solids by Water Pycnometer*. The resins that were dried to establish constant mass were subdivided into duplicate ~2 g aliquots (precisely weighed) and placed in pycnometers. Each pycnometer was partially filled with degassed DI water. During the resin hydration process, a significant quantity of bubbles emanated from the resin. The resin-water slurry was placed under vacuum overnight at ambient temperature and then removed and stirred to help release trapped bubbles. The vacuum/stirring process was repeated until no further bubble-release from the resin was observed. The pycnometer was then brought to volume with de-gassed water and weighed. All masses were measured to the nearest 0.0001 g. The temperature of the resin/water mixture was measured to the nearest 0.1°C with a resistance temperature detector (RTD) thermocouple (PR-11-2-100-1/8-6-E, Omega, Stamford, CT) and readout (HH 612P2C, Omega, Stamford, CT). The skeletal density was then calculated according to Equation 3.7.

$$\delta = \frac{m}{\left(V_p - \frac{m_w}{\delta_w} \right)} \tag{3.7}$$

where δ = skeletal density
m = dry resin mass
V_p = pycnometer volume
m_w = mass of water in pycnometer
δ_w = density of water at temperature.

The overall experimental uncertainty was ~0.3%. The Na-form dried resin probably still contained waters of hydration associated with the Na-ion. (See Section 3.7 for discussion.) Residual water in the Na-form resin will bias the skeletal density results high.

3.9 Ion Exchange Process Testing

The ion exchange column testing was conducted according to test instruction TI-RPP-WTP-377, *Load and Elute Column Testing of Spherical RF Resins*.[(a)] Six ion exchange process systems were available for concurrent testing.[(b)] Columns were color-coded (pink, green, yellow, white, blue, and red) for ease of sample and data tracking. Figure 3.2 shows a schematic of a typical ion exchange column assembly. A system consisted of one glass column containing the ion exchange resin, a small metering pump, three valves, a pressure gauge, and a pressure-relief valve. Valves 1, 2, and 3 were three-way valves that could be turned to the column-flow position or an exhaust position to expel trapped air or fluids from the column input/output lines. Valve 1 was placed at the outlet of the pump and was used to isolate the column from the pump. Valve 3 was primarily used to obtain samples and isolate the system during storage periods.

Columns were prepared at the Kontes Custom Glass Shop (Vineland, NJ). Each column was 10-cm tall with an inside diameter of 2.0 cm (corresponding to a resin volume of 3.1 mL/cm) and a 2.8-mm wall thickness. The glass was safety coated with polyvinyl chloride. Stainless steel, 200-mesh screens, provided by Savannah River Technology Center (SRTC), supported the resin beds. The screens were stabilized with snug-fitting O-rings. The cavity below the screen support was filled with 3-mm-diameter glass beads, reducing the fluid-filled volume below the screen from 11 mL to 6 mL. The height of the resin bed (and thus shrinkage and swelling) was measured with a millimeter-scale ruler (the associated measurement error was estimated to be ±2 mm). The fluid level above the column was maintained at nominally the 10-cm height. Depending on whether the resin was expanded (~6.4 cm tall) or contracted (~5.3 cm tall), the fluid volume above the resin bed varied from ~11 mL to ~15 mL, respectively.

(a) SK Fiskum, July 2004.
(b) Conducting six tests in parallel reduced the overall operating cost per test and speeded the required data-collection process.

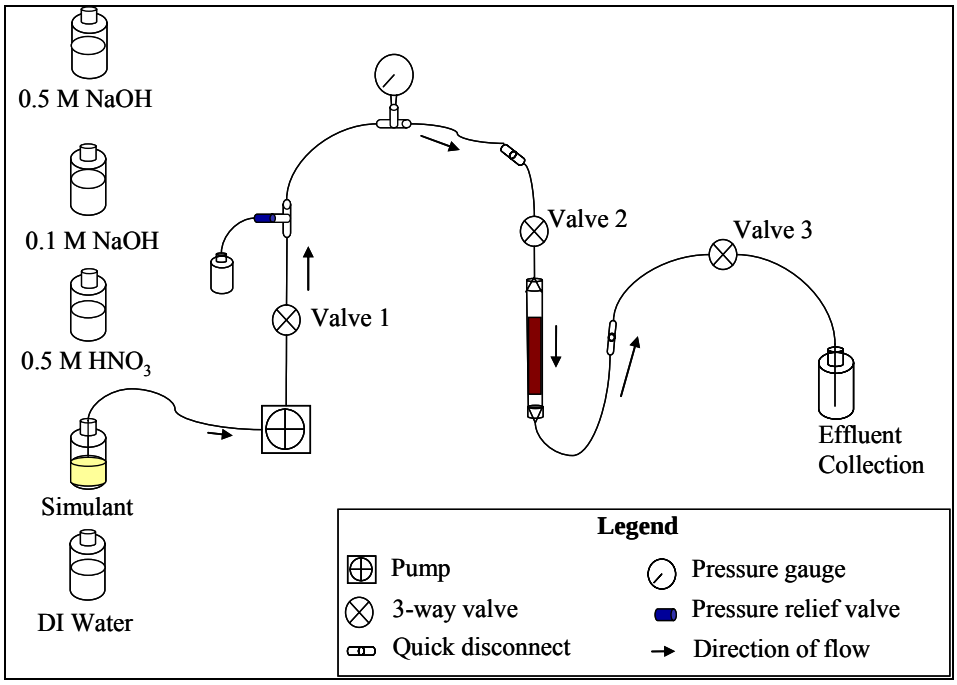


Figure 3.2. Ion Exchange Column Processing System

The polyethylene connecting tubing was $^{1}/_{8}$-in. OD and $^{1}/_{16}$-in. ID. The column end fittings were standard Kontes Chromaflex column end fittings with ethylene tetrafluoroethylene ferrules. The inlet sample line ended at the column fitting. The column assembly contained an in-line Swagelok Poppet pressure relief check valve with a 10-psi trigger (Solon, OH) and a 15-psi pressure gauge (McDaniel Controls Model #SA, Luling, LA). Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column removal and switching. Fluid Metering, Incorporated (FMI) QVG50 pumps (Syosset, NY) equipped with ceramic and Kynar® coated low-flow piston pump heads were used to introduce all fluids. The flowrate was controlled with a remotely operated FMI stroke-rate controller. The pump was set up to deliver flowrates from 0.3- to 1.1-mL/min. The volume actually pumped was determined using the mass of the fluid collected divided by the fluid density.

The holdup volume of the entire ion exchange system, which represented the summed volume of all fluid-filled parts, was ~47 mL. The various fluid holdup volumes for sections of the ion exchange apparatus are shown in Table 3.2. The conversion to bed volume (BV) is also shown where 1 BV is defined as 20 mL.[(a)] The air gap space represents the volume from the input line into the column assembly and the top of the fluid volume. This gap was maintained constant except for the upflow elution condition discussed in Section 12.

(a) One BV was defined as the Na-form resin volume after initial pretreatment cycling in the column (see Section 3.10.1). The 20-mL volume is a nominal value and is used for illustrative purposes. Actual BVs varied from ~17 mL to ~21 mL.

Table 3.2. Apparatus Holdup Volumes

IX Apparatus Section	Fluid Volumes			
	H-form Resin		Na-form Resin	
	mL	BV[(a)]	mL	BV[(a)]
Feed bottle to top of column	8.7	0.43	8.7	0.43
Air gap between column top and fluid surface[(b)]	7.6	0.38	7.6	0.38
Fluid above resin bed	15	0.75	13	0.65
Fluid in resin bed	14	0.70	16	0.80
Bottom of resin bed (support screen) to exit line	9.7	0.49	9.7	0.49

(a) Where 1 BV is defined as 20 mL.
(b) The air gap was filled with fluid only once, during upflow elution (see Section 12).

A photograph of two duplicate apparatuses is shown in Figure 3.3. The appearance of Resin BRF-14 (yellow-taped column) and Resin BRF-15 (green-taped column), both in the Na-form, can be observed. Also shown are the fluid levels above the resin beds, which constituted the mixing areas during feed transitions. The fluid level remained at the same height during resin expansions and contractions.

Figure 3.3. Photograph of Two Ion Exchange Column Assemblies, Na-Form Resin (BRF-14 in Yellow Column, left; BRF-15 in Green Column, right)

The H-form pretreated resin sample taken for column testing was soaked in 1 M NaOH at a 10:1 solution volume to resin volume ratio in a beaker to allow for unconstrained resin expansion. After the

resin was soaked for 2 hours in the caustic medium, the NaOH was decanted, and the resin was slurried with an equivalent volume of DI water. The resin slurry was then transferred to the column. The column was tapped gently to settle the bed. Once process flows started, the resin beds were not disturbed, vibrated, or tapped. Typically the resin bed was successively rinsed downflow with DI water, 0.5 M HNO_3, DI water, and then regenerated with 0.5 M NaOH for follow-on processing. Specific details of in-column pretreatment are provided in the subsequent report sections.

General processing parameters mimicked expected plant operating conditions and are summarized in Table 3.3. Specific parameters for each column test are identified with the test wave results. All processing was conducted downflow.[(a)] The RPP-WTP nominal design condition described in the system description includes a 3-column sequence with feed processing rates at 2.2 BV/h (15 gpm) for Envelope A and C wastes.[(b)] The current testing was limited to single-column tests. Conservatisms were built into the test design to bound the resin performance at a 3-BV/h flow rate (higher than nominal design at 2.2 BV/h) and in a 2-column sequence (shorter than the 3-column sequence design). The AP-101 simulant processing runs were started at 1.5 BV/h simulating the bounding condition of the 2-column sequence at 3 BV/h feed. Thus the cesium breakthrough from the single-column test processing at 1.5 BV/h simulated the 2-column system processing at 3 BV/h. The higher residence time at the expected RPP-WTP flow rate (2.2 BV/h) is expected to provide sharper breakthrough curves with greater volumes processed before reaching initial cesium breakthrough. In addition, the higher superficial flow rates due to the greater column height in the RPP-WTP are expected to provide sharper breakthrough curves even when the residence time is the same as in the test columns. The 1.5 BV/hr initial flow rate also allowed better detection of "cesium bleed" or post elution cesium leakage into the subsequent load cycle. After an initial ~80 BV were processed, the flowrate was increased to 3 BV/hr. Processing with AZ-102 simulant was maintained at 1.5 BV/h.

Table 3.3. Generic Ion Exchange Process Steps

Process step	Feed Solution	Process Volume, BV[(a)]	Process Flowrate, BV/h
Water rinse	DI water	7.5	3
Acid wash	0.5 M HNO_3	8	3
Water rinse	DI water	3	1.4
Regeneration	0.5 M NaOH[(b)]	6	3
Loading column	AP-101 Simulant	0 - 80	1.5
Loading column	AP-101 Simulant	80 - 130	3
Feed displacement	0.1 M NaOH	3	3
Rinse	DI water	3	3
Elution	0.5 M HNO_3	21	1.4
Rinse	DI water	3	1.4

(a) BV = bed volume in regeneration solution (targeted 20 mL in the Na-form; actual volumes varied between 17 mL and 20 mL).
(b) The regeneration solution for SL-644 was 0.25 M NaOH.

(a) One elution process test was conducted in the upflow direction (Wave 3a processing, Section 12.0).
(b) JW Olson, *System Description for Cesium Removal Using Ion Exchange—System CXP*, 24590-PTF-3YD-CXP-00001, 12/2001.

The feed simulant was filtered through a 0.45-μm-pore-size nylon filter before use to remove any trace solids. A suitable amount of simulant, typically 3.5-L per column test, was prepared by spiking with a radioactive Cs tracer. The activity concentrations were nominally 0.113 μCi/mL ^{137}Cs.

Samples were collected periodically during the simulant feed and elution steps to evaluate Cs breakthrough and elution profiles. Feed effluent samples were collected two to three times per day in 10-mL aliquots; between sampling events, the effluent was collected as a composite. Temperature was recorded with each sampling event; the temperature variation during the simulant loading step for each test wave is provided in Appendix A. Where elution profiles were desired, eluate samples were collected in nominal 1- to 2-BV increments. Other process solutions (regeneration, feed displacement, DI water rinses) were collected separately as composite effluents. The Cs loading and elution characteristics were monitored from the sample ^{137}Cs-tracer activity using gamma spectrometry and/or liquid scintillation counting.

After each process cycle, the ion exchange column assembly was disconnected at the quick disconnects and removed from the fume-hood containment. The column assembly was mounted in front of a side-looking gamma energy analysis (GEA) detector for measurement of residual ^{137}Cs. Care was taken to position the resin bed itself in front of the detector face. The column assembly in the counting chamber included the ion exchange resin, the interstitial fluid, glassware, and the fluid head above the resin bed and the fluid immediately below the resin bed. Thus, ^{137}Cs tracer in any of the non-resin areas could shine into the detector and bias the measurement. An approximate geometry correction was applied, although the exact geometry presented to the detector was neither calibrated nor was the Cs location on the system known. The calibration accuracy was checked by recounting one column system at a greater distance from the detector (minimizing geometry effects). Both calculated activities agreed well, lending support to the application of the geometry correction factor. A conservative uncertainty of a factor of two was assigned to the column ^{137}Cs-tracer measurements.

Processing was conducted in multiple waves where up to six columns were operated at one time for a total of 55 individual ion exchange processing tests. The Wave 1 load and elution testing was conducted in a manner that would quickly eliminate the worst-performing RF resin and provide comparative testing with SL-644. The overall test matrix given in Table 3.4 summarizes the test wave, process cycles, resin IDs, column ID (color), and simulant processed.

Wave 1 tested four RF resin formulations and SL-644, three loading and elution cycles, and AP-101 and AZ-102 simulants. Follow-on test waves evaluated one or two process cycles with AP-101 simulant. The AP-101 simulant, containing 0.7 M K, is challenging for Cs ion exchange resins because of the competitive effect K has for Cs exchange sites. Thus, most of the loading/elution performance testing was conducted with AP-101 simulant. Previous testing conducted in support of Scoping Statement A-222 (Fiskum et al. 2004) used AZ-102 simulant. To tie the current test results to previous test results, one Wave 1 column and one Wave 4b column were devoted to testing AZ-102 simulant.

Table 3.4. Overall Ion Exchange Test Matrix

Wave #	Cycle # Start date	AP-101[(a)]		AZ-102	
		Resins	**Column ID**	**Resins**	**Column ID**
Wave 1	Cycle 1 7/20/04	BRF-14, -15, -16, -17	yellow, green, blue, pink	BRF-14	white
	Cycle 2 7/30/04	BRF-14, -15, -17, SL-644	yellow, green, pink blue	BRF-14	white
	Cycle 3[(b)] 8/6/04	BRF-14, -15, -17, SL-644	yellow, green, pink blue	BRF-14	white
	Cycle 3 8/13/04	BRF-14, -15, -17, SL-644	yellow, green, pink blue	BRF-14	white
Wave 1b	Cycle 1 10/17/04	PS-420, PS-424	green, pink	None	NA
Wave 2	Cycle 1 3/29/05	TI394-11, TI394-12, TI394-13, TI394-14, TI394-5, and TI394-8	yellow, green, pink, white, blue, red	None	NA
	Cycle 2 4/6/05	TI394-11, TI394-12, TI394-13, TI394-14, TI394-5, and TI394-8	yellow, green, pink, white, blue, red	None	NA
Wave 2a	Cycle 1 4/18/05	TI394-15, TI394-16	yellow, white	None	NA
Wave 2b	Cycle 1 4/23/05	TI394-17, TI394-18, TI394-19, TI394-20, TI394-21, and TI394-9	yellow, green, pink, white, blue, red	None	NA
Wave 3	Cycle 1 6/13/05	TI394-61, TI394-62	yellow, white	None	NA
	Cycle 2 6/20/05	TI394-61, TI394-62	yellow, white	None	NA
Wave 3a	Cycle 16 1/9/06	TI394-63 (two tests)	yellow, white	None	NA
Wave 4a	Cycle 1 1/9/06	TI-394-64, TI-394-65	green, pink	None	NA
Wave 4b	Cycle 1 5/22/06	TI394-72, TI394-73	white, yellow, pink, blue	TI394-72	Green

(a) The first AP-101 simulant lot (# 135125/1.1) supported Wave 1 through Wave 2a testing. The second AP-101 simulant lot (# 144354/1.1) supported Wave 2b through Wave 4b testing.

(b) Oxygen-saturated reagent processing test. This was an extended regeneration process associated with the third process cycle.

3.10 Ion Exchange Processing Data Analysis

The ion exchange process data manipulations are defined in this section.

3.10.1 Bed Volume

In all cases, one BV was defined as the Na-form resin BV calculated after initial pretreatment cycling in the column. The BV was determined after completing the 0.5 M NaOH preconditioning rinse. All resin beds expanded and contracted ~20% as a function of feed matrix.

3.10.2 Load Profile

The Cs loading profiles are provided for all cycles tested. Each loading profile is plotted as % C/C_o vs. the BVs of feed processed through each column. The abscissa reflects BVs as a function of the resin in the Na-form after the in-column pretreatment (i.e., after the first regeneration step with 0.5 M NaOH or, in the case of SL-644, 0.25 M NaOH).[(a)] The C_o value for the Cs tracer was determined for each feed condition. In most cases, the C/C_o is plotted on a probability scale. A probability scale is the inverse of the Gaussian cumulative distribution function (characteristic of ideal ion exchange theory) such that a graph of the sigmoidally shaped Gaussian cumulative distribution function appears as a straight line (Buckingham 1967). The probability scale has two advantages: 1) making low C/C_o data easily readable such that the initial load performance is discernable and 2) easily estimating extrapolation to 50% breakthrough in the sigmoidal region. Less-than values are recorded on the breakthrough profiles as actual values; the less-than values can be identified from the data-input tables. Also shown on each loading figure is the minimum Cs removal required for the effluent to meet design-basis ^{137}Cs loading in the vitrified glass product (contract limit). For the AZ-102 actual tank waste, the contract limit % C/C_o is 0.025, corresponding to a Cs decontamination factor (DF) of 4000. For the AP-101 actual tank waste, the maximum % C/C_o is 0.091, corresponding to a DF of 1100.[(b)]

Key measures of resin performance can be ascertained from the load profiles. The 50% Cs breakthrough point is indicative of the breakthrough resin capacity. The higher the bed volumes processed before reaching 50% breakthrough, the higher the resin capacity. The onset of Cs breakthrough, defined as the BVs processed before the breakthrough C/C_o starts to increase, is a measure of selectivity and/or kinetics. The sooner the onset Cs breakthrough is observed, the less selective or the slower exchange kinetics the resin has for Cs. Faster Cs breakthrough results in smaller effluent composite volumes meeting RPP-WTP requirements. Ideally, one would want delayed observance of any Cs breakthrough and a very steep breakthrough curve where 50% breakthrough is observed after processing large volumes of feed.

3.10.3 Elution Profile

Each elution profile is plotted as C/C_o versus the BVs of eluant processed through each column. As with the load profile, the BV represents the resin BV in the expanded Na-form (in 0.5 M NaOH). The C/C_o is plotted on a log scale to better discern the low relative Cs concentrations associated with the

(a) The bed volume fluctuated significantly during the various processing steps.

(b) The contract limit is a function of ^{137}Cs and Na concentrations in the feed, the end-product glass Na loading, and the glass density. Because of radioactive decay, the ^{137}Cs feed concentration will decrease with time. The assumptions and calculations for the given AP-101 and AZ-102 contract limits are provided in Appendix B.

elution tailing effect. Ideal elution behavior will cause Cs to be removed quickly with virtually no residual Cs on the column upon completion. The smaller the volume needed to meet this specification, the better for the RPP-WTP plant operations (lower resin regeneration process times and lower resin exposure to dissolved oxygen) and eventual spent resin disposal.

The residual Cs on the resin following elution potentially affects follow-on processing with respect to Cs bleed into the effluent during the subsequent cycle, and the spent resin Cs loading at the end of the resin processing life. A mass balance approach was used to determine the total Cs on the resin bed as a function of the elution and water rinse volumes. The total Cs remaining on the resin bed, normalized to a per gram dry H-form basis, was calculated starting from the residual ^{137}Cs tracer in the resin bed. The Cs quantity on the resin bed as a function of eluate BV processed was calculated by adding the contribution of each eluate sample Cs quantity to the resin in reverse order. Working backward through the water rinse and elution process, the total Cs on the resin was reconstructed and normalized to the mass of dry H-form resin. Thus an alternate elution profile is presented (in selected cases) of total Cs remaining on the resin bed on a per g dry H-form resin basis as a function of the elution and water rinse BVs.

The RPP-WTP design basis residual ^{137}Cs content in spent resin was determined to be 60 μCi/g resin,[(a)] allowing safe handling of the spent resin. Burgeson et al. (2004) calculated the ^{137}Cs loading in spent resin on a volume basis to be 40 μCi/mL, which in turn corresponds to a total Cs concentration of 4.2 μg/g in spherical RF (based on a dry bulk density of 0.44 g/mL) and 2.8 μg/g in SL-644 (based on a bulk density of 0.66 g/mL). These values were based on the isotopic mass fraction of 25% ^{137}Cs. If the ^{137}Cs isotopic fraction increases to 32.8%, consistent with AZ-102 (Fiskum et al. 2003), then the corresponding total Cs limit in spent resin would become 3.2 μg/g for spherical RF and 2.1 μg/g for SL-644 according to Equation 3.8.

$$C = \frac{L_m}{B * SpA * \delta} \tag{3.8}$$

where C = total Cs concentration limit, μg/g H form resin
L_m = ^{137}Cs concentration limit (40 μCi/mL H-form resin; Burgeson et al. 2004)
B = ^{137}Cs isotopic fraction
SpA = specific activity of ^{137}Cs (86.8 μCi/μg)
δ = bulk dry resin density (0.66 g/mL SL-644, 0.44 g/mL spherical RF; Burgeson et al. 2004)

3.11 Test Simulant Selection

Wastes containing high K concentrations, such as found with AP-101 and AW-101, create a more difficult challenge to the RF ion exchange material because the K competes with Cs for active exchange sites. The AP-101 matrix, with 0.7 M K (when diluted to 5 M Na), was considered to bound the worst-case Cs loading condition. Therefore, the AP-101 simulant matrix was selected for evaluating the effects of incremental adjustments in RF resin formulation on Cs loading performance.

(a) Meeting minutes prepared by John Olson for Bechtel National, Inc., CCN 055152, April 24, 2003. "Determine the Baseline Spent Resin Cesium Concentration." Richland, WA.

AZ-102 simulant had previously been used with TSS A-222. The load results with this simulant needed to be evaluated as a tie-in to the previous results. Most of the Hanford tank waste is low in K, as is the AZ-102. Furthermore, AZ-102 contained 52 mg/L Cs (nearly 10-fold higher Cs concentration than AP-101) and provided a good matrix to evaluate the effect of high Cs loading.

3.12 Simulant Preparation and Analysis

Noah Technologies (San Antonio, TX) was contracted to prepare two lots of AP-101 according to the simulant recipe reported by Russell et al. (2003). After allowing the simulant mixture to stand for 24 hours, it was filtered through a 0.5-μm pore size glass fiber filter. The first lot of 160 L (Lot # 135125/1.1) was prepared in May 2004; an 85-L fraction was delivered to the RPL for ion exchange testing (the balance was sent to the Advanced Process Engineering Laboratory [APEL] facility for use in hydraulic testing). Noah Technologies also prepared 50 L of AZ-102 simulant (Lot # 135136/1.1) in May 2004 according to the simulant recipe reported by Hassan and Nash (2002). As with the AP-101 simulant, solids were allowed to form for 24 hours before filtering through a glass-fiber filter with a pore size of 0.5 μm. The entire AZ-102 preparation was delivered to the RPL for ion exchange testing. Noah Technologies prepared a second 100-L lot of AP-101 simulant (# 144354/1.1) in April 2005 for delivery to RPL.[(a)] The AP-101 and AZ-102 simulant recipes are provided in Appendix C.

After arrival at PNWD, subsamples of each simulant were removed, filtered, and analyzed for density. All density determinations were performed in duplicate by measuring the net simulant mass in 25-mL Class A volumetric flasks. Additional subsamples were submitted to the Analytical Support Operations (ASO) under Analytical Services Request (ASR) 7019 (first AP-101 and AZ-102 simulant preparations) and ASR 7273 (second AP-101 simulant preparation) for determination of free hydroxide, metals, total inorganic carbon (TIC, i.e., carbonate), Cs, and anions. The ASO was responsible for generating the appropriate batch and analytical quality control (QC) samples for the analysis as well as providing any additional processing to the sub-samples that might be required. The simulant analytical results are summarized in Table 3.5, Table 3.6, and Table 3.7. Only targeted/major analytes are reported. The simulant also included trace constituents as shown on the preparation recipe in Appendix C.

(a) The first AP-101 simulant lot (# 135125/1.1) supported Wave 1 through Wave 2a testing. The second AP-101 simulant lot (# 144354/1.1) supported Wave 2b through Wave 4b testing. Test waves are discussed later in this section.

Table 3.5. AZ-102 Simulant Composition—Major Analytes

Analyte	Prep Blank µg/mL	Sample µg/mL	Duplicate µg/ mL	Average µg/mL	Average M	Target M	% of Target
$Cs^{(a)}$	NR	52.2	NR	52.2	3.92E-4	3.80E-4	103
Al	<13	1,350	NR	1,350	5.00E-2	5.02E-2	100
Cr	<1.6	1,370	NR	1,370	2.63E-2	2.67E-2	99
K	<330	5,660	NR	5,660	1.45E-1	1.46E-1	99
Na	<70	119,000	NR	119,000	5.18E+0	5.00E+0	104
P	<11	317	NR	317	1.02E-2	9.69E-3	106
Cl^-	[0.12]$^{(b)}$	<2.5	<2.5	<2.5	<7.1E-5	0.00E+0	NA
NO_2^-	<0.025	56,200	55,800	56,000	1.22E+0	1.19E+0	102
NO_3^-	<0.025	29,400	29,300	29,350	4.73E-1	4.93E-1	96
PO_4^{3-}	<0.025	1000	928	964	1.02E-2	9.69E-3	105
SO_4^{2-}	<0.025	29,800	29,600	29,700	3.09E-1	3.01E-1	103
OH^-	0	7,580	7,850	7,715	4.54E-1	3.34E-1	**136**$^{(c)}$
C as CO_3^{2-}	NR	11,400	NR	11,400	9.50E-1	8.76E-1	108
Analyte	**Temp., ºC**	**Sample g/mL**	**Duplicate g/mL**	**Average, g/mL**		**Target g/mL**	**% of Target**
Density	22.9	1.239	1.239	1.239		1.237	100

(a) The Cs results are based on the amended formulation.
(b) The chloride result for the preparation blank is bracketed to indicate that the analyte concentration was less than the estimated quantitation limit (EQL) and greater than the instrument detection limit (IDL).
(c) The hydroxide result exceeded the acceptance criterion of ±15% of target.

Notes:

- NR = not required (direct analysis does not require a preparation blank; inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS) metals and carbonate precision were measured with AP-101 simulant duplicates).
- NA = not applicable
- The overall uncertainty for these analytes of interest is ±15%.
- The total organic carbon (TOC) was also determined opportunistically to be 10.9 mg C per mL.

ASR = 7019 and 7031
Noah Technologies Lot # 135136/1.1

Table 3.6. AP-101 Stock 1 Simulant Composition—Major Analytes, Used with Wave 1 through Wave 2a Testing

Analyte	Prep Blank µg/mL	Sample µg/mL	Duplicate µg/ mL	Average µg/mL	Average M	Target M	% of Target
$Cs^{(a)}$	NR	5.94	5.99	5.97	4.48E-5	4.51E-5	99
Al	<13	6,860	6,980	6,920	2.56E-1	2.59E-1	99
Cr	<1.6	149	151	150	2.88E-3	2.92E-3	99
K	<330	27,900	28,400	28,150	7.20E-1	7.10E-1	101
Na	<70	113,000	116,000	114,500	4.98E+0	5.00E+0	100
P	<11	405	410	408	1.32E-2	1.24E-2	106
Cl^-	[0.12]$^{(b)}$	1,170	NR	1,170	3.30E-2	4.09E-2	**81**$^{(c)}$
NO_2^-	<0.025	32,400	NR	32,400	7.04E-1	7.07E-1	100
NO_3^-	<0.025	105,000	NR	105,000	1.69E+0	1.68E+0	101
PO_4^{3-}	<0.025	1,060	NR	1,060	1.12E-2	1.24E-2	90
SO_4^{2-}	<0.025	3,680	NR	3,680	3.83E-2	3.73E-2	103
OH^-	0	30,600	30,800	30,700	1.81E+0	1.94E+0	93
C as CO_3^{2-}	NR	5,500	5,600	5,550	4.63E-1	4.46E-1	104
Analyte	**Temp. ºC**	**Sample g/mL**	**Duplicate g/mL**	**Average g/mL**		**Target g/mL**	**% of Target**
Density	22.9	1.2587	1.2614	1.260		1.26	100

(a) The Cs results are based on the amended formulation.
(b) The chloride result for the preparation blank is bracketed to indicate that the analyte concentration was less than the estimated quantitation limit (EQL) and greater than the instrument detection limit (IDL).
(c) The chloride result exceeded the acceptance criterion of ±15% of target.

Notes:

- NR = not required (direct analysis does not require a preparation blank; IC batch precision was measured with AZ-102 simulant duplicates)
- NA = not applicable
- The overall uncertainty for these analytes of interest is ±15%.
- The total organic carbon (TOC) was also determined opportunistically to be 1 mg C per mL.

ASR = 7019 and 7031
Noah Technologies Lot # 135125/1.1

The hydroxide, anions, and TIC were determined directly on the simulants. Hydroxide was determined using potentiometric titration with standardized HCl according to procedure RPG-CMC-228, *Determination of Hydroxyl (OH^-) and Alkalinity of Aqueous Solutions, Leachates, and Supernates and Operation of Brinkman 636 Auto-Titrator*. The free hydroxide was defined as the first inflection point on the titration curve. Anions were determined using a Dionix 4500 ion chromatograph (IC) system equipped with a pulsed electrochemical detector according to procedure PNL-ALO-212, *Determination of Inorganic Anions by Ion Chromatography*. The TIC was determined by using silver-catalyzed hot persulfate oxidation according to procedure PNL-ALO-381, *Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method*.

Table 3.7. AP-101 Stock 2 Simulant Composition—Major Analytes Used with Wave 2b through Wave 4b Testing

Analyte	Prep Blank µg/mL	Sample µg/mL	Duplicate µg/ mL	Average µg/mL	Average M	Target M	% of Target
Cs	<0.062	5.90	5.82	5.86	4.40E-5	4.51E-5	98
Al	<1.7	6,540	6,675	6608	2.45E-1	2.59E-1	95
Cr	<0.13	143	146	144	2.77E-3	2.92E-3	95
K	<64	26,300	26,800	26,550	6.79E-1	7.10E-1	96
Na	<6.1	110,000	115,000	112,500	4.89E+0	5.00E+0	98
P	<1.7	376	386	381	1.23E-2	1.24E-2	99
Cl^-	<8	1,770	1,790	1,780	5.02E-2	4.09E-2	**123**[(a)]
NO_2^-	<120	33,900	33,400	33,600	7.32E-1	7.07E-1	104
NO_3^-	<150	107,000	106,000	106,500	1.72E+0	1.68E+0	102
PO_4^{3-}	<1.9	1,120	1,090	1,105	1.16E-2	1.24E-2	94
SO_4^{2-}	<12	3,610	3,550	3,580	3.73E-2	3.73E-2	100
OH^-	0	32,000	32,400	32,200	1.89E+0	1.94E+0	98
C as CO_3^{2-}	NA	5,400	5,600	5,500	4.58E-1	4.46E-1	103
Analyte	**Temp. ºC**	**Sample g/mL**	**Duplicate g/mL**	**Average g/mL**		**Target g/mL**	**% of Target**
Density	22	1.251	1.251	1.251		1.26	99.3

(a) The chloride result exceeded the acceptance criterion of ±15% of target.

Notes:
NA = not applicable, samples are blank corrected.
The overall uncertainty for these analytes of interest is ±15%.
ASR = 7273
Noah Technologies Lot # 144354/1.1

Simulant aliquots (~1.0 mL) were acid-digested in duplicate according to procedure PNL-ALO-128, *HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater*. The acid-digested solutions were brought to a nominal 25-mL volume; absolute volumes were determined based on final solution weights and densities. Along with the sample and duplicate, the ASO processed a digestion preparation blank (PB), two blank spikes (BSs) (one for ICP-AES and one for ICP-MS), and two matrix spikes (MSs) (one for ICP-AES and one for ICP-MS). Aliquots of the BS, MS, and PB, along with aliquots of the duplicate samples, were delivered to the ICP-AES and ICP-MS analytical workstations for analyses. The ICP-AES analysis was conducted according to procedure PNNL-ALO-211, *Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry (ICPAES)*. The ICP MS analysis was conducted according to procedure 329-OP-SC01, Rev. 0, *Inductively-Coupled Plasma Mass Spectrometry (ICP/MS) Analysis*.

All measured analytes were in good agreement with the target compositions (meeting the ±15% allowable tolerance), with three exceptions. The first simulant preparations (AP-101 and AZ-102) contained no measurable Cs. A quantity of $CsNO_3$ (dissolved in minimal water) was added in sufficient

quantity to make the final Cs concentration match the target values of 6 mg/L in the AP-101 simulant[a] and 50 mg/L in the AZ-102 simulant. The amount of water added (< 10 mL) was not sufficient to cause measurable dilution of other analytes. Each simulant was thoroughly mixed and resampled for Cs analysis. The Cs re-analysis was conducted according to ASR 7031. Samples were simply diluted before ICP-MS analysis. The hydroxide in the AZ-102 simulant was 35% high. Adjusting the hydroxide concentration by dilution would have decreased one or more cation concentrations (e.g., Na) significantly. The chloride concentration was 19% low in the first AP-101 simulant preparation and 23% high in the second AP-101 simulant preparation. In both cases, the results exceeded the acceptance criterion (±15%); however, the chloride analytical uncertainty (±15%) overlapped into the acceptable target range. It was decided, in conjunction with the R&T lead, to proceed with ion exchange testing with no simulant matrix modification for hydroxide or chloride.

3.13 Quality Assurance and Quality Control

The following sections describe the quality assurance (QA) program and quality control (QC) measures applied to the conduct of work.

3.13.1 Application of BNI-SP Quality Assurance Requirements

PNWD's Quality Assurance Program is based upon the requirements as defined in DOE Order 414.1A, Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A—Quality Assurance Requirements (a.k.a., the Quality Rule). PNWD chose to implement the requirements of DOE Order 414.1A and 10 CFR 830, Subpart A by integrating them into the laboratory's management systems and daily operating processes. The procedures necessary to implement the requirements are documented through PNWD's Standards-Based Management System (SBMS).

PNWD implemented the BNI-SP quality requirements by performing work in accordance with the PNWD *Bechtel National, Inc. Support Program Quality Assurance Program* (QAP). This work was performed to the quality requirements of NQA-1-1989 Part I, Basic and Supplementary Requirements, NQA-2a-1990, Part 2.7 and DOE/RW-0333P, Rev 13, Quality Assurance Requirements and Descriptions (QARD). These quality requirements were implemented through PNWD's *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements were implemented through WTPSP's Statement of Work (WTPSP-SOW-005) with the RPL ASO.

A matrix that cross-references the NQA-1, NQA-2a and QARD requirements with the PNWD's procedures for this work was given in the test plan, TP-RPP-WTP-368.[b] It included justification for those requirements not implemented.

3.13.2 Conduct of Experimental and Analytical Work

Experiments that were not method-specific were performed in accordance with PNWD's procedures QA-RPP-WTP-1101 "Scientific Investigations" and QA-RPP-WTP-1201 "Calibration Control System,"

(a) Note that only the 85-L fraction delivered to the Radiochemical Processing Laboratory (RPL) was amended with the appropriate amount of $CsNO_3$. The fraction delivered for hydraulic testing had no added Cs.

(b) SK Fiskum. June 2004. *Column Performance Testing of Variations Spherical Resorcinol Formaldehyde Resins, Stage 2*. Rev. 0. Battelle—Pacific Northwest Division, Richland, WA.

verifying that sufficient data were taken with properly calibrated measuring and test equipment (M&TE) to obtain quality results.

As specified in the supporting Test Specification, 24590-PTF-TSP-RT-04-0001, Rev. 0, BNI's QAPjP, PL-24590-QA00001, was not applicable because the work was not performed in support of environmental/regulatory testing, and the data will not be used as such.

The applicable QC parameters for chemical analysis were delineated in the test plan, TP-RPP-WTP-368. Blank spike and/or laboratory control sample QC failures would have resulted in re-analyzing the sample for the particular analyte for which the spike failed. Matrix spike and/or duplicate analysis QC failures would not result in reanalyzing the sample, but probable reasons for the failure would be discussed in the analytical report that is stored in the project files.

Analytical processes were performed in accordance with the requirements in WTPSP's Statement of Work (WTPSP-SOW-005). A ^{137}Cs tracer was used to evaluate Cs loading and elution characteristics in the column process tests. Absolute ^{137}Cs concentrations in the effluent samples and residual ^{137}Cs on the resin bed were determined using a GEA system consisting of a multi-channel analyzer and a suitable detector, such as a high-purity germanium detector. Counting was performed according to the procedure *Gamma Energy Analysis (GEA) and Low Energy Photon Spectroscopy (LEPS)*, PNL-ALO-450. The procedure *Routine Research Operations*, RPL-OP-001, was used to control counting of the elution samples. In this case, absolute counting efficiency and energy calibration were not required because the analyses were comparative. The GEA instrument was monitored for consistent operation by counting known volumes of the ^{137}Cs-traced process batch comparator samples both before and after one day's analysis sequence. The instrument background was counted at least once per day the system is used.

Additional measuring equipment that was used included a thermocouple, ruler, clock, and balances. The clock and ruler were standard laboratory equipment for use as indicators only. Balances are calibrated annually by a certified contractor, QC Services, Portland, Oregon. A balance performance check was conducted each day the balance was used. The thermocouple and meter, used for monitoring ambient temperature, are calibrated annually by the PNNL Instrument Calibration Facility.

3.13.3 Internal Data Verification and Validation

PNWD addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNWD's procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the test plan objectives. This review procedure is part of PNWD's *WTPSP Quality Assurance Requirements and Description Manual*.

4.0 Regeneration Refinement

There are two ion exchange processing objectives for the resin regeneration step in the WTP: 1) shift column fluid pH to basic and 2) convert the resin to the Na-form. The solution pH needs to be basic so that aluminates will not precipitate, and carbonate will not convert to carbon dioxide gas in the resin bed once tank waste is introduced. The resin needs to be in the Na-form so that effective ion exchange with Cs will occur. An ancillary objective is to capture and combine the last three regeneration process bed volumes and recycle the composite through the column as 0.1 M NaOH feed displacement solution.

Regeneration with high NaOH molarity would more quickly convert the resin and adjust the solution pH. However, all sodium used in ion exchange column operations eventually contributes to LAW glass production. Additional Na input, beyond the minimum required, simply increases the waste product volume and increases final disposal costs. Therefore, refinement of the regeneration operation with respect to NaOH concentration and solution volume is desirable.

Bray et al. (1990) used 3 BVs 2 M NaOH as the regeneration solution during column testing of ground-gel RF. Fiskum et al. (2004) conducted comparative testing with ground-gel RF using ~6 BVs 0.25 M NaOH as the regeneration solution (as was typically applied to SL-644 regeneration). These tests resulted in small bubble formation within the resin bed during initial AZ-102 simulant processing (volume expansion was <3%). The bubble formation was attributed to insufficient conversion of the resin from the H-form to the Na-form with the 0.25 M NaOH. It was conjectured that the carbonate in the AZ-102 formulation reacted with residual acid to form gaseous carbon dioxide in the resin bed. Successful processing, that is, lack of bubble formation, was observed with the use of 6 to 7 BVs 1 M NaOH regeneration solution. The optimal NaOH concentration in the regeneration solution for ground-gel RF was thought to be 6 BVs of solution with a concentration range of 0.25 M to 1 M NaOH. Subsequent testing with spherical RF used 6 BVs 1 M NaOH regeneration solution with no observed problems (Fiskum et al. 2004).

Specific regeneration solution testing/optimization had not been conducted on the spherical RF resin. This study was needed to better evaluate the lower limit of NaOH molarity for resin regeneration purposes while still meeting the processing objectives. Three systematic single-column tests were conducted to evaluate the resin regeneration responses with 0.25 M NaOH, 0.5 M NaOH, and 0.8 M NaOH.[(a)] Based on these test results, a NaOH concentration and volume for spherical RF in-column regeneration was to be applied to all subsequent testing.

4.1 Experimental

Testing was initiated before Wave 1 Microbead stocks of spherical RF resin arrived. Therefore, spherical RF test resin, ID #6 (Fiskum et al. 2004), was selected from the suite of resins tested under the A-222 scope. Nominally 7.8 g of as-received (dry stored material) was sub-sampled in triplicate for column testing. At the same time, duplicate F-factor samples were taken to determine dry resin mass. The F-factor samples were dried under vacuum at 50°C to constant mass. The F-factor (F) was calculated according to Equation 4.1,

(a) The regeneration testing was conducted according to TI-RPP-WTP-376, Rev. 0, *Determination of Appropriate RF Resin Preconditioning Steps*, SK Fiskum, June, 2004.

$$F = \frac{M_d}{M} \tag{4.1}$$

where M_d is the dry resin mass, and M is the starting resin mass. The average F-factor was determined to be 0.5851 ±0.0016. The dry resin mass loaded in the columns was calculated by multiplying the resin aliquot mass by the F-factor. The nominal dry H-form resin mass tested in each column system was ~4.56 g.

The resin sampled for column testing was pretreated by soaking each aliquot in 108 mL (a 10:1 liquid volume to resin volume) of 1 M NaOH solution for 2 h. The NaOH was decanted, and 50 mL of DI water was added to the wetted resin. No other open-beaker pretreatment was applied.[(a)]

Each Na-form resin slurry was transferred into one of three columns resulting in 18-mL settled resin bed volumes. The fluid heights above the Na-form resin beds were adjusted to 2.5 cm (7.9 mL). The column ion exchange system (shown in Figure 4.1) was a simplified version of the columns used in subsequent test waves in that the column was not separated by quick-disconnects. The fluid mixing volumes at various process sections are also shown. The fluid head volume changed as the resin bed expanded and contracted. After the in-column pretreatment testing, two of the resins reached an expanded volume of 19 mL; however, for the purposes of this test, the 18-mL Na-form resin volume as loaded and settled in the column was used as the 1-BV basis.

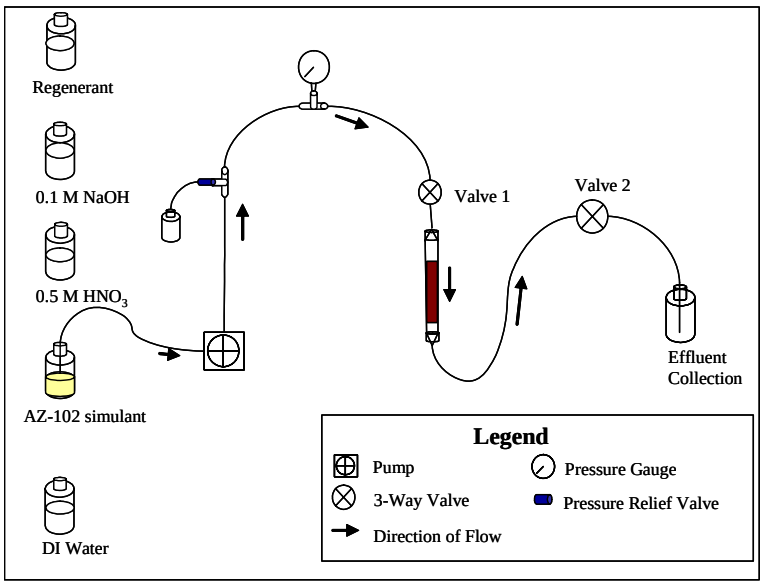


Figure 4.1. Ion Exchange Processing Schematic

Mixing volumes:	Before resin bed	6.0 mL	Resin bed	14 – 19 mL
	Fluid head	7.8 – 13 mL	Fluid below resin bed	8.4 mL

(a) Resin pretreatment and testing were conducted before protocol P1-RF was promulgated.

Each resin bed was pretreated by sequentially rinsing with water, 0.5 M HNO_3, water, and then one of the three concentrations of NaOH. The NaOH solutions were prepared by diluting 10 N NaOH certified solutions (JT Baker, Phillipsburg, NJ).

The RF resin turned dark and expanded as it changed from H-form to Na-form; therefore, the conversion progress could be observed by color and volume change. Resin bed heights and coloration depths were measured during processing. In all cases, the conversion front was flat across the resin bed (i.e., no fingering was observed).

Resin conversion was also assessed from the effluent pH. As the exchange of Na ions for H ions approached completion, the effluent hydroxide concentration approached the influent concentration. The regeneration solution was collected in nominal 1-BV increments. The effluent pH was measured on each increment using an 81-03 ROSS electrode (Thermo Electron Corporation, Beverly, MA) and a Mettler DL21 Titrator (Columbus, OH). The system was operated per manufacturer instructions. The hydroxide concentration in each sample (pH >7) was calculated according to Equation 4.2. Samples with pH >11 were diluted 100× and re-measured.

$$10^{(-14 + pH^{+})} = [OH^{-}] \tag{4.2}$$

After regeneration, ~17 BVs of AZ-102[(a)] simulant were processed. Each resin bed was visually evaluated for in-column bubble formation; none was observed for any of the resin tests. Specific processing parameters for each test column are summarized in Table 4.1.

(a) The AZ-102 simulant processed through the columns had not yet been amended with $CsNO_3$. The Cs-free simulant was not expected to impact the results from this test.

Table 4.1. Preconditioning Testing Process Parameters

Process step	Solution	Total Volume			Flowrate		Time
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h
0.25 M NaOH Pretreatment—4.561 g dry resin mass (H-form)							
Water rinse	DI water	7.97	3.04	143	3.01	0.898	2.65
Acid wash	0.5 M HNO_3	8.19	3.12	147	2.98	0.889	2.75
Water rinse	DI water	3.22	1.23	57.7	1.44	0.431	2.23
Regeneration	0.25 M NaOH	5.91	2.25	106	2.96	0.885	2.00
Loading column	AZ-102 Simulant	16.4	6.23	293	2.91	0.869	5.62
Feed displacement	0.1 M NaOH	3.11	1.19	55.8	3.06	0.914	1.02
Rinse	DI water	3.02	1.15	54.1	3.02	0.902	1.00
0.50 M NaOH Pretreatment—4.563 g dry resin mass (H-form)							
Water rinse	DI water	7.97	3.09	145	3.01	0.914	2.65
Acid wash	0.5 M HNO_3	8.28	3.21	151	3.01	0.915	2.75
Water rinse	DI water	3.20	1.24	58.3	1.42	0.432	2.25
Regeneration	0.5 M NaOH	5.87	2.27	107	2.94	0.894	2.00
Loading column	AZ-102 Simulant	16.57	6.42	302	2.94	0.893	5.63
Feed displacement	0.1 M NaOH	3.10	1.20	56.4	3.10	0.940	1.00
Rinse	DI water	3.06	1.19	55.7	3.06	0.928	1.00
0.80 M NaOH Pretreatment—4.568 g dry resin mass (H-form)							
Water rinse	DI water	8.17	3.11	146	3.38	1.01	2.42
Acid wash	0.5 M HNO_3	8.58	3.27	154	3.08	0.920	2.78
Water rinse	DI water	3.28	1.25	58.8	1.45	0.433	2.27
Regeneration	0.8 M NaOH	6.08	2.32	109	3.05	0.911	2.00
Loading column	AZ-102 Simulant	17.4	6.62	311	3.06	0.912	5.68
Feed displacement	0.1 M NaOH	3.20	1.22	57.3	3.20	0.955	1.00
Rinse	DI water	3.15	1.20	56.5	3.15	0.941	1.00

(a) BV = bed volume (nominally 18 mL in the Na-form volume as loaded in the column).
(b) AV = apparatus volume (nominally 50 mL).
NA = not applicable
NR = not recorded
Process date: 6/17/04

4.2 Results

The changes in NaOH effluent concentration were assessed from a simple mixing model given the assumption no Na is exchanged onto resin and no solution hold-up neutralization is required. This assumed laminar flow (or plug flow) associated with the fluid before the column (0.33 BV, where 1 BV ~18 mL), continuously-stirred mixing volumes in the resin bed and fluid above the column (1.5 BV), and laminar fluid flow below the column (0.47 BV). A graphical presentation of the changing effluent NaOH concentration is shown in Figure 4.2.[(a)] With no Na exchange or acid neutralization, the effluent NaOH concentration would have equilibrated with the influent NaOH concentration after processing ~4 BVs.

(a) The mixing modeling data were provided by the R&T lead, MR Thorson.

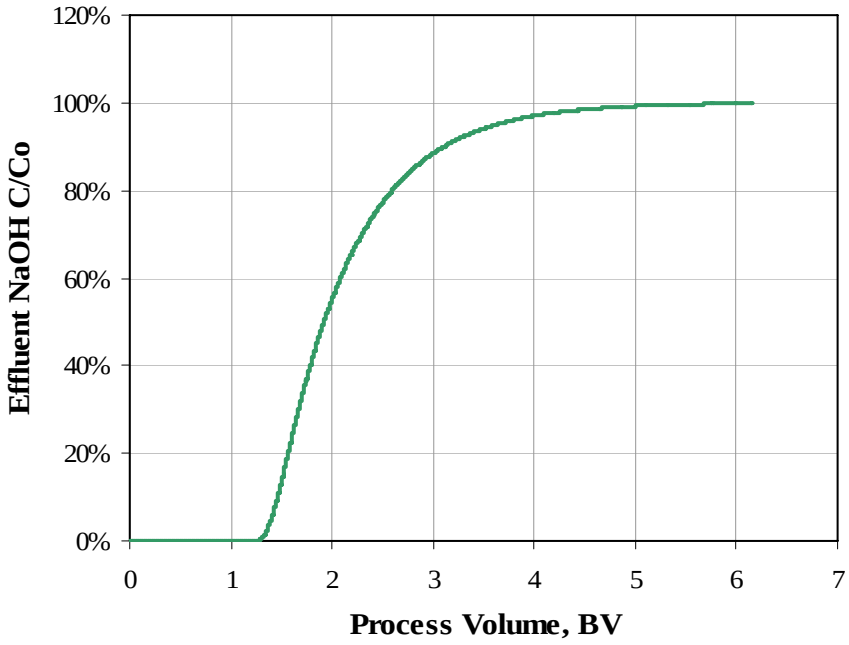


Figure 4.2. Theoretical Process Volume to Equilibrated Effluent—No Sodium Exchange

Two measures of the conversion process to the Na-form for the three regeneration conditions are shown in Figure 4.3. The visual bed conversion (depth of black Na-form resin) is provided as a function of processed BVs. The more quickly the Na-form fraction reached the full resin height, the more quickly conversion was obtained. The effluent pH or hydroxide concentration (calculated from the measured pH) as a function of process volume is also shown.

Six BVs 0.25 M NaOH was insufficient to fully convert the resin bed to the Na form. The 0.25 M NaOH feed converted the resin slowly as noted by the slow resin color change. The fully converted resin bed height should be 6.1 cm; after processing 5.9 BVs 0.25 M NaOH, only 5.5 cm expansion was obtained. Only the final 1-BV increment effluent sample at pH 11.8 was basic (thus, the effluent pH is provided on the secondary y-scale instead of hydroxide concentration). The sum of the final 3 BV increments would have resulted in a combined hydroxide concentration of <0.001 M. There was no observed bubble formation in the column with processing the AZ-102 simulant. Thus, the resin contact solution appeared to have been shifted to caustic sufficient to avoid reaction (CO_2 evolution) with simulant.

The 0.5 M NaOH feed converted the resin more quickly to the Na-form with full resin expansion observed after processing ~4.5 BVs. The effluent pH became basic starting with the fourth process BV, and it continued to rise through the completion of regeneration. The combined last three BVs of regeneration solution were calculated to result in a hydroxide concentration of 0.14 M. This concentration would provide an appropriate feed displacement medium.

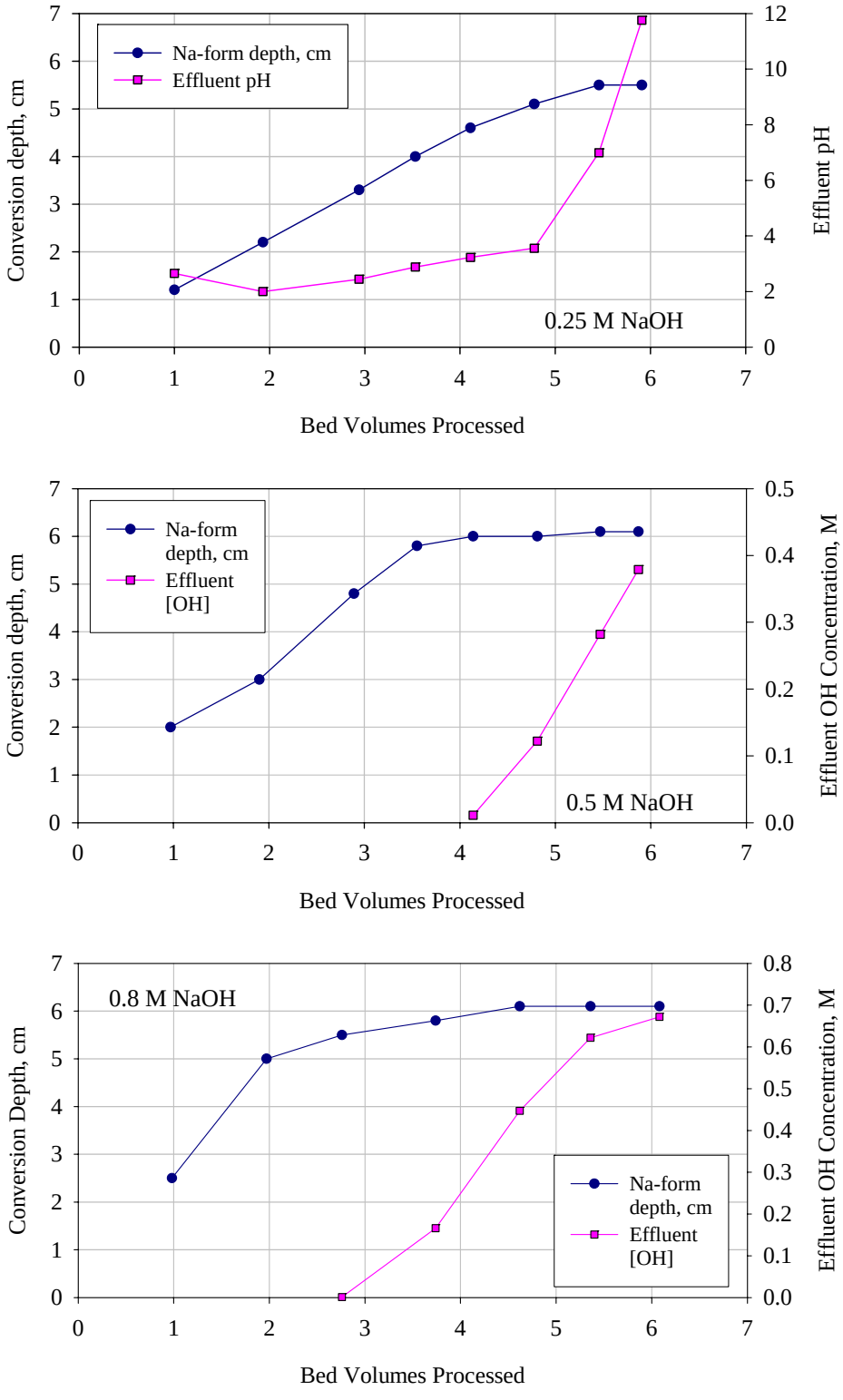


Figure 4.3. Resin Conversion to Na-Form with 0.25 M, 0.50 M, and 0.80 M NaOH (Conversion Fronts and Effluent Hydroxide Concentrations)

Notes: Fluid BV from feed to column: 0.33; in column: 1.5; from column support to exit line: 0.47.

The 0.8 M NaOH feed resulted in a more abrupt color change; however, the maximum bed height was not observed until nearly 4.5 BVs had been processed, virtually equivalent to the processing with 0.5 M NaOH. The effluent became basic more quickly where pH 7 was exceeded after processing 2.7 BVs. Within the uncertainty of the analysis method (pH measurement), the last two BV effluent hydroxide concentrations were equivalent to that of the feed. The combined final 3 BVs of regeneration solution were calculated to result in 0.45 M hydroxide. This concentration was 4.5× that required for the feed displacement concentration.

The resin conversion process to the Na-form was time dependent as well as NaOH concentration dependent. The total resin BV as a function of processed volume over the entire processing cycle is shown in Figure 4.4 for all conditions tested; BV similarities and differences between different regeneration solution uses are apparent. The 0.5 M and 0.8 M NaOH regeneration solutions resulted in full resin expansion after processing ~4.5 BVs (about the same expansion rate). The similarity in expansion rates supported the concept that the RF conversion is, in part, particle diffusion-rate limited. That is, higher (>0.8 M) NaOH concentrations would not be expected to more rapidly convert the resin to Na form. The 6 BVs of 0.25 M NaOH resulted in 90% maximum expansion and a slower expansion rate. The 0.8 M NaOH regeneration solution resulted in complete resin bed darkening sooner than that of the 0.5 M NaOH solution; however, resin expansion (conversion) still continued. This indicated that the resin surface had been converted to the Na-form, but the intra-particle diffusion and conversion were still continuing. These results are consistent with the expansion/conversion test reported in Section 14.1 with TI394-64 (MB 5J-370/686) resin. In this case, resin swelling required a minimum of 60 min contact time before the swelling rate decreased, and increased Na concentration (from 0.5 to 0.6 M) only slightly increased the swelling rate.

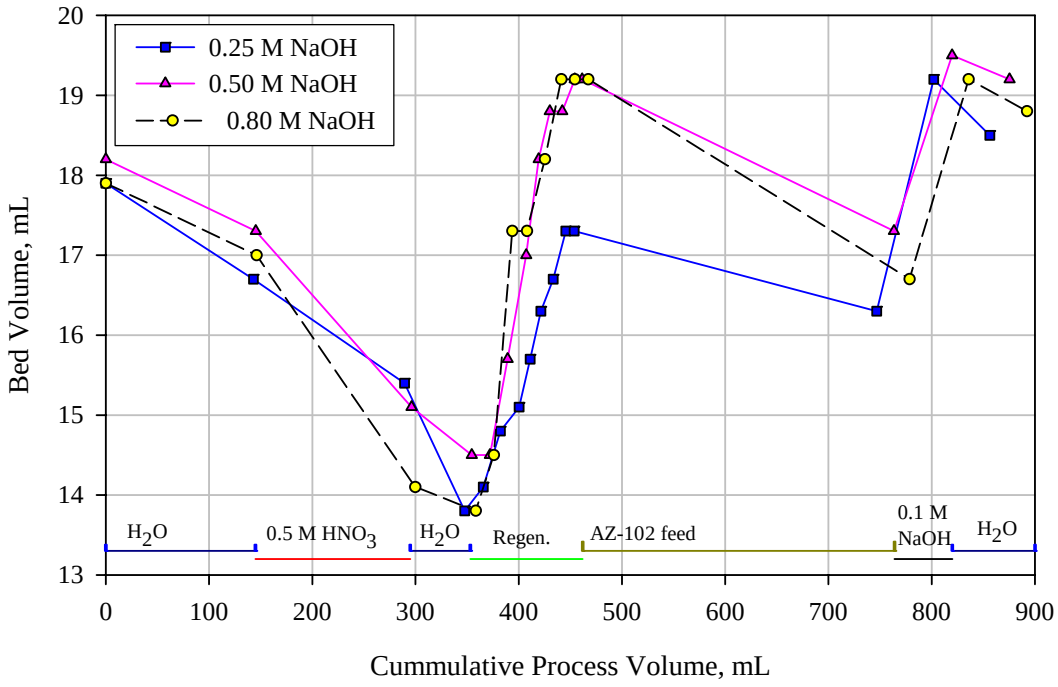


Figure 4.4. Resin Bed Volume Changes as a Function of Process Step

The spherical RF resin conversion using 6 BVs of 0.5 M NaOH was selected as the baseline for all subsequent process testing. Conversion was considered sufficiently complete for follow-on testing, and the combined final 3 BVs of effluent met the plant requirement for use as feed displacement. These parameters may be affected by specific peculiarities of the ion exchange process system. In the current test case, the resin BV represented a nominal 40% of the entire apparatus volume. Changes in the fluid volume above the resin bed and the AV relative to the resin BV may affect the NaOH dilution/ neutralization rates. Compensation for these factors should be evaluated through engineering design.

5.0 Wave 1 Resin Testing

All Wave 1 RF resins were derived from a single batch of seed material prepared by SINTEF (Trondheim, Norway). The seeds were prepared and then subdivided for final functionalization steps where RF polymerization occurs. PNWD, in conjunction with BNI, delineated four different production parameters to apply to the RF polymerization steps. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates, and receipt dates are summarized in Table 5.1. Variations in the molar ratio of resorcinol to formaldehyde, reaction temperature, reaction time, and monomer excess[(a)] were investigated; however, the specific RF processing and polymerization parameters are confidential and were provided directly to the R&T lead in a separate communication from the vendor. The BRF-18 resin spheres were prepared from seeds that cracked during the preparation process.

Table 5.1. Wave 1 Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
BRF-14[(a)]	SINTEF	BRF-14	5 x ~100 g	5/04	7/19/04
BRF-15	SINTEF	BRF-15	5 x ~100 g	5/04	7/19/04
BRF-16	SINTEF	BRF-16 A, B, C	3 x ~100 g	5/04	7/19/04
BRF-17	SINTEF	BRF-17	5 x ~100 g	5/04	7/19/04
BRF-18	SINTEF	BRF-18	5 x ~100 g	5/04	7/19/04
SL-644	IBC Advanced Technology	C-01-11-05-02-35-60	250-gal	11/02	NA
(a) BRF-14 was synthesized under similar conditions as Resin #3 (Fiskum et al. 2004).					

The reaction chamber capacity at SINTEF was restricted relative to the resin production batch size. To create the large volumes (450 g) required for testing, each resin lot production was conducted in five batches except for BRF-16, which only required three production batches. After SINTEF verified consistency between the five batches, the products were combined into the lots shown in Table 5.1. In the case of BRF-16, each batch production resulted in a slightly different particle size; SINTEF chose not to combine these resin batches and submitted them individually (BRF-16A, BRF-16B, and BRF-16C).

Photographs of the as-received resins are provided in Figure 5.1 and Figure 5.2. The BRF-16 resin was noticeably different with a flesh color compared to the maroon colors of the other resins. Because resin BRF-16 was received as three batches (A, B, and C), a test aliquot was obtained by sub-sampling equal portions from each bottle using the core-sampling technique and combining the sample fractions into a composite.

(a) The monomer excess refers to the amount of resorcinol-formaldehyde reacted with the seeds beyond the theoretical maximum required to fill the swollen seed.

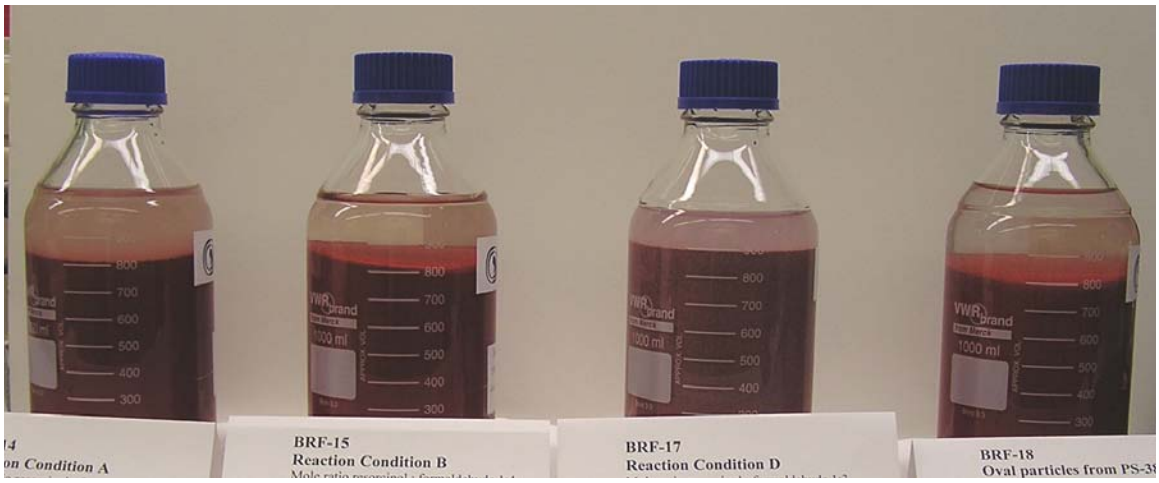


Figure 5.1. Wave 1 Test Resins, As-Received BRF-14, -15, -17, and -18

Figure 5.2. Wave 1 Test Resin, As-Received BRF-16A, -16B, -16C

Two 1-L aliquots of SL-644 from IBC production batch C-01-11-05-02-35-60 (also known as the 250-gal production batch) was received at PNWD from SRTC. The resin had been pretreated at SRTC with batch contacts of water, followed by shrinking in two batch contacts of 0.5 M HNO_3, DI water wash, and re-expansion in two batch contacts of 0.25 M NaOH, and then washed with DI water (Fowley et al. 2003). The resin was then wet-sieved three times through a 38 Tensile Bolt Cloth (TBC) screen at SRTC to provide sample material between 40 mesh and 18 mesh (U.S. Standard mesh sizes), equivalent to resin particles ranging from 425 to 1000 microns with ≤1% by mass of <425 microns and ≤1% by mass >1000 microns. All pretreatment and wet sieving were conducted June–October 2003. The bulk of this material had been used for the 24-in. "all-in" column test reported by Fowley et al. (2004). The two 1-L aliquots PNWD received had not been used in the "all-in" test. The resin was received in 1-L plastic bottles; less than 1 cm water depth covered the resin. No additional pretreatment was conducted on the resin; it was loaded into the column as-received.

5.1 Wave 1 Experimental Specifics

Resin pretreatment and processing evolved as a result of testing. Section 3.0 defines generically the physical property testing, calculations, and data evaluations. This experimental section better defines the specific Wave 1 processing parameters because they were slightly different from the other test waves.

5.1.1 Pretreatment and Sub-Sampling for Wave 1 RF Resins

Wave 1 resin pretreatment used a simple water wash before column loading.[(a)] The pretreatment process volumes discussed below were nominal volumes of settled resin in graduated cylinders; exact (±0.2 mL) volumes were measured. The settled resin bed was determined as the final constant volume obtained by tapping the graduated cylinder with a bung.

Wave 1 resins were processed and split for physical property testing and column performance testing as shown in Figure 5.3.[(b)] The Wave 1 resins were washed with DI water in 1-RV increments for a total of four washes. The first, second, and fourth RV water washes were allowed to contact the resin for 1 to 2 h; the third water wash was allowed to contact the resin for nominally 24 h. After the water washes were complete, the resin aliquot was sub-divided into the appropriate processing fractions as shown.

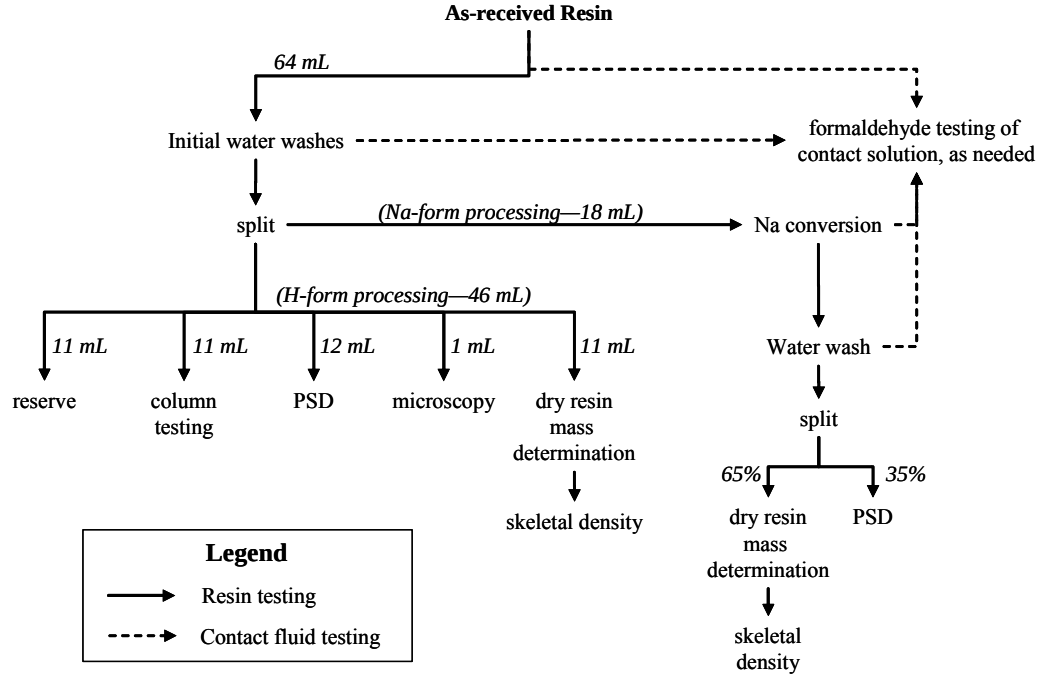


Figure 5.3. Sample Pretreatment and Splitting for Physical Property and Column Performance Testing (Wave 1)

(a) For Wave 1b and subsequent waves, resin pretreatment used a full expansion and contraction cycle in an open beaker format, consistent with the then newly approved protocol, P1-RF, *Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column,* CA Nash, SRNL-RPP-2004-00058, Savannah River National Laboratory, 2004.

(b) Resin processing was conducted according to TI-RPP-WTP-375, Rev. 0, *Physical Properties Evaluation of Spherical RF Resins*, SK Fiskum, 2004.

The Na conversion was accomplished by contacting the resin aliquot with two successive 10 RVs of 1 M NaOH. Each solution was allowed to contact the resin for nominally 2 h. The resin was then washed with two successive contacts of 10 RVs DI water. The first water wash contacted the resin for nominally 2 h; the second water wash contacted the resin for nominally 5 days. The water was removed, and the resin was placed in a final 100-mL volume of storage water. The pH was determined for the second and final water wash solutions using a ROSS pH electrode (Thermo Electron Corp., Beverly, MA). The expanded Na-form resin volumes in water were measured in a graduated cylinder.

5.1.2 Pretreatment for Wave 1 SL-644 Resin

The SL-644 provided by SRNL from Lot C-01-11-05-02-35-60 (250-gal production batch) had already been pretreated and converted to the Na-form at SRNL. An appropriate volume was loaded directly into the column. It was pretreated in-column similarly to the RF resin, except it was regenerated with 0.25 M NaOH.

5.1.3 SL-644 H-form Mass Basis

The RF resin Cs loading and elution performance was normalized to the dry H-form resin mass. To compare the RF resin performance with the SL-644 resin performance, the SL-644 H-form mass basis needed to be determined. The SL-644 was received in the wet Na-form from IBC via SRNL; a known volume of the Na-form resin was measured directly for loading into the ion exchange column (no out-of-column pretreatment). The dry H-form SL-644 resin mass represented in the ion exchange column was estimated from the volumes and masses associated with the SL-644 skeletal density determinations.

Aliquots of water-rinsed Na-form resin taken for Na-form skeletal density were first measured as wet settled volumes and then taken to dryness. Another 48-mL aliquot of water-rinsed Na-form resin was converted to H-form resin, resulting in a settled volume of 28.5 mL. A 21.0-mL volume of the H-form resin was taken to dryness (7.36 g) in preparation for the skeletal density measurement. The settled resin bed densities were calculated and are given in Table 5.2.

Table 5.2. SL-644 Settled Resin Bed Densities

Resin form	Settled Resin Volume, mL	Dried Resin Mass, g	Resin Density, g/mL[(a)]
Na form	16.0	4.2014	0.263
Na-form duplicate	19.8	5.3638	0.271
H-form	21.0	7.3490	0.350
(a) Dry resin mass per wet settled resin volume.			

The mass increase factor (I_{Na}), associated with the resin conversion from H-form to Na-form, was calculated to be 1.28 using the densities and volume relationship according to Equation 5.1.

$$I_{Na} = \frac{V_{Na} \bullet \delta_{Na}}{V_H \bullet \delta_H} \tag{5.1}$$

where V_{Na} = volume of Na-form resin (48 mL)
δ_{Na} = average density of Na-form resin (0.267 g/mL)
V_H = volume of H-form resin (28.5 mL)
δ_H = density of H-form resin (0.350 g/mL)
I_{Na} = mass increase factor (g Na-form resin / g H-form resin.

The H-form mass loaded into the ion exchange column, 4.21 g, was calculated from the measured Na-form volume transferred to the ion exchange column and the duplicate Na-form volume that was taken to dryness according to Equation 5.2.

$$M_{HIX} = \frac{V_{IX} \bullet M_A}{V_A} \bullet \frac{1}{I_{Na}} \tag{5.2}$$

where V_{IX} = volume of Na-form SL-644 loaded into the ion exchange column (19.9 mL)
M_A = dry mass of Na-form resin aliquot (5.3638 g)
V_A = volume of Na-form resin aliquot taken to dryness (19.8 mL)
M_{HIX} = dry H-form SL-644 resin mass in the ion exchange column.

5.1.4 Formaldehyde Testing

Formaldehyde is one of the significant components in RF resin. It was anticipated that some amount of free formaldehyde would exist in the delivered product. Figure 5.3 notes liquid sampling points for formaldehyde testing. Formaldehyde testing was conducted using the Formaldehyde Test Kit (Merck, Darmstadt, Germany) per the manufacturer's instructions for "indication only." In many cases, dilutions were made using DI water to bring the dip-stick reading into the applicable colorimetric range of 0 to 100 μg/g. A small portion of the as-received contact solution was checked for formaldehyde. Each wash solution of DI water was checked at the end of the nominal 2-h contact time. The third wash was again checked at the end of the 24-h contact time. The 1 M NaOH contact solutions were similarly evaluated.

5.1.5 Optical Microscopy

The dried H-form resins forwarded for microscopy testing are shown in Figure 5.4 to delineate the contrasting resin colors.

The resins exhibited different tendencies during cleaving. BRF-14 did not cleave well; only ~1 out of 15 beads produced a center split. BRF-16 was soft and cleaved easily; the outer layer was delicate and de-laminated easily. BRF-18 contained a mixture of dark red and orange beads; the red beads were considerably more brittle than the orange ones.

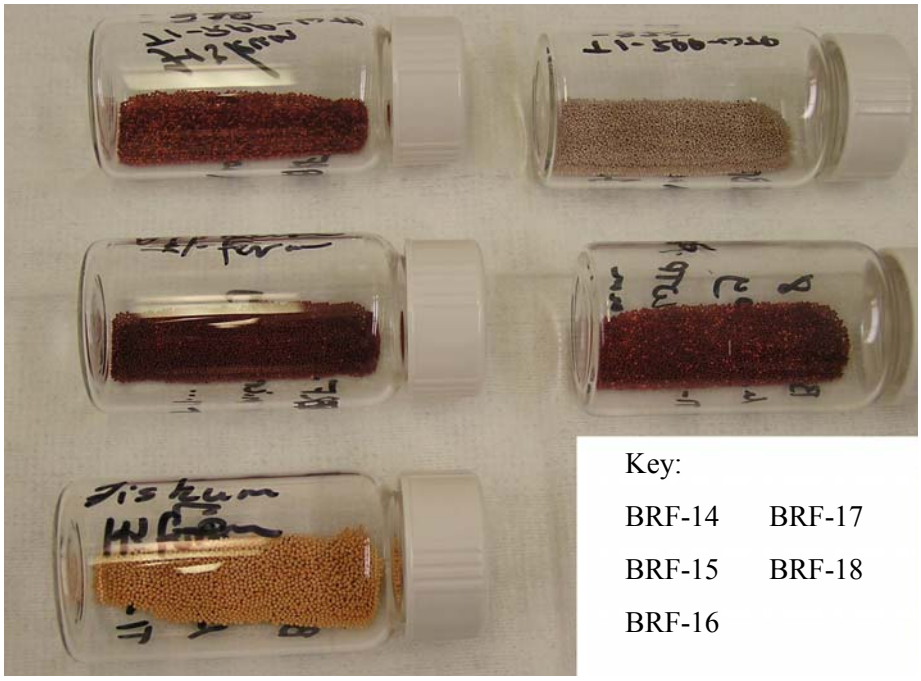


Figure 5.4. Dried H-Form Resins from Wave 1 Submitted for Optical Microscopy

5.1.6 Skeletal Density

The H-form BRF-15 dried resin aliquot released massive amounts of bubbles when water was added (see Figure 5.5). The other resins in Wave 1 were not noted to produce these bubbles. The bubbles had to be knocked out of place with a stirrer to aid in releasing them from the resin.

Figure 5.5. BRF-15 H-Form Resin in Pycnometer Showing Bubble Release from Resin

5.1.7 Column Testing

Wave 1 column testing is summarized in Table 5.3. The first four formulations of RF resin were tested with AP-101 simulant in a single load and elute cycle. The worst-performing resin (BRF-16) was eliminated after the first cycle, and SL-644 resin was placed in the test apparatus. Thus side-by-side comparative testing with SL-644 resin (two cycles) and RF resin (three cycles) was conducted. The BRF-14 resin was also tested with AZ-102 simulant to compare results from the previous test (Fiskum et al. 2004).

Table 5.3. Wave 1 Column Test Summary

	AP-101		AZ-102	
Cycle #	**Resins**	**Column Color Code**	**Resin**	**Column Code**
Cycle 1	BRF-14, -15, -16, -17	yellow, green, blue, pink	BRF-14	white
Cycle 2	BRF-14, -15, -17, SL-644	yellow, green, pink, blue	BRF-14	white
Extended regeneration[(a)]	BRF-14, -15, -17, SL-644	yellow, green, pink, blue	BRF-14	white
Cycle 3	BRF-14, -15, -17, SL-644	yellow, green, pink, blue	BRF-14	white
(a) Oxygen-saturated regeneration solution process test.				

In preparation for column testing, an 11-mL aliquot of pretreated resin was soaked in a 10:1 volume ratio 1 M NaOH to resin for 2 h. The resin was then cycled once in-column before simulant processing. Table 5.4 through Table 5.9 summarize the specific processing conditions for the ion exchange tests. A long (~560 BV) oxygen-rich regeneration test was conducted between Cycle 2 and 3 to evaluate the effects of oxidative attack. Results from this test are provided in Section 6.0.

Table 5.4. Experimental Conditions for BRF-14 (Yellow Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-Situ Preconditioning (7/19/04)								
Water rinse	DI water	8.25	3.20	150	3.17	0.964	2.60	24
Acid wash	0.5 M HNO_3	8.71	3.38	159	3.17	0.962	2.75	24
Water rinse	DI water	3.36	1.30	61.2	1.68	0.510	2.00	23
Cycle 1 (Start 7/20/04)								
Regeneration	0.5 M NaOH	6.86	2.66	125	3.10	0.940	2.17	23
Loading column	AP-101 Simulant	68.7	NA	1252	1.48	0.450	45.5	22-25
Loading column	AP-101 Simulant	75.0	NA	1366	3.20	0.973	23.7	22-28
Feed displacement	0.1 M NaOH	3.14	1.22	57.3	3.14	0.955	1.00	23
Rinse	DI water	2.88	1.12	52.5	2.98	0.906	0.97	24
Elution	0.5 M HNO_3	21.4	NA	389	1.40	0.424	15.5	23
Rinse	DI water	3.09	1.20	56.2	1.38	0.420	2.23	24
Cycle 2 (Start 7/30/04)								
Regeneration	0.5 M NaOH	6.29	2.44	115	3.15	9.55	2.00	23
Loading column	AP-101 Simulant	90.1	NA	1642	1.47	0.446	60.5	22-25
Loading column	AP-101 Simulant	56.9	NA	1037	3.10	0.940	18.6	22-25
Feed displacement	0.1 M NaOH	3.18	1.23	57.9	3.08	0.934	1.03	24
Rinse	DI water	3.16	1.23	57.6	2.87	0.873	1.10	24
Elution	0.5 M HNO_3	21.1	NA	385	1.38	0.419	15.3	23
Rinse	DI water	3.22	1.25	58.7	1.36	0.414	2.37	23
Cycle 3 (Start 8/6/04)								
Regeneration (O_2)	0.5 M NaOH	522	NA	9500	3.06	0.930	170	22-27
Loading column	AP-101 Simulant	82.2	NA	1498	1.50	0.455	54.2	23-28
Loading column	AP-101 Simulant	48.3	NA	880	3.02	0.917	16.0	23-28
Feed displacement	0.1 M NaOH	3.26	1.26	59.4	3.10	0.943	1.05	25
Rinse	DI water	2.94	1.14	53.6	2.80	0.851	1.05	25
Elution	0.5 M HNO_3	22.7	NA	413	1.42	0.430	16.1	22
Rinse	DI water	3.18	1.23	58.0	1.47	0.446	2.17	27

(a) BV = bed volume (18.2 mL in the Na-form).
(b) AV = apparatus volume (47 mL).
NA = not applicable

Table 5.5. Experimental Conditions for BRF-15 (Green Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (7/19/04)								
Water rinse	DI water	8.75	3.39	159	3.60	1.09	2.43	24
Acid wash	0.5 M HNO_3	8.64	3.35	158	3.50	1.06	2.47	24
Water rinse	DI water	3.37	1.31	61.4	2.11	0.639	1.60	23
Cycle 1 (Start 7/20/04)								
Regeneration	0.5 M NaOH	6.49	252	118	3.45	1.05	1.88	23
Loading column	AP-101 Simulant	71.9	NA	1309	1.58	0.480	45.8	22-25
Loading column	AP-101 Simulant	80.1	NA	1461	3.44	1.04	23.4	22-28
Feed displacement	0.1 M NaOH	3.05	1.18	55.6	3.27	0.993	0.93	23
Rinse	DI water	3.05	1.18	55.6	3.05	0.927	1.00	24
Elution	0.5 M HNO_3	22.0	NA	400	1.42	0.432	15.5	23
Rinse	DI water	3.09	1.20	56.2	1.39	0.423	2.22	24
Cycle 2 (Start 7/30/04)								
Regeneration	0.5 M NaOH	7.05	2.73	128	3.31	1.00	2.13	23
Loading column	AP-101 Simulant	88.6	NA	1615	1.45	0.441	60.6	22-25
Loading column	AP-101 Simulant	59.9	NA	1091	3.23	0.981	18.3	22-25
Feed displacement	0.1 M NaOH	3.15	1.22	57.4	3.05	0.925	1.03	24
Rinse	DI water	3.14	1.22	57.1	2.85	0.866	1.10	24
Elution	0.5 M HNO_3	21.3	NA	389	1.39	0.423	15.3	23
Rinse	DI water	3.27	1.27	59.6	1.38	0.420	2.37	23
Cycle 3 (Start 8/6/04)								
Regeneration (O_2)	0.5 M NaOH	566	NA	10,296	3.34	1.01	169	22-27
Loading column	AP-101 Simulant	82.0	NA	1494	1.49	0.454	54.7	23-28
Loading column	AP-101 Simulant	54.8	NA	998	3.18	0.964	17.1	23-28
Feed displacement	0.1 M NaOH	3.23	1.25	58.8	3.02	0.918	1.07	25
Rinse	DI water	2.99	1.16	54.4	2.85	0.864	1.05	25
Elution[(c)]	0.5 M HNO_3	27.2	NA	495	1.48	0.451	18.3	22
Rinse	DI water	3.39	1.31	61.7	1.56	0.475	2.17	27

(a) BV = bed volume (18.2 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Column accidentally ran dry during elution after processing 22 BVs of eluate resulting in significant air gaps in the resin bed. No adverse effect to data analysis and interpretation was expected.
NA = not applicable

Table 5.6. Experimental Conditions for BRF-16 (Blue Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (7/19/04)								
Water rinse	DI water	8.19	3.28	154	3.07	0.965	2.67	24
Acid wash	0.5 M HNO_3	9.08	3.64	171	3.03	0.951	3.00	24
Water rinse	DI water	3.32	1.33	62.6	1.59	0.501	2.08	23
Cycle 1 (Start 7/20/04)								
Regeneration	0.5 M NaOH	6.40	2.57	121	3.02	0.949	2.12	23
Loading column	AP-101 Simulant	70.2	NA	1324	1.52	0.478	45.8	22-25
Feed displacement	0.1 M NaOH	3.01	1.21	56.7	2.91	0.915	1.03	23
Rinse	DI water	3.29	1.32	62.0	3.03	0.953	1.08	23
Elution	0.5 M HNO_3	21.8	NA	410	1.43	0.450	15.2	23
Rinse	DI water	3.14	1.26	59.2	1.42	0.445	2.22	24

(a) BV = bed volume (18.8 in mL Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable

Table 5.7. Experimental Conditions for SL-644 (Blue Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (7/29/04)								
Water rinse	DI water	7.67	3.23	152	3.07	1.01	2.50	23
Acid wash	0.5 M HNO_3	8.29	3.49	164	2.94	0.971	2.82	23
Water rinse	DI water	2.95	1.24	58.5	1.26	0.415	2.35	24
Cycle 1 (Start 7/29/04)								
Regeneration	0.25 M NaOH	5.90	2.49	117	2.90	0.957	2.03	25
Loading column	AP-101 Simulant	89.1	NA	1763	1.38	0.455	64.2	22-26
Loading column	AP-101 Simulant	133	NA	2634	2.90	0.956	45.8	22-26
Feed displacement	0.1 M NaOH	3.00	1.18	59.4	2.91	0.959	1.03	22
Rinse	DI water	2.90	1.03	57.4	2.85	0.941	1.02	23
Elution	0.5 M HNO_3	20.8	NA	412	1.31	0.430	16.0	23
Rinse	DI water	3.06	1.29	60.6	1.35	0.446	2.27	23
Cycle 2 (Start 8/6/04)								
Regeneration (O_2)	0.25 M NaOH	329	NA	6511	2.90	0.956	113.5	22-26
Loading column	AP-101 Simulant	71.3	NA	1411	1.41	0.466	49.0	22-25
Loading column	AP-101 Simulant	55.3	NA	1094	2.92	0.963	19.0	22-25
Feed displacement	0.1 M NaOH	3.15	1.33	62.4	3.00	0.991	1.05	22
Rinse	DI water	3.07	1.29	60.8	2.93	0.965	1.05	22
Elution	0.5 M HNO_3	21.3	NA	421	1.36	0.449	15.6	22
Rinse	DI water	2.89	1.22	57.3	1.36	0.447	2.13	22

(a) BV = bed volume (19.8 mL in Na form).
(b) AV = apparatus volume (47 mL).
NA = not applicable

Table 5.8. Experimental Conditions for BRF-17 (Pink Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (7/19/04)								
Water rinse	DI water	9.03	3.56	167	3.47	1.07	2.60	24
Acid wash	0.5 M HNO_3	9.14	3.61	170	3.31	1.02	2.77	24
Water rinse	DI water	3.20	1.26	59.3	1.67	0.516	1.92	23
Cycle 1 (Start 7/20/04)								
Regeneration	0.5 M NaOH	6.38	2.52	118	3.06	0.947	2.08	23
Loading column	AP-101 Simulant	68.9	NA	1277	1.50	0.464	45.8	22-25
Loading column	AP-101 Simulant	75.3	NA	1396	3.15	0.974	23.8	22-28
Feed displacement	0.1 M NaOH	2.96	1.17	54.8	3.17	0.979	0.93	23
Rinse	DI water	3.02	1.19	55.9	3.02	0.932	1.00	24
Elution	0.5 M HNO_3	21.6	NA	400	1.42	0.438	15.2	23
Rinse	DI water	3.07	1.21	56.9	1.41	0.434	2.18	24
Cycle 2 (Start 7/30/04)								
Regeneration	0.5 M NaOH	11.4[c]	4.51	212	3.05	0.942	3.75	23
Loading column	AP-101 Simulant	87.3	NA	1619	1.45	0.449	59.0	22-25
Loading column	AP-101 Simulant	54.4	NA	1008	3.00	0.927	17.9	22-25
Feed displacement	0.1 M NaOH	3.48	1.37	64.5	3.26	1.01	1.07	25
Rinse	DI water	3.11	1.23	57.6	2.92	0.901	1.07	24
Elution	0.5 M HNO_3	20.7	NA	384	1.36	0.419	14.6	23
Rinse	DI water	3.07	1.21	56.8	1.34	0.415	2.28	23
Cycle 3 (Start 8/6/04)								
Regeneration (O_2)	0.5 M NaOH	442	NA	8173	2.60	0.803	170	22-27
Loading column	AP-101 Simulant	78.3	NA	1452	1.42	0.439	54.7	23-28
Loading column	AP-101 Simulant	49.5	NA	917	2.79	0.861	17.6	23-28
Feed displacement	0.1 M NaOH	3.49	1.38	64.7	3.08	0.951	1.13	25
Rinse	DI water	3.06	1.21	56.8	2.83	0.874	1.08	25
Elution	0.5 M HNO_3	23.3	NA	432	1.30	0.420	17.1	22
Rinse	DI water	3.01	1.19	55.9	1.40	0.433	2.15	27

(a) BV = bed volume (18.5 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) The regeneration volume was larger than nominal plant operating conditions. Color striations indicated that channeling may be occurring, and the larger volume was required to visibly convert the resin to the dark Na form.
NA = not applicable

Table 5.9. Experimental Conditions for BRF-14 in AZ-102 Simulant (White Column) Wave 1

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (7/19/04)								
Water rinse	DI water	9.08	3.52	165	4.16	1.26	2.18	24
Acid wash	0.5 M HNO_3	9.09	3.52	166	3.25	0.986	2.80	24
Water rinse	DI water	3.45	1.34	62.8	2.01	0.609	1.72	23
Cycle 1 (Start 7/20/04)								
Regeneration	1 M NaOH	6.53	2.53	119	3.21	0.975	2.03	23
Loading column	AZ-102 Simulant	173.8	NA	3166	1.50	0.457	115.9	22-28
Feed displacement	0.1 M NaOH	2.98	1.16	54.4	3.03	0.921	0.98	23
Rinse	DI water	3.08	1.19	56.1	2.98	0.905	1.03	23
Elution	0.5 M HNO_3	20.9	NA	381	1.35	0.410	15.5	23
Rinse	DI water	3.05	1.18	55.6	1.38	0.418	2.22	24
Cycle 2 (Start 7/29/04)								
Regeneration	1 M NaOH	6.29	2.44	115	3.04	0.924	2.07	23
Loading column	AZ-102 Simulant	177	NA	3230	1.52	0.462	117.6	22-26
Feed displacement	0.1 M NaOH	3.10	1.20	56.5	2.39	0.724	1.30	23
Rinse	DI water	2.91	1.13	53.1	2.86	0.870	1.02	23
Elution	0.5 M HNO_3	21.1	NA	384	1.43	0.434	14.7	23
Rinse	DI water	3.20	1.24	58.3	1.42	0.432	2.25	23
Cycle 3 (Start 8/6/04)								
Regeneration	1 M NaOH	523	NA	9526	3.07	0.934	170	22-27
Loading column	AZ-102 Simulant	167	NA	3044	1.49	0.452	111.9	23-28
Feed displacement	0.1 M NaOH	3.26	1.26	59.4	2.92	0.886	1.12	23
Rinse	DI water	3.34	1.30	60.9	3.19	0.967	1.05	22
Elution	0.5 M HNO_3	22.2	NA	405	1.30	0.394	17.1	22
Rinse	DI water	3.00	1.16	54.6	1.39	0.423	2.15	22

(a) BV = bed volume (18.2 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable

Column processing through the RF resins resulted in generally sharp conversion fronts (H-form to Na-form and vice versa) with a few exceptions. The BRF-16 resin demonstrated evidence of channeling during the Na-conversion pretreatment and elution steps. BRF-17 demonstrated channeling during the Cycle 2 regeneration process. The observed channeling from conversion fronts may be associated with channeling during the simulant feed and Cs ion exchange (which is discerned visually). After the extended oxidative regeneration process, the elution step demonstrated un-even resin conversion or fingering. Photographs of the various resins in process are shown in Figure 5.6 through Figure 5.9. The photographs were selected such that the transition zones and fingering can be observed.

a) Pretreatment, H-conversion b) Elution (Cycle 2) c) Elution (Cycle 3)

Figure 5.6. BRF-14 During Various Process Stages

a) Pretreatment, H-conversion b) Elution (Cycle 2) c) Elution (Cycle 3)

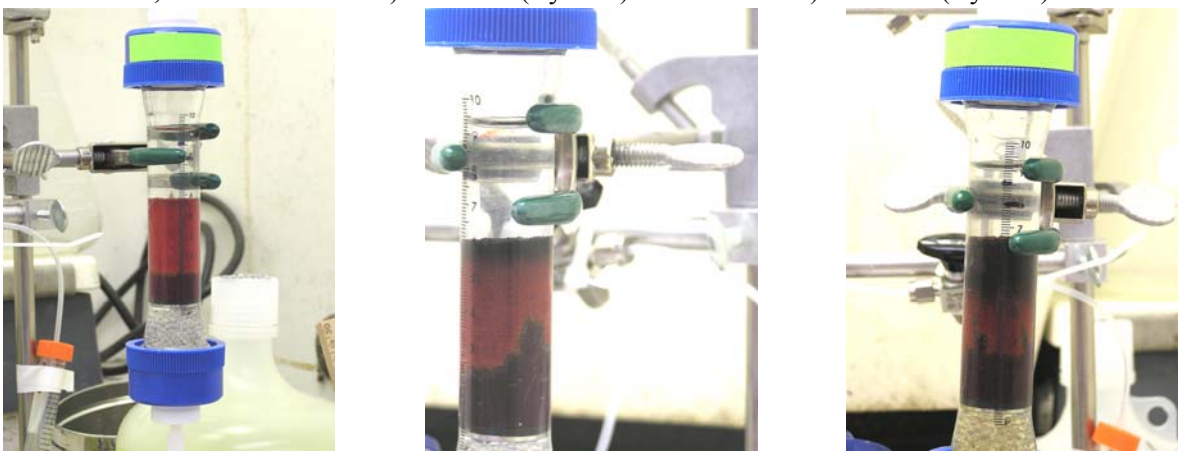


Figure 5.7. BRF-15 During Various Process Stages

a) Pretreatment, H-conversion b) Pretreatment, Na-conversion c) Elution (Cycle 1)

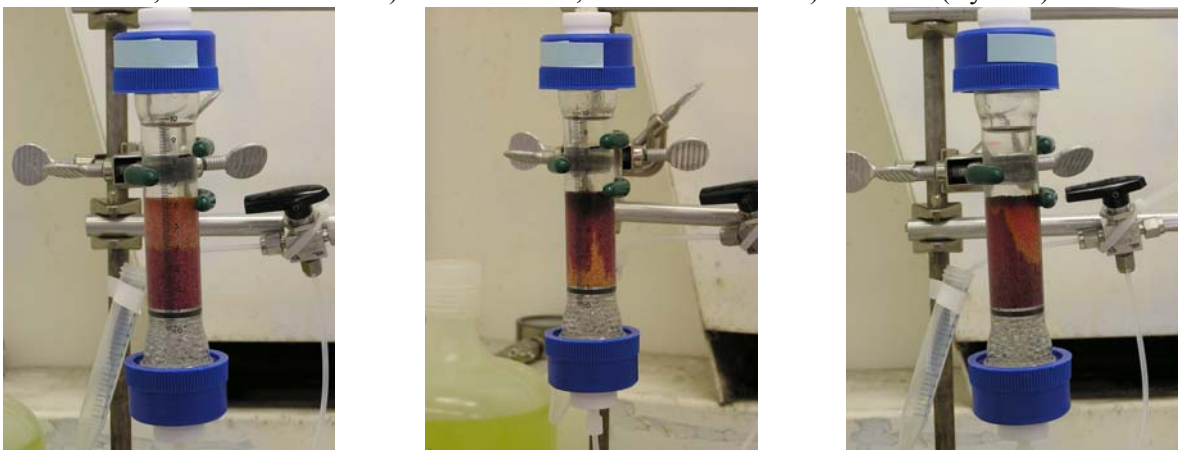


Figure 5.8. BRF-16 During Various Process Stages

a) Pretreatment, H-conversion

b) Regeneration (Cycle 2)

c) Elution (Cycle 2)

d) Elution (Cycle 3)

Figure 5.9. BRF-17 During Various Process Cycles

The small black band observed on the top of the resin bed during Cycle 2 elution had expanded significantly during the course of the long regeneration process; the expanded condition is shown in the Cycle 3 elution photographs.

The RF eluate samples did not have any significant coloration; a small amount of precipitate was observed in the 4- to 5-BV process samples. The first three eluate samples from Cycle 3 RF processing were intensely colored whereas the SL-644 eluate samples were only slightly colored.

5.2 Physical Property Test Results

This section summarizes physical property test results, including resin morphology, PSDs in hydrated H-form and Na-form, bed density, and skeletal density.

5.2.1 Optical Microscopy

The micrographs of pretreated resin per Figure 5.3 are shown in Figure 5.11 through Figure 5.14. All resins appeared spherical and ranged from brick red to tan in color. The general morphology is easily discerned in the pictures. Resin BRF-16 was the largest of the group and appeared pitted or mottled on the surface. The pitting was a result of the extra monomer adhering to the surface of the resin, especially apparent in the cross-section. The other resins exhibited smooth surface areas. The black specks visible on the cross-section resin portions are carbon tape contamination, not characteristics of the particles.

In all cases, a distinct resin bead core was apparent. This may be attributed to incomplete RF polymerization.

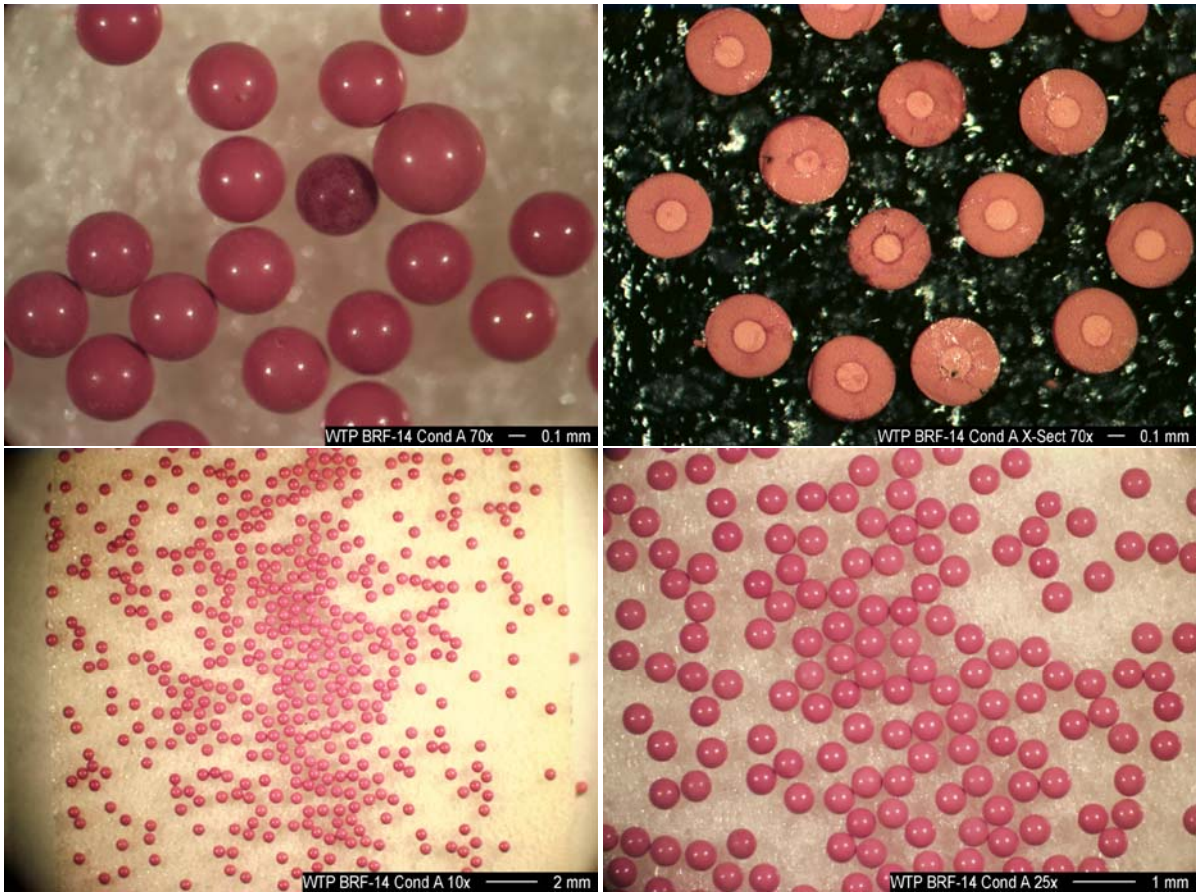


Figure 5.10. Micrographs of BRF-14 H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

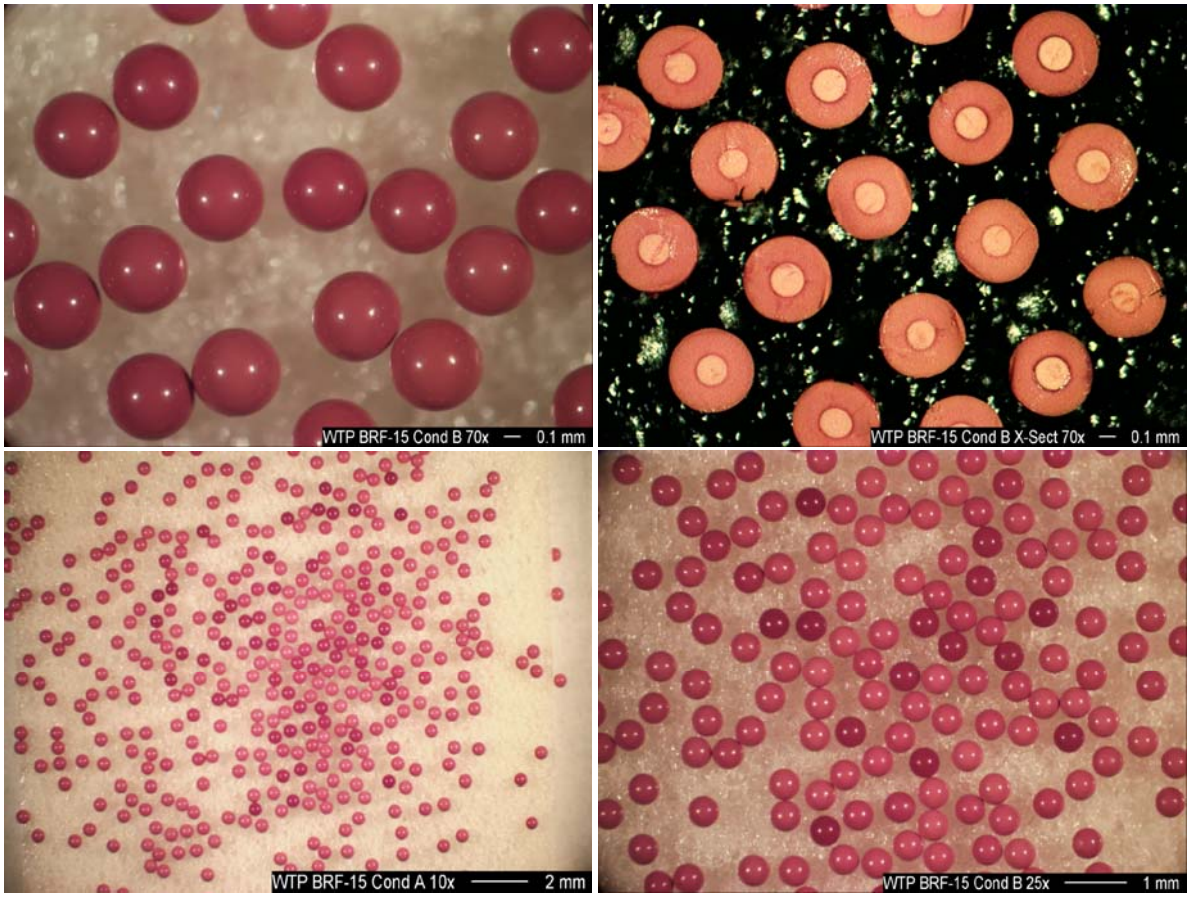


Figure 5.11. Micrographs of BRF-15 H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

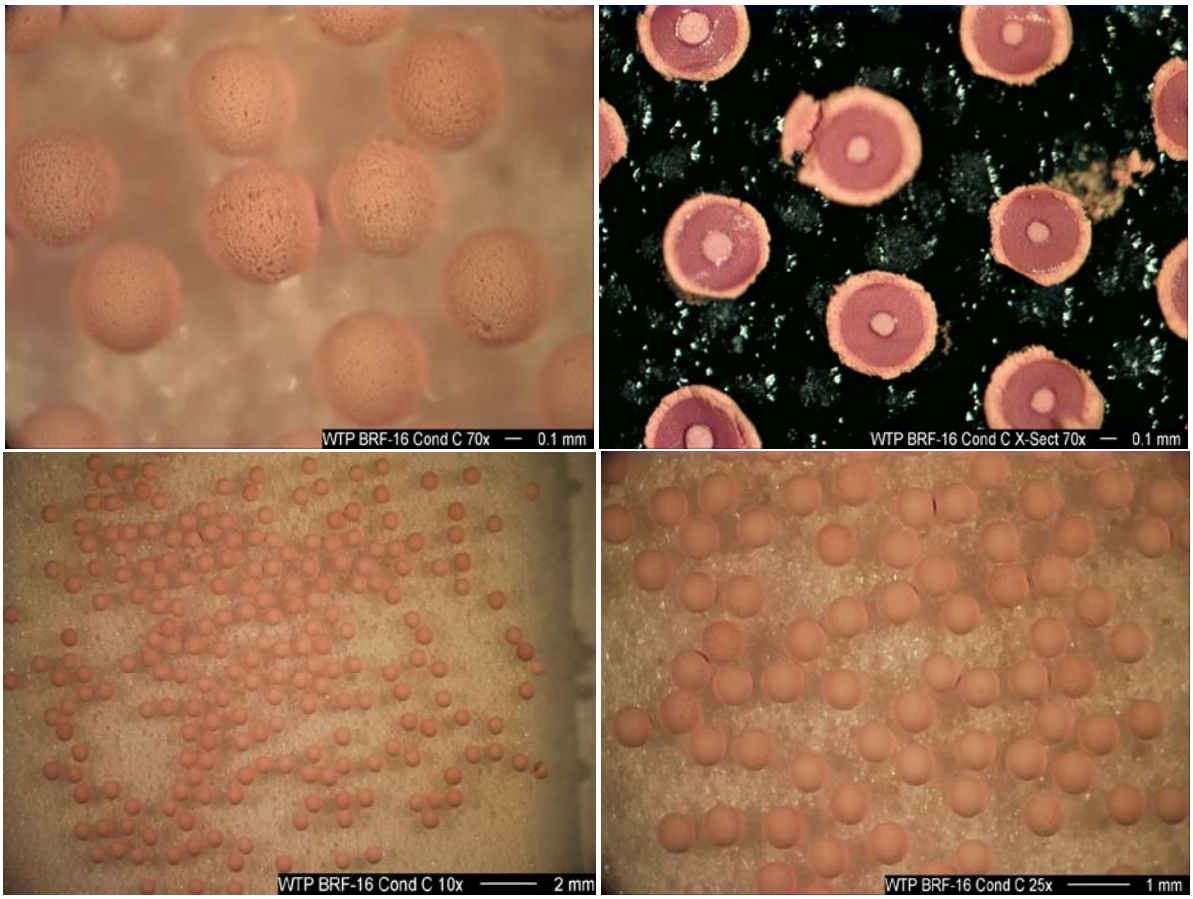


Figure 5.12. Micrographs of BRF-16 H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

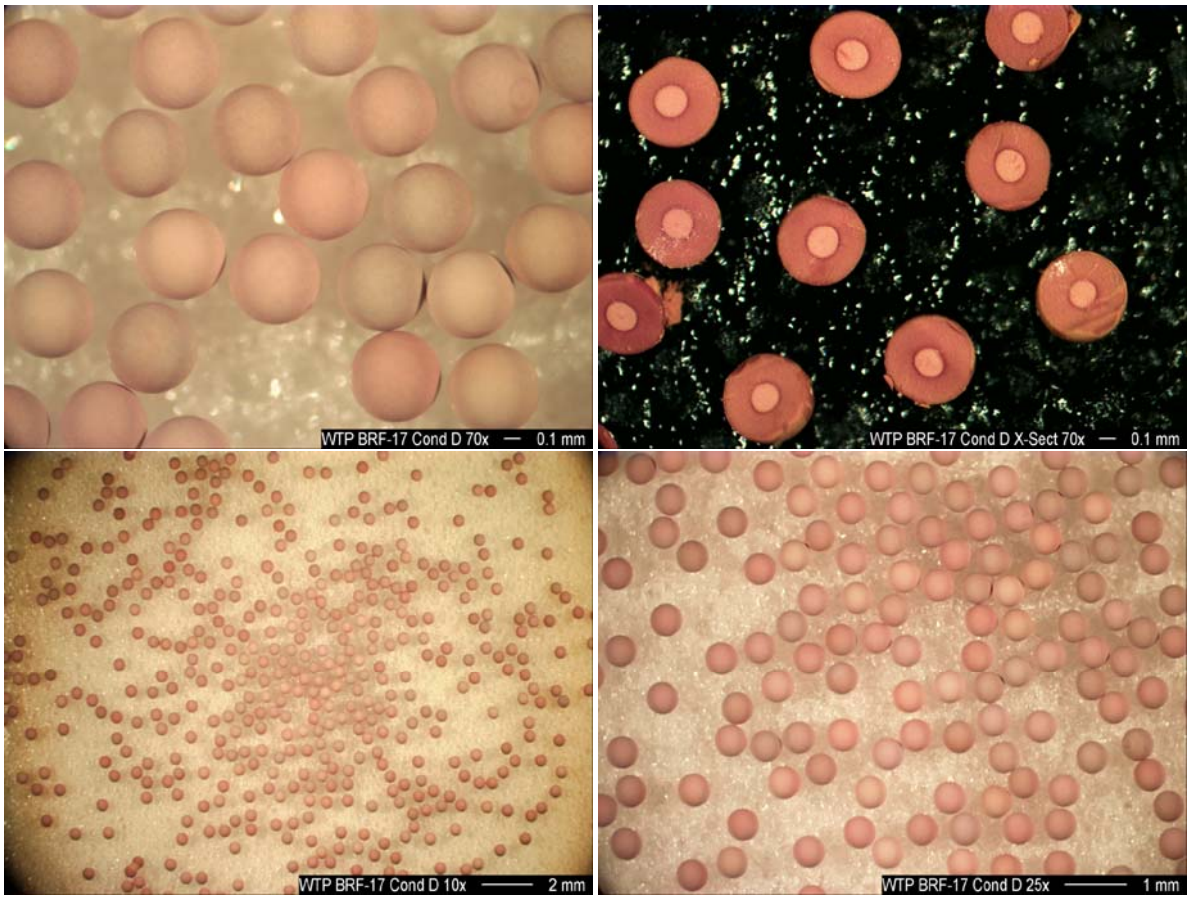


Figure 5.13. Micrographs of BRF-17 H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

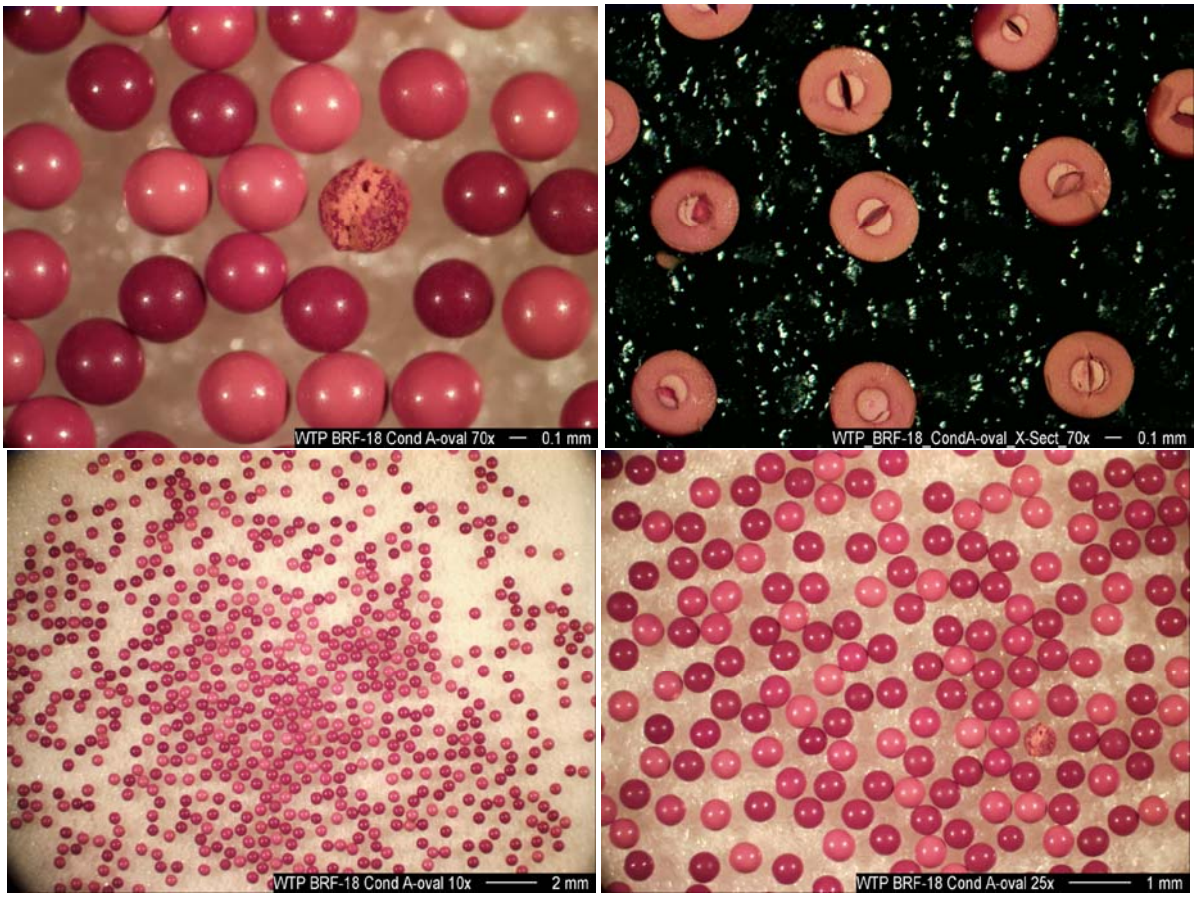


Figure 5.14. Micrographs of BRF-18 H-Form Resin (oval). Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The effect of processing with cracked seeds was evident in the cross-section of BRF-18. The cross section shows the center crack, and the spheres were slightly out of round (oval). The external appearance was only slightly oval.

5.2.2 Particle-Size Distribution

Figure 5.15 shows the comparison between the average particle sizes for the resins in the H-form and in the Na-form. The mean diameters are reported on a volume basis. The bars above and below each mean represent the particle-size distribution obtained from the lower 5% to upper 90% on a volume basis. Thus, 85% of the particles (volume basis) have a diameter in the indicated range.

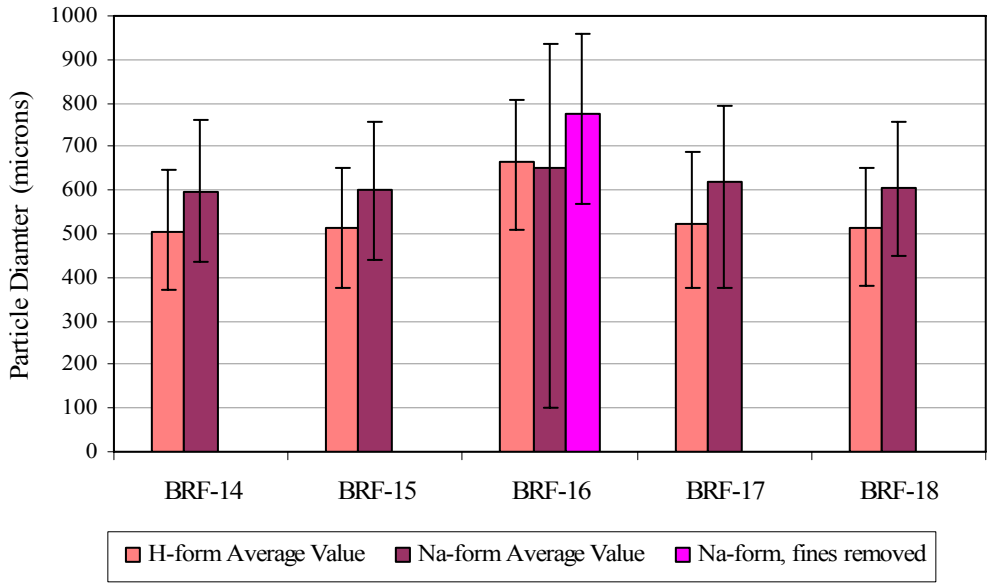


Figure 5.15. Average Particle Diameter (Volume Distribution) with 5% to 90% Volume Percentile Distribution

The relative diameter expansion from the H-form to the Na-form is also apparent. The average diameter expansion from the H-form to the Na-form for the Wave 1 resins (BRF-14 through BRF-18) was nominally 20%. The expansion for Waves 2 to 4 resins was nominally 7 to 10%. Because the diameter is directly related to volume, the corresponding volume expansion could be calculated. The Wave 1 resin volume expansion (from H-form to Na-form) was typically 60% whereas the Wave 2 to 4 resins expanded 25 to 30%. The difference between the test waves was a direct result of the limited pretreatment of the Wave 1 resins. The Wave 1 H-form particle size was essentially determined on the as-received condition. Subsequent resin test waves were conducted after a "bead relaxation process" as a consequence of the initial swell-shrink cycle.

The BRF-16 Na-form resin manifested a bi-modal particle distribution with diameter means at 777 μm (~90%) and 12 μm (~10%). The smaller particle size fraction was consistent with the presence of fines shed from the particle surface. The contribution of fines was mathematically removed to provide a true evaluation of the bead particle diameter, which is shown in Figure 5.15.

A summary of average particle sizes (volume distribution mean—m_v, number distribution mean—m_n, and area distribution mean—m_a) are tabulated in Table 5.10. The better the agreement between the m_v, m_n, and m_a values, the more uniform is the overall particle distribution. The tested spherical resins manifested monodisperse particle population with one exception of the BRF-16 Na-form resin. In that case, the bimodal spread represented a population of spheres and a population of fines, presumably shed from the sphere surface.

Table 5.10. Wave 1 Resins Particle-Size-Distribution Summary

Resin ID	Form	Volume distribution (microns)				Number distribution (microns)				Area dist. (microns)
		m_v	sd	Low 5%	High 90%	m_n	sd	Low 5%	High 90%	m_a
BRF-14	H-form	506	95	370	647	455	72	348	554	486
	Na-form	598	115	435	762	538	91	406	664	575
BRF-15	H-form	512	96	374	652	461	76	351	564	493
	Na-form	601	109	442	755	545	91	413	668	580
BRF-16	H-form	663	104	508	806	617	92	478	737	647
	Na-form, w/fines[(a)]	650	266	101	937	15	6	9	26	179
	Na-form, w/o fines[(a)]	777	137	569	958	708	128	512	892	754
BRF-17	H-form	522	105	376	687	462	74	354	565	498
	Na-form	619	122	446	794	555	97	418	688	595
BRF-18	H-form	515	93	380	650	467	75	355	569	497
	Na-form	607	107	449	758	553	91	420	675	587

(a) BRF-16 Na-form resin resulted in a bi-modal (volume distribution basis). The second series results stripped the fines contribution from the spectra.

m_v = mean diameter volume distribution

m_n = mean diameter number distribution

m_a = mean diameter area distribution

Low 5% = 5% of particles (volume distribution) are below this value.

High 90% = 10% of particles (volume distribution) are greater than this value.

5.2.3 Skeletal Density

The averages and relative percent differences (RPDs) of the H-form and Na-form skeletal densities are summarized in Table 5.11. The RF resin H-form densities were essentially equivalent at around 1.44 to 1.48 g/mL; the Na-form densities were similar, ranging from 1.58 g/mL to 1.63 g/mL. These results compared well to previously reported values of similar resin (but smaller particle size) by King et al. (2004) where the RF resin H-form skeletal density was reported to be 1.47 g/mL, and the Na form resin was 1.63 g/mL. Note that Wave 1 RF resins H-form densities appeared to be a little lower than subsequent test waves. This difference may be driven by the different manufacturing conditions of the latter resins where higher RF loading and polymerization into the spheres were attempted. Comparing the cross-section micrographs clearly indicated that the Wave 1 resins contained a core that may be less densely loaded with RF relative to the Wave 2 through 4 resins.

Table 5.11. Wave 1 Resins Skeletal Densities

Resin ID	H-form		Na-form	
	Average, g/mL	RPD	Average, g/mL	RPD
BRF-14	1.442	0.29	1.577	0.12
BRF-15	1.441	0.20	1.632	0.15
BRF-16	1.440	0.03	1.632	0.16
BRF-17	1.440	0.22	1.608	0.07
BRF-18	1.425	0.41	1.604	0.07
SL-644	1.341	1.4	1.518[(a)]	0.01[(a)]
(a) The Na-form SL-644 skeletal density was determined in quadruplicate; the standard deviation is shown.				

5.3 Formaldehyde in Pretreatment Contact Solutions

The wash solution formaldehyde concentration results are summarized in Figure 5.16. Initial formaldehyde concentrations varied from 1000 mg/L to 3000 mg/L. Water rinses brought the concentrations down significantly. However, simply soaking the resin overnight in water apparently released additional formaldehyde. Soaking in NaOH solution resulted in barely detectable formaldehyde. This could have been caused in large part by the much higher dilution that occurred with the 10-RV contact as opposed to the previous 1-RV contact volumes. The method detection limit was estimated to be 5 mg/L and was reached with the second NaOH soak.

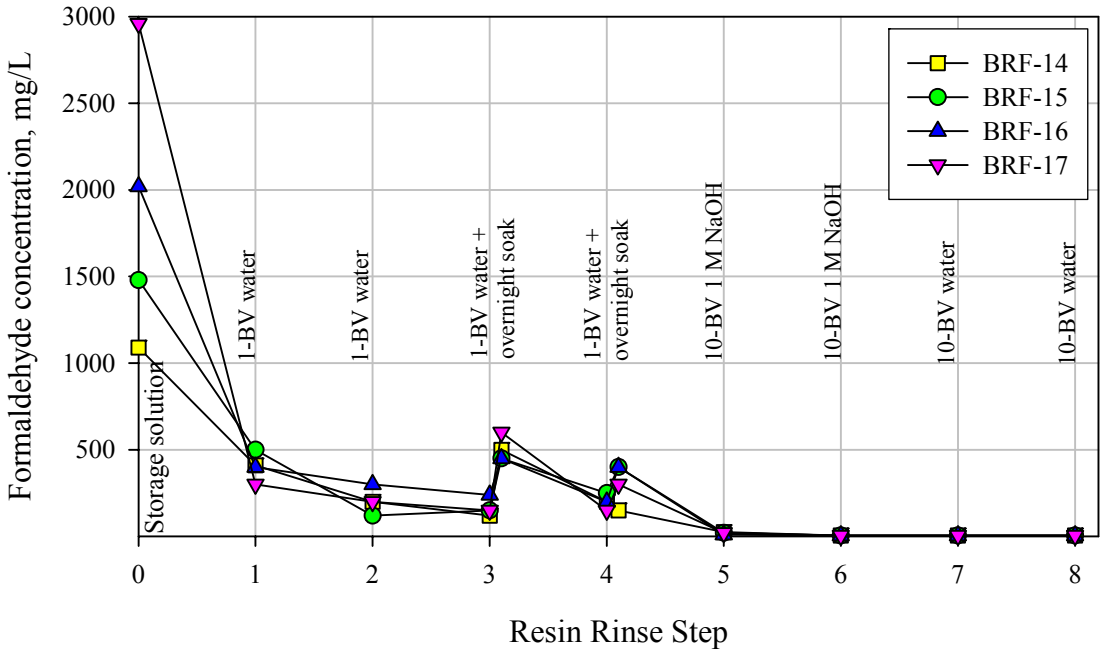


Figure 5.16. Free Formaldehyde in Solution, Indication-Only

5.4 Column Performance Testing

The Wave 1 Cs loading and elution characteristics for the all process cycles are provided in this section. A discussion of the oxygen-saturated regeneration processing and results conducted between Cycle 2 and Cycle 3 are discussed in Section 6.

5.4.1 BRF-14 with AZ-102 Simulant

The white column test was identical to tests conducted under the A-222 scoping statement (Fiskum et al. 2004, Resin #3) and served as a performance tie or link to the current work scope. The BRF-14 resin was manufactured identically to Resin #3 except for the particle size; the Resin #3 average diameter was 501 μm (Na-form, volume basis) compared to the BRF-14 average diameter of 598 μm (Na-form, volume basis).

The Cs loading results are presented in Figure 5.17 and Table 5.12. The loading profiles from the three process cycles were linear on the probability scale indicating ideal loading behavior. The onset of breakthrough was observed at ~45 BVs for Cycles 1 and 2; the Cycle 3 breakthrough onset occurred sooner at ~30 BVs. The contract limit breakthrough of 0.025% C/C_o was observed at 68 BVs, 68 BVs, and 49 BVs for Cycles 1, 2, and 3, respectively. The first and second process cycles resulted in virtually identical profiles where 50% breakthrough occurred at 170 BVs; the third process cycle resulted in 50% breakthrough at 140 BVs. Nominally 26 mg Cs/g dry H-form resin were loaded on the resin bed in Cycles 1 and 2; 23 mg Cs/g dry H-form resin were loaded in Cycle 3. The effects of bleed from one process cycle into the next were difficult to evaluate because sample ^{137}Cs concentrations through the first 30 BVs were at or close to the instrument detection limit.

Also shown in Figure 5.17 are the data from AZ-102 testing with Resin #3. The kinetics and/or selectivity of Resin #3 appeared to be superior based on the delayed onset of Cs breakthrough and the steeper (sharper) breakthrough slope. This was attributed to the smaller particle size of the Resin #3 (501 μm Na-form) relative to the BRF-14 resin (598 μm Na-form) and ion exchange theory where smaller particle radii sharpens the breakthrough curve (Hardy et al. 2004; Helfferich 1962). The 50% breakthrough capacity of BRF-14 was greater than that of Resin #3, 170 BVs versus 143 BVs, which was not expected to be influenced by increased particle size.

The elution profiles for the three process cycles are provided in Figure 5.18 and Table 5.12. The elution profile previously reported for Resin #3, the first process cycle (Fiskum et al. 2004), is also shown for comparison. The appearance of a shoulder on the Resin #3 elution profile is the consequence of a reduction in flowrate from 2 BV/h to 1.4 BV/h. A constant eluant flowrate was applied to the current suite of tests. Significant Cs elution began after processing 5 BVs 0.5 M HNO_3 with diminishing returns as elution continued through the 21 BVs tested. The calculated total Cs remaining on the resin per g dry H-form resin as a function of the elution volume is shown in Figure 5.19.

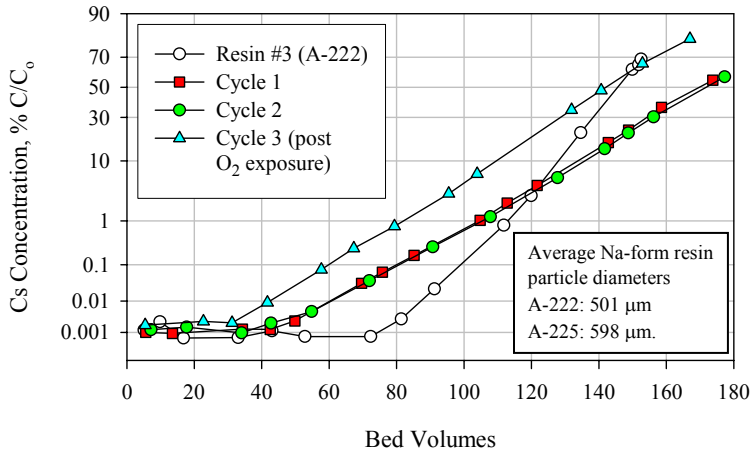


Figure 5.17. BRF-14 Cs Loading Profiles with AZ-102 Simulant

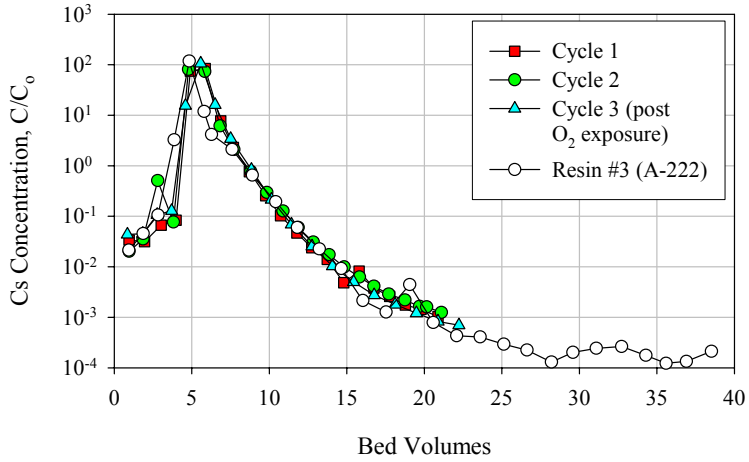


Figure 5.18. BRF-14 Cs Elution Profiles (Following AZ-102 Simulant Load)

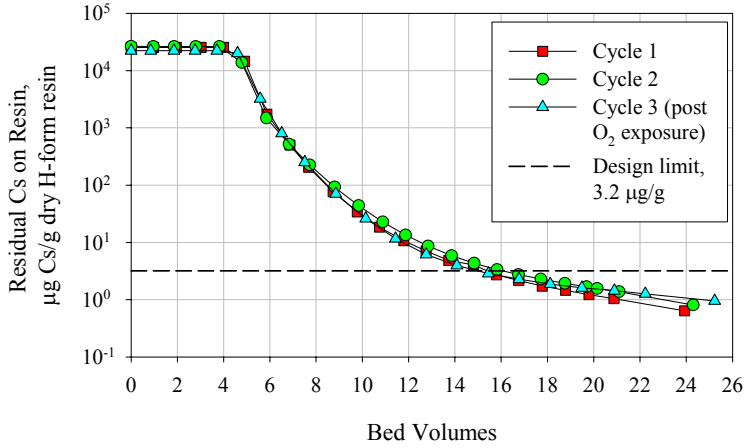


Figure 5.19. Residual Cs on BRF-14 as a Function of Elution Volume (AZ-102 Simulant Load)

Table 5.12. BRF-14 (White Column) Effluent Cs Concentration During AZ-102 Simulant Loading and Elution

Cycle 1					Cycle 2					Cycle 3 (Post O_2 Exposure)[b]				
Load		Elution		Resin Cs Conc.[a]	Load		Elution		Resin Cs Conc.[a]	Load		Elution		Resin Cs Conc.[a]
BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g
5.6	<9.97E-4	0.97	3.39E-2	2.54E+4	7.1	1.21E-3	0.96	2.02E-2	2.63E+4	5.4	1.74E-3	0.85	4.37E-2	2.23E+4
13.5	<9.22E-4	1.96	3.15E-2	2.54E+4	17.7	1.49E-3	1.86	3.56E-2	2.63E+4	22.7	2.28E-3	1.85	4.52E-2	2.23E+4
34.3	<1.28E-3	3.03	6.65E-2	2.54E+4	34.0	9.57E-4	2.80	5.05E-1	2.63E+4	31.2	2.06E-3	2.77	1.07E-1	2.22E+4
42.5	<1.25E-3	3.99	8.35E-2	2.54E+4	42.7	2.00E-3	3.82	7.68E-2	2.63E+4	41.7	9.00E-3	3.71	1.29E-1	2.22E+4
49.8	2.37E-3	4.91	7.48E+1	1.45E+4	54.8	4.75E-3	4.79	8.08E+1	1.38E+4	57.7	7.57E-2	4.60	1.55E+1	2.00E+4
69.6	3.24E-2	5.87	8.40E+1	1.73E+3	71.9	3.79E-2	5.85	7.34E+1	1.48E+3	67.3	2.54E-1	5.58	1.06E+2	3.22E+3
75.8	6.42E-2	6.87	7.72E+0	5.01E+2	90.7	2.74E-1	6.83	6.18E+0	5.21E+2	79.4	7.80E-1	6.51	1.61E+1	8.04E+2
85.2	1.71E-1	7.68	2.30E+0	2.04E+2	107.8	1.21E+0	7.72	2.10E+0	2.25E+2	95.5	3.21E+0	7.51	3.43E+0	2.51E+2
104.8	1.03E+0	8.73	7.67E-1	7.68E+1	127.7	5.78E+0	8.80	7.72E-1	9.25E+1	103.9	6.58E+0	8.85	8.32E-1	7.05E+1
112.8	2.20E+0	9.78	2.56E-1	3.41E+1	141.7	1.42E+1	9.84	2.95E-1	4.38E+1	131.9	3.47E+1	10.14	2.14E-1	2.61E+1
121.7	4.36E+0	10.74	1.03E-1	1.85E+1	148.7	2.13E+1	10.88	1.27E-1	2.28E+1	140.7	4.79E+1	11.43	6.93E-2	1.16E+1
142.8	1.68E+1	11.79	4.67E-2	1.07E+1	156.2	3.03E+1	11.87	6.00E-2	1.34E+1	152.9	6.61E+1	12.76	2.54E-2	6.17E+0
148.8	2.28E+1	12.75	2.38E-2	7.06E+0	177.3	5.72E+1	12.84	3.09E-2	8.66E+0	167.0	8.00E+1	14.09	1.03E-2	3.96E+0
158.6	3.63E+1	13.74	1.40E-2	4.85E+0	FD	4.02E+1	13.86	1.73E-2	5.86E+0	FD	5.76E+1	15.45	5.00E-3	2.86E+0
173.8	5.50E+1	14.80	4.84E-3	4.04E+0	FDI	3.39E+0	14.83	9.92E-3	4.33E+0	FDI	4.73E+0	16.77	2.75E-3	2.28E+0
FD	4.15E+1	15.80	8.22E-3	2.73E+0			15.82	6.25E-3	3.35E+0			18.12	1.80E-3	1.88E+0
FDI	3.16E+0	16.76	3.81E-3	2.15E+0			16.75	4.13E-3	2.74E+0			19.49	1.21E-3	1.61E+0
		17.77	2.61E-3	1.73E+0			17.71	2.90E-3	2.30E+0			20.89	8.31E-4	1.43E+0
		18.78	1.75E-3	1.45E+0			18.75	2.20E-3	1.93E+0			22.23	7.01E-4	1.27E+0
		19.79	1.44E-3	1.22E+0			19.70	1.65E-3	1.68E+0			EDI	6.70E-4	9.55E-1[c]
		20.88	1.04E-3	1.04E+0			20.15	1.61E-3	1.57E+0					
		EDI	8.31E-4	6.37E-1[c]			21.10	1.25E-3	1.38E+0					
							EDI	1.14E-3	8.09E-1[c]					

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) The O_2 exposure is discussed in Section 6.0.
(c) The final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution.

The elution profiles reproduced well between the three process cycles. After a nominal 21-BV elution, the eluate Cs concentration reached ~1E-3 C/C_o. The design limit of 3.2 μg residual Cs per g dry H-form resin was reached after processing 16 BVs 0.5 M HNO_3. The residual Cs on the resin bed (determined from direct column counting by GEA) increased from one cycle to the next (0.64 μg/g, 0.81 μg/g, and 0.96 μg/g, respectively) but did not contribute significant Cs bleed onto the next process cycle. Final post-elution Cs loading on the resin was 3.8, 4.8, and 5.7 μg for Cycles 1 to 3, respectively. In the 6-g resin bed mass (H-form), the Cs concentration corresponded to 1 μg Cs per g resin (worst case). If 32.8% of the Cs was ^{137}Cs (consistent with Envelope B AZ-102 waste), then the residual ^{137}Cs activity concentration on the resin would be 29 μCi/g.

5.4.2 BRF-14, BRF-15, BRF-17 with AP-101 Simulant

The BRF-14, BRF-15, and BRF-17 test loading and elution results were similar; the overall comparative summary of specific performance parameters is provided in Table 5.13. The load curves are presented in Figure 5.20 through Figure 5.22. In all cases, the loading profiles from the three process cycles were slightly bowed on the probability scale, indicating non-ideal loading behavior, most probably attributed to the K interference in the Cs ion exchange process. In each case, the first loading cycle curve resulted in a more pronounced bow at nominally 70 BVs that was coincident with the flowrate increase from 1.5 BV/h to 3 BV/h. The second and third loading cycles did not demonstrate a similar bow-effect in the loading profiles. It is possible that the cycling in the column, as a result of processing the first feed, better conditioned the resin, allowing the second cycle to perform better with the high K waste simulant. However, the first and second process cycle Cs loading profiles with the AZ-102 simulant were virtually identical, indicating that the single in-column shrink-swell pretreatment was sufficient for use with the AZ-102 waste.

Table 5.13. Comparative Performance Summary for Three Process Cycles of BRF-14, BRF-15 and BRF-17 with AP-101 Simulant

	BRF-14			BRF-15			BRF-17		
Cycle #	1	2	3	1	2	3	1	2	3
Contract limit, BV	28	31	24	24	30	22	22	27	23
50% breakthrough, BV	114	121	111	107	120	107	108	122	105
Loading, mg Cs/g resin	2.0	2.1	1.8	1.9	2.0	1.8	1.9	2.0	1.8
Residual, μg Cs/g resin	0.13	0.14	0.099	0.13	0.13	0.084	0.16	0.16	0.12

The onset of Cs breakthrough was nearly immediate with the AP-101 matrix. The early onset was attributed to the competing K ion, which is the highest concentration in AP-101 of all expected feeds to the WTP plant. The contract limit of 0.091% C/C_o was reached after processing 22 to 31 BVs. The first and third process cycles resulted in virtually identical 50% breakthroughs; the second process cycle 50% breakthrough occurred after processing slightly more BVs. A small amount of Cs bleed from Cycle 1 into Cycle 2 (≥3× Cycle 1) was observed for BRF-14 and BRF-15, which only affected the first 15 BVs. After the first 15 BV, the Cs breakthrough from the AP-101 simulant processing dominated the effluent, and no further effects of Cs bleed could be quantified. Because Cs broke through rapidly during the first process cycle of BRF-17, no Cs bleed effect on the second process cycle was discerned. The extended fingering or channeling observed during the BRF-17 Cycle 2 regeneration process did not appear to have any adverse effect on the Cs loading profile.

In all tests, nominally 2 mg Cs per g dry H-form resin were loaded onto the RF. This was one order of magnitude less Cs loading than was observed with AZ-102, a low-K waste simulant.

The elution profiles for the three process cycles are provided in Figure 5.23 through Figure 5.28. The elution profiles from the first two cycles tracked well with each other; the third process cycle appeared to be slightly broader than the other two cycles. All cycles demonstrated an eluate Cs concentration of ~1E-3 C/C_o after processing ~21 BVs eluant. The design limit of 4.2 μg residual Cs per gram H-form resin was reached after processing ≤12 BVs 0.5 M HNO_3. The residual Cs on the resin bed remained nearly constant from one cycle to the next, ranging from 0.1 to 0.16 μg Cs per g resin. The variability could be related to the experimental/analysis variability associated with direct measurement of the column assembly. The residual Cs concentration on the resin was less than one fourth of the residual Cs observed with the AZ-102 process test (0.64 μg/g). However, the AP-101 process test loaded less than a factor of 10 total Cs relative to the AZ-102 process test.

The event in which air was inadvertently pumped into the BRF-15 resin bed during Cycle 3 elution (after processing 22 BVs eluate) resulted in void spaces throughout the bed. The elution profile may have changed slightly after the air exposure as evidenced by the slight rise in C/C_o at the elution tail (see process volume at 25.6 BV, Figure 5.24); however, virtually all Cs was already eluted. The air-isolated beads were briefly insulated from the eluant such that Cs was not swept away. During this time, the Cs diffusion from the center of the particle continued, re-establishing an equilibrium condition within the bead. With reestablishment of eluant flow around the bead, the Cs was released in slight excess of the original eluate concentration gradient.

The data graphed in the figures are provided in Table 5.14 through Table 5.16.

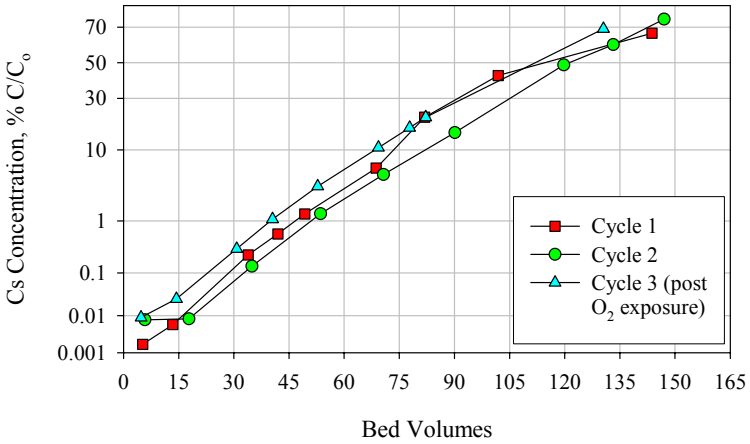


Figure 5.20. BRF-14 Cs Loading Profiles with AP-101 Simulant

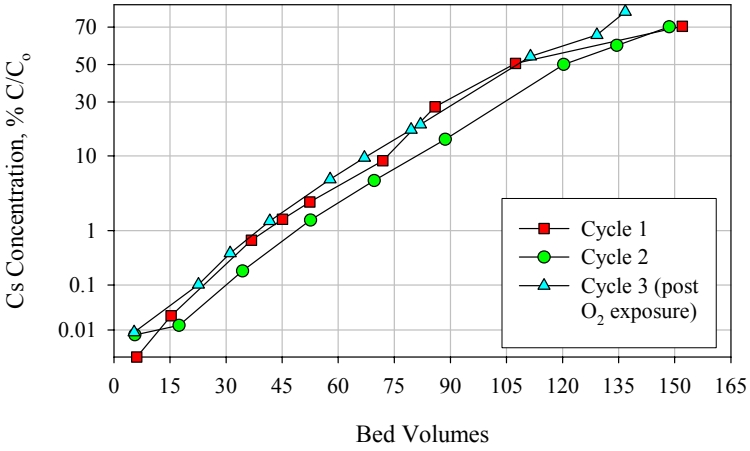


Figure 5.21. BRF-15 Cs Loading Profiles with AP-101 Simulant

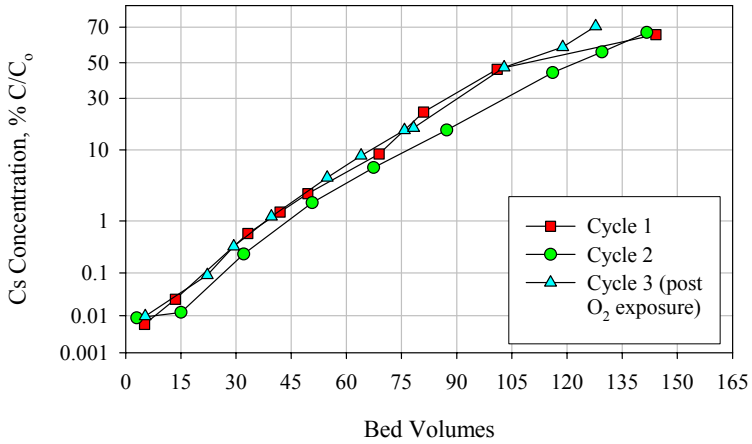


Figure 5.22. BRF-17 Cs Loading Profiles with AP-101 Simulant

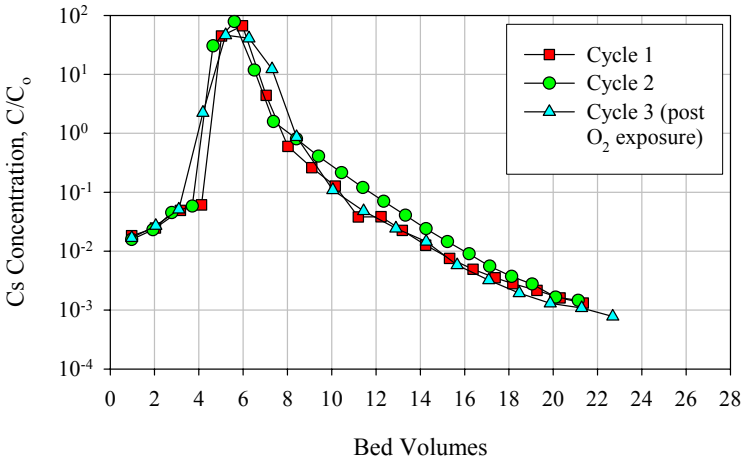


Figure 5.23. BRF-14 Cs Elution Profiles

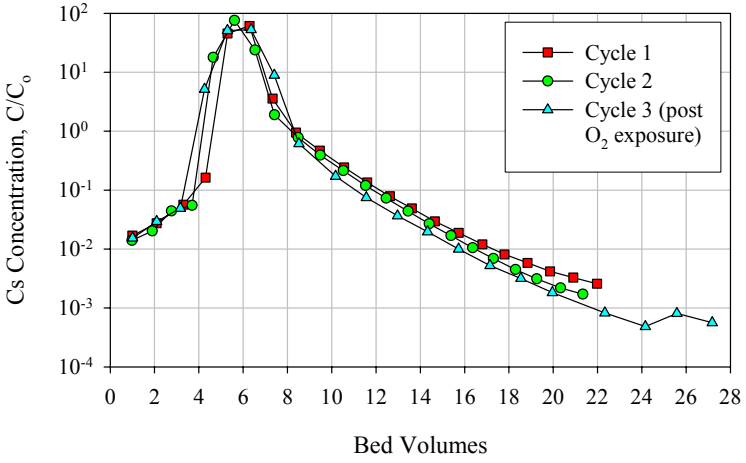


Figure 5.24. BRF-15 Cs Elution Profiles

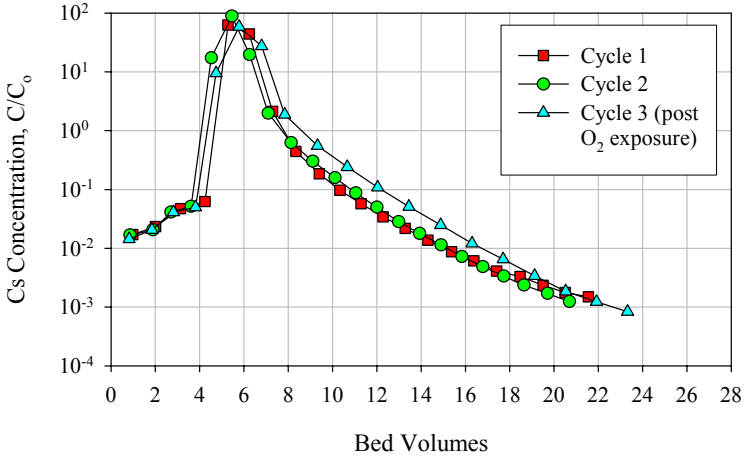


Figure 5.25. BRF-17 Cs Elution Profiles

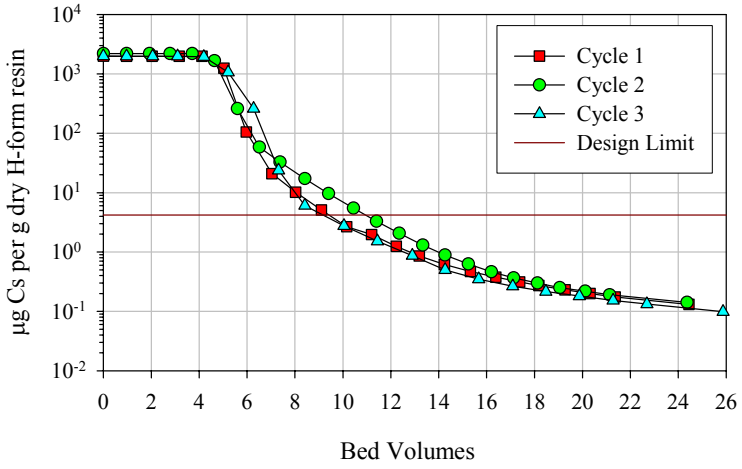


Figure 5.26. Residual Cs as a Function of Elution Volume, BRF-14 (AP-101 Simulant Load)

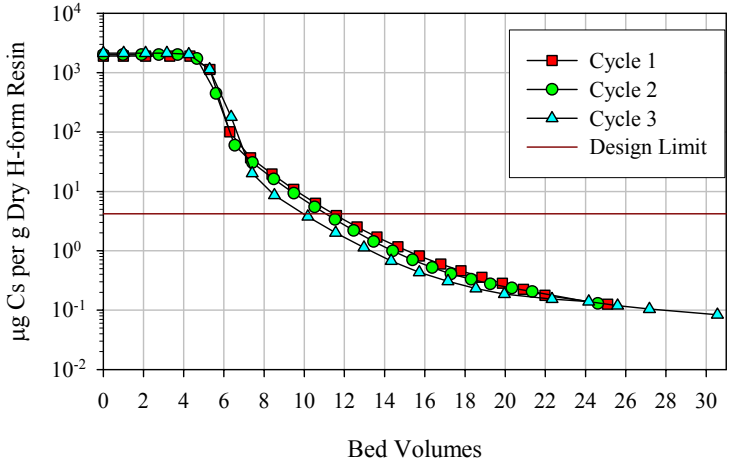


Figure 5.27. Residual Cs as a Function of Elution Volume, BRF-15

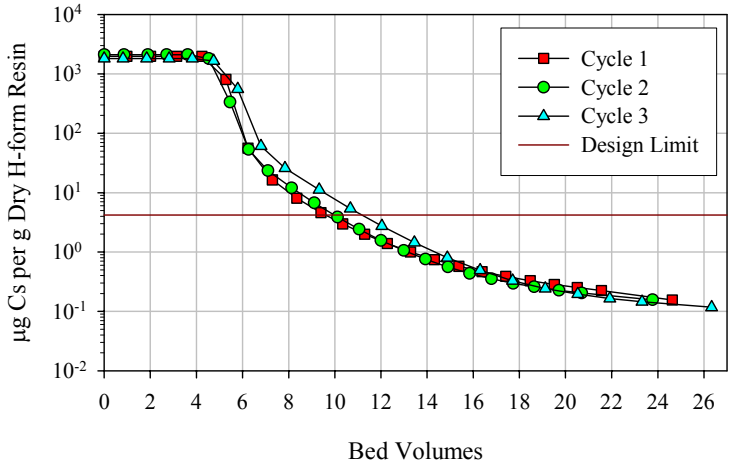


Figure 5.28. Residual Cs as a Function of Elution Volume, BRF-17

Table 5.14. BRF-14 (Yellow Column) Effluent Cs Concentration During AP-101 Simulant Loading and Elution

Cycle 1					Cycle 2					Cycle 3 (Post O_2 Exposure)[b]				
Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]
BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g
5.2	<1.73E-3	0.97	1.83E-2	1.98E+3	5.8	7.79E-3	0.97	1.58E-2	2.19E+3	4.8	8.97E-3	0.97	1.68E-2	1.98E+3
13.4	5.89E-3	2.04	2.47E-2	1.98E+3	17.8	8.27E-3	1.93	2.31E-2	2.19E+3	14.4	2.59E-2	2.06	2.71E-2	1.98E+3
33.9	2.35E-1	3.17	4.94E-2	1.98E+3	34.9	1.39E-1	2.78	4.51E-2	2.19E+3	30.8	3.11E-1	3.10	5.09E-2	1.98E+3
42.0	5.92E-1	4.12	6.09E-2	1.98E+3	53.6	1.32E+0	3.71	5.80E-2	2.19E+3	40.5	1.07E+0	4.19	2.23E+0	1.94E+3
49.3	1.31E+0	5.03	4.46E+1	1.25E+3	70.7	5.01E+0	4.64	3.03E+1	1.66E+3	52.8	3.45E+0	5.21	4.63E+1	1.06E+3
68.7	6.07E+0	5.97	6.69E+1	1.05E+2	90.1	1.52E+1	5.60	7.80E+1	2.60E+2	69.3	1.06E+1	6.27	4.08E+1	2.60E+2
81.9	2.11E+1	7.04	4.39E+0	2.09E+1	119.7	4.87E+1	6.51	1.18E+1	5.83E+1	77.8	1.70E+1	7.31	1.23E+1	2.38E+1
101.9	4.25E+1	8.03	6.01E-1	1.02E+1	133.2	6.05E+1	7.37	1.58E+0	3.28E+1	82.2	2.11E+1	8.41	8.68E-1	6.07E+0
143.7	6.68E+1	9.10	2.61E-1	5.11E+0	147.0	7.39E+1	8.41	8.02E-1	1.72E+1	130.5	6.91E+1	10.05	1.09E-1	2.76E+0
FD	4.54E+1	10.16	1.27E-1	2.67E+0	FD	5.95E+1	9.40	4.09E-1	9.63E+0	FD	5.30E+1	11.44	4.80E-2	1.52E+0
FDI	2.53E+0	11.20	3.81E-2	1.96E+0	FDI	2.62E+0	10.44	2.14E-1	5.47E+0	FDI	3.19E+0	12.90	2.43E-2	8.68E-1
		12.23	3.83E-2	1.25E+0			11.40	1.20E-1	3.30E+0			14.27	1.45E-2	5.00E-1
		13.19	2.26E-2	8.56E-1			12.35	7.00E-2	2.06E+0			15.67	5.80E-3	3.49E-1
		14.24	1.26E-2	6.18E-1			13.33	4.06E-2	1.31E+0			17.09	3.21E-3	2.65E-1
		15.32	7.51E-3	4.72E-1			14.26	2.40E-2	8.91E-1			18.47	1.94E-3	2.15E-1
		16.38	4.94E-3	3.77E-1			15.23	1.45E-2	6.28E-1			19.87	1.30E-3	1.81E-1
		17.38	3.55E-3	3.13E-1			16.20	9.02E-3	4.64E-1			21.28	1.09E-3	1.53E-1
		18.18	2.81E-3	2.73E-1			17.13	5.54E-3	3.68E-1			22.69	7.79E-4	1.33E-1
		19.27	2.15E-3	2.31E-1			18.12	3.71E-3	3.00E-1			EDI	5.96E-4	9.85E-2[c]
		20.31	1.61E-3	2.00E-1			19.05	2.76E-3	2.51E-1					
		21.36	1.32E-3	1.75E-1			20.11	1.66E-3	2.18E-1					
		EDI	7.94E-4	1.31E-1[c]			21.13	1.46E-3	1.90E-1					
							EDI	8.43E-4	1.41E-1[c]					

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) The O_2 exposure is discussed in Section 6.0.
(c) Final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution.

Table 5.15. BRF-15 (Green Column) Effluent Cs Concentration During AP-101 Simulant Loading and Elution

Cycle 1					Cycle 2					Cycle 3 (Post O_2 Exposure)[b]				
Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]
BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g
6.1	2.10E-3	1.00	1.69E-2	1.90E+3	5.6	7.57E-3	0.98	1.40E-2	2.01E+3	5.4	8.78E-3	1.01	1.53E-2	2.13E+3
15.3	2.17E-2	2.10	2.74E-2	1.90E+3	17.4	1.29E-2	1.90	2.02E-2	2.01E+3	22.6	1.03E-1	2.10	2.93E-2	2.13E+3
36.8	6.93E-1	3.31	5.64E-2	1.90E+3	34.4	1.91E-1	2.76	4.40E-2	2.01E+3	31.1	4.10E-1	3.17	4.94E-2	2.13E+3
45.1	1.51E+0	4.31	1.63E-1	1.90E+3	52.6	1.47E+0	3.70	5.51E-2	2.01E+3	41.7	1.42E+0	4.26	5.13E+0	2.03E+3
52.4	2.72E+0	5.30	4.53E+1	1.13E+3	69.6	5.19E+0	4.65	1.79E+1	1.72E+3	57.8	5.40E+0	5.29	5.10E+1	1.14E+3
71.9	8.91E+0	6.29	6.11E+1	1.01E+2	88.6	1.47E+1	5.61	7.58E+1	4.45E+2	67.0	9.60E+0	6.36	5.27E+1	1.79E+2
85.9	2.77E+1	7.33	3.56E+0	3.67E+1	120.3	5.00E+1	6.54	2.38E+1	5.95E+1	79.5	1.81E+1	7.41	8.86E+0	2.02E+1
107.4	5.06E+1	8.39	9.46E-1	1.96E+1	134.5	6.06E+1	7.42	1.89E+0	3.06E+1	82.0	2.01E+1	8.52	6.11E-1	8.62E+0
152.0	7.04E+1	9.47	4.69E-1	1.08E+1	148.5	7.02E+1	8.48	7.83E-1	1.62E+1	111.4	5.46E+1	10.18	1.72E-1	3.77E+0
FD	4.86E+1	10.56	2.43E-1	6.32E+0	FD	6.42E+1	9.48	3.92E-1	9.32E+0	129.2	6.61E+1	11.56	7.47E-2	2.01E+0
FDI	2.58E+0	11.60	1.35E-1	3.92E+0	FDI	2.52E+0	10.52	2.13E-1	5.46E+0	136.8	7.71E+1	12.97	3.67E-2	1.13E+0
		12.63	7.88E-2	2.52E+0			11.53	1.19E-1	3.37E+0	FD	4.95E+1	14.33	1.96E-2	6.73E-1
		13.62	4.90E-2	1.70E+0			12.46	7.30E-2	2.19E+0	FDI	2.59E+0	15.73	1.00E-2	4.33E-1
		14.66	2.95E-2	1.17E+0			13.45	4.38E-2	1.43E+0			17.15	5.26E-3	3.06E-1
		15.73	1.89E-2	8.19E-1			14.40	2.68E-2	9.89E-1			18.54	3.17E-3	2.31E-1
		16.80	1.20E-2	6.00E-1			15.39	1.69E-2	7.00E-1			19.96	1.83E-3	1.87E-1
		17.80	8.14E-3	4.61E-1			16.36	1.05E-2	5.22E-1			22.33	8.26E-4	1.54E-1
		18.84	5.81E-3	3.57E-1			17.30	6.93E-3	4.08E-1			24.16	4.84E-4	1.39E-1
		19.86	4.18E-3	2.84E-1			18.30	4.49E-3	3.30E-1			25.59	8.06E-4	1.19E-1
		20.91	3.28E-3	2.25E-1			19.26	3.12E-3	2.77E-1			27.18	5.63E-4	1.04E-1
		21.98	2.58E-3	1.78E-1			20.33	2.18E-3	2.36E-1			EDI	3.42E-4	8.37E-2[c]
		EDI	9.74E-4	1.26E-1[c]			21.34	1.72E-3	2.06E-1					
							EDI	1.36E-3	1.29E-1[c]					

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) The O_2 exposure is discussed in Section 6.0.
(c) Final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution.

Table 5.16. BRF-17 (Pink Column) Effluent Cs Concentration During AP-101 Simulant Loading and Elution

Cycle 1					Cycle 2					Cycle 3 (Post O_2 Exposure)[b]				
Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]
BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g
5.1	5.97E-3	0.98	1.71E-2	1.98E+3	3.0	8.78E-3	0.86	1.68E-2	2.10E+3	5.3	9.67E-3	0.82	1.43E-2	1.81E+3
13.5	2.54E-2	2.01	2.33E-2	1.98E+3	15.0	1.21E-2	1.89	2.05E-2	2.10E+3	22.2	8.96E-2	1.85	2.06E-2	1.81E+3
33.2	6.02E-1	3.14	4.74E-2	1.98E+3	32.1	2.45E-1	2.71	4.13E-2	2.10E+3	29.4	3.47E-1	2.82	4.13E-2	1.81E+3
42.0	1.41E+0	4.24	6.24E-2	1.98E+3	50.7	1.98E+0	3.62	5.18E-2	2.10E+3	39.6	1.19E+0	3.82	4.99E-2	1.81E+3
49.5	2.72E+0	5.28	6.29E+1	8.08E+2	67.4	6.16E+0	4.53	1.73E+1	1.81E+3	54.8	4.56E+0	4.75	9.54E+0	1.65E+3
68.9	8.96E+0	6.24	4.40E+1	5.62E+1	87.3	1.61E+1	5.46	8.87E+1	3.36E+2	64.0	8.60E+0	5.79	5.87E+1	5.56E+2
81.0	2.34E+1	7.29	2.12E+0	1.64E+1	116.1	4.42E+1	6.26	1.97E+1	5.34E+1	75.8	1.61E+1	6.80	2.74E+1	6.09E+1
101.0	4.61E+1	8.35	4.42E-1	8.02E+0	129.5	5.63E+1	7.10	1.99E+0	2.35E+1	78.3	1.69E+1	7.84	1.88E+0	2.59E+1
144.2	6.61E+1	9.40	1.84E-1	4.59E+0	141.7	6.72E+1	8.13	6.25E-1	1.20E+1	102.9	4.73E+1	9.32	5.54E-1	1.12E+1
FD	4.77E+1	10.34	9.67E-2	2.97E+0	FD	4.25E+1	9.11	3.02E-1	6.72E+0	118.8	5.91E+1	10.67	2.41E-1	5.39E+0
FDI	2.54E+0	11.29	5.75E-2	1.99E+0	FDI	1.89E+0	10.12	1.58E-1	3.87E+0	127.8	7.04E+1	12.04	1.08E-1	2.73E+0
		12.28	3.41E-2	1.39E+0			11.05	8.75E-2	2.41E+0	FD	4.30E+1	13.45	5.11E-2	1.44E+0
		13.28	2.18E-2	9.96E-1			12.00	4.99E-2	1.56E+0	FDI	2.56E+0	14.88	2.49E-2	8.01E-1
		14.31	1.37E-2	7.42E-1			12.99	2.83E-2	1.06E+0			16.30	1.21E-2	4.93E-1
		15.38	8.75E-3	5.76E-1			13.93	1.79E-2	7.56E-1			17.71	6.49E-3	3.29E-1
		16.38	6.10E-3	4.66E-1			14.90	1.14E-2	5.58E-1			19.12	3.40E-3	2.43E-1
		17.41	4.11E-3	3.91E-1			15.84	7.20E-3	4.37E-1			20.53	1.85E-3	1.96E-1
		18.47	3.31E-3	3.28E-1			16.79	4.88E-3	3.54E-1			21.92	1.23E-3	1.65E-1
		19.51	2.34E-3	2.85E-1			17.73	3.35E-3	2.97E-1			23.32	8.32E-4	1.45E-1
		20.51	1.77E-3	2.53E-1			18.64	2.36E-3	2.59E-1			EDI	5.18E-4	1.17E-1[c]
		21.56	1.49E-3	2.25E-1			19.71	1.71E-3	2.26E-1					
		EDI	1.25E-3	1.56E-1[c]			20.70	1.24E-3	2.04E-1					
							EDI	8.49E-4	1.57E-1[c]					

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) The O_2 exposure is discussed in Section 6.0.
(c) Final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution

5.4.3 BRF-16 with AP-101 Simulant

The BRF-16 test (single process cycle) loading results with AP-101 simulant are presented in Table 5.17 and Figure 5.29 to Figure 5.31. The BRF-16 resin loading resulted in rapid Cs breakthrough; after one process cycle, it was eliminated from the testing matrix. The onset of Cs breakthrough was immediate with the first sample collected exceeding the contract limit of 0.091% C/C_o. The total Cs loading was ~1.6 mg/g. The elution profile is provided in Figure 5.24 and Figure 5.27 (data are provided in Table 5.17).

The poor performance was attributed largely to leakage of the exterior ion exchange material coating the resin surface (see Figure 5.12) and channeling.

Table 5.17. BRF-16 (Blue Column) Effluent Cs Concentration During AP-101 Simulant Loading and Elution

Cycle 1				
Load		Elute		Resin Cs Conc.[(a)]
BV	% C/C_o	BV	C/C_o	µg/g
5.3	5.51E-1	0.96	7.66E-3	1.61E+3
13.5	3.83E+0	1.99	1.76E-2	1.61E+3
33.7	2.71E+1	3.12	4.41E-2	1.61E+3
42.8	3.89E+1	4.25	2.66E+1	6.24E+2
50.2	4.44E+1	5.29	1.68E+1	5.35E+1
70.2	6.00E+1	6.22	1.37E+0	1.16E+1
FD	4.96E+1	7.29	2.37E-1	3.24E+0
FDI	1.65E+0	8.37	5.27E-2	1.37E+0
		9.44	1.61E-2	8.05E-1
		10.41	7.00E-3	5.83E-1
		11.38	4.62E-3	4.36E-1
		12.41	2.89E-3	3.38E-1
		13.40	1.87E-3	2.77E-1
		14.45	1.42E-3	2.29E-1
		15.53	1.08E-3	1.91E-1
		16.54	8.32E-4	1.63E-1
		17.59	6.06E-4	1.42E-1
		18.66	7.31E-4	1.16E-1
		19.71	6.98E-4	9.22E-2
		20.73	6.13E-4	7.17E-2
		21.77	5.39E-4	5.34E-2
		EDI	4.92E-4	4.86E-3[(b)]

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) Final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution.

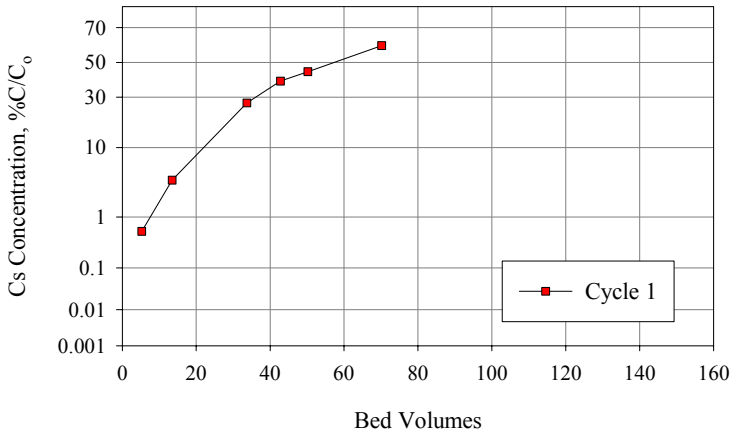


Figure 5.29. BRF-16 Cs Loading Profile with AP-101 Simulant, 1 Cycle Only

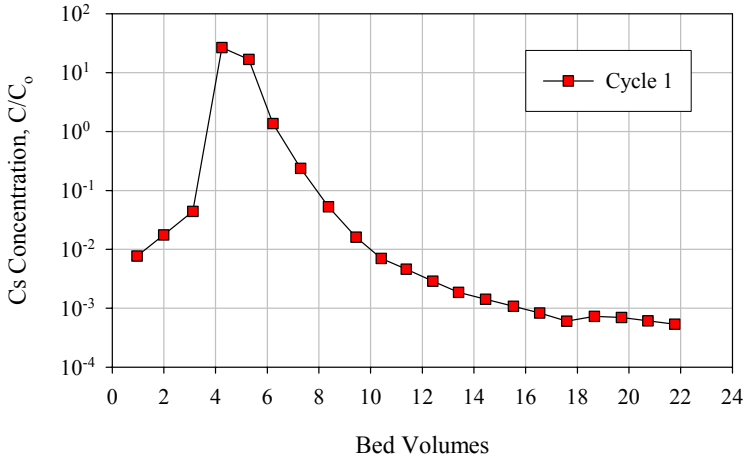


Figure 5.30. BRF-16 Cs Elution Profile, 1 Cycle Only

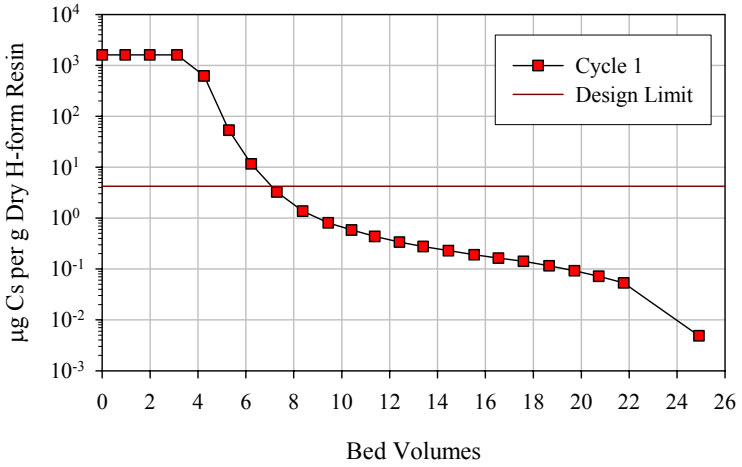


Figure 5.31. Residual Cs as a Function of Elution Volume, BRF-16, 1 Cycle Only

5.4.4 SL-644 with AP-101 Simulant

Only two process cycles were tested with SL-644, one cycle before (Cycle 1) and one cycle after (Cycle 2) oxygen exposure. The loading process cycles are shown in Figure 5.32, and the data are presented in Table 5.18. The increase in flowrate at 89 BVs from 1.4 to 2.9 BV/h resulted in a slight increase in the effluent Cs concentration (note the bow in the loading curve). This indicated that a slower flowrate for this waste matrix (high K waste) provided favorable Cs exchange.

The onset of Cs breakthrough was nearly immediate with the AP-101 matrix. The contract limit of 0.091% C/C_o was reached after processing 73 BVs and 38 BVs for process Cycles 1 and 2, respectively. The 50% breakthrough occurred at 220 BVs and was extrapolated to ~140 BVs for the second cycle.

Nominally 5.4 and 3.3 mg Cs per g dry H-form resin were loaded onto the SL-644 during the first and second process cycles, respectively. Even after the oxidative attack, more Cs was loaded on the SL-644 than was loaded onto the fresh RF resin.

The SL-644 elution results are presented in Figure 5.33, Figure 5.34, and Table 5.18. The elution profile shapes did not change between the pre- and post-oxygen exposure; the magnitude of the peak Cs concentration did change, however, consistent with the reduced Cs loading. The effluent Cs concentration of 1E-3 C/C_o was extrapolated to be reached after processing ~25 BVs.

The contract (mass basis) limit for residual Cs in SL-644 (2.8 μg/g) (Burgeson et al. 2004) was less than that of the RF resin (4.2 μg/g) (see Section 3.11). The mass limit difference was associated with the difference in H-form bulk densities of RF (0.43 g/mL) and SL-644 (0.66 g/mL) that were previously reported (Fiskum et al. 2004). The contract limit was reached after processing 11 BVs eluate. Residual Cs concentrations on the SL-644 resin bed, 0.75 and 0.53 μg/g, were ~3 to ~5× below the contract limit. In contrast, the residual Cs on RF was ~15× below the contract limit.

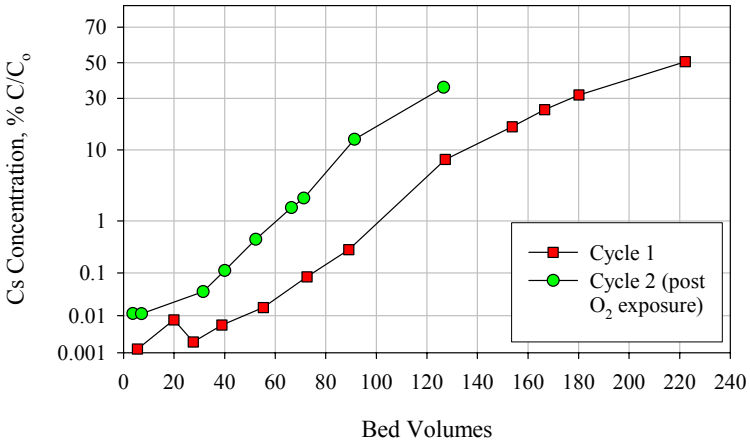


Figure 5.32. SL-644 Cs Loading Profiles with AP-101 Simulant

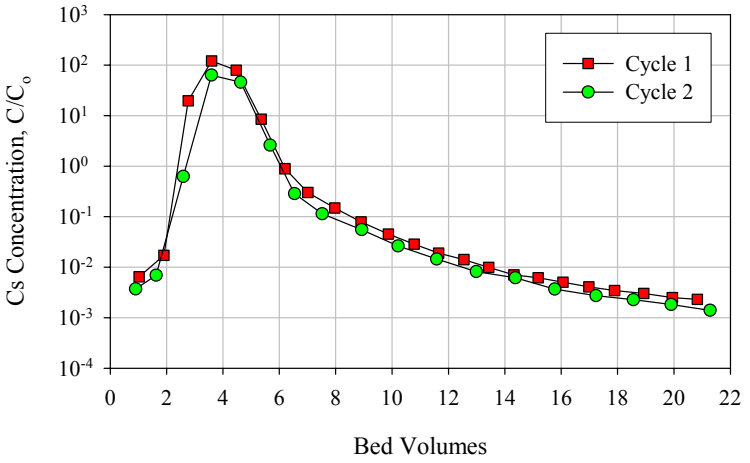


Figure 5.33. SL-644 Cs Elution Profiles

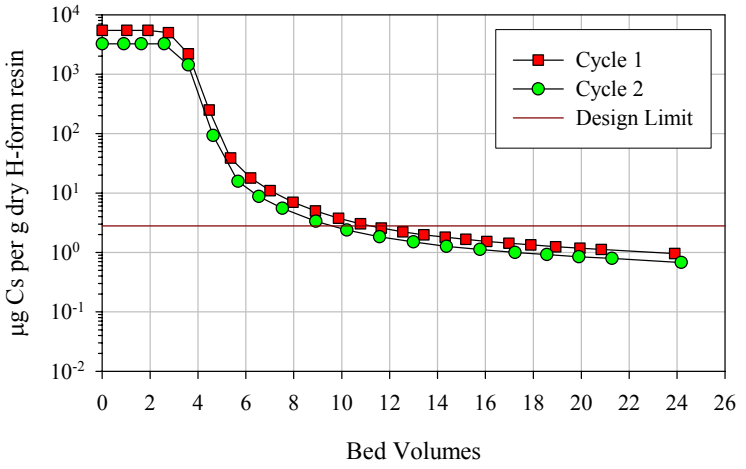


Figure 5.34. Residual Cs as a Function of Elution Volume, SL-644

Table 5.18. SL-644 (Blue Column) Effluent Cs Concentration During AP-101 Simulant Loading and Elution

Cycle 1					Cycle 2 (Post O_2 Exposure)[b]				
Load		Elute		Resin Cs Conc.[a]	Load		Elute		Resin Cs Conc.[a]
BV	% C/C_o	BV	C/C_o	µg/g	BV	% C/C_o	BV	C/C_o	µg/g
5.5	1.28E-3	1.03	6.49E-3	5.47E+3	3.7	1.13E-2	0.90	3.72E-3	3.24E+3
19.9	7.81E-3	1.91	1.72E-2	5.47E+3	7.1	1.12E-2	1.63	6.89E-3	3.24E+3
27.6	2.01E-3	2.77	1.97E+1	5.00E+3	31.5	3.78E-2	2.59	6.27E-1	3.23E+3
38.9	5.75E-3	3.60	1.21E+2	2.19E+3	40.0	1.12E-1	3.59	6.33E+1	1.43E+3
55.3	1.60E-2	4.47	7.96E+1	2.50E+2	52.3	4.73E-1	4.63	4.57E+1	9.27E+1
72.6	8.25E-2	5.36	8.50E+0	3.89E+1	66.4	1.65E+0	5.67	2.61E+0	1.57E+1
89.1	2.98E-1	6.20	8.91E-1	1.79E+1	71.3	2.33E+0	6.54	2.86E-1	8.71E+0
127.3	7.75E+0	7.01	3.02E-1	1.10E+1	91.4	1.30E+1	7.52	1.14E-1	5.54E+0
153.8	1.73E+1	7.97	1.49E-1	7.03E+0	126.6	3.58E+1	8.92	5.51E-2	3.35E+0
166.6	2.45E+1	8.90	7.90E-2	4.99E+0	FD	3.61E+1	10.21	2.63E-2	2.39E+0
180.2	3.17E+1	9.87	4.52E-2	3.76E+0	FDI	1.23E+0	11.58	1.45E-2	1.83E+0
222.2	5.06E+1	10.78	2.86E-2	3.04E+0			12.99	8.15E-3	1.50E+0
FD	3.59E+1	11.65	1.90E-2	2.58E+0			14.37	6.16E-3	1.26E+0
FDI	1.89E+0	12.55	1.41E-2	2.23E+0			15.77	3.67E-3	1.12E+0
		13.43	9.94E-3	1.98E+0			17.23	2.74E-3	1.00E+0
		14.32	7.05E-3	1.81E+0			18.56	2.27E-3	9.19E-1
		15.18	6.16E-3	1.66E+0			19.90	1.83E-3	8.49E-1
		16.06	5.06E-3	1.53E+0			21.28	1.40E-3	7.95E-1
		16.98	4.08E-3	1.43E+0			EDI	1.43E-3	6.79E-1[c]
		17.89	3.44E-3	1.34E+0					
		18.93	3.04E-3	1.25E+0					
		19.94	2.51E-3	1.18E+0					
		20.83	2.30E-3	1.12E+0					
		EDI	1.94E-3	9.59E-1[c]					

(a) Concentration of Cs remaining on resin as micrograms Cs/g dry H-form resin after elution aliquot processed.
(b) The O_2 exposure is discussed in Section 6.0.
(c) Final residual Cs on the resin bed after elution and water rinse.
Notes: FD = feed displacement; FDI = water rinse following feed displacement; EDI = water rinse following elution

5.5 Summary of Resin Performance

Figure 5.35 summarizes the process loading performance characteristics, and Figure 5.36 summarizes the process elution performance of the Wave 1 resins. The ground-gel RF resin performance (third process cycle) was previously reported (Fiskum et al. 2004) and was included for information only. The spherical RF resin has high Cs capacity based on the AZ-102 matrix test, but the high K matrix (AP-101) challenged the Cs selectivity. The BRF-14, -15, and -17 RF formulations performed nearly the same in the AP-101 Cs exchange and elution.

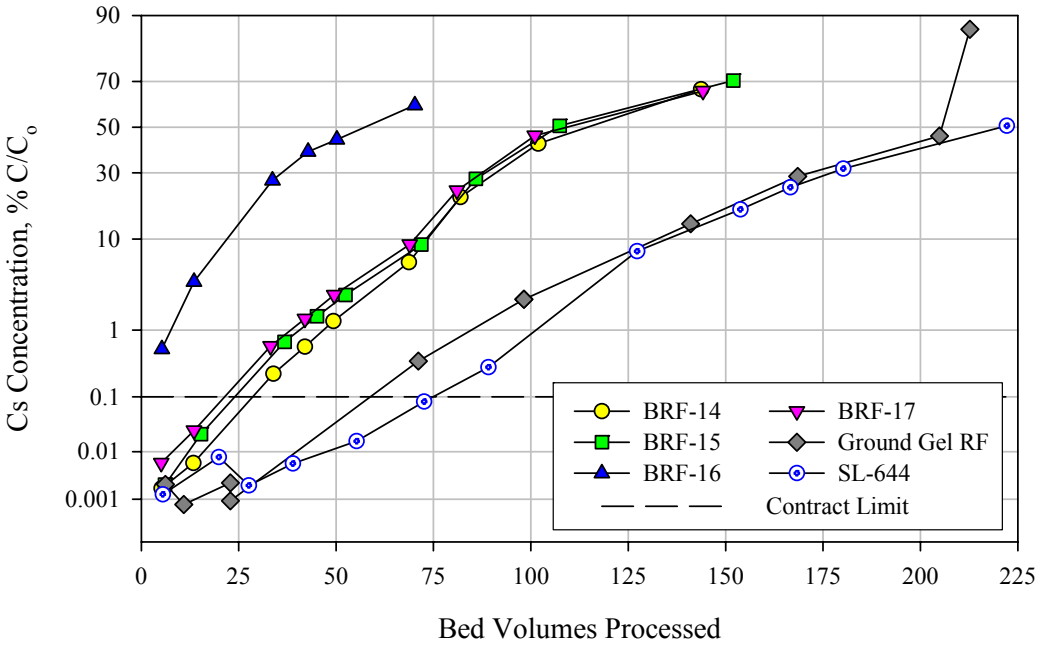


Figure 5.35. Comparative Loading Profiles for Wave 1 Resins, First Process Cycle with AP-101 Simulant

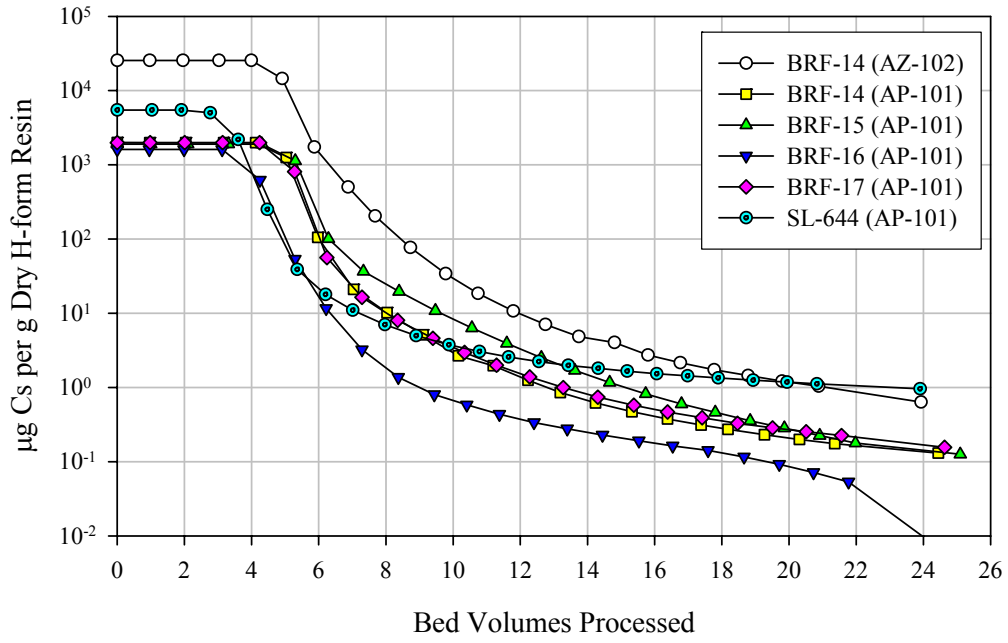


Figure 5.36. Comparative Residual Cs Elution Profiles for Wave 1 Resins, First Process Cycle

Process results (discussed previously) are compared in Table 5.19. The SL-644 AZ-102 simulant (Fiskum et al. 2004) and actual waste test processing (Fiskum et al. 2003) results are also included for reference. However, direct comparison of the actual AZ-102 waste testing with SL-644 and simulant AZ-102 testing with RF were confounded since the actual waste test used a different SL-644 production lot and sieve fraction (smaller size cut).[(a)] Furthermore, the actual waste feed had 40% higher Cs concentration than the simulant (70 μg/mL versus 50 μg/mL).

Table 5.19. Comparative Performance Summary of Wave 1 Resins

Resin	Cs load, mg Cs per g H-form resin	Breakthrough in BV			Post-Elution Residual Cs, μg/g
		Onset[(f)]	Contract Limit	50%	
AZ-102					
BRF-14[(a)]	26	40	68	170	0.72
SL-644[(b)]	40	77	102	187	3.9
SL-644[(c)]	40[(e)]	68	70	93	na
AP-101					
BRF-14[(a)]	2.0	<5	30	115	0.13
BRF-15[(a)]	2.0	<5	27	113	0.13
BRF-16[(d)]	1.6	<1	<1	55	0.005
BRF-17[(a)]	2.0	<5	25	113	0.16
SL-644[(d)]	5.4	<20	73	220	0.96

(a) Values for Cycles 1 and 2 were averaged.
(b) Simulant AZ-102 process test (Fiskum et al. 2004) using Lot # C-01-11-05-02-35-60 18 to 40 mesh.
(c) Actual AZ-102 waste concentrated to 4.61 M Na where total Cs was 70 μg/mL (Fiskum et al. 2003). The SL-644 was a small particle size fraction (212- to 425-μm dry sieved fraction) of Lot 010319SMC-IV-73.
(d) Cycle 1 process values.
(e) Calculated based on eluate composition: 0.508 mg/mL Cs ×151 mL/1.9 g H-form resin.
(f) The onset of breakthrough was defined as the BVs processed before the C/C_o starts to increase.
na = not applicable

5.6 Shrink-Swell Characteristics

The relative resin expansion and contraction characteristics are summarized in Figure 5.37, and actual BVs are provided in Table 5.20. The process step number defined in Figure 5.37 is defined in Table 5.20. The in-column pre-treated resin Na-form (Step 4) was used as the relative volume basis.

All spherical RF resins resulted in similar expansion factors of 1.2. The SL-644 demonstrated the highest volume swings of 30% before oxygen attack. The resin bed initially expanded to the normal Na-form volume of 20.7 mL during the initial phases of oxidative attack (discussed in detail in Section 6.0).

(a) Actual waste testing used SL-644 dry-screened (in the as-received condition) to 212 to 425 microns. Later SL-644 productions increased particle size in response to hydraulic issues. The current test was conducted with wet Na-form resin screened to 18 to 40 mesh (425 to 1000 microns). Other SL-644 production parameters were also being modified by the manufacturer during the testing phases and therefore were subject to lot-to-lot variation.

As oxidative attack continued, the resin bed expansion continued to 27.0 mL. Eventually the resin bed compressed or “crushed” to 21.0 mL at which point the pressure-relief valve was triggered and flow through the bed ceased. The resin bed relaxed to the pre-crushed volume within 2 h after the cessation of flow and gradual pressure relief.

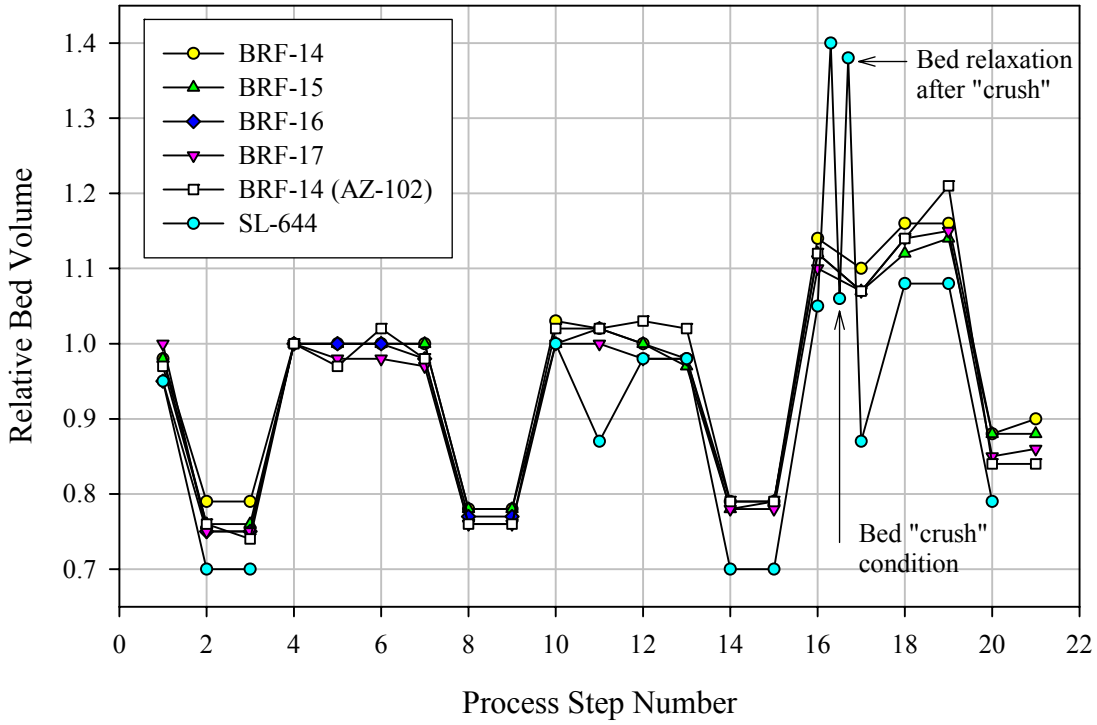


Figure 5.37. Relative BV Changes, Wave 1 Resins (AP-101 Simulant, Except as Noted)

Table 5.20. Actual Bed Volumes as a Function of Feed Matrix, Wave 1 Resins

Feed matrix	Process Step Number	Resin Bed Volume, mL					
		Yellow BRF-14	Green BRF-15	Blue BRF-16	Pink BRF-17	White BRF-14[a]	Blue SL-644[b]
Pretreatment							
Baseline volume, mL	NA	18.2	18.2	18.8	18.5	18.2	19.8
1 M NaOH soak/DI rinse	1	17.9	17.9	18.8	18.5	17.6	18.8
0.5 M HNO_3	2	14.5	13.8	14.1	13.8	13.8	13.8
DI water	3	14.5	13.8	14.1	13.8	13.5	13.8
Cycle 2 Processing							
0.25 M/0.5 M NaOH[c]	4	18.2	18.2	18.8	18.5	18.2	NA
feed, AP101 simulant	5	18.2	18.2	18.8	18.2	17.6	NA
feed displacement	6	18.2	18.2	18.8	18.2	18.5	NA
DI water	7	18.2	18.2	18.5	17.9	17.9	NA
0.5M HNO_3	8	14.1	14.1	14.5	14.1	13.8	NA
DI water	9	14.1	14.1	14.5	14.1	13.8	NA
Cycle 2 Processing							
0.25 M/0.5 M NaOH[c]	10	18.8	18.2	NA	18.5	18.5	19.8
feed, AP101 simulant	11	18.5	18.5	NA	18.5	18.5	17.3
feed displacement	12	18.2	18.2	NA	18.2	18.8	19.5
DI water	13	17.9	17.6	NA	18.2	18.5	19.5
0.5 M HNO_3	14	14.5	14.1	NA	14.5	14.5	13.8
DI water	15	14.5	14.5	NA	14.5	14.5	13.8
Regeneration/Oxidation test							
0.25 M/0.5 M NaOH[c]	16	20.7	20.4	NA	20.4	20.4	20.7/27.6/21.0[d]
Cycle 3 Processing							
feed, AP101 simulant	17	20.1	19.5	NA	19.8	19.5	17.3
feed displacement	18	21.0	20.4	NA	21.0	20.7	21.4
DI water	19	21.0	20.7	NA	21.4	22.0	21.4
0.5 M HNO_3	20	16.0	16.0	NA	15.7	15.4	15.7
DI water	21	16.3	16.0	NA	16.0	15.4	NR

(a) White column tested AZ-102 simulant instead of AP-101 simulant.
(b) SL-644 from production batch C-01-05-28-02-35-60 (250-gal production batch).
(c) The 0.25 M NaOH regeneration solution was used for SL-644.
(d) The three volumes are associated with the expanded resin bed form: initial expansion to 20.7 mL, oxidative attack caused resin bed to swell to 27.6 mL, and the 10 psig pressure crushed the resin bed to 21.0 mL. When pressure equilibrated, the resin bed expanded back to 27.6 mL.
NA = not applicable; NR = not recorded

5.7 Resin Dry-Bed Densities

The dry-bed densities of the H-form and Na-form resins were derived from the ion exchange testing. The densities are summarized in Table 5.21. The bed density in water was dependent on the resin ionic form. In the case of Na-form resin, the bed density was equivalent to the density in 0.1 M NaOH. In the case of H-form resin, the bed density was equivalent to the density in the eluant. In all cases, the dry-bed

density mass was based on the calculated dry H-form resin mass loaded in the column. The overall error was estimated to be ±5%. To calculate the Na-form resin dry-bed density relative to the Na-form mass, the resin-specific mass increase factor (I_{Na}) will need to be determined.[(a)] Once loaded and tapped to constant volume, the resin BVs in the actual column tests were not disturbed, vibrated, or tapped; packing resulted only from resin-bed shrinking and swelling in the course of normal in-column pre-conditioning and processing steps.

Table 5.21. Dry-Bed Resin Densities (H-form Mass Basis)

Resin ID	H-form Dry Resin Mass, g	Average Dry-Bed Density[(a)]			
		Na-form			H-form
		0.5 M NaOH, g/mL	AP-101 Simulant, g/mL	0.1M NaOH, g/mL	0.5 M HNO_3, g/mL
BRF-14[(b)]	5.99	0.33/0.29	0.32/0.30	0.33/0.28	0.41/0.37
BRF-14[(b)]	5.99	0.33/0.29	0.33/031[(c)]	0.32/0.29	0.42/0.39
BRF-15[(b)]	6.23	0.34/0.31	0.34/0.32	0.34/0.31	0.44/0.39
BRF-16	3.57	0.19	0.19	0.19	0.25
BRF-17[(b)]	6.15	0.33/0.30	0.34/0.31	0.34/0.29	0.43/0.39
SL-644[(b)]	4.21[(d)]	0.21/0.15[(e)]	0.24/0.24	0.22/0.20	0.30/0.27

(a) The resin mass was based on the calculated dry H-form resin.
(b) The second value is the post-oxygenated regeneration solution processing.
(c) The simulant was AZ-102.
(d) Based on the dried Na-form mass of 5.36 g, and conversion to the estimated H-form mass of 4.21 g using the I_{Na}-factor = 1.28; this factor may be artificially high from waters of hydration associated with the Na ion (Arm and Blanchard, 2004) and therefore subject to uncertainty.
(e) The SL-644 regeneration solution was 0.25 M NaOH.

(a) Determination of the I_{Na} mass increase factor was beyond the scope of the current testing.

6.0 Effect of Resin Oxidation on Cs Ion Exchange

Oxygen exposure was shown to adversely affect SL-644 Cs ion exchange effectiveness (Brown et al. 1995b). Furthermore, SL-644 was shown to take up O_2 more rapidly from gaseous headspace than ground-gel RF resin (Brown et al. 1995b). A comparative test was conducted to evaluate relative damage effects from oxygen consumption based on follow-on Cs ion exchange load performance for both the spherical RF resin and SL-644 resin. The test was embedded with the Wave 1 Cycle 3 processing by incorporating an extended regeneration condition (~9 L) with oxygen-rich solution in an effort to create a worst-case or bounding oxidation condition. Resin oxidation effects were monitored by visual resin bed observations, measuring effluent dissolved oxygen (DO), and comparing follow-on Cs ion exchange performance (Cycle 3) with Cycle 2 performance. This report section details the oxygen consumption/degradation test and results.

6.1 Experimental

The Wave 1 resins, BRF-14, -15, -17, and SL-644, were tested. Overall process parameters were described in Section 5.0. Specific (oxidative) regeneration process parameters are summarized in Table 6.1.

Table 6.1. Resin Oxidative Regeneration Test Parameters

Parameter	BRF-14 (white, AZ-102)	BRF-14 (yellow, AP-101)	BRF-15	BRF-17	SL-644
Resin mass, g[(a)]	5.99	5.99	6.23	6.15	4.21[(b)]
Resin BV, mL	18.2	18.2	18.2	18.5	19.8
Regeneration solution	0.5 M NaOH	0.5 M NaOH	0.5 M NaOH	0.5 M NaOH	0.25 M NaOH
Total process time, h	170	170[(c)]	170	169	113
Process volume, mL	9,530	9,500	10,300	8,170	6,510
Flow rate, mL/min	0.93	0.93[(c)]	1.0	0.80	0.96
Flow rate, BV/h	3.07	3.06[(c)]	3.34	2.60	2.90
Processed volume, BV	524	522	566	442	329
Estimated dissolved O_2 supplied, mmoles	4.7	5.5	5.9	4.0	3.3

(a) Dry H-form resin mass
(b) The H-form mass was calculated by dividing the dry Na-form mass of 5.36 by the I_{Na} factor of 1.28.
(c) Flow was interrupted with a stop-flow condition; see Section 6.1. The time and flow rates reflect the interval associated with solution flow through the ion exchanger and did not include the time associated with the stop-flow condition.

Large reservoir volumes of regeneration solutions were prepared. The BRF-17 (pink column) and BRF-14 (white column) shared a single recirculation bottle containing 3.0 L 0.5 M NaOH. The BRF-14 (yellow column) and BRF-15 (green column) shared a single recirculation bottle containing 3.0 L 0.5 M NaOH. The SL-644 (blue column) regeneration solution contained a pro-rated volume of 1.5 L 0.25 M NaOH.

Oxygen saturation was obtained in each regeneration solution before processing by bubbling 99% pure oxygen into the feed solution with stirring. The oxygen saturation was maintained for the duration of the test except for one flow interruption condition when the oxygen feed tank had emptied.

The regeneration solution was processed at the nominal flowrate of 3 BV/h. The first 6 BVs processed were collected separately and discarded. During the course of the remaining test, the effluent solution was re-circulated back into the (regeneration) feed bottle.

The BRF-14 (yellow column) was subjected to an unplanned stopflow condition for 18 hours after processing the regeneration solution for 141 hours (433 circulated BVs) because a valve was inadvertently turned to the wrong position. No other columns were affected. The valve closure caused a 0.99 L (54 BV) loss of 0.5 M NaOH from the regeneration bottle for the duration of the test. The regeneration bottle also supplied the BRF-15 (green column); however, its experimental integrity was unaffected despite the fact that a smaller reservoir volume was available for recirculation through the BRF-15 resin bed. Because the problem occurred near the end of the test, the regeneration solution reservoir was not re-supplied to the original 3-L working volume. The process time for the yellow column was extended 18 hours such that the total O_2 exposure of the resin would be equivalent to that of the other RF resins.

The DO was measured in the feed and effluent using an Orion Model 850A meter equipped with an Orion Model 083005D polarographic probe (Thermo Electron Corp., Beverly MA). Calibration was conducted according to the manufacturer directions using a single point source of water-saturated air. Feed samples were taken from the feed bottle for DO measurement. Effluent samples were collected from the effluent line. The effluent sampling process took nominally 10 min to obtain a 9-mL sample. The effluent line was placed at the bottom of a 20-mL collection vial such that the volume collected first would be displaced upward. This provided the best chance to minimize air contact (and thus O_2 exchange) in the last half of the collected sample. Care was taken to not perturb the sample any more than necessary during sampling and transit to the analysis workstation. The analysis workstation was located in the same fume hood as the BRF-14 (yellow) and BRF-15 columns. Samples collected from the SL-644 and BRF-14 (white) and BRF-17 columns had a longer time interval between collection and analysis because of the requisite steps required to transport the samples in compliance with radiological control procedures. The DO probe was promptly and gently placed at the bottom of the sample in an effort to measure the sample fraction with the least potential for contact with air. Because of the inherent limitations of the method, all DO measurements were taken for indication only.

The working range of the DO measurement system was 0 to 20 µg/mL. In several cases, the feed DO concentration exceeded the upper range. In these cases, the maximum oxygen concentration was calculated based on Lange and Zander (1986) estimated Bunsen coefficients for the given NaOH concentration. Lange and Zander reported a linear relationship between the log of the ratio of Bunsen coefficients of oxygen in water (a_o) and in NaOH solutions from 1 to 4 M (a). Using the best-fit linear relationship provided in Equation 6.1, the Bunsen coefficients for 0.5 M and 0.25 M NaOH were calculated, where C is the molar NaOH concentration.

$$\log\left(\frac{a_o}{a}\right) = C * 0.169 \tag{6.1}$$

The maximum oxygen concentrations (μg/g) were calculated from the Bunsen coefficients according to Equation 6.2.

$$\mu g/mL\ O_2 = \frac{a * p_o}{R} * \left(\frac{P}{T}\right) * \frac{1 L}{1000\,mL} * \frac{32\,g}{mole} * \frac{10^6\ \mu g}{g} \qquad (6.2)$$

where R = 0.082056 L-atm/(K-mole)
p_o = partial pressure of oxygen
P = ambient pressure, 1 atm
T = temperature, 298 K.

The DO saturation for 0.5 M NaOH was calculated to be 26.0 μg/mL, and for 0.25 M NaOH, the saturated value was 28.6 μg/mL. In several instances, the feed DO measurements were measured as >20 μg/mL. Selected samples were serially diluted in an effort to better define the DO concentration; however, the dilution-corrected DO values were higher than the saturated values. If the regeneration solution was supersaturated at the sampling point, the oxygen was thought to attain the saturated value through off-gassing by the time the solution was pumped to the column. Indeed, an ever-increasing air gap (with corresponding decrease in solution height) above the resin bed was observed, supporting this assumption. The column systems were therefore periodically opened at the top fittings (so as not to disturb the resin beds), and the fluid levels above the resin beds were topped off to prevent the gradual displacement of the headspace fluid.

Occasionally, the feed solution oxygen was below the saturated value. Gas flowrate inconsistencies occurred as the oxygen bottle pressure decreased. The oxygen gas bottle emptied once (after ~375 BVs were processed), resulting in a feed DO of ~9 μg/mL. The feed DO concentrations are shown in Figure 6.1 as a function of time.

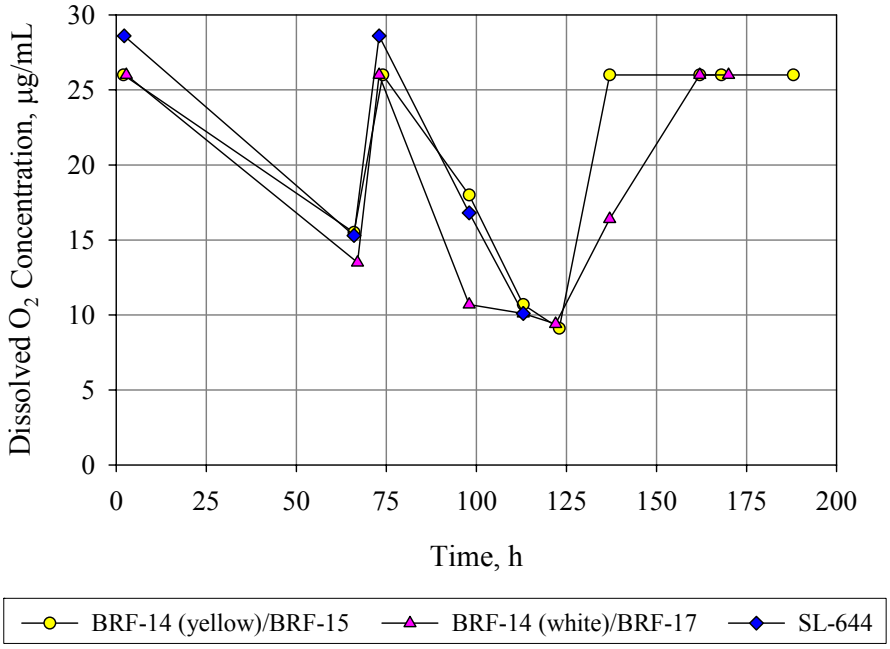


Figure 6.1. Feed DO Concentration as a Function of Time

The µmoles O_2 consumed during the oxidative regeneration process was estimated from the influent, effluent O_2 concentrations, and process volumes according to Equation 6.3.

$$\frac{(O_F - O_E) * V_i}{FW} = \mu\text{moles } O_2 \qquad (6.3)$$

where O_F = feed O_2 concentration, µg/mL
O_E = effluent O_2 concentration, µg/mL
V_i = interval process volume, mL
FW = formula weight of O_2, 32 µg/µmole.

All measurements were taken for indication only. The measurement system was limited in concentration range (0 to 20 µg O_2 per mL) and with regard to sample handling issues. More accuracy would have been associated with in-line measurement systems. Therefore, the data set should not be used as a basis of exact values. However, comparisons, trends, and inferences can be made despite the data limitations.

6.2 Physical Results

The SL-644 manifested dramatic physical responses to the oxidative regeneration test. The resin continually expanded and bled intense color (presumably from organics) into the effluent. The Na-form resin bed expanded from 19.8 mL to 27.6 mL (39% expansion) during the oxidative feed re-cycle. Catastrophic failure occurred after processing 330 BVs through the bed, manifested in plugging with a

pressure drop ≥10 psi.[(a)] The increased pressure caused the resin bed to compact or crush from 28 mL to 21 mL. Once pressure was relieved, the resin bed expanded, and in ≤2 h reached the previously-observed 27.6 mL volume. Photographs of the SL-644 resin bed before and after oxidative regeneration testing are shown in Figure 6.2; note the difference in resin bed height and intense coloration of the regeneration solution.

a) (b)

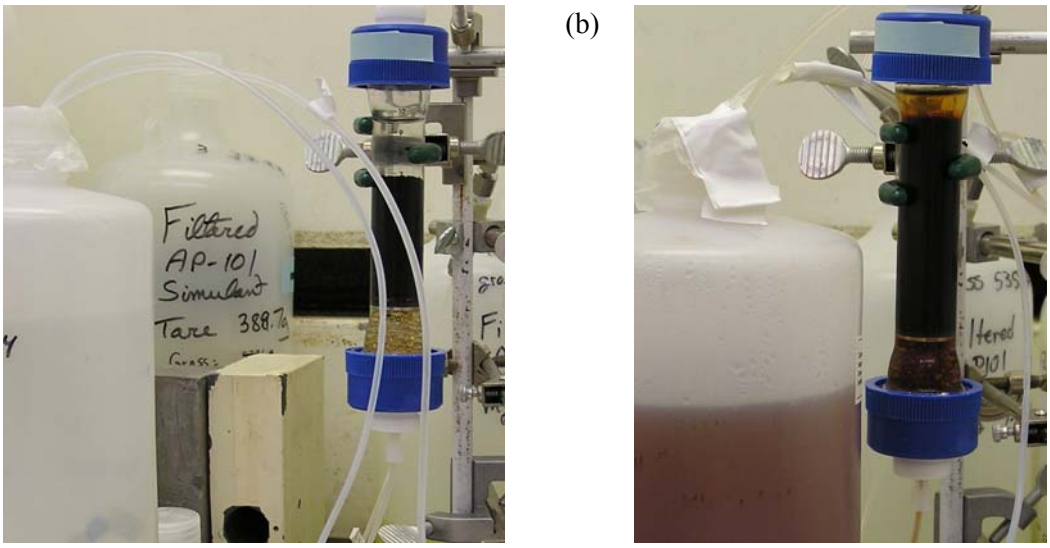


Figure 6.2. SL-644 Resin Bed and Regeneration Solution
(a) Before and (b) After Dissolved Oxygen Exposure

Subsequent processing through the SL-644 resin bed with fresh 0.25 M NaOH continued pressure buildup with slow solution transport across the bed. Flow through the bed was re-established when processing was switched to AP-101 simulant. This was accompanied with resin bed contraction to 17.3 mL, a 37% contraction from 27.6 mL. After initial processing with AP-101 simulant, clumping of the resin was noticed. The top ¼ of the resin bed contracted slightly from the column walls. The resin sides expanded to fill the gap during the water rinse. The eluted resin bed showed evidence of the clumps as the top surface was no longer flat. Figure 6.3 shows photographs of the resin bed at various process stages.

(a) The calculated bed permeability was reduced from 1E-10 m^2 (as reported by Arm et al. [2006] under similar conditions) to <5E-14 m^2, where viscosity was assumed to equal that of water (1 centipoise), the crushed bed height was 6.7 cm, the flowrate was 2.9 BV/h, and the pressure drop was 10 psi.

a) b) c)

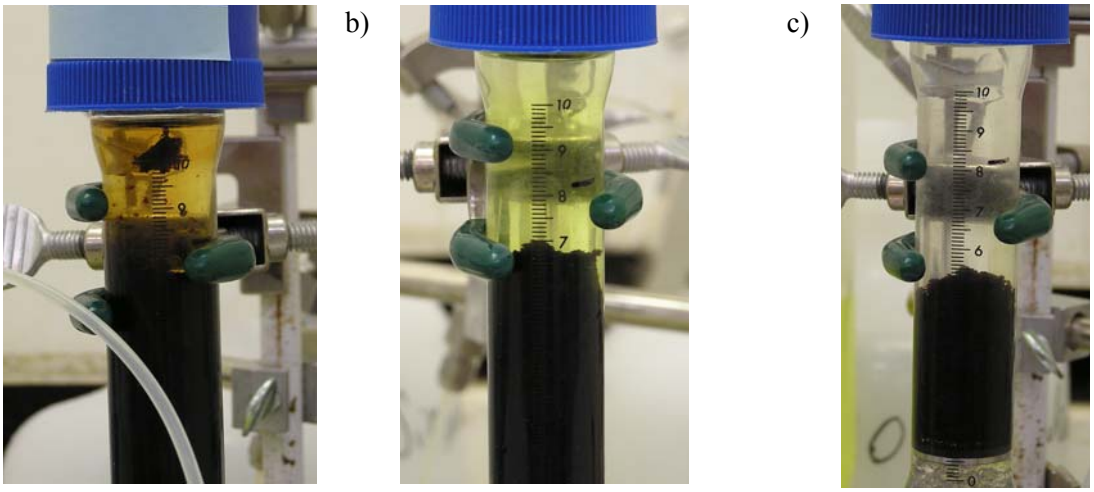


Figure 6.3. SL-644 Resin, Processing After Oxidative Regeneration
a) initial AP-101 load, b) mid-process AP-101 load, c) post-elution

In contrast, the RF resins only expanded 10% with the oxygen exposure, and process flow continued without system pressurization. The BRF-14 (white) and BRF-17 systems after 7 days processing are shown in Figure 6.4. The color intensity of the regeneration solution was significantly less than that of the SL-644 regeneration solution, indicating less organic dissolution or breakdown.

Figure 6.4. BRF-14 (Left) and BRF-17 (Right) with Regeneration Solution after 7 Days Processing

A dark (black or very dark red) band developed from the top of the RF resin bed, expanding downward as processing with the regeneration solution continued. By the end of processing, the dark band depth was nominally 2 to 2.5 cm (nearly 40% of the resin height). The dark band appearance is illustrated in Figure 6.5 from the eluted BRF-14 (yellow) column following the third process cycle, post-elution.[(a)] All RF resins tested developed a dark band at the top. In all cases, this was attributed to oxidative attack from dissolved oxygen in the feedstocks. In other processing scenarios (test Waves 2, 3, and 4a), the band was typically only 0.5 cm deep.

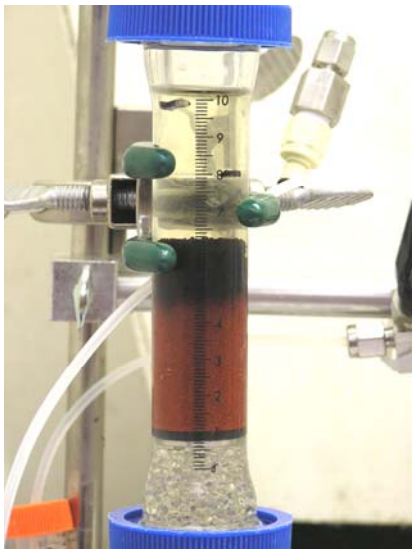


Figure 6.5. BRF-14 (Yellow Column), Post-Elution Following Third Process Cycle (Note the dark band at the top of the resin bed.)

Adamson et al. (2006) reported the darkened resin was associated with conversion of the aromatic ring to a quinone-like structure as determined through infrared analysis and an increase in total oxygen content. Hubler et al. (1995) also reported ground-gel RF structural modification related to oxidation as the formation of ketone and quinone structures. The oxidative changes in the ground-gel RF formulation are expected to be similar to the changes found in the spherical form, despite the different manufacturing conditions. A proposed mechanism for RF resin oxidation is shown in Figure 6.6.[(b)] Based on observations reported by Hubler et al. (1995), other oxidative side reactions may be possible.

(a) The band was most striking when the resin was in the lighter color H-form and was difficult to discern from the Na-form resin photographs.

(b) The chemistry and mechanism of RF oxidation was supplied by RL Albright, WTP Consultant, personal communication, 2005.

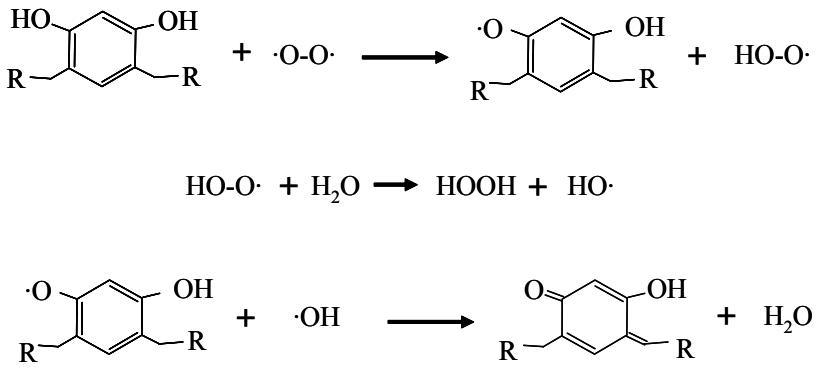


Figure 6.6. Proposed Chemistry and Mechanism for RF Oxidation

This mechanism implies that one mole of dissolved oxygen is required to destroy one mole of RF. Converting to a gram resin basis, 1 mmole DO will destroy 0.122 g H-form resin. This corresponds to a theoretical maximum (under the proposed mechanism) of 12% resin destruction on a 1-mmole DO exposure/g H-form resin basis. The hydrogen peroxide byproduct is likely to cause additional RF resin oxidation. The maximum destruction from the combined oxygen and hydrogen peroxide pathways would result in a 2:1 mole ratio (two moles RF destroyed : 1 mole O_2), which is equivalent to 24% destruction per mole O_2/g H-form resin.

6.3 Dissolved Oxygen Consumption

The RF and SL-644 resins were shown to consume oxygen during processing with the oxygen-saturated regeneration solution. The effluent DO concentrations are summarized in Table 6.2. In most cases, the effluent DO concentrations were significantly less than the influent DO concentrations. Samples taken early appeared to have higher effluent DO concentrations than those taken during later processing. The difference in effluent DO readings was attributed to constraints in the experimental design. The SL-644, BRF-14 (white), and BRF-17 column tests were conducted in fume hoods separate from the DO meter. The sample travel time between fume hoods probably resulted in slightly elevated effluent DO readings, which in turn would reduce the calculated oxygen consumption. Better efficiencies in sample handling were achieved toward the end of test. The three cases where >20 μg/mL DO was measured in the effluent may be attributed to probable cross-contamination with the feed and/or a trapped air bubble at the measurement surface.

Table 6.2. Effluent Dissolved Oxygen Concentrations

BRF-14 (yellow)		BRF-14 (white)		BRF-15		BRF-17		SL-644	
Sample Time, h	DO, µg/mL	Sample Time, h	DO, µg/mL	Sample Time, h	DO, µg/mL	Sample Time, h	DO, µg/mL	Sample Time, h	DO, µg/mL
2.2	<5.3	2.1	8.1	2.2	<3.2	2.4	20.6	2.4	>20[(a)]
66	1.2	66	5.0	66	2.6	66	7.3	66	2.2
74	>20[(a)]	72	8.3	74	>20[(a)]	72	6.7	73	5.3
98	8.2	97	8.7	98	8.5	97	2.0	98	2.0
113	9.0	113	8.8	113	8.9	113	9.0	113	1.4
122	8.2	122	4.6	122	8.0	122	4.7		
136	1.4	136	2.7	136	1.5	136	2.6		
162	3.2	162	1.8	162	3.4	162	2.8		
168	2.2	170	3.8	170	2.2	169	3.4		
188	2.1								

(a) Effluent concentrations > 20 µg/mL were considered to be outliers.

The O_2 consumption was evaluated for each resin. Most interval measurements indicated that >80% of the input O_2 was consumed. The observed drop in the RF O_2 consumption (~90 to ~122 h) was correlated to the decrease in the feed DO concentration. After the O_2 feed concentration was corrected, the consumption appeared to markedly increase. This observation may be an artifact of the measurement constraints. Online measurements (that avoided room air bias) conducted at SRNL using a significantly higher flowrate through an RF resin bed resulted in effluent DO concentrations less than or near the detection limit (Adamson et al. 2006).

The calculated SL-644 resin O_2 consumption rate was ~140 µmoles O_2 g^{-1} h^{-1}. Because of the inherent issues associated with the measurement method, this consumption rate is expected to be biased low. Brown et al. (1995b) reported a consumption rate of 780 µmoles O_2 g^{-1} h^{-1} (which decreased as DO concentration decreased) in a 1 M NaOH contact solution, and Arm et al. (2003) reported a consumption rate of 1.5 µmoles O_2 g^{-1} h^{-1} (for unsaturated 0.25 M NaOH feed). Similarly, Brown et al. reported 335 µmoles O_2 g^{-1} h^{-1} consumption by RF resin (ground gel from BSC), whereas the current study resulted in ~160 µmoles O_2 g^{-1} h^{-1}. The disparate measurements are attributed to the available DO in each test case; as the contact medium DO concentration increased, the rate of consumption increased. In summary, both the RF and SL-644 resins appeared to consume all dissolved oxygen to which they were exposed under the given test conditions.

6.4 Cs Ion Exchange Performance

Relative performance impacts from the oxidation were evaluated from the post-exposure process cycle. The number of BVs processed to reach the contract limit and 50% Cs breakthrough, as well as effects on the elution profile, were compared to the pre-exposure cycle. The summary of breakthrough impacts is provided in Table 6.3. The breakthrough and elution profiles are provided in Section 5.0; specific figure references are included in the table.

Table 6.3. Effect on Cs Breakthrough and Elution from Oxygen Attack

	BRF-14 (AP-101)	BRF-14 (AZ-102)	BRF-15	BRF-17	SL-644
Pre-exposure					
Contract limit, BVs	33	68	31	26	73
50% breakthrough, BVs	121	170	120	123	220
Oxygen Exposure					
Estimated O_2 exposure, mmoles/mL resin bed	0.30	0.26	0.32	0.22	0.17
Estimated O_2 exposure, mmoles/g H-form resin	0.92	0.78	0.95	0.65	0.78
Post exposure					
Contract limit, BVs	24	50	23	22	39
Contract limit relative reduction	27%	26%	26%	15%	47%
50% breakthrough, BVs	110	142	107	105	143
50% breakthrough relative reduction	9%	16%	11%	15%	35%
Loading Figure reference (Section 5.0)	5.20	5.17	5.21	5.22	5.32
Elution characteristics	slight broadening	no change	slight broadening	slight shift	no change
Elution Figure reference (Section 5.0)	5.20	5.17	5.21	5.22	5.32

The spherical RF resins demonstrated a 15% to 27% reduction in the contract limit breakthrough and a 9% to 16% reduction in the 50% breakthrough (BV basis). The corresponding SL-644 breakthrough reductions were about twice the factors obtained for RF. Evaluation of the elution profiles and residual Cs post-elution showed no significant change from the pre-oxidized condition. All resins processed more than 100-BV before 50% Cs breakthrough, even after the severe exposure to oxidizing conditions.

The oxidative attack on the RF resin appeared to be limited to the top layer, progressing downward with increasing exposure. Since oxidation was associated with destruction of the Cs exchange sites (Hubler et al. 1995, Adamson, et al 2006), the effective active resin bed height and volume decreased as the oxidation progressed from the top of the resin bed downward. Therefore, the Cs residence time during the subsequent feed step was proportionately decreased (shortened effective ion exchange bed height and volume with no adjustment to flowrate). Changing the residence time (flowrate) was shown to impact the contract limit breakthrough with virtually no effect on the 50% breakthrough (Hardy et al. 2004). The follow-on simulant feeds were therefore processed through shorter bed heights (effectively shorter residence times) and smaller bed volumes (effectively faster flowrate and decreased L/D). Both conditions effectively decreased the contact-limit breakthrough and the 50% breakthrough proportional to the reduced bed size.

The total mmoles of O_2 were evaluated against the mmoles RF in the column.[(a)] For simplicity, the entire mass H-form resin loaded in the column was assumed to be 100% RF (the supporting bead structure is a minor component of the resin). With a molecular weight of 122.1 g/mole (reported by

(a) The same evaluation could not be performed for SL-644 because its formulation is proprietary.

Hardy et al. 2004), the total RF quantity loaded in the column and the oxidative exposure could be evaluated on a mole basis. The results are summarized in Table 6.4.

Table 6.4. Estimated Relative Effect of Oxygen Exposure on RF Resin

Parameter	BRF-14 (AP-101)	BRF-14 (AZ-102)	BRF-15	BRF-17
RF (mmoles)[(a)]	49	49	51	50
Estimated O_2 exposure (mmoles)	5.5	4.7	5.9	4.0
Contract Limit Breakthrough Effects				
Relative reduction in contract limit breakthrough (%)	27	26	26	15
Contract limit reduction / (mmoles O_2/mmoles RF)	27/(5.5/49) = 240%	26/(4.7/49) = 270%	26/(5.9/51) = 220%	15/(4.0/50) = 190%
Average	230 (± 34) %			
Estimated reduction factor (contract limit reduction) / (mmole O_2/g H-form resin)	27/(5.5/5.99) = 29%	26/(4.7/5.99) = 33%	26/(5.9/6.23) = 27%	15/(4.0/6.15) = 23%
Average	28 (± 4) %			
50% Breakthrough Effects				
Relative reduction 50% breakthrough (%)	9	16	11	15
50% breakthrough reduction / (mmoles O_2/mmoles RF)	9/(5.5/49) = 82%	16/(4.7/49) = 170%	11/(5.9/51) = 92%	15/(4.0/50) = 190%
Average	133 (± 54) %			
Estimated reduction factor (reduced 50% breakthrough) / (mmole O_2/g H-form resin)	9/(5.5/5.99) = 10%	16/(4.7/5.99) = 20%	11/(5.9/6.23) = 12%	15/(4.0/6.15) = 23%
Average	16 (± 6) %			
(a) Mass of RF/FW; FW = 122.1 g/mole.				

The 50% breakthrough capacity would be expected to decline in direct proportion to the fraction of resin damage. A 1:1 mole ratio of oxygen attack on the resin would result in 12% (by mass) resin damage; a 1:2 mole ratio of oxygen attack on the resin would result in 24% resin damage. The 50% Cs-breakthrough reduction appeared to result in two populations (n=2) of damage from DO exposure. Two tests (BRF-14 [AP-101] and BRF-15) resulted in nearly a 1:1 ratio of reduced performance per mole of oxygen exposure, averaging to 11% damage. Two tests (BRF-14 [AZ-102] and BRF-17) resulted in a reduced performance correlated to an oxidative attack of 1 mole O_2: 2 mole RF, averaging to ~22% RF damage. The overall average for the 50% breakthrough reduction was 16 ± 6 % (n=4). The variation in oxidation damage results may be attributed in large part to uncertainties in the DO measurements (based on the large variation determined for one resin, BRF-14).

The oxidative damage effect on the contract limit[(a)] was more pronounced than that on the 50% breakthrough limit, 28% versus 16% reduction/mmole O_2/g resin, respectively. The difference was attributed to the shortened Cs residence time in the resin bed and its more significant impact on the contract-limit breakthrough. Had the resin damage been uniformly distributed throughout the bed, the contract-limit breakthrough reduction would be expected to be similar to the 50%-breakthrough reduction where residence time is constant. Indeed, a later test on Microbeads RF Lot #5E-370/641 resin (Section 12.0) supported this assertion. In this case, Adamson et al. (2006) reported about 12% (7.2 kg damaged in a 58.9 kg resin bed) resin damage from oxidation after exposure to 16 process cycles. A fraction of the damaged resin bed was delivered to PNWD for testing where reductions of ~8% at the contract limit breakthrough and ~10% at the 50% Cs breakthrough were observed. Given an oxygen exposure of 0.50 to 0.63 mmoles/g H-form resin (Adamson et al. 2006), the relative performance reduction factors for both the contract limit and 50% breakthroughs were calculated to range from 14 to 18 % per mmole O_2 exposure per g resin.[(b)] These results were within the 50%-breakthrough reduction range (10% to 23%) provided in Table 6.4.

(a) The contract-limit reduction relative to functional resin reduction was consistent with a destructive ratio of ~1 mole O_2 : 2.3 mole RF.

(b) The estimated reduction factor of 14% to 18% per mmole O_2 per g resin was calculated by dividing the measured average 9% performance reduction by the estimated 0.63 to 0.5 mmole O_2/g resin exposure, respectively.

7.0 Wave 1b Testing

Wave 1b testing was conducted with resins of two different bead diameters prepared by Microbeads under identical process conditions.[a] Neither the production dates nor the lot sizes were reported. Wet resin samples were received at PNWD on 10/8/04. The Microbeads lot number was used as the internal PNWD tracking ID. Table 7.1 summarizes the PNWD ID, resin bead sizes, and receipt quantities. The bead diameter represented the as-received H-form condition, as reported by Microbeads. Results from this testing allowed the BNI R&T lead to define the preferred particle size for future RF lot preparations.

Table 7.1. Wave 1b Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Bead Diameter, μm	Receipt Quantity, mL	Receipt Date
PS-420	Microbeads	PS-420	352	30	10/8/04
PS-424	Microbeads	PS-424	439	40	10/8/04

Wave 1b resins were pretreated consistent with Protocol P1-RF. The resin pretreatment and processing for column performance testing is shown in the unshaded areas of Figure 7.1.[b] The shaded areas (typically applied to other process waves) are shown for comparison.

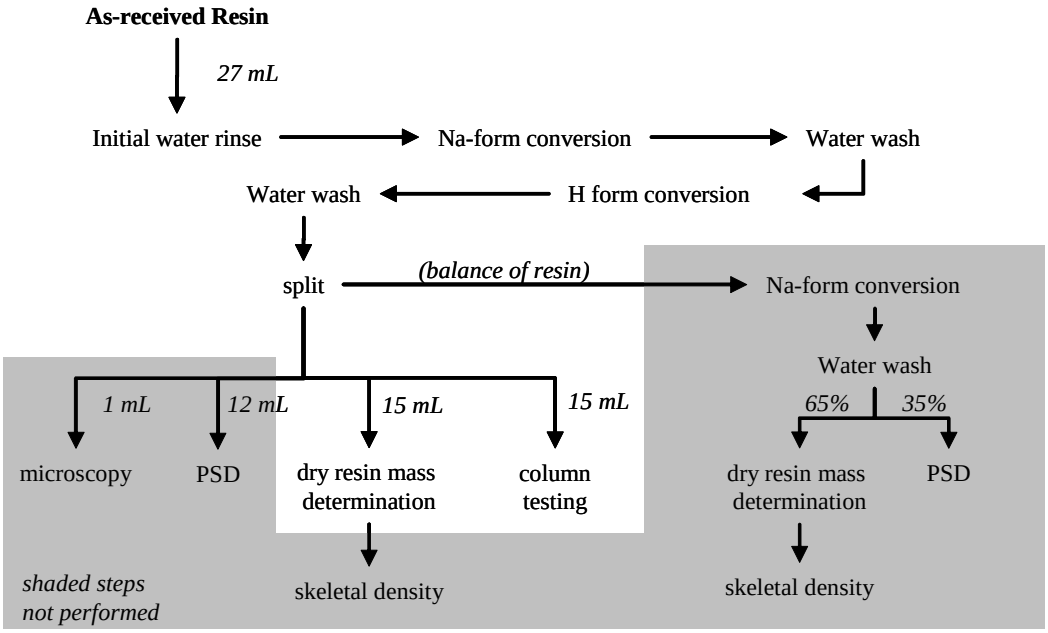


Figure 7.1 Sample Pretreatment and Splitting for Physical Property and Column Performance Testing (Wave 1b)

(a) Production parameters are confidential and were provided directly from Microbeads to the BNI R&T Lead.

(b) Resin processing was conducted according to TI-RPP-WTP-394, Rev. 0, *Physical Properties Evaluation of Spherical RF Resins*.

The only scope of work associated with these resins was to evaluate the Cs loading performance. Physical property testing was specifically not included in the Wave 1b resin evaluation; however, some minimal physical properties were determined during pretreatment processing.

7.1 Resin Swell and Bed Densities

Table 7.2 summarizes the unconstrained resin volume expansion as a result of the swell-shrink cycle during pre-treatment. Both resins expanded ~30% from the as-received condition.

Table 7.2. Wave 1b Resins Pretreatment Swell Data Summary

	1st Cycle Expansion		
PNWD ID	As-received H-form, mL	Pretreated H-form, mL	Expansion Factor
PS-420	27	36	1.3
PS-424	27	36	1.3

Table 7.3 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed densities determined after the in-column pretreatment shrink-swell were slightly higher than the unconstrained bed densities. The Microbead product dry bed densities were typical of previously tested resins.

Table 7.3. Wave 1b Resins Dry Bed Densities

Resin ID	Resin Form	Settled Vol., mL[(a)]	Dry Mass, g	Settled Resin Density, g/mL[(a)]	Column Processing Bed Density, g/mL[(b)]
PS-420	H-form	15	5.86	0.391	0.38
PS-424	H-form	15	5.89	0.393	0.38

(a) Dry resin mass per unit wet volume.
(b) Measured during column processing; only the dry H-form mass placed in the column was determined.

7.2 Column Testing

Column testing was conducted similarly to the Wave 1 tests. The specific column test parameters are summarized in Table 7.4 and Table 7.5. Only one process cycle was evaluated for Cs loading characteristics.

Table 7.4. Experimental Conditions for PS-420 (Green Column) Wave 1b

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (10/15/04)								
Water rinse	DI water	6.90	2.86	134	2.02	0.656	3.42	21
Acid wash	0.5 M HNO_3	7.30	3.03	142	2.32	0.752	3.15	23
Water rinse	DI water	2.88	1.19	56.1	1.09	0.353	2.65	22
Cycle 1 (Start 10/17/04)								
Regeneration	0.5 M NaOH	6.21	2.57	121	2.78	0.903	2.23	22
Loading column	AP-101 Simulant	78.8	NA	1535	1.10	0.356	71.7	22
Loading column	AP-101 Simulant	60.4	NA	1176	3.06	0.995	19.7	22
Feed displacement	0.1 M NaOH	3.06	1.27	59.6	3.06	0.994	1.00	22
Rinse	DI water	3.07	1.27	59.9	3.07	0.998	1.00	22
Elution	0.5 M HNO_3	11.4	4.71	222	1.29	0.418	8.83	22
Rinse	DI water	2.96	1.23	57.7	1.38	0.447	2.15	22

(a) BV = bed volume (19.5 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 7.5. Experimental Conditions for PS-424 (Pink Column) Wave 1b

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (10/15/04)								
Water rinse	DI water	7.27	3.06	144	2.57	0.847	2.83	21
Acid wash	0.5 M HNO_3	8.23	3.47	163	2.84	0.936	2.90	23
Water rinse	DI water	3.11	1.31	61.6	1.37	0.453	2.27	22
Cycle 1 (Start 10/17/04)								
Regeneration	0.5 M NaOH	6.16	2.59	122	2.76	0.912	2.23	22
Loading column	AP-101 Simulant	76.3	NA	1509	1.07	0.355	71.8	22
Loading column	AP-101 Simulant	59.1	NA	1170	3.00	0.991	19.7	22
Feed displacement	0.1 M NaOH	3.01	1.27	59.5	3.01	0.992	1.00	22
Rinse	DI water	3.00	1.26	59.4	3.00	0.990	1.00	22
Elution	0.5 M HNO_3	11.5	4.85	228	1.25	0.411	9.23	22
Rinse	DI water	2.93	1.23	57.9	1.32	0.435	2.22	22

(a) BV = bed volume (19.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Photographs of selected process phases and conversion fronts are shown in Figure 7.2 and Figure 7.3. In these pictures, the dark resin color is the Na-form resin, the burnt orange color is the H-form resin. Column processing resulted in generally sharp conversion fronts (H-form to Na-form) during the in-column pretreatment stage. The Na-form conversion of PS-424 showed evidence of channeling during conversion from the H-form to the Na-form (Figure 7.3b). The conversion fronts during the elution processing appeared more ragged with some distinct finger-like projections. The PS-424 elution conversion front was particularly uneven and could not be traced to experimental variables. The top surface layer of each resin bed was black after elution. The black surface was typical during RF processing and was attributed to be oxidative attack.

a) b) c)

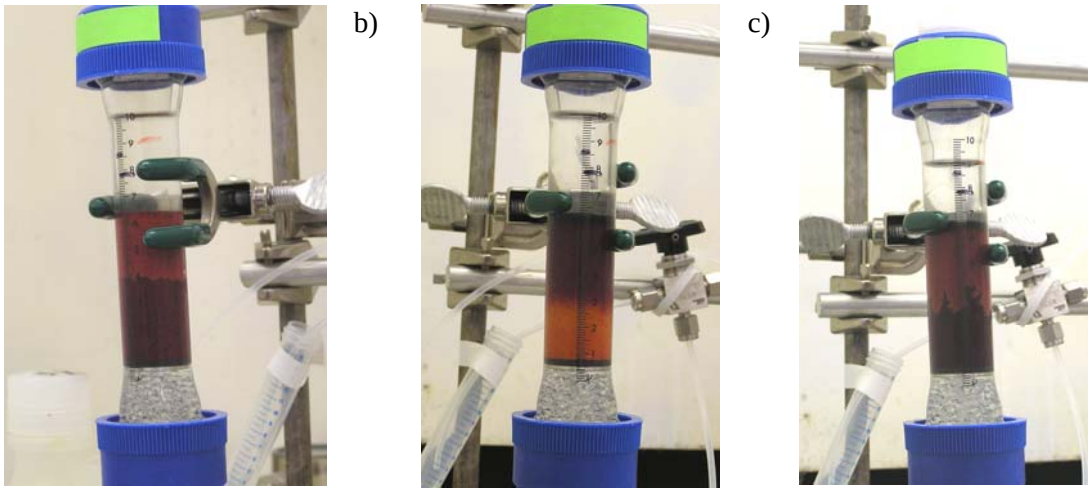


Figure 7.2. PS-420 Resin Transitions a) Pretreatment Na-Form to H-Form b) Pretreatment H-Form to Na-Form, and c) Elution

a) b) c)

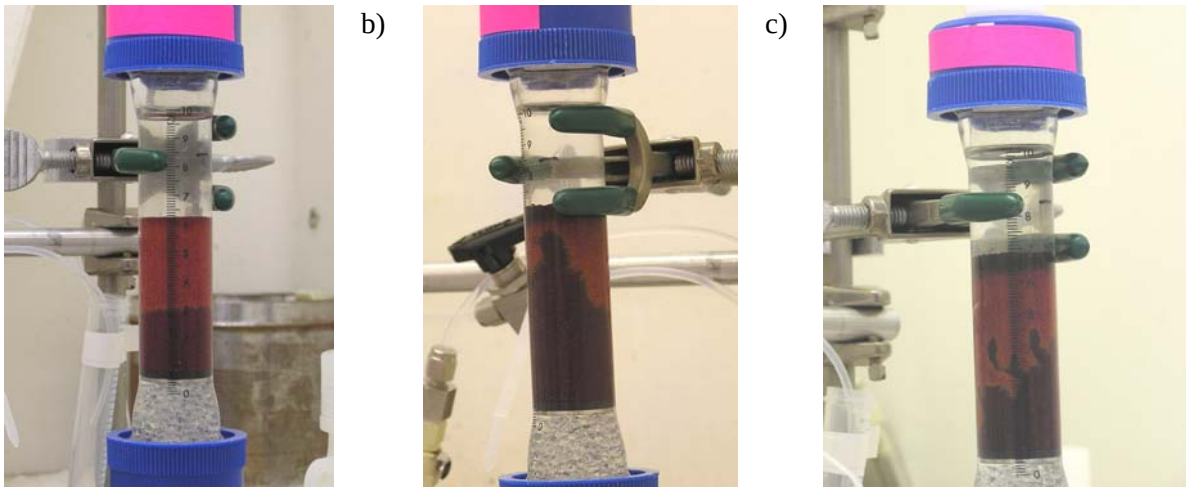


Figure 7.3. PS-424 Resin Transitions a) Pretreatment Na-Form to H-Form b) Pretreatment H-Form to Na-Form, and c) Elution

The Cs loading profile for each resin test is shown in Figure 7.4; the plotted points are given in Table 7.6. The change in flowrate from 1.5 BV/h to 3.0 BV/h was associated with a slight change in the linearity of the loading profile on the probability plot (at ~78 BVs).

The PS-420 resulted in slightly improved Cs loading parameters relative to PS-424, which was attributed to its smaller bead diameter. The Cs breakthrough onset occurred between 20 and 30 BVs and was virtually the same for both resins. The contract limit breakthrough differed by less than 5 BVs. The 50% Cs breakthroughs were ~100 BVs for PS-424 and ~110 BVs for PS-420.

The residual Cs concentrations on the resins, following elution with 11.5 BV of 0.5 M HNO_3, were determined to be 0.47 μg/g for PS-420 and 0.40 μg/g for PS-424.

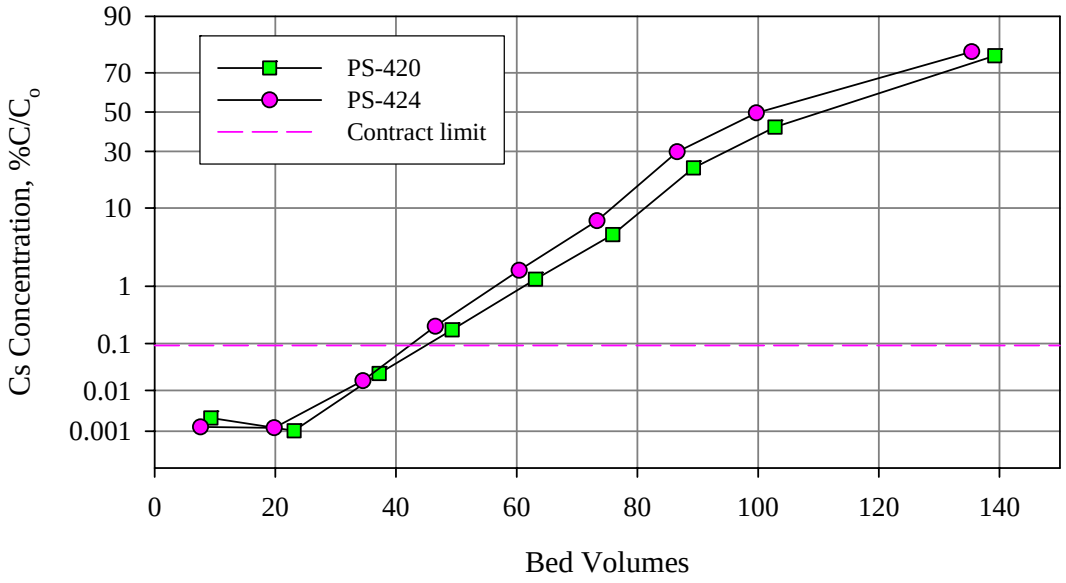


Figure 7.4. PS-420 and PS-424 Cs Loading Profiles

Table 7.6. PS-420 and PS-424 Effluent Cs Concentrations During AP-101 Simulant Loading

PS-420		PS-424	
Cum. BV	**% C/C$_o$**	**Cum. BV**	**% C/C$_o$**
9.3	<2.20E-3	7.6	<1.30E-3
23.1	1.04E-3	19.8	<1.23E-3
37.2	2.40E-2	34.5	1.67E-2
49.3	1.82E-1	46.5	2.11E-1
63.1	1.28E+0	60.4	1.73E+0
75.9	5.10E+0	73.3	7.32E+0
89.3	2.28E+1	86.6	2.98E+1
102.8	4.21E+1	99.7	4.96E+1
139.2	7.75E+1	135.4	7.90E+1

Figure 7.5 displays the in-column shrink-swell characteristics of the Wave 1b resins. The actual measured volumes (and matrix IDs) are provided in Table 7.7. The relative BV shown is the BV observed divided by the BV in 0.5 M NaOH (matrix ID #5). The PS-420 resulted in a ~15% shrink-swell volume variation, and the PS-424 was slightly higher at ~20%.

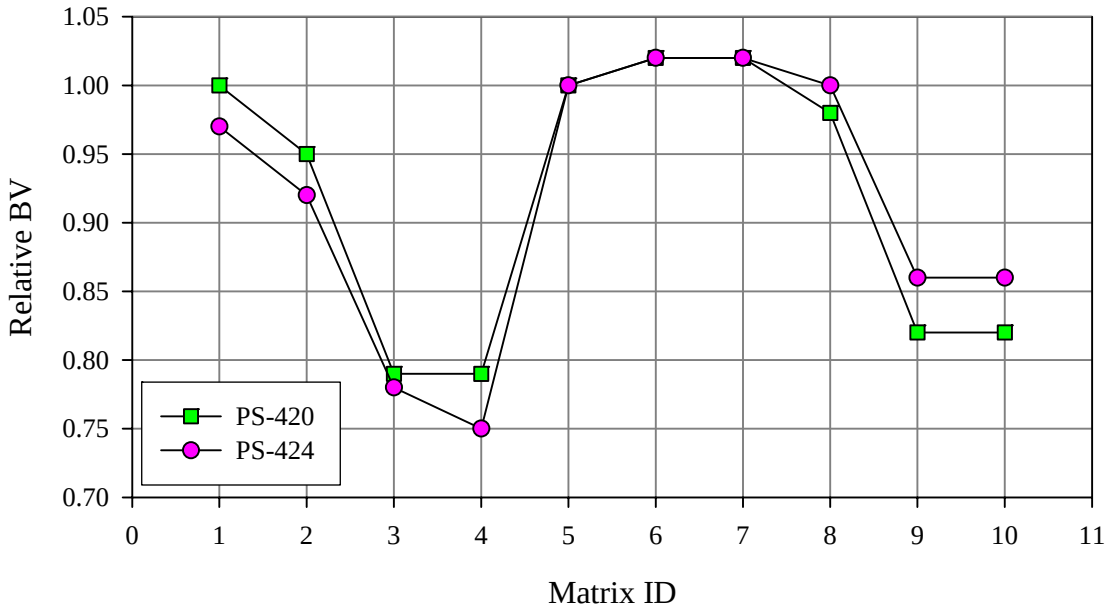


Figure 7.5. Wave 1b Resins Shrink-Swell Characteristics

Table 7.7. Actual Bed Volumes as a Function of Feed Matrix, Wave 1b

Feed Matrix	Matrix ID	PS-420 Green, mL	PS-424 Pink, mL
As-loaded (Na-form)	1	19.5	19.2
DI rinse	2	18.5	18.2
0.5 M HNO_3	3	15.4	15.4
DI water	4	15.4	14.8
0.5 M NaOH	5	19.5[(a)]	19.8[(a)]
feed, AP101 simulant	6	19.8	20.1
feed displacement	7	19.8	20.1
DI water	8	19.2	19.8
0.5 M HNO_3	9	16.0	17.0
DI water	10	16.0	17.0
Dry H-form resin mass	na	5.86 g	5.89 g
(a) Reference volume defined as 1-BV.			

8.0 Wave 2 Resin Testing

Wave 2 testing encompassed spherical RF resin supplied by a second manufacturer, BSC, as well as permutations of the resin production process. All Wave 2 resins used the seed material prepared from one production batch by Microbeads. A subset of the seeds was forwarded to BSC for functionalization using various curing temperatures and times for a total of four manufacturing conditions. Wave 2 testing also included two resins prepared at Microbeads. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates, and receipt dates are cross-referenced in Table 8.1.

Table 8.1. Wave 2 Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-5	Microbeads	PS-493	0.15-L	3/05	3/14/05
TI394-8	Microbeads	PS-501	0.15-L	3/05	3/14/05
TI394-11[(a)]	BSC	3380-2P-0100	0.5-gal	3/05	3/18/05
TI394-12	BSC	3380-2P-0101	0.5-gal	3/05	3/18/05
TI394-13	BSC	3380-2P-0102	0.5-gal	3/05	3/18/05
TI394-14	BSC	3380-2P-0103	0.5-gal	3/05	3/18/05
(a) TI394-11 was synthesized similarly to Microbeads BRF-14 (Wave 1).					

8.1 Wave 2 Experimental Specifics

The four resin lots received from BSC required wet sieving.[(a)] The sieving was performed in a sieve shaker (AS200 Basic, Retsch Inc. Newtown, PA) using 8-in.-diameter sieves in a cascade array of 600-µm, 500-µm, 300-µm, and 150-µm aperture sizes, respectively. Each lot was sub-divided in 500-mL batches. The resin batch was loaded onto the largest sieve before the assembly was purged with nitrogen gas in an effort to minimize oxygen exposure to the resin. Wet sieving was accomplished with a continuous flow of DI water running at approximately 1 L/minute with the shaker switched on for 15 min at an amplitude setting of 50. The dissolved oxygen content of the recirculation water was reduced approximately 90% by bubbling nitrogen gas through the contents of the stock bottle. Greater than 98% by volume of the as-received resin was collected on the 300-µm sieve and, therefore, in the size range 500 µm to 300 µm. Each batch processing was combined into the parent lot according to sieve size. The fraction representing 300-µm to 500-µm material was used for performance testing.

Wave 2 resins were pretreated consistent with Protocol P1-RF. The resin pretreatment and processing for physical property testing and column performance testing is summarized in Figure 8.1.[(b)]

(a) At the production time, BSC did not have the appropriate screening equipment.

(b) Resin processing was conducted according to TI-RPP-WTP-394, Rev. 0, *Physical Properties Evaluation of Spherical RF Resins*.

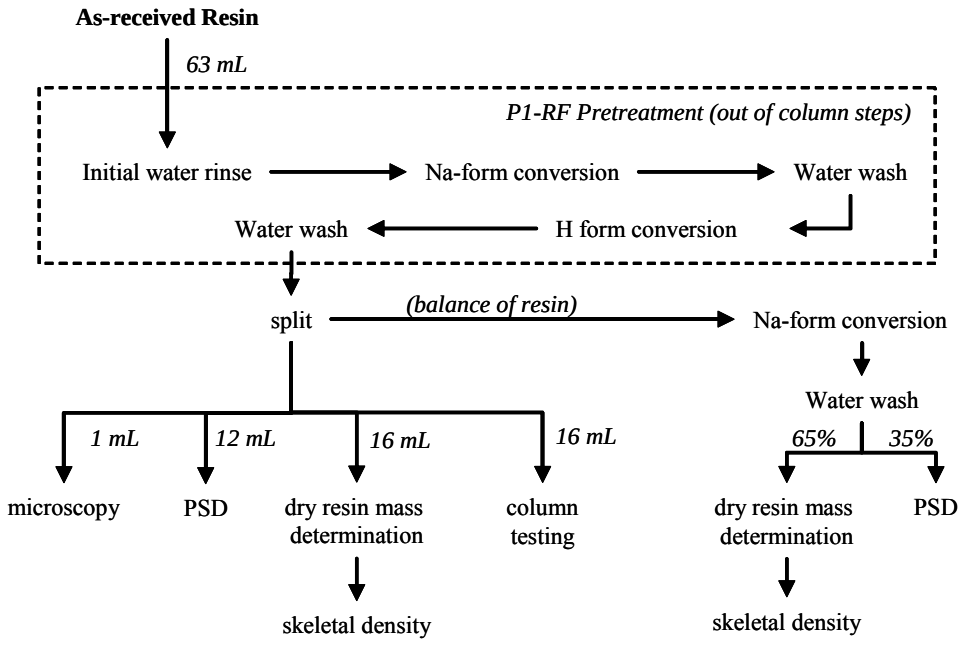


Figure 8.1. Resin Pretreatment and Splitting for Physical Property and Column Performance Testing (Wave 2)

The BSC resins bled a considerable color into the 1 M NaOH contact solution during the initial overnight soak, whereas the color bleed from the Microbeads resins was minimal. Figure 8.2 shows the relative color variations in the contact solutions.

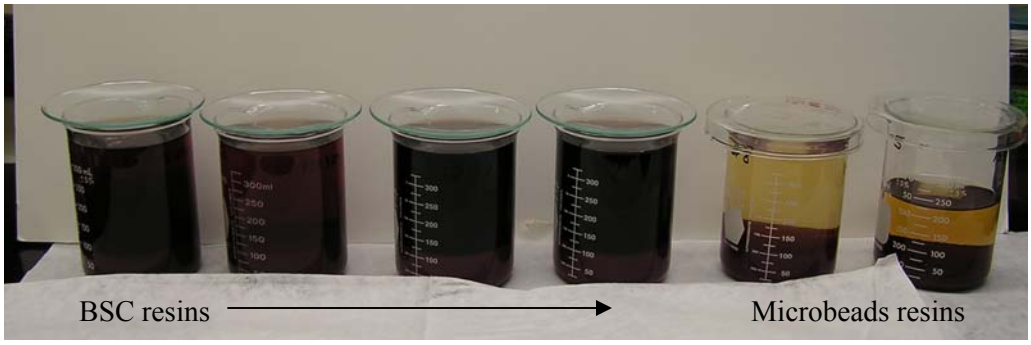


Figure 8.2. Resins with 1 M NaOH Contact Solutions after Overnight Soak (resins from left to right: TI394-11, TI394-12, TI394-13, TI394-14, TI394-8, and TI394-5)

All resin formulations were tested with AP-101 simulant in two complete loading and elution cycles. Table 8.2 through Table 8.7 summarize the specific processing conditions for the ion exchange tests. An unplanned stop-flow condition occurred after processing ~75 BVs AP-101 simulant during the first loading cycle. The building power went out on 3/31/05 at 10:35 a.m.; power to the pumps was resumed 50 minutes later at 11:25 a.m.

Table 8.2. Experimental Conditions for TI394-5 (Microbeads Lot PS-493) (Blue Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	9.13	3.48	164	4.98	1.49	1.83	22
Acid wash	0.5 M HNO_3	8.86	3.38	159	3.15	0.939	2.82	22
Water rinse	DI water	3.58	1.37	64.2	1.56	0.465	2.30	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	6.35	2.42	114	3.12	0.932	2.03	22
Loading column	AP-101 Simulant[(c)]	81.1	NA	1453	1.51	0.452	53.8	22
Loading column	AP-101 Simulant	51.3	NA	918	2.89	0.864	17.7	22
Feed displacement	0.1 M NaOH	2.91	1.11	52.0	2.60	0.777	1.12	22
Rinse	DI water	3.20	1.22	57.2	2.82	0.842	1.13	22
Elution	0.5 M HNO_3	21.6	8.23	387	1.44	0.430	14.9	23
Rinse	DI water	3.32	1.27	59.5	1.45	0.434	2.28	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	7.10	2.71	127	2.98	0.889	2.38	22
Loading column	AP-101 Simulant	82.8	NA	1483	1.51	0.450	54.4	22
Loading column	AP-101 Simulant	40.8	NA	730	2.97	0.886	13.8	22
Feed displacement	0.1 M NaOH	3.50	1.34	62.7	3.05	0.909	1.15	21
Rinse	DI water	3.43	1.31	61.5	2.90	0.866	1.18	21
Elution	0.5 M HNO_3	20.9	7.96	374	1.40	0.419	14.8	22
Rinse	DI water	3.21	1.22	57.5	1.41	0.419	2.28	22

(a) BV = bed volume (17.9 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 69 and 77 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

Table 8.3. Experimental Conditions for TI394-8 (Microbeads Lot PS-501) (Red Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	8.50	3.29	155	3.15	0.956	2.70	22
Acid wash	0.5 M HNO_3	7.66	2.97	140	3.15	0.956	2.43	22
Water rinse	DI water	3.32	1.29	60.5	1.18	0.358	2.82	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	6.32	2.45	115	3.19	0.968	2.00	22
Loading column	AP-101 Simulant[(c)]	76.2	NA	1388	1.40	0.425	54.0	22
Loading column	AP-101 Simulant	50.8	NA	926	2.86	0.868	17.6	22
Feed displacement	0.1 M NaOH	3.15	1.21	57.3	3.72	1.13	0.70	22
Rinse	DI water	3.03	1.18	55.3	2.84	0.864	1.07	22
Elution	0.5 M HNO_3	21.3	8.28	389	1.43	0.435	14.8	23
Rinse	DI water	3.19	1.24	58.2	1.44	0.437	2.22	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	7.09	2.75	129	3.08	0.936	2.30	22
Loading column	AP-101 Simulant	81.4	NA	1484	1.49	0.452	54.4	22
Loading column	AP-101 Simulant	41.9	NA	762	3.03	0.920	13.8	22
Feed displacement	0.1 M NaOH	3.34	1.29	60.8	3.08	0.935	1.08	21
Rinse	DI water	3.36	1.30	61.2	2.96	0.900	1.13	21
Elution	0.5 M HNO_3	21.3	8.26	388	1.43	0.435	14.8	22
Rinse	DI water	3.24	1.26	59.0	1.43	0.434	2.27	22

(a) BV = bed volume (18.2 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 64 and 72 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

Table 8.4. Experimental Conditions for TI394-11 (BSC Lot 3380-2P-0100) (Green Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	8.81	3.30	155	4.07	1.19	2.17	22
Acid wash	0.5 M HNO_3	8.52	3.19	150	3.68	1.08	2.32	22
Water rinse	DI water	3.42	1.28	60.2	1.48	0.433	2.32	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	6.76	2.53	119	3.59	1.05	1.88	22
Loading column	AP-101 Simulant[(c)]	81.4	NA	1432	1.50	0.440	54.0	22
Loading column	AP-101 Simulant	56.6	NA	996	3.10	0.908	18.2	22
Feed displacement	0.1 M NaOH	3.23	1.21	56.9	2.94	0.861	1.10	22
Rinse	DI water	3.00	1.12	52.8	2.81	0.825	1.07	22
Elution	0.5 M HNO_3	21.1	7.89	370	1.41	0.412	15.0	23
Rinse	DI water	3.1	1.16	54.5	1.41	0.413	2.20	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	6.73	2.52	118	3.08	0.904	2.18	22
Loading column	AP-101 Simulant	82.3	NA	1447	1.50	0.439	54.3	22
Loading column	AP-101 Simulant	48.1	NA	848	3.03	0.883	16.0	22
Feed displacement	0.1 M NaOH	3.60	1.35	63.3	3.13	0.917	1.15	21
Rinse	DI water	3.45	1.29	60.7	3.00	0.880	1.15	21
Elution	0.5 M HNO_3	20.9	7.82	367	1.40	0.41	14.9	22
Rinse	DI water	3.30	1.24	58.1	1.38	0.406	2.38	22

(a) BV = bed volume (17.6 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 69 and 77 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

Table 8.5. Experimental Conditions for TI394-12 (BSC Lot 3380-2P-0101) (Pink Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	8.31	3.28	154	2.82	0.870	2.95	22
Acid wash	0.5 M HNO_3	7.47	2.95	138	2.82	0.871	2.65	22
Water rinse	DI water	3.40	1.34	63.1	1.41	0.435	2.42	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	8.06	3.18	149	2.72	0.839	2.97	22
Loading column	AP-101 Simulant[(c)]	77	NA	1428	1.42	0.440	53.6	22
Loading column	AP-101 Simulant	57.4	NA	1064	3.14	0.971	18.2	22
Feed displacement	0.1 M NaOH	2.97	1.17	55.1	2.74	0.847	1.08	22
Rinse	DI water	3.03	1.20	56.2	2.84	0.878	1.07	22
Elution	0.5 M HNO_3	20.7	8.15	384	1.38	0.427	15.0	23
Rinse	DI water	3.04	1.20	56.4	1.37	0.424	2.22	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	7.43	2.93	138	2.62	0.810	2.83	22
Loading column	AP-101 Simulant	79.8	NA	1480	1.48	0.457	53.7	22
Loading column	AP-101 Simulant	47.9	NA	888	3.01	0.930	16.0	22
Feed displacement	0.1 M NaOH	3.42	1.35	63.4	3.06	0.946	1.12	21
Rinse	DI water	3.19	1.26	59.2	2.90	0.897	1.10	21
Elution	0.5 M HNO_3	21.1	8.31	390	1.41	0.437	14.9	22
Rinse	DI water	3.22	1.27	59.7	1.40	0.433	2.30	22

(a) BV = bed volume (18.5 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 65 and 73 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

Table 8.6. Experimental Conditions for TI394-13 (BSC Lot 3380-2P-0102) (White Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	8.35	3.35	157	3.13	0.983	2.67	22
Acid wash	0.5 M HNO_3	8.55	3.43	161	3.18	1.00	2.68	22
Water rinse	DI water	3.16	1.27	59.5	1.52	0.476	2.08	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	5.95	2.39	112	3.13	0.984	1.90	22
Loading column	AP-101 Simulant[(c)]	80.7	NA	1522	1.50	0.471	53.9	22
Loading column	AP-101 Simulant	50.8	NA	957	2.99	0.940	16.9	22
Feed displacement	0.1 M NaOH	3.06	1.23	57.8	3.01	0.947	1.02	22
Rinse	DI water	3.22	1.29	60.7	2.84	0.892	1.13	22
Elution	0.5 M HNO_3	21.3	8.53	401	1.42	0.446	14.9	23
Rinse	DI water	3.1	1.24	58.4	1.44	0.453	2.15	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	7.25	2.91	137	2.81	0.882	2.58	22
Loading column	AP-101 Simulant	81.4	NA	1534	1.49	0.467	54.3	22
Loading column	AP-101 Simulant	40.4	NA	763	2.98	0.938	13.6	22
Feed displacement	0.1 M NaOH	3.21	1.29	60.5	3.06	0.960	1.05	21
Rinse	DI water	3.24	1.30	61.0	2.94	0.924	1.10	21
Elution	0.5 M HNO_3	21.3	8.55	402	1.41	0.444	15.0	22
Rinse	DI water	3.07	1.23	57.8	1.44	0.452	2.13	22

(a) BV = bed volume (18.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 69 and 77 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

Table 8.7. Experimental Conditions for TI394-14 (BSC Lot 3380-2P-0103) (Yellow Column) in AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (3/28/05)								
Water rinse	DI water	8.09	3.24	152	3.28	1.03	2.47	22
Acid wash	0.5 M HNO_3	8.30	3.33	156	3.28	1.03	2.53	22
Water rinse	DI water	3.40	1.36	64.0	1.31	0.413	2.58	23
Cycle 1 (Start 3/29/05)								
Regeneration	1 M NaOH	6.06	2.43	114	3.19	1.002	1.90	22
Loading column	AP-101 Simulant[(c)]	74.9	NA	1412	1.39	0.437	54.0	22
Loading column	AP-101 Simulant	34.9	NA	658	1.89	0.594	18.6	22
Feed displacement	0.1 M NaOH	2.88	1.16	54.4	2.58	0.811	1.12	22
Rinse	DI water	3.00	1.20	56.5	2.65	0.832	1.13	22
Elution	0.5 M HNO_3	20.4	8.19	385	1.37	0.429	14.9	23
Rinse	DI water	2.99	1.20	56.4	1.50	0.470	2.00	23
Cycle 2 (Start 4/6/05)								
Regeneration	1 M NaOH	6.52	2.62	123	2.52	0.793	2.58	22
Loading column	AP-101 Simulant	78.3	NA	1476	1.43	0.450	54.3	22
Loading column	AP-101 Simulant	40.5	NA	763	2.97	0.933	13.6	22
Feed displacement	0.1 M NaOH	3.13	1.26	59.1	3.08	0.968	1.02	21
Rinse	DI water	3.21	1.29	60.6	2.92	0.918	1.10	21
Elution	0.5 M HNO_3	20.7	8.28	390	1.37	0.431	15.0	22
Rinse	DI water	3.12	1.25	58.8	1.35	0.423	2.32	22

(a) BV = bed volume (18.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Between 63 and 71 BVs, a power outage caused a 0.6-h stop-flow condition.
NA = not applicable

8.2 Physical Properties

This section summarizes physical property test results, including resin morphology, PSDs in hydrated H-form and Na-form, bed density, and skeletal density.

8.2.1 Pretreatment Shrink-Swell

The pretreatment shrink-swell data are summarized in Table 8.8. The resins expanded ~30% after one cycle pretreatment in the open beaker format relative to the as-received condition. The follow-on expansion to Na-form resin varied from 24% to 32%. The overall expansion from the as-received H-form resin to the pretreated Na-form resin was nominally 70%.

Table 8.8. Wave 2 Resins Pretreatment Swell Data Summary

PNWD ID	1st Cycle Expansion			H-form to Na-form Expansion[a]			Total Expansion
	As-received H-form, mL[b]	Pretreated H-form, mL	Expansion Factor	H-form, mL	Na-form, mL	Expansion Factor	
TI394-5	35	45.5	1.30	5.5	7.2	1.31	1.70
TI394-8	60	76.5	1.28	35.3	46	1.30	1.66
TI394-11	61	82	1.34	41	51	1.24	1.67
TI394-12	63.5	85	1.34	42.5	53	1.25	1.67
TI394-13	62	79.5	1.28	38.5	51	1.32	1.70
TI394-14	62	81	1.31	40	52	1.30	1.70

(a) Unconstrained expansion after first pretreatment cycle.
(b) Aliquot removed for testing.

8.2.2 Optical Microscopy

Optical micrographs of surfaces (10×, 25×, and 70×) and cross-sections (70×) were taken of each of the dried resins in the H-form. The micrographs are shown in Figure 8.3 through Figure 8.8. All resins appeared spherical and were generally brick red. The general morphology is easily discerned in the pictures.

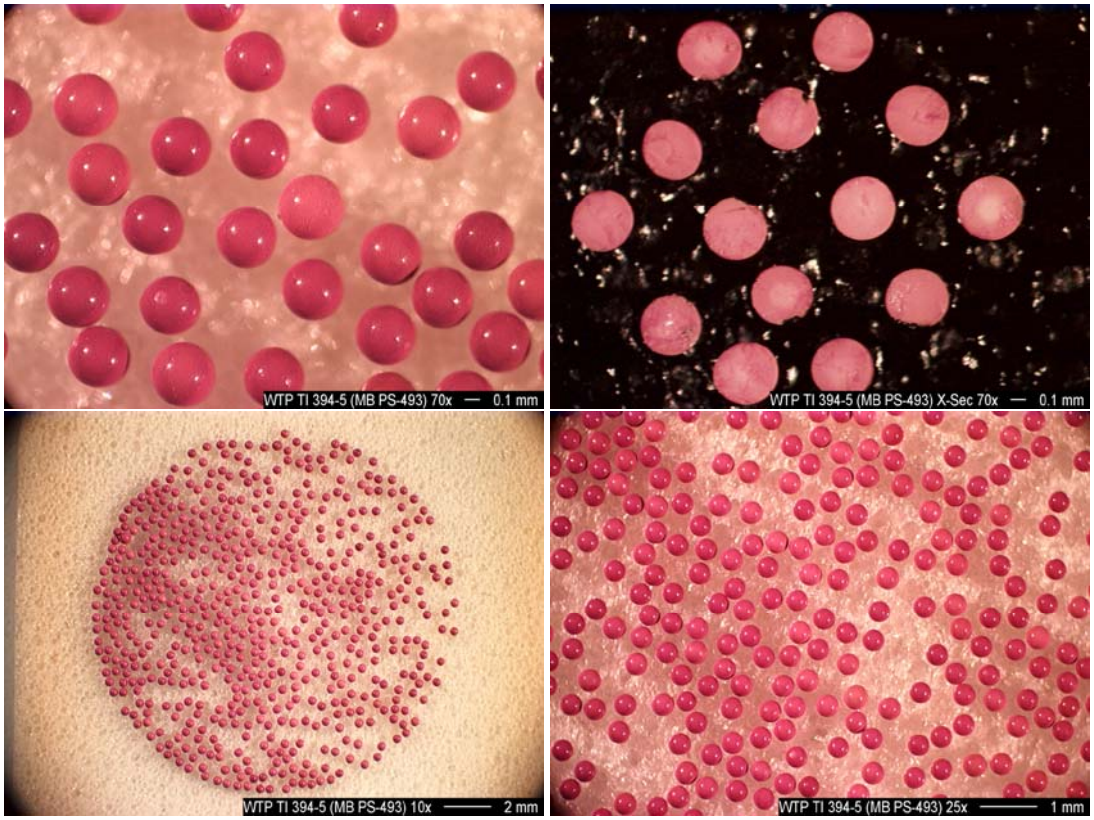


Figure 8.3. Micrographs of TI394-5 (Microbeads PS-493) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

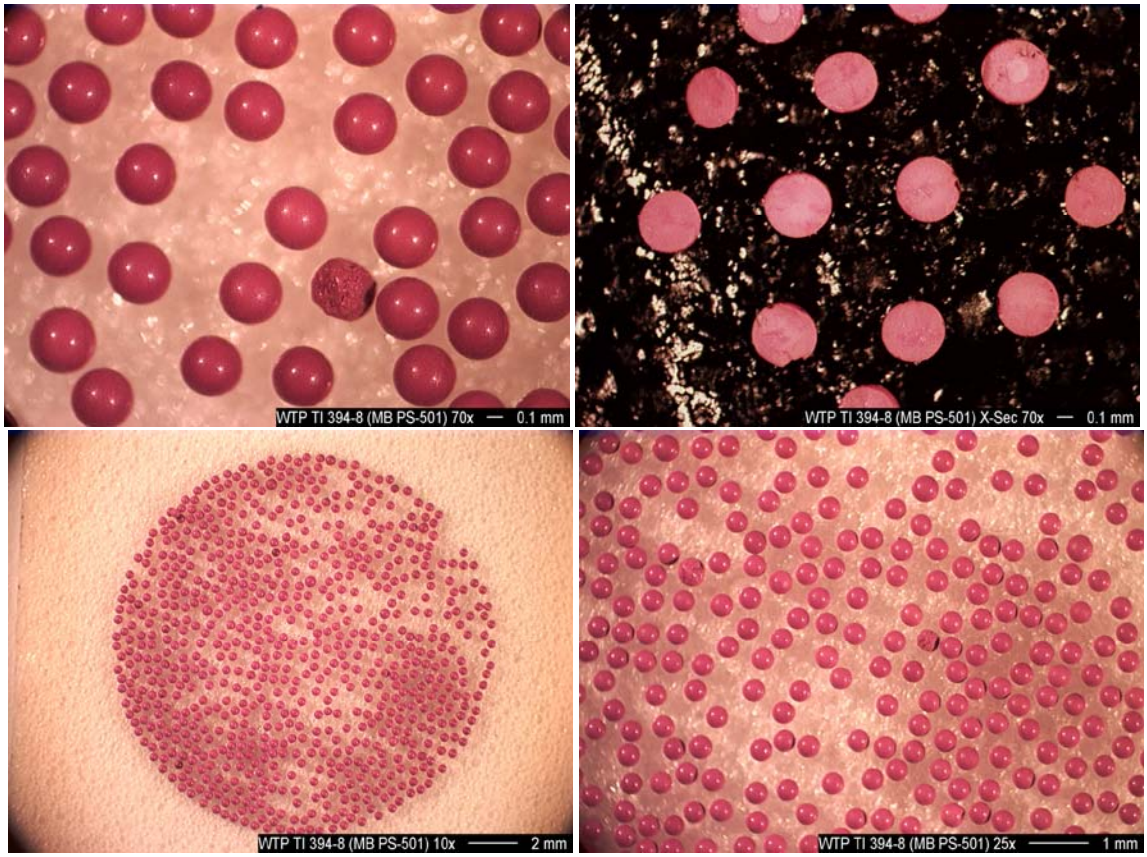


Figure 8.4. Micrographs of TI394-8 (Microbeads PS-501) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The TI394-5 and TI394-8 Microbeads resins were generally uniform in shape and size. Only a couple of anomalous amorphous clumps were found in the TI394-8 sample. They were nominally the size of a typical bead but were non-spherical.

Some of the resin beads cross-sectional photographs showed a core of slightly lighter coloration than the rest of the material. The lighter coloration may have been caused by incomplete polymerization or monomer loading. The number of beads cut in two was small and did not represent a statistically significant fraction. Therefore, it is not clear what fraction of the resin was affected by this phenomenon.

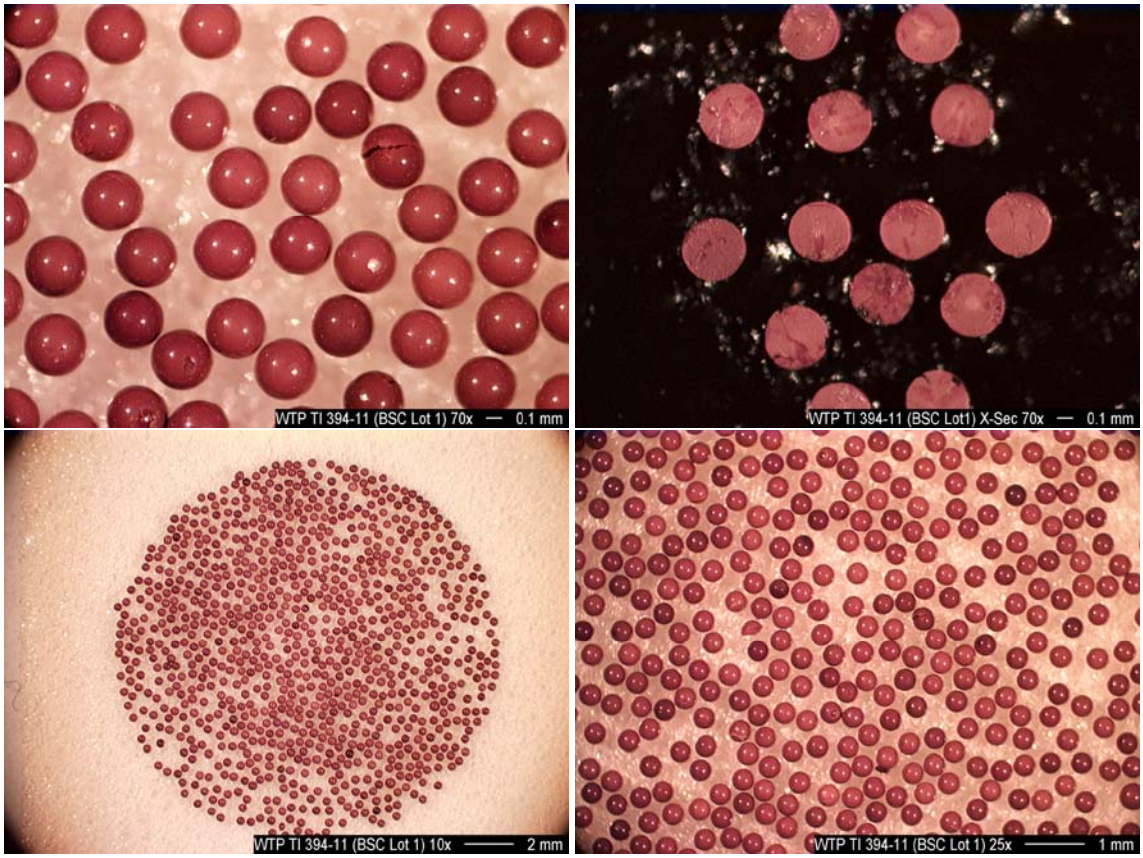


Figure 8.5. Micrographs of TI394-11 (BSC 3380-2P-0100) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The TI394-11 resin was generally uniform in shape and size. Some of the bead cross-section cores appeared to be lighter in color than the outer surfaces. The core volume affected was about half that observed with the Microbeads resins. Several broken resin pieces were observed. Several spheres were noted to have imperfections such as dimples and dents.

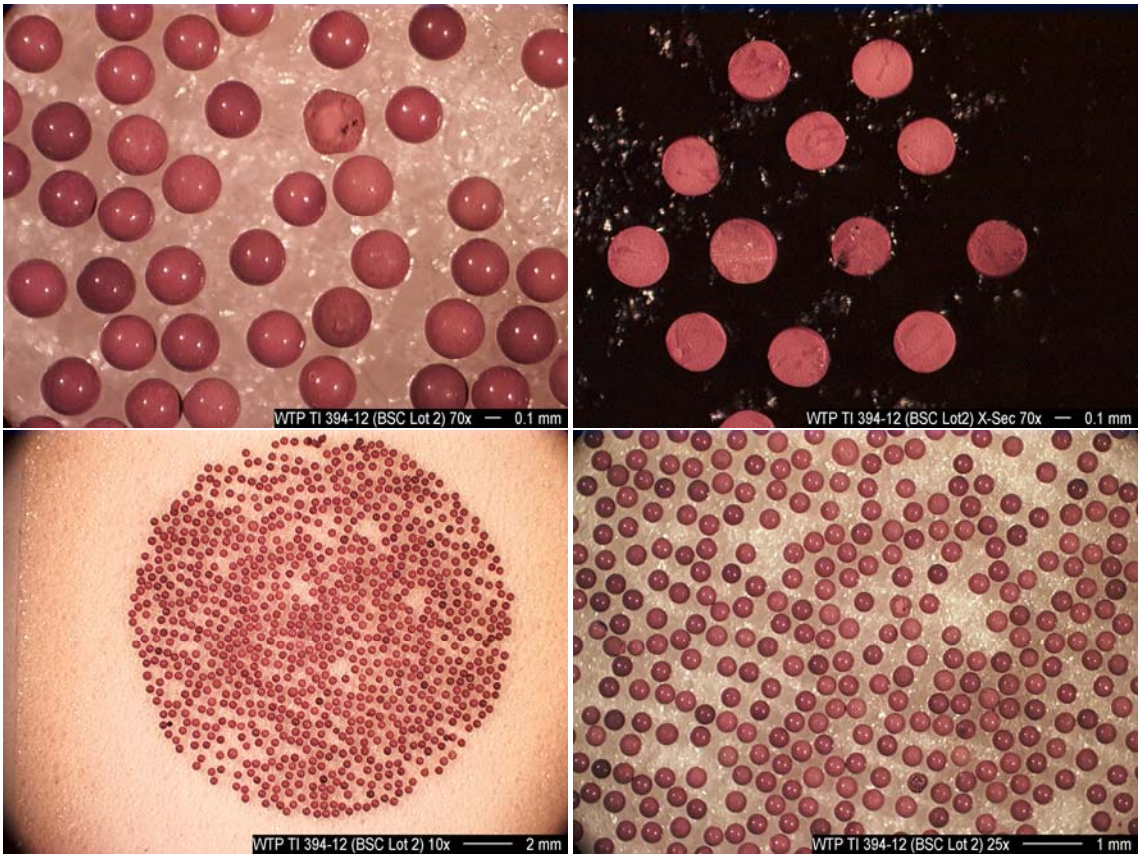


Figure 8.6. Micrographs of TI394-12 (BSC 3380-2P-0101) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The TI394-12 resin was generally uniform in shape and size. It also contained a small percentage of amorphous bead-sized chunks. Some beads were significantly darker than other beads. The cross-section photographs show that the interior was uniform.

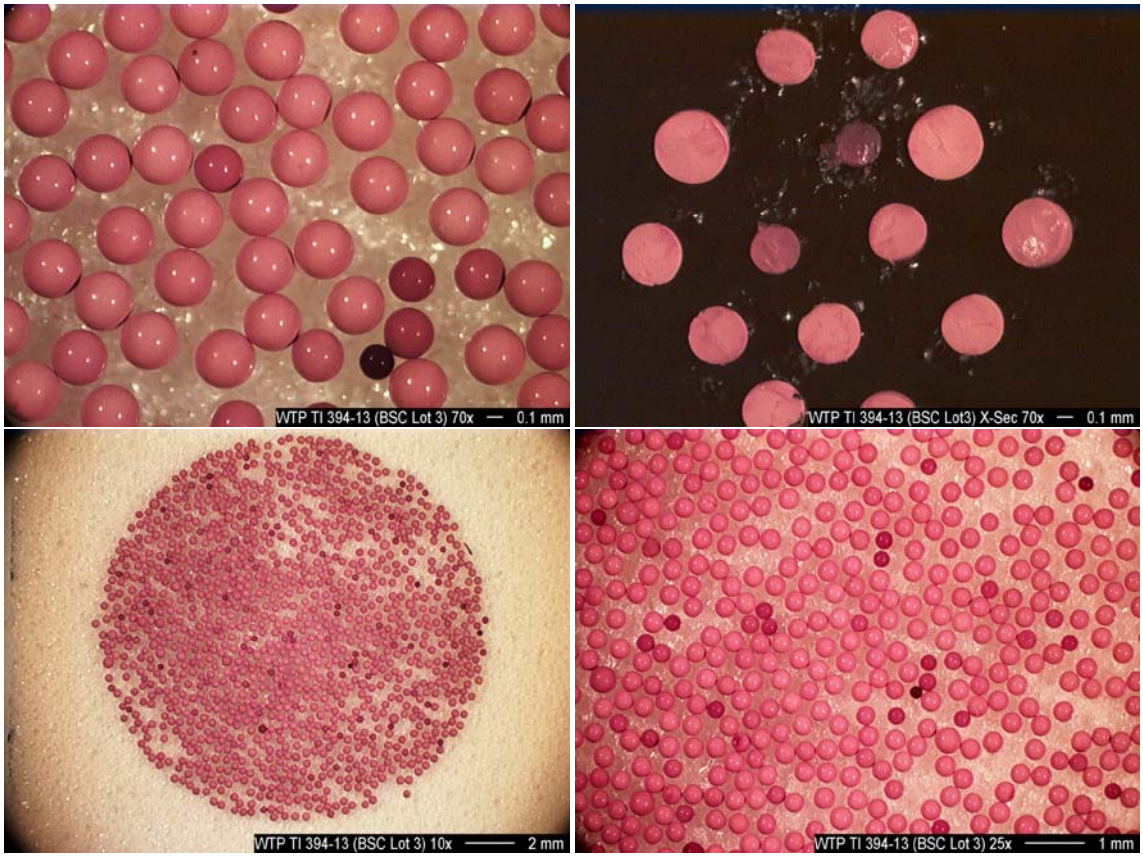


Figure 8.7. Micrographs of TI394-13 (BSC 3380-2P-0102) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The TI394-13 resin appeared to be composed of three different bead populations. The lighter color beads were largest (~0.36 mm dia.), the maroon beads were slightly smaller (~0.29 mm dia), and a few very small black beads (~0.18 mm dia) were observed.

Of the several cores examined in cross-section, all appeared to be uniformly filled with RF.

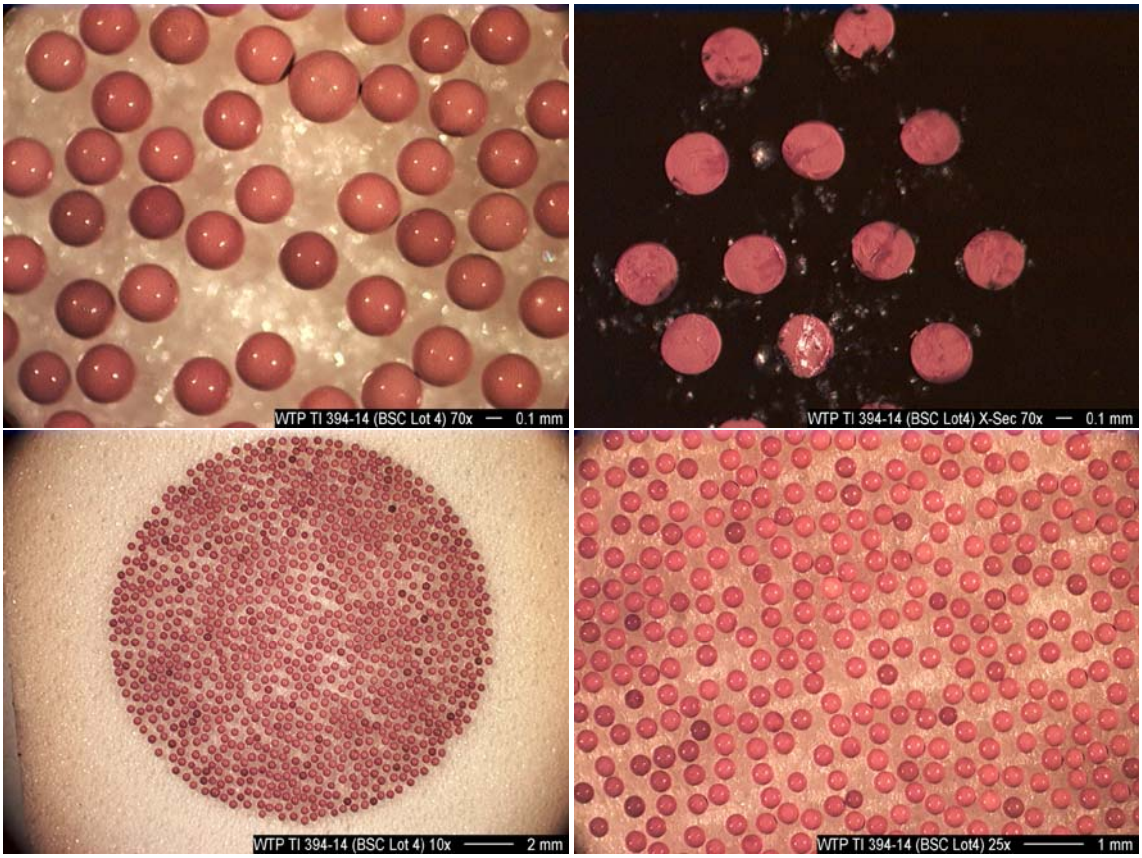


Figure 8.8. Micrographs of TI394-14 (BSC 3380-2P-0103) H-Form Resin. Clockwise from top left: 70×, 70× cross-section, 25×, and 10×.

The TI394-14 resin bead morphology appeared generally uniform across the surface and the cross-section. Generally two colors were obtained: light and dark maroon.

8.2.3 Particle-Size Distribution

Two resins, TI394-11 (BSC 3380-2P-0100) and TI394-12 (BSC 3380-2P-0101), were selected for additional PSD analysis using the Microtrac. Figure 8.9 shows the comparison between the average particle sizes for the pretreated resins in the H-form and in the Na-form (pretreatment as shown in Figure 8.1). The mean diameters are reported on a volume basis. The bars above and below each mean represent the particle-size distribution obtained from the lower 5% to upper 90% on a volume basis. Thus, 85% of the particles (volume basis) have a diameter in the indicated range.

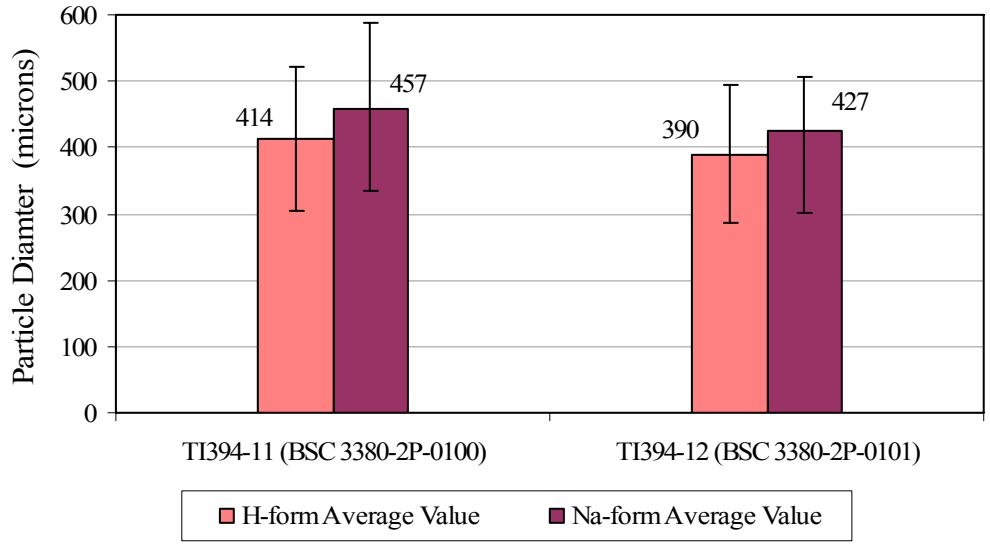


Figure 8.9. Average Particle Diameters (Volume Distribution) with 5% to 90% Volume Percentile Distribution

A summary of average particle sizes (volume distribution mean—m_v, number distribution mean—m_n, and area distribution mean—m_a) is tabulated in Table 8.9. The greater the agreement between the m_v, m_n, and m_a values, the more uniform is the overall particle distribution. Both resins manifested a fairly monodisperse particle population with generally good agreement between m_v, m_n, and m_a. Both resins tested resulted in ~30% calculated volume expansion based on the volume mean diameter change.

Table 8.9. TI394-11 and TI394-12 Particle-Size-Distribution Summary

Resin ID BSC Lot #	Resin form	Volume Distribution (microns) m_v	sd	Low 5%[a]	High 90%[b]	Number Distribution (microns) m_n	sd	Low 5%[a]	High 90%[b]	Area Dist. (microns) m_a
TI394-11	H-form	414	75	305	522	375	60	285	455	399
3380-2P-0100	Na-form	457	86	335	587	408	64	309	494	437
TI394-12	H-form	390	71	287	494	367	56	263	425	375
3380-2P-0101	Na-form	427	70	302	508	382	68	302	499	409
	Resin ID	**Calculated Average Sphere Volume, mm^3**								
TI394-11	H-form	0.0372				0.0276				0.0333
3380-2P-0100	Na-form	0.0500				0.0354				0.0437
Expansion factor >		*35%*				*29%*				*31%*
TI394-12	H-form	0.0311				0.0259				0.0275
3380-2P-0101	Na-form	0.0406				0.0293				0.0358
Expansion factor>		*31%*				*13%*				*30%*

(a) 5% of the particles are less than the given diameter.
(b) 10% of the particles are greater than the given diameter.
m_v = mean diameter volume distribution
m_n = mean diameter number distribution
m_a = mean diameter area distribution

8.2.4 Bed Densities

Table 8.10 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated H-form dry-bed density determined after the in-column pretreatment shrink-swell agreed well with the unconstrained bed density. The Na-form dry bed density in the ion exchange column was not determined.

Table 8.10. Wave 2 Resins Dry Bed Densities

Resin ID (Lot #)	Resin Form	Settled Resin Vol., mL[(a)]	Dry Resin Mass, g[(a)]	Settled Resin Density, g/mL[(b)]	Column Processing Bed Density, g/mL[(c)]
TI394-5	H-form	14.0	5.0924	0.364	0.36
(PS-493)	Na-form	--	--	--	--
TI395-8	H-form	14.4	4.3408	0.301	0.30
(PS-501)	Na-form	32.0	8.9441	0.280	na
TI394-11	H-form	14.4	6.1566	0.428	0.41
(3380-2P-0100)	Na-form	36.5	15.7080	0.430	na
TI394-12	H-form	14.3	6.0979	0.426	0.42
(3380-2P-0101)	Na-form	36.5	15.6842	0.430	na
TI394-13	H-form	14.6	4.8349	0.331	0.33
(3380-2P-0102)	Na-form	36.0	11.7722	0.327	na
TI394-14	H-form	14.2	5.3662	0.378	0.38
(3380-2P-0103)	Na-form	40.0	9.536[(d)]	0.238[(d)]	na

(a) Measured for skeletal density.
(b) Dry resin mass per unit wet volume.
(c) Measured during column processing; only the dry H-form mass placed in the column was determined.
(d) Data appeared suspect because of the very low Na-form density.
na = not applicable, Na-form mass was not determined.

8.3 Skeletal Density

The resin skeletal densities are provided in Table 8.11. The H-form densities were generally at 1.47 g/mL with the one exception for TI394-12 where duplicate measures of 1.49 and 1.53 g/mL were obtained. These results were consistent with those reported by King et al. (2004). The Wave 1 H-form densities were lower at about 1.44 g/mL; the lower values may be associated with the Wave 1 resin core structures being less dense than the Wave 2 resin cores. The Na-form skeletal densities were nominally equivalent at 1.60 g/mL and were about the same for Wave 1 and Wave 2 resins. The Na-form resin density could not be determined for TI394-11. This resin formed agglomerated rock-hard chunks upon drying, unlike the other resins, which produced dark free-flowing beads.

Table 8.11. Wave 2 Resins Skeletal Densities

Resin ID	Lot #	H-Form		Na-Form	
		g/mL	RPD	g/mL	RPD
TI394-5	PS-493	1.463	0.26%	--	--
TI394-8	PS-501	1.472	0.12%	1.593	0.57%
TI394-11	3380-2P-0100	1.497	0.01%	(a)	(a)
TI394-12	3380-2P-0101	1.511	2.53%	1.605	0.09%
TI394-13	3380-2P-0102	1.467	0.01%	1.610	0.11%
TI394-14	3380-2P-0103	1.479	0.24%	1.618	0.08%
(a) Not analyzed, dried Na-form resin resulted in rock-hard clumps.					

8.4 Column Performance Testing

The Cs loading characteristics for cycles one and two are shown in Figure 8.11 through Figure 8.16. The actual plotted data are summarized in Table 8.12 and Table 8.13. All resins were processed to >65% C/C_o Cs breakthrough. Only cycle one Cs elution profile characteristics were evaluated; these are shown in Figure 8.17 and Table 8.14.

8.4.1 TI394-5 (Microbeads PS-493)

The pretreatment processing resulted in a sharp conversion front from Na-form to H-form. The Cycle 1 elution resulted in a slightly ragged conversion front. The Cycle 2 elution conversion front was slightly more ragged than that of Cycle 1. Figure 8.10 shows the conversion-front characteristics through the different cycle elution processing operations.

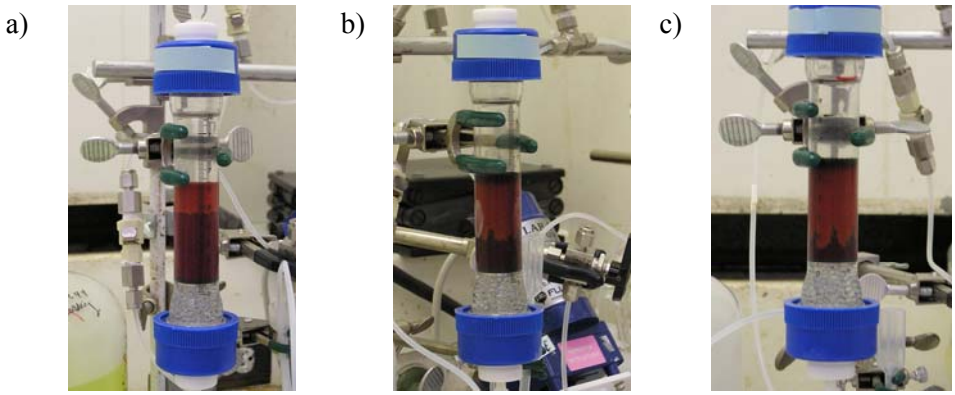


Figure 8.10. TI394-5 Elution Processing with 0.5 M HNO_3
(a) Pretreatment, (b) Cycle 1 Elution, (c) Cycle 2 Elution

The TI394-5 resin resulted in Cs breakthrough between 12 and 33 BVs. The contract limit (0.091% C/C_o) was reached after processing 33 BVs. The 50% C/C_o was attained after processing ~100 BVs. The first 20 BVs of Cycle 2 resulted in immediate Cs breakthrough at one order of magnitude below the contract limit. After processing 30 BVs, the Cycle 2 loading profile was virtually identical to the Cycle 1 loading profile.

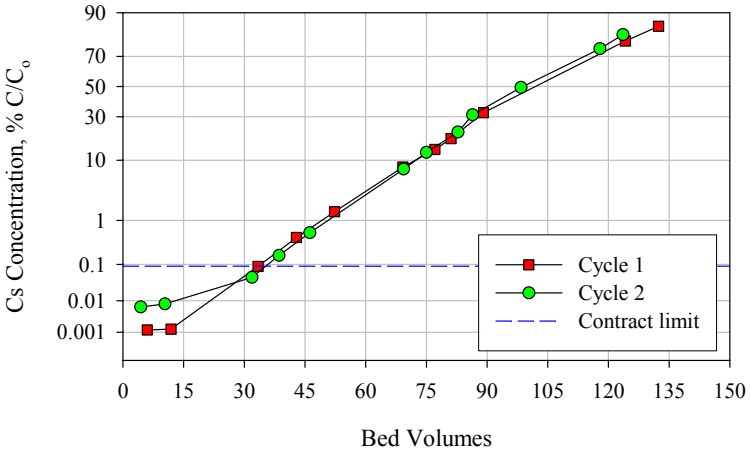


Figure 8.11. TI394-5 (Microbeads PS-493) Cs Loading Profiles with AP-101 Simulant

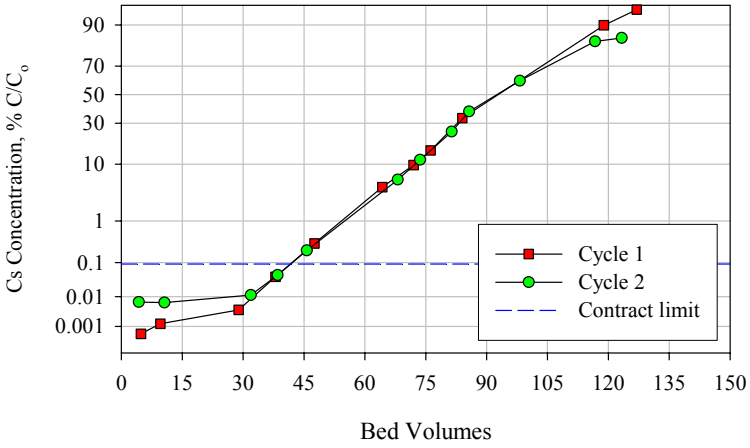


Figure 8.12. TI394-8 (Microbeads PS-501) Cs Loading Profiles with AP-101 Simulant

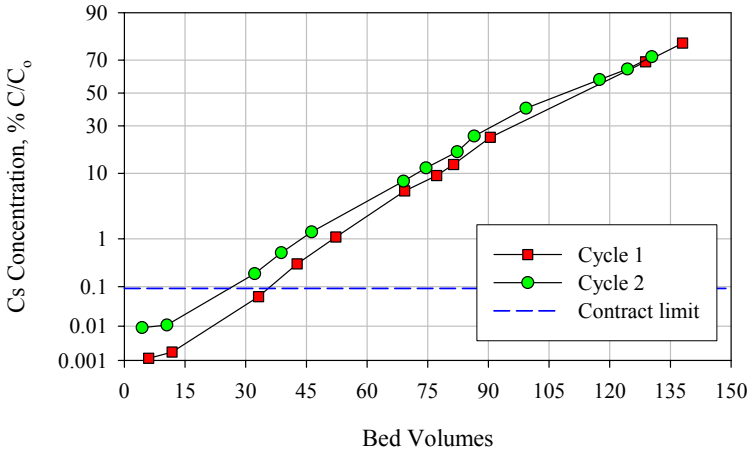


Figure 8.13. TI394-11 (BSC 3380-2P-0100), Cs Loading Profiles with AP-101 Simulant

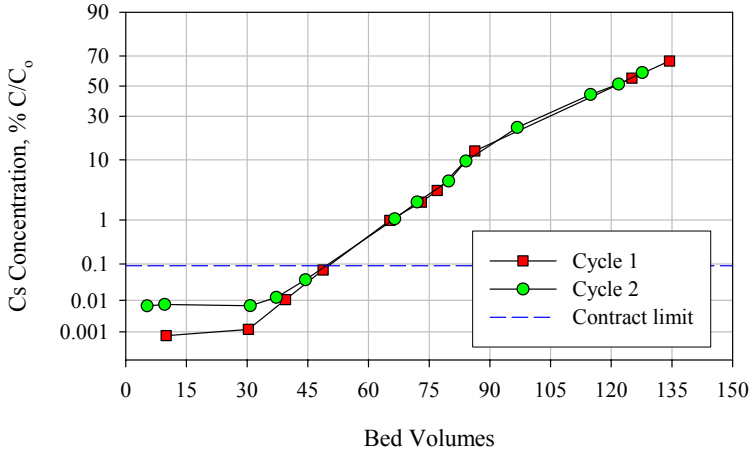


Figure 8.14. TI394-12 (BSC 3380-2P-0101), Cs Loading Profiles with AP-101 Simulant

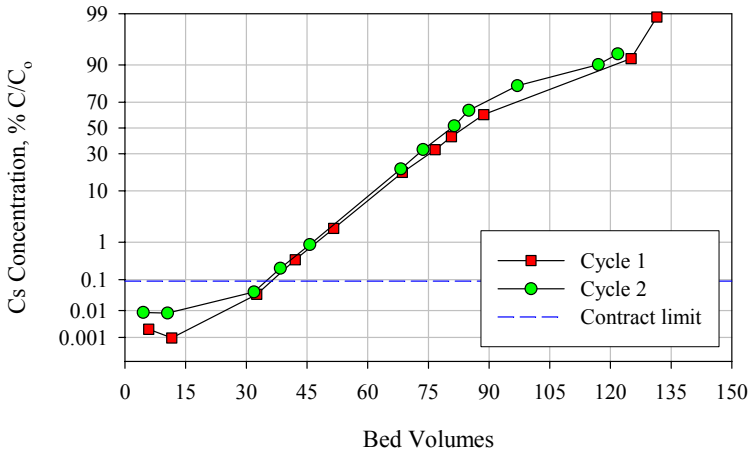


Figure 8.15. TI394-13 (BSC 3380-2P-0102) Cs Loading Profiles with AP-101 Simulant

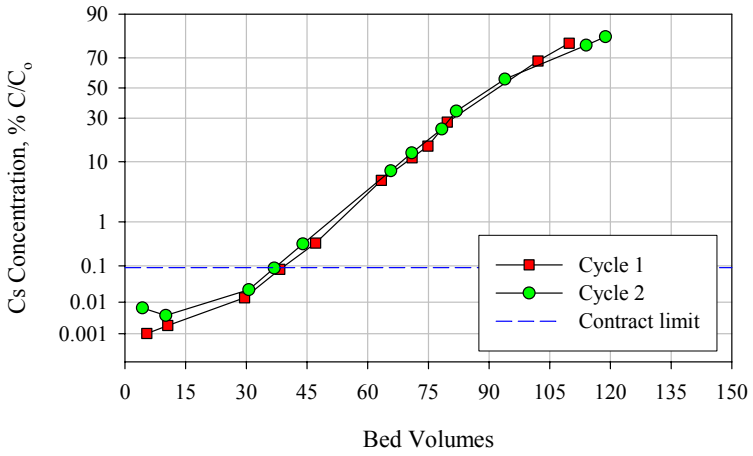


Figure 8.16. TI394-14 (BSC 3380-2P-0103) Cs Loading Profiles with AP-101 Simulant

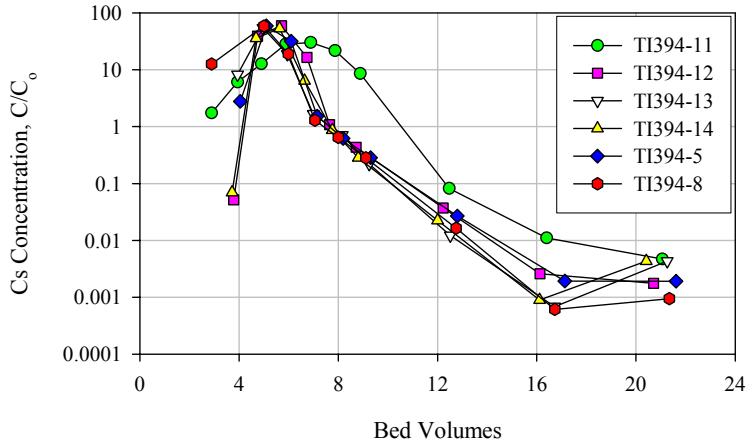


Figure 8.17. Cycle 1 Elution Profiles From Wave 2 Testing

Table 8.12. Effluent Cs Concentrations During AP-101 Simulant Feed Processing (TI394-5, TI394-8, TI394-11)

TI394-5 (Microbeads PS-493)				TI394-8 (Microbeads PS-501)				TI394-11 (BSC 3380-2P-0100)			
Cycle 1		Cycle 2		Cycle 1		Cycle 2		Cycle 1		Cycle 2	
Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o
6.0	<1.18E-3	4.4	6.47E-3	4.8	<5.33E-4	4.3	6.74E-3	6.0	<1.16E-3	4.4	9.11E-3
11.9	<1.26E-3	10.4	8.04E-3	9.6	<1.23E-3	10.6	6.48E-3	11.8	<1.80E-3	10.5	1.06E-2
33.4	9.01E-2	31.9	4.61E-2	28.9	3.73E-3	31.9	1.13E-2	33.2	5.83E-2	32.2	1.98E-1
42.9	4.38E-1	38.6	1.68E-1	38.0	4.01E-2	38.5	4.46E-2	42.7	3.19E-1	38.8	5.39E-1
52.3	1.48E+0	46.2	5.55E-1	47.6	3.07E-1	45.7	2.08E-1	52.3	1.08E+0	46.3	1.33E+0
69.2	8.09E+0	69.4	7.62E+0	64.3	4.42E+0	68.1	5.87E+0	69.3	5.93E+0	69.0	7.99E+0
77.1	1.37E+1	75.0	1.26E+1	72.0	9.73E+0	73.6	1.15E+1	77.2	9.39E+0	74.6	1.16E+1
81.1	1.83E+1	82.8	2.14E+1	76.2	1.52E+1	81.4	2.47E+1	81.4	1.27E+1	82.3	1.74E+1
89.1	3.25E+1	86.4	3.12E+1	84.0	3.33E+1	85.7	3.78E+1	90.5	2.39E+1	86.5	2.45E+1
124.2	7.85E+1	98.4	4.93E+1	118.9	8.99E+1	98.2	6.01E+1	128.9	6.91E+1	99.3	4.03E+1
132.4	8.52E+1	117.9	7.44E+1	127.0	9.41E+1	116.7	8.36E+1	138.0	7.87E+1	117.5	5.84E+1
		123.6	8.16E+1			123.3	8.51E+1			124.4	6.49E+1
										130.4	7.19E+1

Table 8.13. Effluent Cs Concentrations During AP-101 Simulant Feed Processing (TI394-12, TI394-13, TI394-14)

TI394-12 (BSC 3380-2P-0101)				TI394-13 (BSC 3380-2P-0102)				TI394-14 (BSC 3380-2P-0103)			
Cycle 1		Cycle 2		Cycle 1		Cycle 2		Cycle 1		Cycle 2	
Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o
4.9	<1.40E-3	5.3	6.80E-3	5.9	<2.07E-3	4.5	8.55E-3	5.4	<1.01E-3	4.3	6.66E-3
10.0	7.41E-4	9.6	7.50E-3	11.6	<9.51E-4	10.5	8.11E-3	10.6	<1.87E-3	10.1	3.94E-3
30.3	1.20E-3	30.8	6.89E-3	32.6	3.50E-2	31.9	4.13E-2	29.6	1.36E-2	30.6	2.31E-2
39.5	1.05E-2	37.2	1.22E-2	42.1	3.65E-1	38.4	2.13E-1	38.3	8.15E-2	36.9	8.80E-2
48.8	7.08E-2	44.4	3.84E-2	51.6	2.03E+0	45.7	8.73E-1	47.1	3.54E-1	44.0	3.37E-1
65.3	9.88E-1	66.5	1.06E+0	68.6	1.82E+1	68.2	2.01E+1	63.4	5.42E+0	65.7	7.53E+0
73.1	2.20E+0	72.0	2.20E+0	76.7	3.29E+1	73.7	3.29E+1	71.0	1.13E+1	70.9	1.30E+1
77.0	3.49E+0	79.8	4.94E+0	80.7	4.29E+1	81.4	5.16E+1	74.9	1.56E+1	78.3	2.37E+1
86.3	1.30E+1	84.1	9.64E+0	88.7	6.07E+1	85.0	6.40E+1	79.7	2.76E+1	81.9	3.43E+1
125.1	5.54E+1	96.8	2.36E+1	125.1	9.21E+1	97.0	8.05E+1	102.1	6.80E+1	93.9	5.61E+1
134.4	6.69E+1	114.9	4.42E+1	131.5	9.88E+1	117.0	9.01E+1	109.8	7.81E+1	114.0	7.70E+1
		121.8	5.13E+1			121.8	9.34E+1			118.8	8.13E+1
		127.7	5.92E+1								

Table 8.14. Effluent Cs Concentrations During Elution Processing Cycle 1

TI394-5 (PS-493)		TI394-8 (PS-501)		TI394-11 (BSC 3380-2P-0100)		TI394-12 (BSC 3380-2P-0101)		TI394-12 (BSC 3380-2P-0102)		TI394-14 (BSC 3380-2P-0103)	
Cum. BV	C/C_o	Cum. BV	C/C_o	Cum. BV	C/C_o	Cum. BV	C/C_o	Cum. BV	C/C_o	Cum. BV	C/C_o
4.05	2.78E+0	2.89	1.26E+1	2.89	1.74E+0	3.78	5.18E-2	3.94	8.22E+0	3.72	6.94E-2
5.10	5.91E+1	5.00	5.86E+1	3.94	6.09E+0	4.74	3.90E+1	4.96	5.41E+1	4.69	3.58E+1
6.10	3.17E+1	5.99	1.90E+1	4.90	1.27E+1	5.71	5.92E+1	5.94	1.88E+1	5.63	5.31E+1
7.15	1.53E+0	7.06	1.29E+0	5.88	2.81E+1	6.74	1.64E+1	6.99	1.67E+0	6.64	6.42E+0
8.19	6.22E-1	8.00	6.46E-1	6.89	3.02E+1	7.65	1.09E+0	8.16	7.09E-1	7.76	8.71E-1
9.30	2.85E-1	9.11	2.83E-1	7.87	2.17E+1	8.72	4.35E-1	9.25	2.21E-1	8.81	2.85E-1
12.80	2.69E-2	12.75	1.64E-2	8.88	8.57E+0	12.23	3.70E-2	12.52	1.22E-2	12.00	2.24E-2
17.13	1.93E-3	16.73	6.15E-4	12.47	8.19E-2	16.13	2.59E-3	16.72	6.85E-4	16.12	8.98E-4
21.62	1.91E-3	21.35	9.49E-4	16.40	1.11E-2	20.71	1.77E-3	21.26	4.34E-3	20.42	4.33E-3
				21.06	4.69E-3						

8.4.2 TI394-8 (Microbeads PS-501)

The pretreatment processing resulted in a sharp conversion front from Na-form to H-form. The Cycle 1 elution resulted in a slightly ragged conversion front, similar to that observed with TI394-5. The eluted resin bed manifested a thickening of the black band to one side. Follow-on processing appeared to be affected by this phenomenon. The regeneration processing favored the opposite side, as though the thickened black band region shielded the solution downward flow. Figure 8.18 shows the thickened black band and the associated effect with the Cycle 2 regeneration.

a) b)

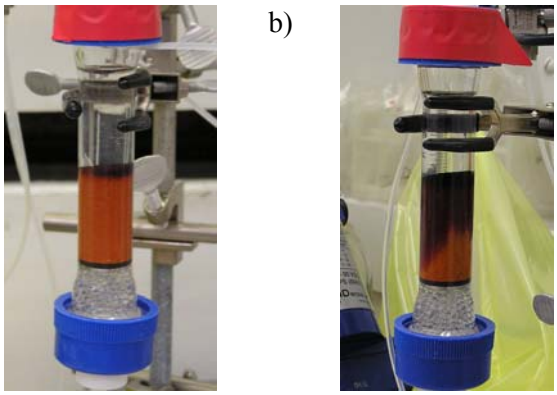


Figure 8.18. TI394-8 Cycle 2 (a) Just Before Regeneration, and (b) During Regeneration

The resin resulted in Cs breakthrough onset between 10 and 30 BVs. The contract limit (0.091% C/C_o) was reached at ~41 BVs. The 50% C/C_o was attained after processing ~100 BVs. The first 20 BVs of Cycle 2 resulted in immediate Cs breakthrough at one order of magnitude below the contract limit. After processing 30 BVs, Cycle 2 loading profile was virtually identical to the Cycle 1 loading profile.

8.4.3 TI394-11 (BSC 3380-2P-0100)

The pretreatment processing resulted in sharp conversion front from Na-form to H-form. The Cycle 1 elution manifested severe channeling based on the nearly vertical section of resin bed remaining black during the elution process (see Figure 8.19b). The channeling was further manifested in the broad elution profile (see Figure 8.17) relative to the other resins. After elution was completed, a dark band was found extending down ⅓ of resin bed length. Elution during Cycle 2 showed severe fingering as well as the persistent, irregular, dark black band. Pictures of these conditions are shown in Figure 8.20. (Note that this resin lot formed rock-hard agglomerated chunks when dried as the Na-form resin in preparation for skeletal density measurement.)

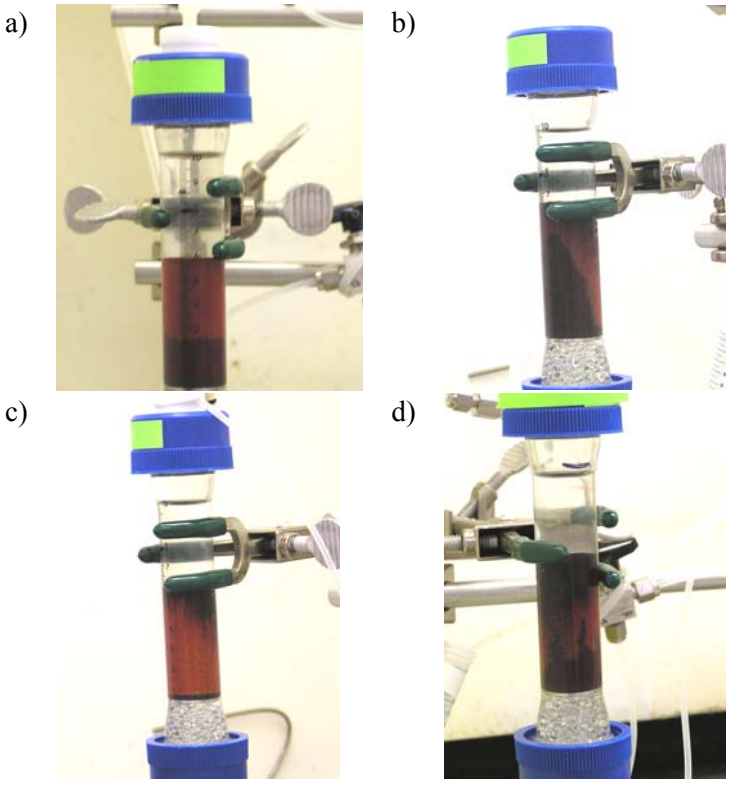


Figure 8.19. TI394-11 Elution Conditions (a) Resin Pretreatment, (b) Cycle 1 Elution, and (c) Cycle 1 Post-elution, (d) Cycle 2 Elution

The resin resulted in Cs breakthrough onset between 12 and 30 BVs. The contract limit was reached at ~36 BVs for Cycle 1 and ~26 BVs for Cycle 2. The 50% C/C_o was attained after processing ~112 BVs. The first 20 BVs of Cycle 2 resulted in immediate Cs breakthrough at one order of magnitude below the contract limit. The loading profile for Cycle 2 demonstrated earlier Cs breakthrough relative to the Cycle 1 loading profile. The Cycle 2 offset was attributed to the probable channeling caused by the irregularly oxidized (blackened) resin.

8.4.4 TI394-12 (BSC 3380-2P-0101)

The pretreatment processing resulted in a sharp conversion front from Na-form to H-form. The follow-on regeneration conversion front displayed channeling as one small section of the resin bed converted more slowly than the rest, therefore, 8 BVs (instead of 6 BVs) of 0.5 M NaOH regeneration solution was required to complete the conversion. The Cycle 1 elution resulted in a slightly ragged conversion front, as did the Cycle 2 regeneration and elution.

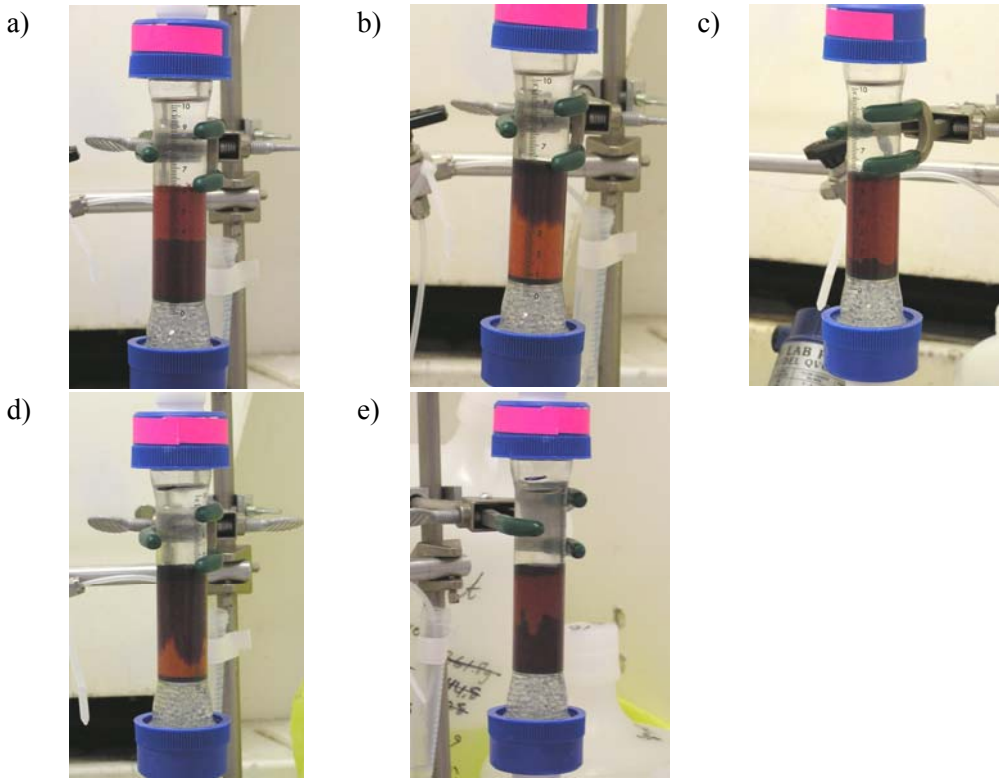


Figure 8.20. TI394-12 Conversion Fronts (a) Resin Pretreatment Na-Form to H-Form, (b) Resin Pretreatment H-Form to Na-Form, (c) Cycle 1 Elution, (d) Cycle 2 Regeneration (e) Cycle 2 Elution

The resin resulted in Cs breakthrough onset at 10 BVs, two orders of magnitude below the contract limit. The contract limit was reached at ~50 BVs. The 50% C/C_o was attained after processing ~121 BVs. The first 31 BVs of Cycle 2 resulted in immediate (but constant) Cs breakthrough at one order of magnitude below the contract limit. After processing 45 BVs, the Cycle 2 loading profile was virtually identical to the Cycle 1 loading profile.

8.4.5 TI394-13 (BSC 3380-2P-0102)

The pretreatment processing resulted in a sharp conversion front from Na-form to H-form. The Cycle 1 elution manifested a flat conversion front through the first 1/3 resin bed; after this portion was converted, fingering (maximum ~1 cm peak to valley) began to develop. The second cycle conversions were similar to those of the first cycle.

The resin resulted in Cs breakthrough onset between 12 and 30 BVs. The contract limit was reached at ~36 BVs. The 50% C/C_o was attained after processing ~82 BVs. The first 15 BVs of Cycle 2 resulted in immediate Cs breakthrough at one order of magnitude below the contract limit. After processing 32 BVs, the Cycle 2 loading profile was virtually identical to the Cycle 1 loading profile.

8.4.6 TI394-14 (BSC 3380-2P-0103)

The pretreatment processing resulted in a sharp conversion front from Na-form to H-form. The Cycle 1 elution resulted in a ragged conversion front. The second cycle conversion fronts were similar to those of the first cycle.

The resin resulted in Cs breakthrough onset between 11 and 30 BVs. The contract limit was reached at ~38 BVs. The 50% C/C_o was attained after processing ~91 BVs. The first 15 BVs of Cycle 2 resulted in immediate Cs breakthrough at one order of magnitude below the contract limit. After processing 30 BVs, the Cycle 2 loading profile was virtually identical to the Cycle 1 loading profile.

8.4.7 Cs Loading and Residual Cs Summary

The total Cs loading and residual Cs remaining after elution on each resin bed are summarized in Table 8.15. The TI394-12 resin loaded the largest Cs mass and highest concentration per unit volume. The TI394-8 resin loaded the highest Cs per unit resin mass.

Table 8.15. Cs Load and Residuals Summary, Wave 2

Resin ID	**TI394-5**	**TI394-8**	**TI394-11**	**TI394-12**	**TI394-13**	**TI394-14**
Manufacturer	**Microbeads**		**BSC**			
Lot #	**PS-493**	**PS-501**	**3380-2P-0100**	**3380-2P-0101**	**3380-2P-0102**	**3380-2P-0103**
Cycle 1						
Contract limit, BV	33	41	36	50	37	39
50% breakthrough, BV	102	100	113	121	83	92
Net mg Cs loaded	10.7	10.3	11.5	12.5	9.57	10.2
mg Cs/mL resin	0.60	0.57	0.65	0.68	0.51	0.54
mg Cs/g resin	2.11	2.41	1.88	2.05	2.04	1.87
Eluate 0.5 M HNO_3, BV	21.6	21.3	21.1	20.7	21.3	20.4
residual μg Cs/g resin	0.150	0.117	0.155	0.171	0.154	0.148
% Cs remaining on resin	7.1E-3	4.9E-3	8.2E-3	8.3E-3	7.5E-3	7.9E-3
Cycle 2						
Contract limit, BV	36	41	26	50	35	37
50% breakthrough, BV	99	98	109	121	81	90
Net mg Cs loaded	10.4	10.2	11.1	12.4	9.30	10.3
mg Cs/mL resin	0.58	0.56	0.63	0.67	0.49	0.55
mg Cs/g resin	2.04	2.38	1.81	2.03	1.98	1.89
Eluate 0.5 M HNO_3, BV	20.9	21.3	20.9	21.1	21.3	20.7
residual μg Cs/g resin	0.145	0.158	0.141	0.145	0.141	0.144
% Cs remaining	7.1E-3	6.6E-3	7.8E-3	7.1E-3	7.1E-3	7.6E-3

Typically, 7.2E-3% of the total Cs loaded on the resin bed remained on the resin after elution. The residual Cs on TI394-11 resin, which manifested channeling, was not significantly different

than the other samples. In all cases, the residual Cs content was well below (a factor of 30) the design limit of 4.2 μg/g.

8.5 Shrink-Swell Characteristics

The relative in-column expansion and contraction characteristics of the Wave 2 resins are summarized in Figure 8.21 along with SL-644 as a reference point (Wave 1 testing). The process steps (x-axis) are provided in Table 8.16. The relative volume changes were similar for the suite of resins tested, except for TI394-11. This resin may not have been fully converted to the H-form (Step 8) during elution because of channeling problems. This resin also expanded in Cycle 2 to a higher extent than any other resin.

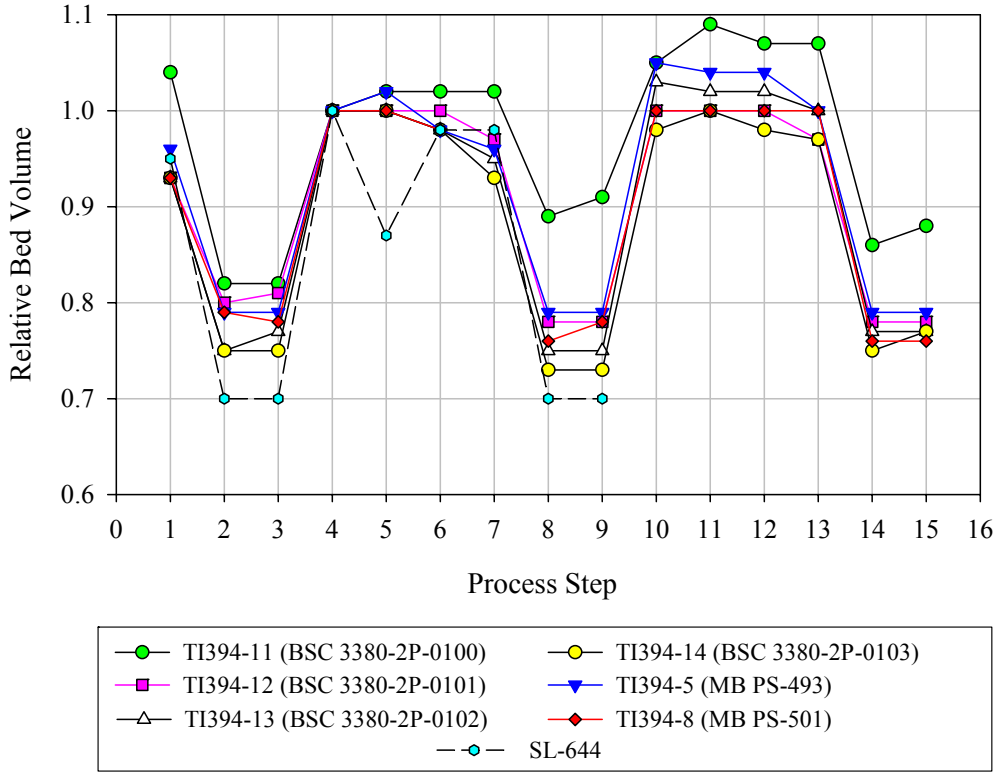


Figure 8.21. Wave 2 Resins Shrink-Swell Characteristics

Table 8.16. Actual Bed Volumes as a Function of Feed Matrix, Wave 2

Feed matrix	Step No.	TI394-5 PS-493 mL	TI394-8 PS-501 mL	TI394-11 3380-2P-0100 mL	TI394-12 3380-2P-0101 mL	TI394-13 3380-2P-0102 mL	TI394-14 3380-2P-0103 mL
1M NaOH soak/DI rinse	1	17.3	17.0	18.2	17.3	17.6	17.6
0.5M HNO_3	2	14.1	14.5	14.5	14.8	14.1	14.1
DI water	3	14.1	14.1	14.5	15.1	14.5	14.1
0.5M NaOH[(a)]	4	17.9	18.2	17.6	18.5	18.8	18.8
Feed, AP101 simulant	5	18.2	18.2	17.9	18.5	18.8	18.8
Feed displacement	6	17.6	17.9	17.9	18.5	18.5	18.5
DI water	7	17.3	nr	17.9	17.9	17.9	17.6
0.5M HNO_3	8	14.1	13.8	15.7	14.5	14.1	13.8
DI water	9	14.1	14.1	16.0	14.5	14.1	13.8
0.5M NaOH	10	18.8	18.2	18.5	18.5	19.5	18.5
Feed, AP101 simulant	11	18.5	18.2	19.2	18.5	19.2	18.8
Feed displacement	12	18.5	18.2	18.8	18.5	19.2	18.5
DI water	13	17.9	18.2	18.8	17.9	18.8	18.2
0.5M HNO_3	14	14.1	13.8	15.1	14.5	14.5	14.1
DI water	15	14.1	13.8	15.4	14.5	14.5	14.5
Dry resin mass (H-form)	na	5.09 g	4.28 g	6.11 g	6.10 g	4.70 g	5.44 g

(a) Reference volume for 1-BV.

9.0 Wave 2a Testing

Wave 2a testing was conducted with two resins prepared in the 200-L pilot reactor under different manufacturing conditions by Microbeads in April 2005. Samples of ~2 L were received at PNWD on 04/12/05. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates, and receipt dates are cross-referenced in Table 9.1.

Table 9.1. Wave 2a Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-15[(a)]	Microbeads	5C-370/522	2-gal	4/05	4/12/05
TI394-16	Microbeads	5C-370/523	2-gal	4/05	4/12/05

(a) TI394-15 was synthesized similarly to Microbeads BRF-14 (Wave 1 testing) and TI-394-11 (BSC resin 3380-2P-0100, Wave 2 testing).

9.1 Physical Properties

The pre-treatment swell data are summarized in Table 9.2. Both resins expanded ~30% from the as-received condition after one cycle pretreatment in the open beaker format. Micrographs were not taken, and PSD and skeletal densities were not determined.

Table 9.2. Wave 2a Resins Pretreatment Swell Data Summary

		1st Cycle Expansion		
PNWD ID	Lot No.	As-received H-form, mL	Pretreated H-form, mL	Expansion Factor
TI394-15	5C-370/522	61.0	82	1.34
TI394-16	5C-370/523	60.5	79	1.31

Table 9.3 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed densities determined after the in-column pretreatment shrink-swell were slightly higher than the unconstrained bed densities. The Microbead product dry bed densities were typical of previously-tested resins.

Table 9.3. Wave 2a Resins Dry Bed Densities

Resin ID	Resin Form	Settled Resin Vol., mL[(a)]	Dry Resin Mass, g[(a)]	Settled Resin Density, g/mL[(b)]	Column Processing Bed Density, g/mL[(c)]
TI394-15	H-form	14.4	4.9463	0.343	0.36
TI394-16	H-form	13.9	4.4000	0.317	0.33

(a) Measured for dry resin mass determination.
(b) Dry H-form resin mass per unit wet H-form resin volume.
(c) Measured during column processing; only the dry H-form mass placed in the column was determined.

9.2 Column Testing

Column testing was conducted similarly to the previous test waves. The specific column test parameters are summarized in Table 9.4 and Table 9.5. One process cycle was evaluated for Cs load characteristics only. The resin beds were eluted in a batch mode (i.e., no samples were collected). Following elution, the columns were removed for direct counting by GEA to assess the residual Cs on the resin beds.

Table 9.4. Experimental Conditions for TI394-15 (Microbeads Lot 5C-370/522, Yellow Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/14/05)								
Water rinse	DI water	8.07	2.97	140	3.19	0.918	2.53	22
Acid wash	0.5 M HNO_3	9.08	3.34	157	3.19	0.917	2.85	22
Water rinse	DI water	3.43	1.26	59.3	1.49	0.430	2.30	22
Cycle 1 (Start 4/18/05)								
Regeneration	0.5 M NaOH	6.28	2.31	109	3.14	0.905	2.00	22
Loading column	AP-101 Simulant	82.1	NA	1418	1.51	0.436	54.9	22-23
Loading column	AP-101 Simulant	45.0	NA	778	2.94	0.848	15.4	22-23
Feed displacement	0.1 M NaOH	3.11	1.15	53.8	2.88	0.828	1.08	22
Rinse	DI water	3.41	1.25	58.8	2.92	0.841	1.17	22
Elution	0.5 M HNO_3	23.0	8.45	397	1.1 - 1.5	0.30 - 0.44	19.6	22-23
Rinse	DI water	3.44	1.26	59.4	1.41	0.407	2.43	22

(a) BV = bed volume (17.3 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 9.5. Experimental Conditions for TI394-16 (Microbeads Lot 5C-370/523, White Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/14/05)								
Water rinse	DI water	8.25	3.03	143	3.28	0.944	2.52	22
Acid wash	0.5 M HNO_3	9.20	3.38	159	3.25	0.935	2.83	22
Water rinse	DI water	3.42	1.26	59.1	1.51	0.435	2.27	22
Cycle 1 (Start 4/18/05)								
Regeneration	0.5 M NaOH	6.37	2.34	110	3.19	0.917	2.00	22
Loading column	AP-101 Simulant	82.4	NA	1424	1.53	0.440	53.9	22-23
Loading column	AP-101 Simulant	46.0	NA	794	3.00	0.864	15.3	22-23
Feed displacement	0.1 M NaOH	3.23	1.19	55.8	2.98	0.858	1.08	22
Rinse	DI water	3.32	1.22	57.3	3.02	0.868	1.10	22
Elution	0.5 M HNO_3	22.5	8.26	388	1.38	0.398	19.6	22-23
Rinse	DI water	3.41	1.26	59.0	1.42	0.410	2.4	22

(a) BV = bed volume (17.3 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Column processing resulted in generally sharp conversion fronts (H-form to Na-form and vice versa). During preconditioning, the conversion fronts were generally level across the beds. Photographs of the yellow column (TI394-15) during the pretreatment 0.5 M HNO_3 wash and Cycle 1 elution are shown in Figure 9.1. In these pictures, the resins are converting from the Na-form (dark color) to the H-form (lighter color). The conversion front during the elution processing with 0.5 M HNO_3 appeared more ragged than the pretreatment process with some distinct finger-like projections. The conversion front during elution of the white column was slightly more even than that of the yellow column. After processing, the top surface layer on each resin bed was black. The black surface was typical during RF processing and was attributed to oxidative attack.

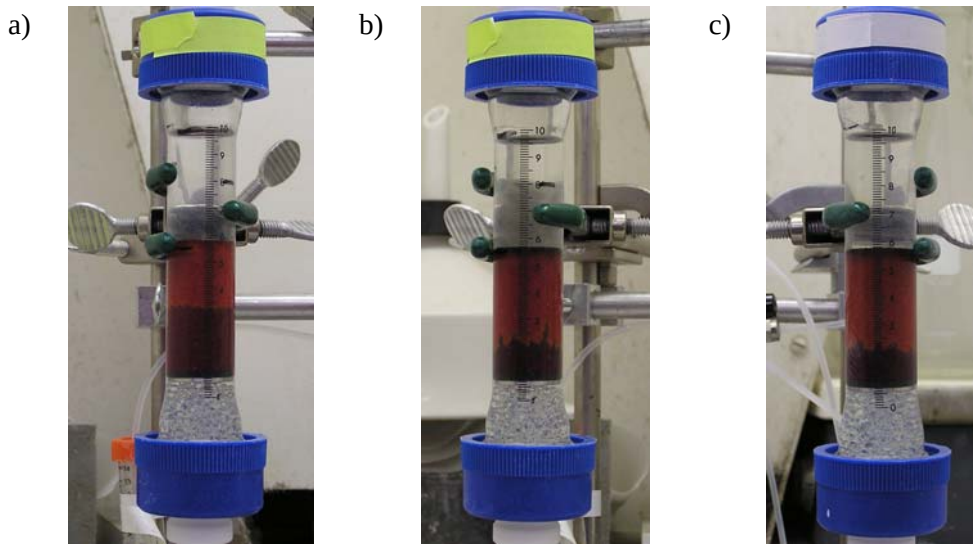


Figure 9.1. Resin Transitions from Na-form to H-form a) TI394-15 (5C-370/522) During Pretreatment and b) During Elution, c) TI394-16 (5C-370/523) During Elution

The Cs loading profile for each resin test is shown in Figure 9.2, and the plotted points are given in Table 9.6. The change in flowrate from 1.5 BV/h to 3.0 BV/h was associated with a slight change in the linearity of the load profile on the probability plot (at ~80 BVs).

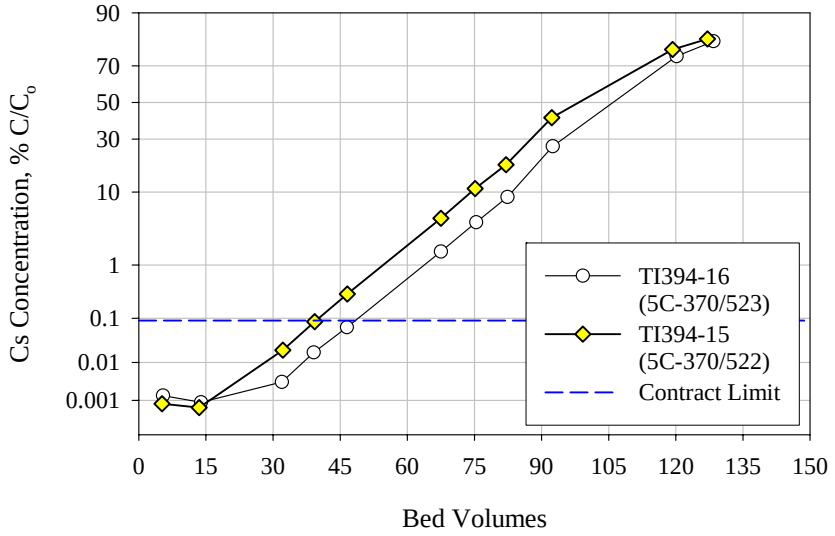


Figure 9.2. TI394-15 and TI394-16 Cs Loading Profiles with AP-101 Simulant

Manufacturing conditions in the large reactor successfully produced spherical RF. The manufacturing conditions associated with TI394-16 resulted in improved performance relative to TI394-15. In both cases, the Cs breakthrough onset occurred between 15 and 30 BVs. The 50% breakthrough was ~98 BVs for TI394-15 (5C-370/522) and ~106 BVs for TI394-16

(5C-370/523). The total Cs loaded on the TI394-15 resin was 9.90 mg, equivalent to 2.1 mg Cs/g dry H-form resin. The total Cs loaded on TI394-16 resin was 10.8 mg, equivalent to 2.4 mg Cs/g dry H-form resin. The residual Cs on the eluted resin beds were 0.141 μg/g and 0.163 μg/g for resins TI394-15 and TI394-16, respectively.

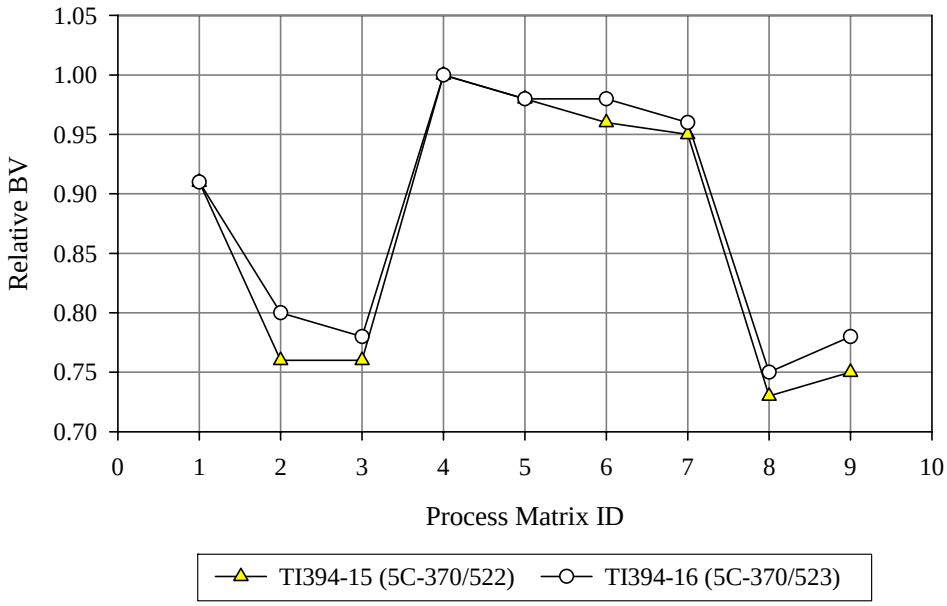
Table 9.6. Effluent Cs Concentrations During AP-101 Simulant Loading

TI394-15 (5C-370/522)		TI394-16 (5C-370/523)	
Cum. BV	**% C/C_o**	**Cum. BV**	**% C/C_o**
5.2	8.16E-4	5.4	<1.37E-3
13.5	<6.37E-4	13.9	<8.91E-4
32.2	1.97E-2	32.0	<3.18E-3
39.3	8.58E-2	39.1	1.73E-2
46.6	3.07E-1	46.5	6.42E-2
67.5	4.88E+0	67.5	1.64E+0
75.2	1.09E+1	75.4	4.33E+0
82.1	1.86E+1	82.4	8.82E+0
92.3	4.14E+1	92.5	2.65E+1
119.3	7.75E+1	120.2	7.46E+1
127.1	8.18E+1	128.4	8.09E+1

The actual measured resin volumes are provided in Table 9.7. Figure 9.3 displays the relative in-column shrink-swell characteristics of the Wave 2a resins. The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing. The relative BV shown (y-axis) is the BV observed divided by the BV in 0.5 M NaOH. Both resins displayed ~25% volume contraction from Na-form to H-form.

Table 9.7. Actual Bed Volumes in Each Feed Matrix, Wave 2a

		TI394-15	TI394-16
		5C-370/522	5C-370/523
Feed Matrix	**Matrix ID**	**mL**	**mL**
1 M NaOH soak/DI rinse	1	15.7	15.7
0.5 M HNO_3	2	13.2	13.8
DI water	3	13.2	13.5
0.5 M NaOH[(a)]	4	17.3	17.3
Feed, AP101 simulant	5	17.0	17.0
0.1 M NaOH	6	16.7	17.0
DI water	7	16.3	16.7
0.5 M HNO_3	8	12.6	12.9
DI water	9	12.9	13.5
Dry H-form resin mass	na	4.81 g	4.49 g
(a) Reference volume, 1-BV.			



Matrix IDs

1: Loaded in column	4: 0.5 M NaOH	7: DI water
2: 0.5M HNO_3	5: AP101 simulant	8: 0.5 M HNO_3
3: DI water rinse	6: 0.1 M NaOH	9: DI water

Figure 9.3. Wave 2a Resins Shrink-Swell Characteristics

10.0 Wave 2b Testing

Wave 2b testing was conducted with six resins prepared from a variety of production conditions by Microbeads. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates, and receipt dates are cross-referenced in Table 10.1. Note that four resins were prepared as continued curing of a separate aliquot of previously-prepared and tested resin.[a] The Microbeads resin 5C-370/522 was used as feedstock for resins PS-513 and PS-517; the Microbeads resin 5C-370/523 was used as feedstock for resins PS-514 and PS-515. In two of these cases, additional resorcinol and formaldehyde chemicals were added during the additional cure time. The preparation of PS-518 (TI394-21) at Microbeads duplicated the BSC Lot 3380-2P-0101 preparation.

Table 10.1. Wave 2b Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-9	Microbeads	PS-502	150-mL	3/05	3/14/05
TI394-21[a]	Microbeads	PS-518	150-mL	4/05	4/20/05
TI394-15[b,c]	*Microbeads*	*5C-370/522*	*Feedstock*	*4/05*	na
TI394-17	Microbeads	PS-513	0.5-L	4/05	4/20/05
TI394-20	Microbeads	PS-517	0.5-L	4/05	4/20/05
TI394-16[b,d]	*Microbeads*	*5C-370/523*	*Feedstock*	*4/05*	na
TI394-18	Microbeads	PS-514	0.5-L	4/05	4/20/05
TI394-19	Microbeads	PS-515	0.5-L	4/05	4/20/05

(a) The PS-518 resin was prepared under identical conditions to the BSC Lot 3380-2P-0101.
(b) Samples from resins shown in italics were tested in Wave 2a. Microbeads further processed these resins to provide four new test resins.
(c) The 5C-370/522 resin was used as the stock material for the PS-513 and PS-517 production.
(d) The 5C-370/523 resin was used as the stock material for the PS-514 and PS-515 production.

10.1 Physical Properties

Physical property testing during Wave 2b was limited to shrink-swell and bed density data. Micrographs were not taken, and particle size was not determined.

The pre-treatment shrink-swell data are summarized in Table 10.2. All resins expanded ~30% after one cycle pretreatment in the open beaker format. No follow-on expansion to Na-form resin testing was conducted in the unconstrained (open beaker) format.

(a) The processing feedstock resins (5C-370/522 and 5C-370/523) were previously tested in Wave 2a, see Section 9.0.

Table 10.2. Wave 2b Resins Pretreatment Swell Data Summary

PNWD ID	Lot #	1st Cycle Expansion		
		As-received H-form, mL	Pretreated H-form, mL	Expansion Factor
TI394-9	PS-502	26.8	35.0	1.31
TI394-21	PS-518	46	61.5	1.34
TI394-17	PS-513	49	62.5	1.28
TI394-20	PS-517	49	65.5	1.34
TI394-18	PS-514	49	64.5	1.32
TI394-19	PS-515	48	62.0	1.29

Table 10.3 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed density determined after the in-column pretreatment shrink-swell agreed well with the unconstrained bed density. The resins prepared from the 5C-370/522 feedstock (TI394-17 and TI394-20) resulted in higher densities than the resins prepared from the 5C-370/523 feedstock.

Table 10.3. Wave 2b Resins Dry Bed Densities

Resin ID	Lot #	Resin Form	Settled Vol., mL	Dry Mass, g	Settled Resin Density, g/mL[(a)]	Column Processing Bed Density, g/mL[(b)]
TI394-9	PS-502	H-form	14.6	4.7165	0.323	0.31
TI394-21	PS-518	H-form	14.5	4.9005	0.338[(c)]	0.32
TI394-17	PS-513	H-form	14.4	5.3169	0.369	0.37
TI394-20	PS-517	H-form	14.5	5.4071	0.373	0.36
TI394-18	PS-514	H-form	14.5	4.8331	0.333	0.32
TI394-19	PS-515	H-form	14.4	4.8439	0.336	0.34

(a) Dry resin mass per unit wet volume
(b) Measured during column processing; only the dry H-form mass placed in the column was determined.
(c) The TI394-12 resin (BSC 3380-2P-0101 product prepared under same manufacturing conditions) resulted in a settled resin density of 0.426 g/mL, 26% higher than the TI394-21.

10.2 Column Testing

Column testing for Wave 2b only evaluated Cs loading profiles. The elution was conducted as a bulk collection under normal process conditions without sample evaluation. Therefore, elution profiles are not reported. The ion exchange columns were counted after testing to determine residual Cs on the resin and thus the efficiency of elution. The column test parameters are summarized in Table 10.4 through Table 10.9.

Table 10.4. Experimental Conditions for TI394-9 (Microbeads Lot PS-502, Red Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/23/05)								
Water rinse	DI water	7.95	3.08	145	3.06	0.928	2.60	23
Acid wash	0.5 M HNO_3	8.74	3.39	159	3.05	0.926	2.87	23
Water rinse	DI water	3.63	1.41	66.1	1.43	0.435	2.53	22
Cycle 1 (Start 4/24/05)								
Regeneration	0.5 M NaOH	6.82	2.64	124	3.03	0.921	2.25	23
Loading column	AP-101 Simulant	82.0	NA	1494	1.48	0.448	55.6	22-23
Loading column	AP-101 Simulant	43.9	NA	801	3.01	0.914	14.6	22-23
Feed displacement	0.1 M NaOH	3.33	1.29	60.7	2.98	0.906	1.12	23
Rinse	DI water	3.50	1.36	63.7	3.08	0.937	1.13	23
Elution	0.5 M HNO_3	21.6	8.36	393	1.47	0.446	14.6	22-23
Rinse	DI water	3.08	1.19	56.1	1.50	0.456	2.05	23

(a) BV = bed volume (18.2 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 10.5. Experimental Conditions for TI394-17 (Microbeads Lot PS-513, Blue Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/22/05)								
Water rinse	DI water	7.70	3.09	145	2.84	0.891	2.72	22
Acid wash	0.5 M HNO_3	8.39	3.36	158	2.80	0.878	3.00	22
Water rinse	DI water	3.02	1.21	56.9	1.34	0.421	2.25	22
Cycle 1 (Start 4/23/05)								
Regeneration	0.5 M NaOH	6.57	2.63	124	2.86	0.897	2.30	23
Loading column	AP-101 Simulant	80.6	NA	1519	1.48	0.464	53.7	22-23
Loading column	AP-101 Simulant	49.9	NA	940	2.91	0.916	17.2	22-23
Feed displacement	0.1 M NaOH	3.16	1.27	59.6	2.83	0.889	1.12	22
Rinse	DI water	3.10	1.25	58.5	2.87	0.900	1.08	22
Elution[c]	0.5 M HNO_3	18.2	7.28	342	1.37	0.431	14.8	22-23
Rinse	DI water	2.81	1.13	53.0	1.40	0.441	2.00	23

(a) BV = bed volume (18.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Flow was stopped at 13 BVs for 2 hours because valve was inadvertently left closed.
NA = not applicable
All processing was downflow.

Table 10.6. Experimental Conditions for TI394-18 (Microbeads Lot PS-514, Yellow Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/22/05)								
Water rinse	DI water	8.01	3.16	148	3.00	0.927	2.67	22
Acid wash	0.5 M HNO_3	8.51	3.35	158	3.13	0.967	2.72	22
Water rinse	DI water	3.11	1.23	57.6	1.39	0.43	2.23	22
Cycle 1 (Start 4/23/05)								
Regeneration	0.5 M NaOH	6.41	2.53	119	2.89	0.894	2.22	23
Loading column	AP-101 Simulant	79.1	NA	1467	1.48	0.456	53.7	22-23
Loading column	AP-101 Simulant	50.9	NA	942	2.96	0.914	17.2	22-23
Feed displacement	0.1 M NaOH	3.22	1.27	59.6	2.88	0.890	1.12	22
Rinse	DI water	3.06	1.21	56.7	2.87	0.886	1.07	22
Elution	0.5 M HNO_3	20.6	8.11	381	1.38	0.426	14.9	22-23
Rinse	DI water	2.83	1.12	52.5	1.42	0.437	2.00	23

(a) BV = bed volume (18.5 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 10.7. Experimental Conditions for TI394-19 (Microbeads Lot PS-515, White Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/22/05)								
Water rinse	DI water	8.23	3.19	150	3.07	0.931	2.68	23
Acid wash	0.5 M HNO_3	8.65	3.35	158	3.11	0.944	2.78	22
Water rinse	DI water	3.31	1.28	60.4	1.51	0.457	2.20	22
Cycle 1 (Start 4/23/05)								
Regeneration	0.5 M NaOH	6.55	2.54	119	3.05	0.925	2.15	23
Loading column	AP-101 Simulant	79.4	NA	1447	1.46	0.444	53.7	22-23
Loading column	AP-101 Simulant	52.1	NA	949	3.04	0.923	17.2	22-23
Feed displacement	0.1 M NaOH	3.31	1.28	60.3	2.96	0.899	1.12	22
Rinse	DI water	3.14	1.22	57.3	2.95	0.895	1.07	22
Elution[c]	0.5 M HNO_3	18.3	7.09	333	1.36	0.414	14.9	22-23
Rinse	DI water	2.74	1.06	49.9	1.37	0.416	2.00	23

(a) BV = bed volume (18.2 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Flow was stopped at 13 BVs for 2 hours because valve was inadvertently left closed.
NA = not applicable
All processing was downflow.

Table 10.8. Experimental Conditions for TI394-20 (Microbeads Lot PS-517, Pink Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/22/05)								
Water rinse	DI water	8.14	3.26	153	3.01	0.947	2.70	22
Acid wash	0.5 M HNO_3	8.53	3.42	161	3.20	1.005	2.67	22
Water rinse	DI water	3.13	1.26	59.1	1.39	0.438	2.25	22
Cycle 1 (Start 4/23/05)								
Regeneration	0.5 M NaOH	6.53	2.62	123	2.90	0.912	2.25	23
Loading column	AP-101 Simulant	81.6	NA	1539	1.50	0.470	53.9	22-23
Loading column	AP-101 Simulant	50.0	NA	942	2.98	0.937	16.8	22-23
Feed displacement	0.1 M NaOH	3.15	1.26	59.3	2.95	0.927	1.07	22
Rinse	DI water	3.27	1.31	61.6	2.84	0.893	1.15	22
Elution[c]	0.5 M HNO_3	18.4	7.40	348	1.40	0.440	14.7	22-23
Rinse	DI water	2.89	1.16	54.4	1.43	0.450	2.02	23

(a) BV = bed volume (18.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
(c) Flow was stopped at 13 BVs for 2 hours because valve was inadvertently left closed.
NA = not applicable
All processing was downflow.

Table 10.9. Experimental Conditions for TI394-21 (Microbeads Lot PS-518, Green Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[a]	AV[b]	mL	BV/h	mL/min	h	
In-situ Preconditioning (4/22/05)								
Water rinse	DI water	7.84	3.14	148	2.87	0.901	2.73	23
Acid wash	0.5 M HNO_3	8.25	3.31	156	2.85	0.894	2.90	22
Water rinse	DI water	2.96	1.19	55.8	1.33	0.417	2.23	22
Cycle 1 (Start 4/23/05)								
Regeneration	0.5 M NaOH	6.57	2.63	124	2.59	0.814	2.53	23
Loading column	AP-101 Simulant	79.5	NA	1499	1.45	0.456	53.9	22-23
Loading column	AP-101 Simulant	48.4	NA	912	2.88	0.905	16.8	22-23
Feed displacement	0.1 M NaOH	3.16	1.27	59.5	2.91	0.915	1.08	22
Rinse	DI water	3.23	1.29	60.8	2.81	0.881	1.15	22
Elution	0.5 M HNO_3	20.7	8.30	390	1.41	0.443	14.7	22-23
Rinse	DI water	2.87	1.15	54.0	1.42	0.447	2.02	23

(a) BV = bed volume (18.8 mL in Na form as loaded in column)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

The resin conversion from H-form to Na-form resulted in generally diffuse conversion fronts. In contrast, resin conversion from Na-form to H-form resulted in sharp conversion fronts. Examples of the two types of conversion fronts are shown in Figure 10.1. In these pictures, the H-form resin is light-colored, and the Na-form resin is dark.

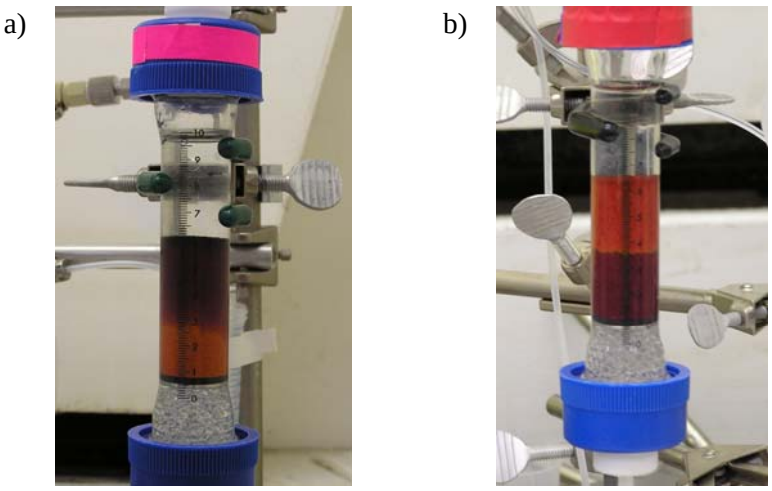


Figure 10.1. Typical Resin Conversion Fronts (a) TI394-20 (PS-517) Converting to Na-form and (b) TI394-9 (PS-502) Converting to H-form

The Cs loading profiles for each resin test are shown in Figure 10.2 and Figure 10.3. As shown previously, the change in flowrate from 1.5 BV/h to 3.0 BV/h was associated with a slight change in the linearity of the load profile on the probability plot (at ~80 BVs). The Cs breakthrough onset occurred at ~35 BVs for all resins except TI394-17 (PS-513), which appeared to break through at ~30 BVs. The 50% breakthrough points were similar for the all resins ranging from 105 BVs to 110 BVs. The data plotted in Figure 10.2 and Figure 10.3 are given in Table 10.10.

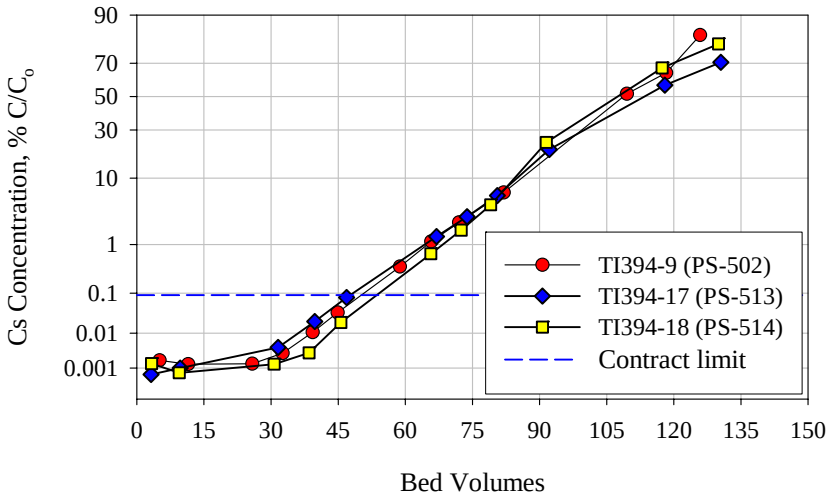


Figure 10.2. Cs Breakthrough Loading Profiles of TI394-9, -17, and -18 (Microbeads PS-502, PS-513, PS-514) with AP-101 Simulant

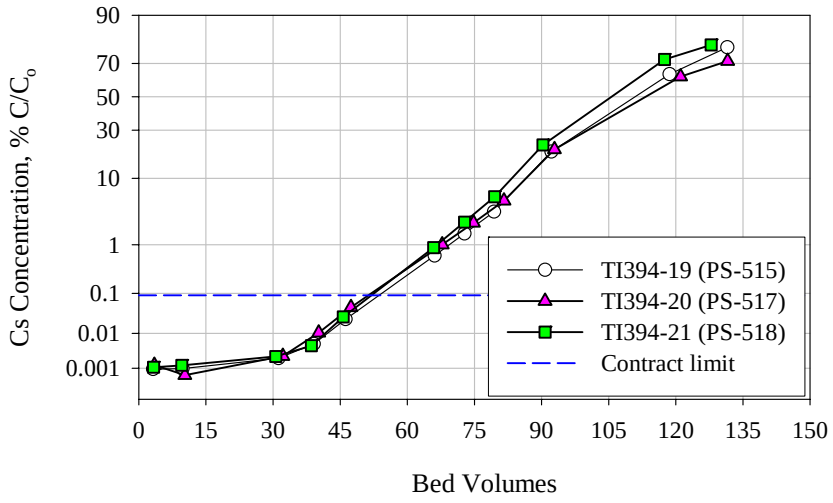


Figure 10.3. Cs Breakthrough Loading Profiles of TI394-19, -20, and -21 (Microbeads PS-515, PS-517, PS-518) with AP-101 Simulant

Table 10.10. Effluent Cs Concentration During Loading

TI394-9		TI394-17		TI394-18		TI394-19		TI394-20		TI394-21	
PS-502		PS-513		PS-514		PS-515		PS-517		PS-518	
Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o	Cum. BV	% C/C_o
5.1	<1.69E-3	3.2	<6.21E-4	3.3	<1.36E-3	3.2	<9.32E-4	3.5	<1.24E-3	3.3	<1.06E-3
11.5	<1.30E-3	9.7	<9.98E-4	9.5	<7.11E-4	9.8	<9.70E-4	10.3	<6.10E-4	9.6	<1.22E-3
25.8	<1.34E-3	31.6	4.01E-3	30.7	<1.30E-3	31.2	<1.99E-3	32.2	2.29E-3	30.6	<2.22E-3
32.6	2.75E-3	39.8	2.04E-2	38.5	2.81E-3	39.1	5.13E-3	40.2	1.05E-2	38.6	4.55E-3
39.3	1.06E-2	46.9	7.95E-2	45.6	1.92E-2	46.2	2.37E-2	47.4	4.67E-2	45.7	2.70E-2
44.9	3.45E-2	67.0	1.40E+0	65.7	6.77E-1	66.1	6.27E-1	67.8	1.01E+0	65.9	8.88E-1
58.8	3.75E-1	73.8	2.96E+0	72.5	1.79E+0	72.8	1.58E+0	74.8	2.38E+0	72.8	2.45E+0
65.8	1.14E+0	80.6	6.00E+0	79.1	4.46E+0	79.4	3.55E+0	81.6	5.10E+0	79.5	5.82E+0
72.0	2.40E+0	92.2	2.03E+1	91.5	2.36E+1	92.2	1.94E+1	92.9	2.04E+1	90.3	2.25E+1
82.0	6.58E+0	118.0	5.71E+1	117.4	6.74E+1	118.6	6.39E+1	121.1	6.25E+1	117.5	7.21E+1
109.5	5.17E+1	130.5	7.05E+1	130.0	7.96E+1	131.5	7.81E+1	131.6	7.13E+1	127.9	7.93E+1
118.3	6.45E+1										
125.9	8.33E+1										

Figure 10.4 compares the Cs loading performances of TI394-21 (Microbeads PS-518) with TI394-12 (BSC Lot 3380-2P-0101). The initial breakthrough performances were virtually identical. The discrepancy found between the resins was associated with the 50% C/C_o breakthrough. The BSC resin performance exceeded 120 BVs processed before reaching 50% breakthrough whereas the PS-518 resin processed 105 BVs at 50% breakthrough. The BSC resin had a higher settled resin bed density and thus may have had higher RF density loading within the resin bead, thus giving it more active Cs exchanges sites.

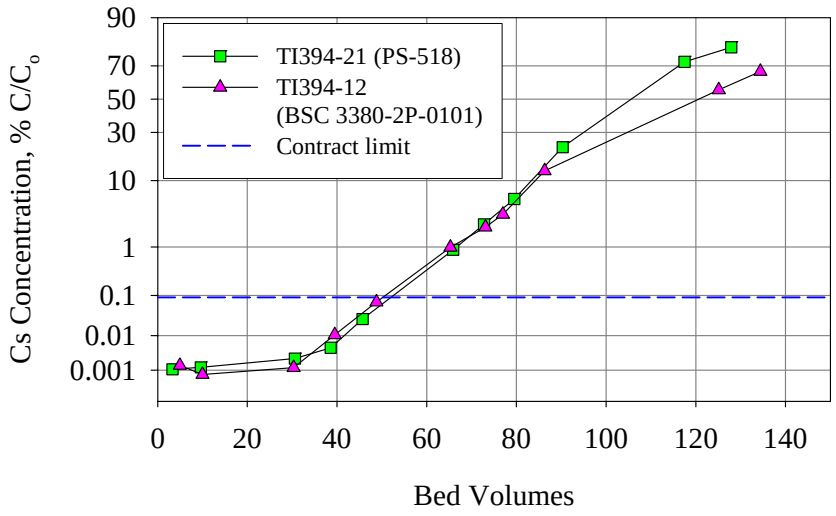


Figure 10.4. Comparison of TI394-21 (PS-518) with TI394-12 (BSC 3380-2P-0101) (Same Production Conditions)

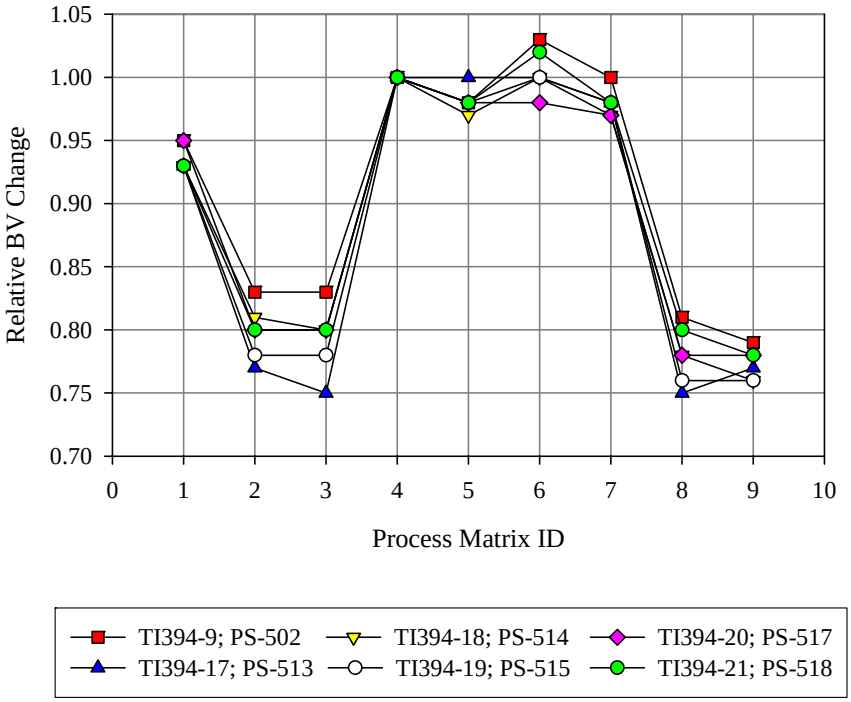
The total Cs load concentration and residual Cs on the eluted ion exchange resins were evaluated. The summarized results are presented in Table 10.11 along with the BVs to the contract limit and 50% Cs breakthroughs. The load density varied from 2.25 to 2.46 mg Cs/g dry H-form resin. The residual Cs varied between 0.14 and 0.21 μg/g.

Table 10.11. Wave 2b Resins Cs Load and Residual Summary

Resin ID	Lot #	Cs Breakthrough, BV		Total Cs Load			Eluant Volume, BV	Residual Cs, μg/g[a, c]
		Contract Limit	50%	mg	mg/g[a]	mg/mL[b]		
TI394-9	PS-502	51	109	11.5	2.46	0.63	21.6	0.21
TI394-21	PS-518	53	105	11.7	2.38[d]	0.62[d]	20.7	0.18
TI394-17	PS-513	48	113	12.1	2.25	0.64	18.2	0.16
TI394-20	PS-517	52	113	12.3	2.27	0.65	18.4	0.14
TI394-18	PS-514	54	107	11.6	2.41	0.63	20.6	0.19
TI394-19	PS-515	54	110	11.7	2.42	0.64	18.3	0.16

(a) Cs per gram dry H-form resin.
(b) Volume in the expanded Na-form.
(c) Residual Cs was measured post-elution.
(d) The BSC resin, prepared under identical conditions, resulted in a Cs load concentration of 2.05 mg/g resin and 0.68 mg/mL resin.

Figure 10.5 displays the in-column shrink-swell characteristics of the Wave 2b resins. The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing. The relative BV shown is the BV observed divided by the BV in 0.5 M NaOH. The TI394-17 (PS-513) showed the widest volume contraction at 25%, and the TI394-9 (PS-502) and TI394-21 (PS-518) resulted in the smallest volume variations of ~20%.



Matrices	1: Loaded in column (Na-form)	4: 0.5 M NaOH	7: DI water
	2: 0.5M HNO_3	5: AP101 simulant	8: 0.5M HNO_3
	3: DI water	6: 0.1 M NaOH feed displacement	9: DI water

Figure 10.5. Shrink-Swell Characteristics of Wave 2b Resins

Table 10.12 summarizes the measured resin volumes and the dry H-form resin masses loaded in each column.

Table 10.12. Actual Bed Volumes as a Function of Feed Matrix, Wave 2b

		TI394-9	TI394-17	TI394-18	TI394-19	TI394-20	TI394-21
	Matrix	PS-502	PS-513	PS-514	PS-515	PS-517	PS-518
Feed matrix	ID	mL	mL	mL	mL	mL	mL
1 M NaOH soak/DI rinse	1	17.3	17.6	17.3	17.0	17.9	17.6
0.5 M HNO_3	2	15.1	14.5	15.1	14.1	15.1	15.1
DI water	3	15.1	14.1	14.8	14.1	15.1	15.1
0.5 M NaOH[(a)]	4	18.2	18.8	18.5	18.2	18.8	18.8
Feed, AP101 simulant	5	17.9	18.8	17.9	17.9	18.5	18.5
0.1 M NaOH	6	18.8	18.8	18.5	18.2	18.5	19.2
DI water	7	18.2	18.5	17.9	17.9	18.2	18.5
0.5 M HNO_3	8	14.8	14.1	14.5	13.8	14.8	15.1
DI water	9	14.5	14.5	14.1	13.8	14.8	14.8
Dry resin mass (H-form)	na	4.68 g	5.35 g	4.83 g	4.84 g	5.41 g	4.90 g
(a) Reference volume, defined as 1-BV.							

11.0 Wave 3 Testing

Wave 3 testing was conducted with two resins produced in 50-gallon production batches by Microbeads in May 2005. These resins were the first received from a scale-up production process. PNWD received ~1.5 L from each batch for testing. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates, and receipt dates are cross-referenced in Table 11.1.

Table 11.1. Wave 3 Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-61[(a)]	Microbeads	5E-370/639	50-gal	5/05	6/6/05
TI394-62	Microbeads	5E-370/641	75-gal	5/05	6/6/05
(a) TI394-61 was synthesized under similar conditions as TI394-16 (Microbeads 5C-370/523, Wave 2a testing).					

11.1 Physical Properties

The pre-treatment shrink-swell data are summarized in Table 11.2. Both resins expanded ~26% after the initial (first cycle) swell-shrink cycle pretreatment in the open beaker format relative to the as-received condition. The follow-on expansion to Na-form resin was measured at 21% for the TI394-61 resin and 24% for the TI394-62 resin. The overall expansion from the as-received H-form resin to the pretreated Na-form resin was nominally 56%.

Table 11.2. Wave 3 Resins Pretreatment Swell Data Summary

	1st Cycle Expansion			H-form to Na-form Expansion			
PNWD ID	As-received H-form, mL	Pretreated H-form, mL	Expansion Factor	H-form,[(a)] mL	Na-form, mL	Expansion Factor	Total Expansion
TI394-61	53.5	68.2	1.27	25	30.3	1.21	1.55
TI394-62	54.2	68.0	1.25	25	31.0	1.24	1.56
(a) After first pretreatment cycle.							

Micrographs of the two RF resins are shown in Figure 11.1 and Figure 11.2. The 50-gal scaleup process resulted in uniformly spherical beads. The color difference between the two resins shown in the micrographs was consistent with macroscopic observations of the dried material. In contrast, the as-received, wet materials appeared similar in hue. The TI394-62 resin displayed greater inter-particle differences in color intensity than the TI394-61 resin. The material density and color appeared uniform through the sphere cross-sections, indicating that both production parameters successfully loaded RF into the entire resin bead. Although some anomalies in resin form or size were observed, both productions were generally uniformly spherical.

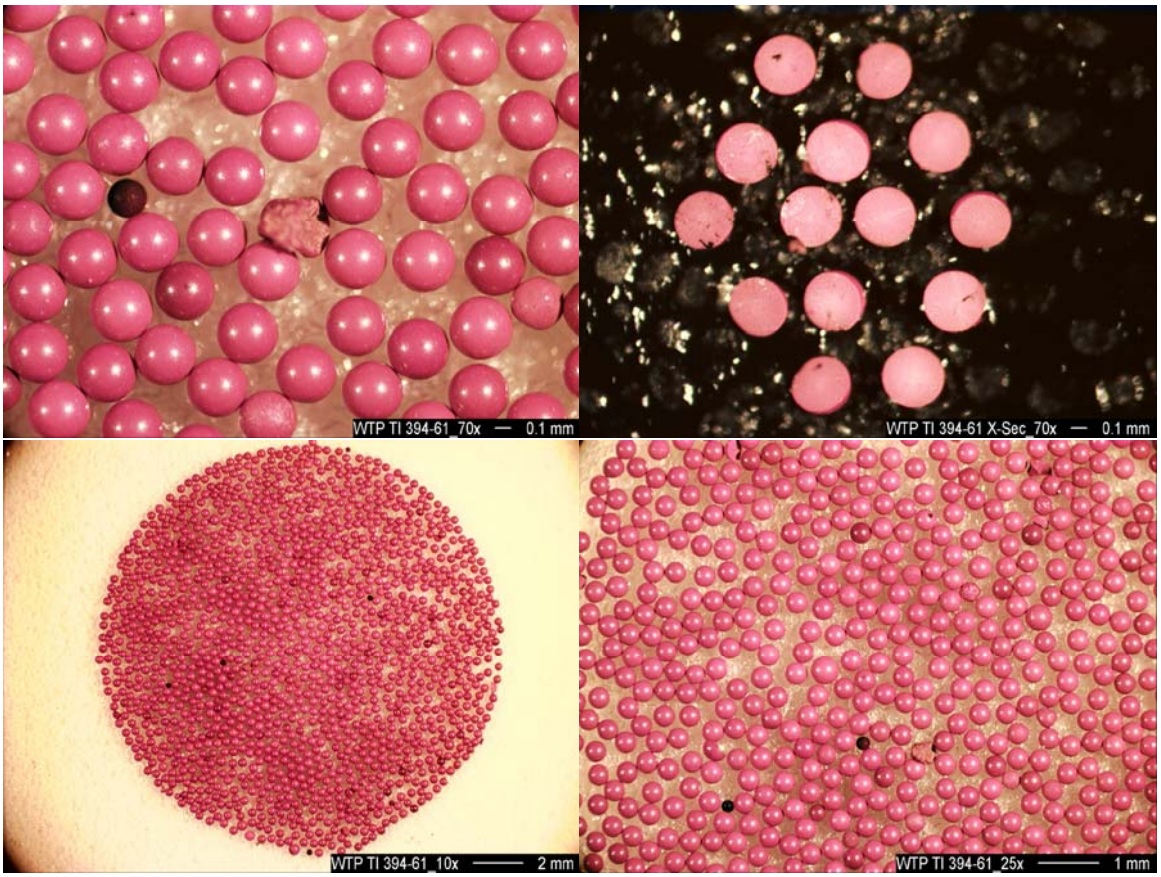


Figure 11.1. Micrographs of TI394-61 (Microbeads 5E-370/639). Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

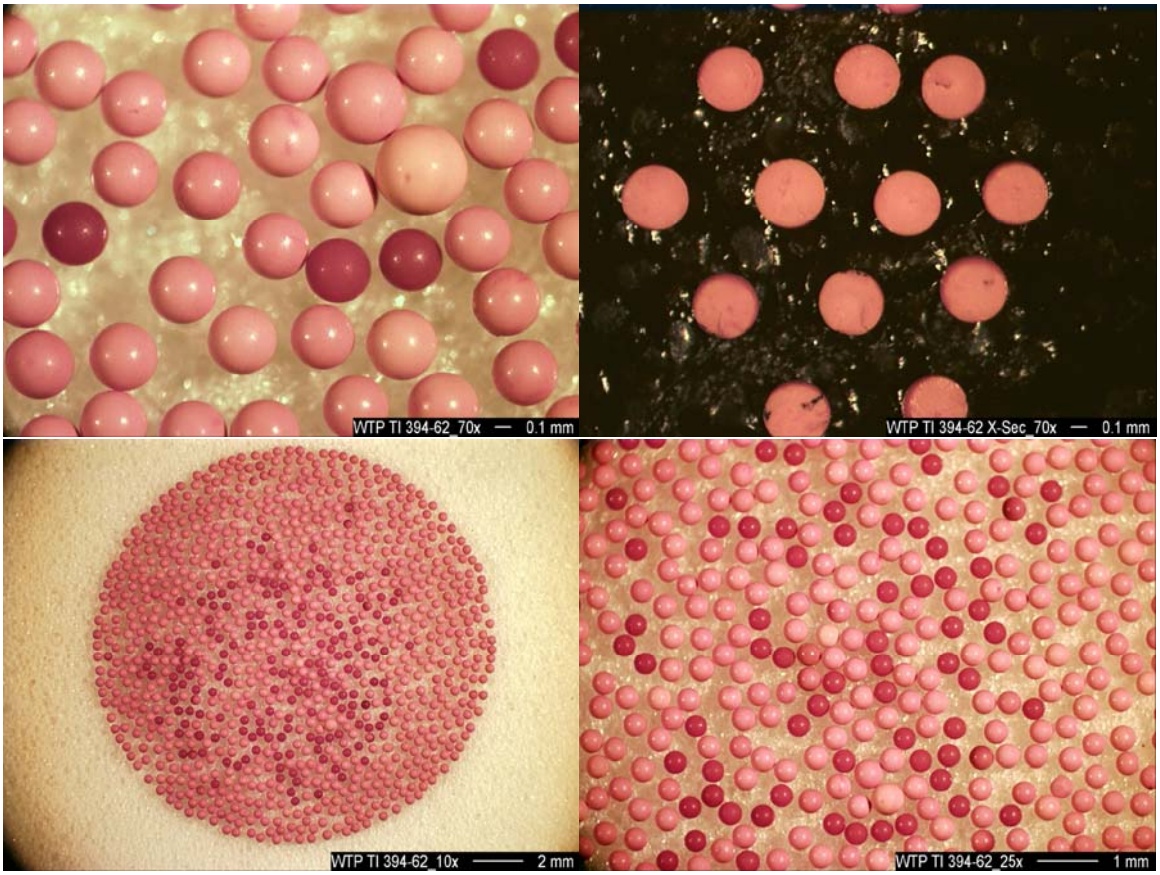


Figure 11.2. Micrographs of TI394-62 (Microbeads 5E-370/641). Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

The PSD results for the pretreated resins are shown in Figure 11.3 and Table 11.3. The PSDs based on volume, number, and area were in good agreement, indicating minimal fines and tight size distribution in each sample. The bead volume difference from the H-form to Na-form (calculated average ~25%) was consistent with the observed bulk resin volume expansions and contractions in the pretreatment steps and column processing. The detailed PSD results for TI394-62 resin are provided in Appendix D.

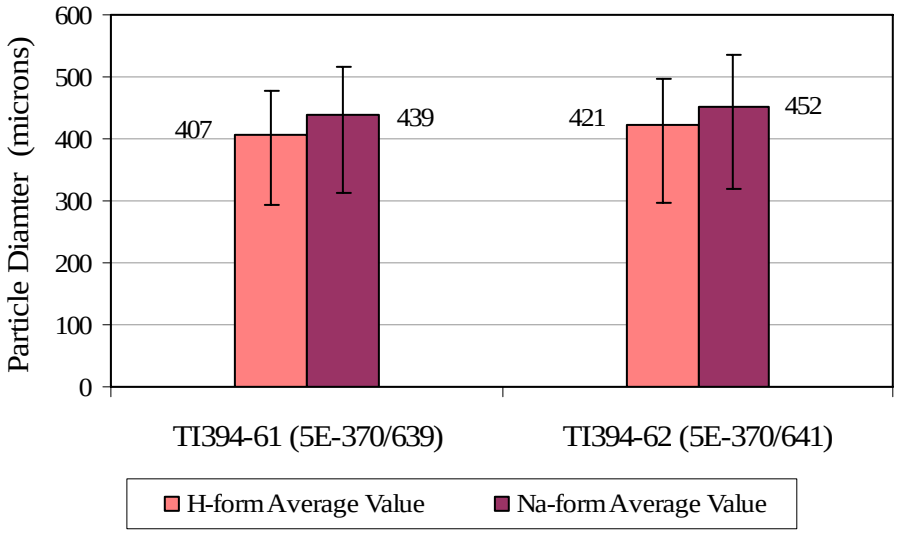


Figure 11.3. Wave 3 Resins PSD Showing Average Values and Low 5% to High 90% Spread (Volume basis)

Table 11.3. Wave 3 Resins Particle-Size-Distribution Summary

Resin ID Lot #	Resin Form	Volume Distribution (microns) m_v	sd	Low 5%[a]	High 90%[b]	Number Distribution (microns) m_n	sd	Low 5%[a]	High 90%[b]	Area Dist. (microns) m_a
TI394-61	H-form	407	73	302	511	369	56	283	445	392
5E-370/639	Na-form	439	79	323	559	394	60	301	476	421
TI394-62	H-form	421	78	307	538	377	59	288	457	403
5E-370/641	Na-form	452	83	333	575	406	70	308	793	434
Resin ID		**Calculated Average Sphere Volume, mm^3**								
TI394-61	H-form	0.0353				0.0263				0.0316
5E-370/639	Na-form	0.0444				0.0320				0.0391
Expansion factor >		*26%*				*22%*				*24%*
TI394-62	H-form	0.0391				0.0280				0.0343
5E-370/641	Na-form	0.0483				0.0351				0.0428
Expansion factor >		*24%*				*25%*				*25%*

(a) 5% of the particles are less than the given diameter.
(b) 10% of the particles are greater than the given diameter.

Table 11.4 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed density determined after the in-column pretreatment shrink-swell agreed well with the unconstrained bed density. The dry bed densities were typical of previously tested Microbeads resins.

Table 11.4. Wave 3 Resins Dry Bed Densities

Resin ID Lot #	Resin Form	Settled Vol., mL[(a)]	Dry Mass, g[(a)]	Settled Resin Density, g/mL[(b)]	Column Processing Bed Density, g/mL[(c)]
TI394-61	H-form	14.6	5.199	0.356	0.36
5E-370/639	Na-form	20.0	7.311	0.366	na
TI394-62	H-form	14.4	5.285	0.367	0.37
5E-370/641	Na-form	21.0	7.734	0.368	na

(a) Measured for skeletal density.
(b) Dry resin mass per unit wet volume
(c) Measured during column processing; only the dry H-form mass placed in the column was determined.
na = not applicable, Na-form mass was not determined.

The resin skeletal densities are provided in Table 11.5. Skeletal densities for both forms were consistent with previous measurements.

Table 11.5. Wave 3 Resins Skeletal Densities

		H-form		Na-form	
Resin ID	Lot #	g/mL	RPD	g/mL	RPD
TI394-61	5E-370/639	1.477	0.10%	1.614	0.09%
TI394-62	5E-370/641	1.477	0.09%	1.628	0.13%

11.2 Column Testing

Column testing was conducted similarly to the previous test waves with the exception of elution conditions. Elution was conducted with 19 BVs of 0.25 M nitric acid during one process cycle and 16 BVs of 0.4 M nitric acid during the other process cycle. The column test parameters are summarized in Table 11.6 and Table 11.7.

Table 11.6. Experimental Conditions for TI394-61 (Microbeads Lot 5E-370/639, Yellow Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (6/9/05)								
Water rinse	DI water	8.49	3.18	149	3.07	0.900	2.77	22
Acid wash	0.5 M HNO_3	9.21	3.45	162	3.03	0.890	3.03	22
Water rinse	DI water	3.37	1.26	59.3	1.51	0.442	2.23	22
Cycle 1 (Start 6/13/05)								
Regeneration	0.5 M NaOH	7.93	2.97	140	4.21	1.23	1.88	18
Loading column	AP-101 Simulant	79.3	NA	1395	1.47	0.431	53.6	19
Loading column	AP-101 Simulant	60.7	NA	1068	2.89	0.847	20.8	19
Feed displacement	0.1 M NaOH	3.11	1.16	54.6	3.01	0.881	1.03	20
Rinse	DI water	2.98	1.11	52.3	2.93	0.858	1.02	20
Elution	0.25 M HNO_3	19.0	7.13	335	1.41	0.414	13.5	20
Rinse	DI water	3.27	1.22	57.5	1.41	0.414	2.32	20
Cycle 2 (Start 6/20/05)								
Regeneration	0.5 M NaOH	6.50	2.43	114	2.98	0.873	2.18	20
Loading column	AP-101 Simulant	80.5	NA	1417	1.51	0.442	52.4	20-23
Loading column	AP-101 Simulant	65.6	NA	1154	3.03	0.888	22.1	20
Feed displacement	0.1 M NaOH	3.22	1.21	56.7	2.98	0.873	1.08	20
Rinse	DI water	3.26	1.22	57.3	2.96	0.869	1.10	20
Elution	0.4 M HNO_3	16.0	6.00	282	1.43	0.420	11.2	20
Rinse	DI water	3.17	1.19	55.8	1.41	0.413	2.25	20

(a) BV = bed volume (17.6 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 11.7. Experimental Conditions for TI394-62 (Microbeads Lot 5E-370/641, White Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (6/9/05)								
Water rinse	DI water	8.46	3.22	152	3.06	0.913	2.77	22
Acid wash	0.5 M HNO_3	8.87	3.38	159	3.02	0.902	2.93	22
Water rinse	DI water	3.22	1.22	57.6	1.44	0.430	2.23	22
Cycle 1 (Start 6/13/05)								
Regeneration	0.5 M NaOH	6.79	2.59	122	3.06	0.915	2.22	18
Loading column	AP-101 Simulant	80.8	NA	1447	1.50	0.447	53.6	19
Loading column	AP-101 Simulant	63.3	NA	1134	3.05	0.910	20.8	19
Feed displacement	0.1 M NaOH	3.16	1.21	56.7	3.06	0.914	1.03	20
Rinse	DI water	3.03	1.16	54.3	2.89	0.862	1.05	20
Elution	0.40 M HNO_3	16.0	6.09	286	1.41	0.421	11.3	20
Rinse	DI water	3.24	1.23	58.0	1.38	0.411	2.35	20
Cycle 2 (Start 6/20/05)								
Regeneration	0.5 M NaOH	6.51	2.48	116	2.98	0.890	2.18	20
Loading column	AP-101 Simulant	80.0	NA	1432	1.49	0.446	53.1	20-23
Loading column	AP-101 Simulant	65.2	NA	1168	3.02	0.900	21.7	20
Feed displacement	0.1 M NaOH	3.29	1.25	58.9	2.99	0.893	1.10	20
Rinse	DI water	3.23	1.23	57.8	2.93	0.876	1.10	20
Elution	0.25 M HNO_3	19.0	7.26	341	1.40	0.417	13.6	20
Rinse	DI water	3.22	1.23	57.6	1.35	0.403	2.38	20

(a) BV = bed volume (17.9 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Column processing resulted in generally sharp conversion fronts (H-form to Na-form and vice versa). The yellow (TI394-61) and white (TI394-62) column second cycle elution processing is shown in Figure 11.4. In this picture, the resin was converting from the Na-form (dark color) to the H-form (lighter color). The conversion front had a crisp edge and was only slightly ragged. The top surface layer on each resin bed was black. The black surface was typical during RF processing and was attributed to be oxidative attack. The final depth of the black layer, after two process cycles, was about 3 mm.

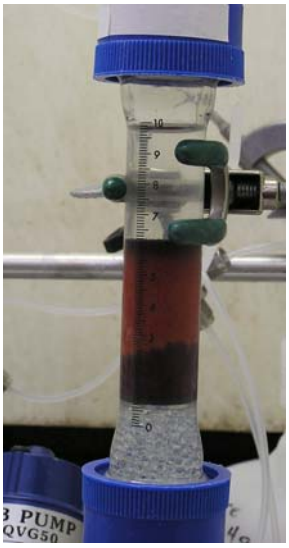
(a) (b) 

Figure 11.4. TI394-61 (a) and TI394-62 (b) Columns 2nd Cycle Elution

The Cs loading and elution profiles for each resin test are shown in Figure 11.5 through Figure 11.8. The change in flowrate from 1.5 BV/h to 3.0 BV/h was associated with a slight change in the linearity of the load profile on the probability plot (at ~80 BVs). The Cs breakthrough onset occurred at ~35 BVs for TI394-61 resin and at about 30 BVs for the TI394-62 resin. The 50% breakthrough points were similar for the two resins at about 140 BVs. The slight deviation from this value was expected to be within the uncertainty of the process. A summary of the Cs loading characteristics is provided in Table 11.8.

Table 11.8. Cs Load Summary for Wave 3 Resins

Process Parameter	TI394-61 5E-370/639		TI394-62 5E-370/641	
	Cycle 1	Cycle 2	Cycle 1	Cycle 2
Contract limit, BV	67	64	57	57
50% breakthrough, BV	139	139	135	137
Net mg Cs loaded	13.3	13.4	13.3	13.4
mg Cs/mL resin	0.76	0.76	0.74	0.75
mg Cs/g resin	2.57	2.59	2.50	2.51

In each case, the second loading profile resulted in Cs bleed into the effluent, evidenced by the raised baseline $\%C/C_o$ through the first 30 BV processed. The Wave 3 test bleed was ~3.5× higher than observed with previous tests. Wave 1 Cycle 2 processing with BRF-14 resulted in ~8E-3% C/C_o, and Wave 2 Cycle 2 processing with TI394-12 resulted in ~7 E-3% C/C_o. The higher Cs bleed during the Wave 3 processing was associated with lower eluate acid concentrations and volumes, which in turn left higher residual Cs on the resin bed. The Wave 1 testing used 192 mmoles H^+ during elution (21 BVs of 0.5 M HNO_3) whereas the Wave 3 elution processing used 84 mmoles (19 BVs of 0.25 M HNO_3) and 113 mmoles (16 BVs of 0.4 M HNO_3) H^+. Increasing the elution volumes of the lower acid strength eluates should decrease the residual Cs on the resin.

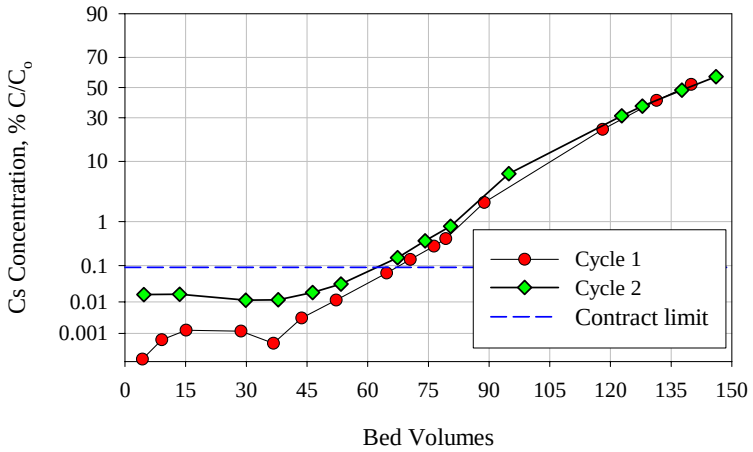


Figure 11.5. TI394-61 (5E-370/639) Loading Profiles with AP-101 Simulant

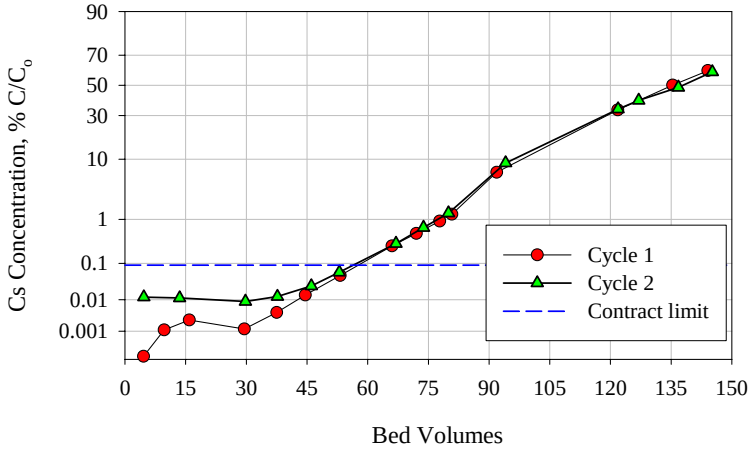


Figure 11.6. TI394-62 (5E-370/641) Loading Profiles with AP-101 Simulant

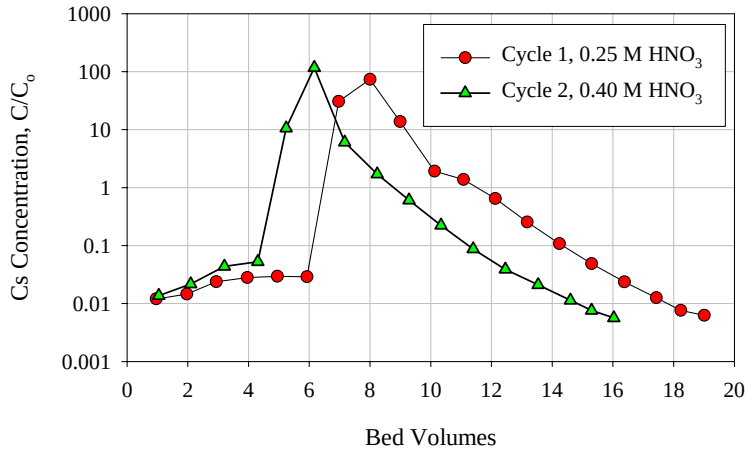


Figure 11.7. TI394-61 (5E-370/639) Elution, Different Acid Concentrations

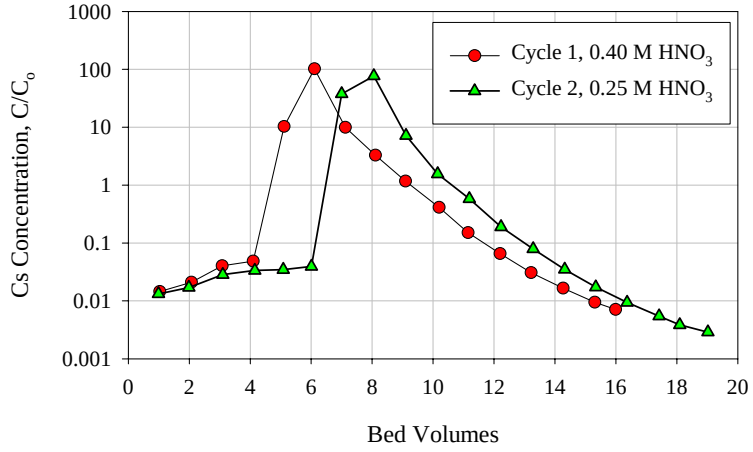


Figure 11.8. TI394-62 (5E-370/641) Elution, Different Acid Concentrations

The lower acid strength affected the elution profile shapes by delaying and broadening the Cs elution peak. Despite these changes, the elution peaks were relatively sharp. The final Cs remaining on the resin bed after processing 19 BVs 0.25 M HNO_3 was similar to the amount remaining after processing with the 0.4 M HNO_3 elution of 16 BVs. Using either of these eluates appears to be a viable alternative in the WTP. Salient comparison parameters between the average results from 0.25, 0.4, and 0.5M HNO_3 elution processing are provided in Table 11.9. The lower acid strengths and volumes left about twice the residual Cs on the RF resin.

Table 11.9. Elution Performance Comparison as a Function of Eluate Concentration

HNO_3 M	BV Processed	mmole H^+ per mL Resin[(a)]	Cs Peak, BV	Residual Cs, µg Cs/g resin
0.25	19	4.7	8	0.36
0.4	16	6.4	6	0.34
0.5[(b)]	21	10	5	0.17

(a) Na-form resin volume basis.
(b) The 0.5 M HNO_3 elution data was obtained from BRF-14 processing (Wave 1, Section 5.0).

Residual resin Cs concentration as a function of the elution bed volume is summarized in Figure 11.9 and Figure 11.10. The design limit Cs concentration (4.2 µg/g resin) was reached within 13 BVs.

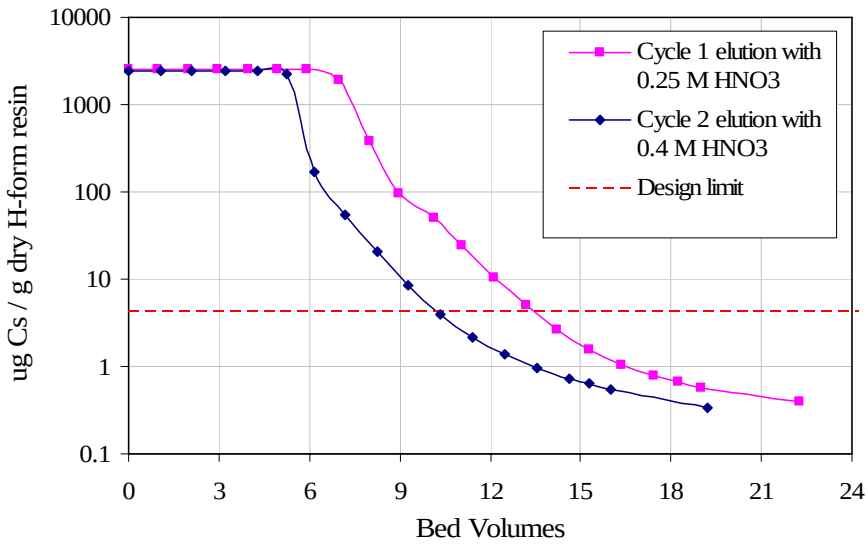


Figure 11.9. Residual Cs as a Function of Elution Volume TI394-61 (5E-370/639)

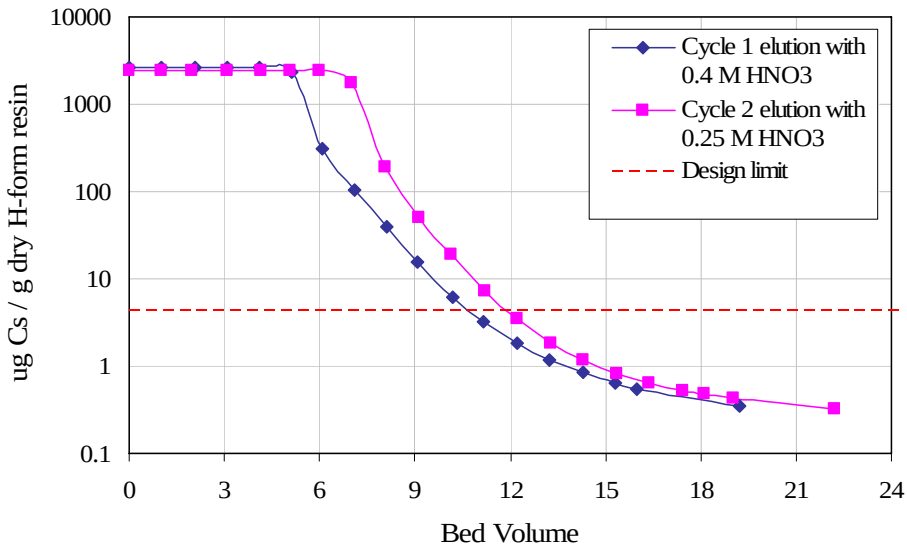


Figure 11.10. Residual Cs as a Function of Elution Volume TI394-62 (5E-370/641)

The data plotted in the preceding figures are given in Table 11.10 and Table 11.11.

Table 11.10. Effluent Cs Concentration During Loading and Elution, TI394-61 (5E-370/639)

Cycle 1					Cycle 2				
Feed		Elution with 0.25 M HNO_3			Feed		Elution with 0.4 M HNO_3		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc. [a]
4.3	1.22E-4	0.96	1.20E-2	2.56E+3	4.7	1.63E-2	1.05	1.37E-2	2.44E+3
9.1	<5.99E-4	1.97	1.45E-2	2.56E+3	13.5	1.67E-2	2.10	2.17E-2	2.44E+3
15.1	1.26E-3	2.94	2.37E-2	2.56E+3	29.9	1.13E-2	3.21	4.35E-2	2.44E+3
28.7	<1.18E-3	3.96	2.81E-2	2.56E+3	37.9	1.15E-2	4.31	5.25E-2	2.43E+3
36.7	4.53E-4	4.95	2.94E-2	2.56E+3	46.4	1.88E-2	5.24[b]	1.06E+1	2.24E+3
43.7	3.15E-3	5.93[b]	2.88E-2	2.56E+3	53.4	3.28E-2	6.17	1.17E+2	1.72E+2
52.2	1.12E-2	6.97[b]	3.07E+1	1.91E+3	67.4	1.58E-1	7.17	6.03E+0	5.56E+1
64.7	6.44E-2	8.00	7.38E+1	3.74E+2	74.2	3.73E-1	8.24	1.71E+0	2.07E+1
70.5	1.42E-1	8.99	1.38E+1	9.48E+1	80.5	8.06E-1	9.29	6.08E-1	8.39E+0
76.4	2.94E-1	10.13	1.92E+0	5.06E+1	94.9	6.72E+0	10.34	2.22E-1	3.95E+0
79.3	4.33E-1	11.08	1.38E+0	2.41E+1	122.8	3.12E+1	11.40	8.69E-2	2.19E+0
88.8	2.29E+0	12.13	6.44E-1	1.03E+1	127.9	3.74E+1	12.46	3.88E-2	1.39E+0
118.1	2.34E+1	13.18	2.54E-1	4.94E+0	137.7	4.82E+1	13.54	2.09E-2	9.62E-1
131.4	4.11E+1	14.24	1.08E-1	2.62E+0	146.1	5.75E+1	14.60	1.13E-2	7.33E-1
140.0	5.21E+1	15.30	4.86E-2	1.58E+0			15.30	7.59E-3	6.31E-1
		16.38	2.35E-2	1.06E+0			16.03	5.64E-3	5.53E-1
		17.43	1.26E-2	7.94E-1			EDI[c]	3.56E-3	3.37E-1
		18.24	7.57E-3	6.71E-1					
		19.01	6.27E-3	5.72E-1					
		EDI[c]	2.55E-3	3.98E-1					

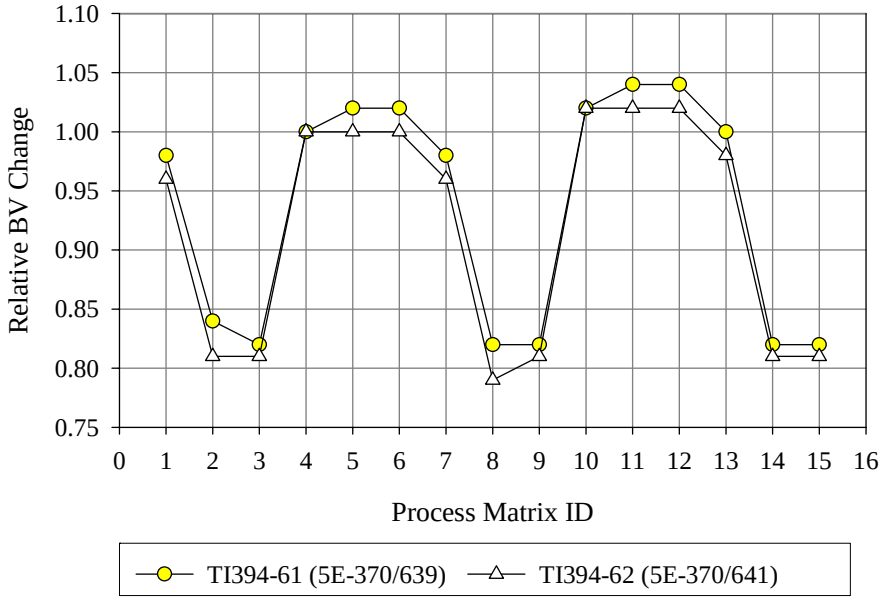
(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate observed in eluate.
(c) Post elution water rinse.

Table 11.11. Effluent Cs Concentration During Loading and Elution, TI394-62 (5E-370/641)

Cycle 1					Cycle 2				
Feed		Elution			Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc. [a]
4.6	1.28E-4	1.03	1.45E-2	2.60E+3	4.7	1.19E-2	0.99	1.32E-2	2.46E+3
9.7	<1.10E-3	2.07	2.10E-2	2.60E+3	13.5	1.13E-2	1.99	1.71E-2	2.46E+3
15.9	2.33E-3	3.08	4.03E-2	2.60E+3	29.8	8.84E-3	3.10	2.85E-2	2.46E+3
29.5	<1.19E-3	4.10	4.88E-2	2.59E+3	37.7	1.23E-2	4.15	3.36E-2	2.46E+3
37.5	4.02E-3	5.11[b]	1.03E+1	2.38E+3	46.1	2.49E-2	5.09	3.47E-2	2.46E+3
44.6	1.37E-2	6.11	1.02E+2	3.11E+2	53.0	5.94E-2	6.01	3.93E-2	2.46E+3
53.2	4.82E-2	7.12	9.94E+0	1.05E+2	67.0	3.03E-1	7.00[b]	3.77E+1	1.74E+3
66.0	2.65E-1	8.11	3.29E+0	3.89E+1	73.8	6.79E-1	8.06	7.66E+1	1.94E+2
72.0	5.09E-1	9.10	1.17E+0	1.54E+1	80.0	1.33E+0	9.11	7.18E+0	4.94E+1
77.8	9.24E-1	10.20	4.11E-1	6.17E+0	94.1	8.47E+0	10.15	1.56E+0	1.88E+1
80.8	1.26E+0	11.15	1.51E-1	3.24E+0	121.9	3.40E+1	11.19	5.80E-1	7.22E+0
91.9	6.55E+0	12.20	6.57E-2	1.83E+0	127.0	3.97E+1	12.23	1.90E-1	3.43E+0
121.8	3.34E+1	13.22	3.06E-2	1.20E+0	136.8	4.95E+1	13.29	7.89E-2	1.85E+0
135.4	5.02E+1	14.27	1.66E-2	8.41E-1	145.2	5.85E+1	14.32	3.50E-2	1.16E+0
144.1	6.00E+1	15.31	9.49E-3	6.41E-1			15.34	1.73E-2	8.22E-1
		15.99	7.10E-3	5.42E-1			16.37	9.44E-3	6.36E-1
		EDI[c]	2.98E-3	3.46E-1			17.42	5.45E-3	5.28E-1
							18.10	3.86E-3	4.77E-1
							19.02	2.88E-3	4.27E-1
							EDI[c]	1.77E-3	3.19E-1

(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate observed in eluate.
(c) Post-elution water rinse.

Figure 11.11 displays the in-column shrink-swell characteristics of the Wave 3 resins. The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing. The relative BV shown is the BV observed divided by the BV in 0.5 M NaOH. Both resins displayed ~20% volume contraction from Na-form to H-form. The second process cycle for both resins showed a slight Na-form volume creep. The measured resin volumes and dry H-form resin masses are provided in Table 11.12.



Matrix IDs

1: Loaded in column (Na-form)
2: 0.5M HNO_3
3: DI water rinse
4, 10: 0.5 M NaOH
5, 11: AP101 simulant
6, 12: 0.1 M NaOH feed displacement
7, 13: DI water
8, 14: 0.25 M or 0.4 M HNO_3
9, 15: DI water

Figure 11.11. Wave 3 Resins Shrink-Swell Characteristics

Table 11.12. Actual Resin Volumes as a Function of Feed Matrix, Wave 3

Feed matrix	Matrix ID	TI394-61 5E-370/639 mL	TI394-62 5E-370/641 mL
1 M NaOH soak/DI rinse	1	17.3	17.3
0.5 M HNO_3	2	14.8	14.5
DI water	3	14.5	14.5
0.5 M NaOH	4	17.6[(a)]	17.9[(a)]
Feed, AP101 simulant	5	17.9	17.9
0.1 M NaOH	6	17.9	17.9
DI water	7	17.3	17.3
0.5 M HNO_3	8	14.5	14.1
DI water	9	14.5	14.5
0.5 M NaOH	10	17.9	18.2
Feed, AP101 simulant	11	18.2	18.2
0.1 M NaOH	12	18.2	18.2
DI water	13	17.6	17.6
0.5 M HNO_3	14	14.5	14.5
DI water	15	14.5	14.5
Dry resin mass (H-form)	na	5.16 g	5.32 g

(a) Reference volume, defined as 1-BV.

11.3 Eluate Analysis

The TI394-62 0.4 M HNO_3 eluate was selected for additional characterization according to TI-RPP-WTP-432.[(a)] The individual sample pH was determined using narrow-range pH paper. Pro-rated volumes of the individual samples were combined to prepare a representative composite sample. An aliquot of the composite was submitted to the ASO for metals analysis, ion analysis (by ion chromatography), and total carbon analysis according to ASR 7489.

The eluate sample pH as a function of BV is plotted in Figure 11.12. The effluent pH changed from basic to acidic between the fifth and sixth BV processed, coincident with the Cs elution. Precipitate was observed in the fifth BV eluate sample and was probably associated with aluminates that were insoluble at pH 11.5 (most probably gibbsite).

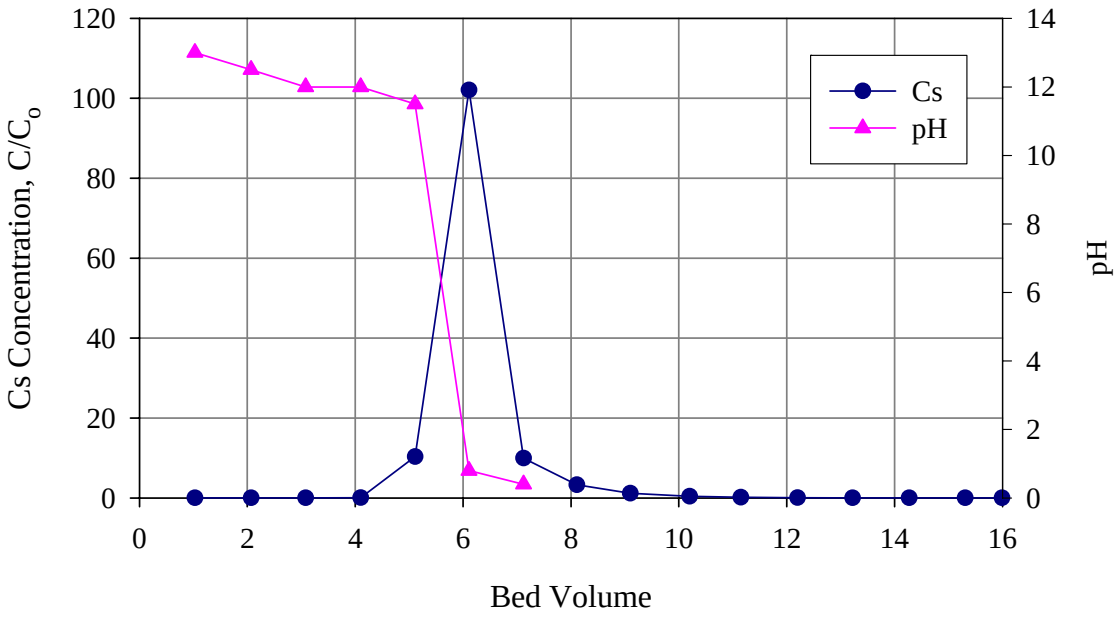


Figure 11.12. Eluate Sample pH as a Function of Process BV (0.4 M HNO_3 Eluant)

Apparatus volumes:

Process Location	mL	BV
Na-form resin bed	17.9	1
Feed bottle to top of column	8.7	0.49
Fluid above Na-form resin bed	13.5	0.75
Fluid in Na-form resin bead and resin bed interstices	15	0.84
Fluid below resin bed to exit line	9.7	0.54

The metal, carbon, and anion analytical results are summarized in Table 11.13. The total metal analyte masses, fractional recoveries, and milliequivalents (meq) recovered are also provided.

(a) SK Fiskum, October 2005. *Eluate pH Testing and Compositing for Sample Submission Supporting A-225 AP-101 Simulant Cesium Ion Exchange Processing on Wave 3 Test.*

Major cations eluted from the resin were (in order of decreasing mass) Na, K, Cs, Pb, and Al. Nominally 31.7 total meq were recovered in the eluate, which corresponded to 1.77 meq/mL Na-form resin and 5.96 meq/g H-form resin. The K meq represented 28% of the total recovered in the eluate (relative to 7% of the meq fraction in the feed); the Na meq essentially made up the balance (72%) in the eluate.

Table 11.13. Analytical Results from Composite Eluate

	Concentration		Recovery		
Analyte	µg/mL[(a)]	M	Mass, µg[(a)]	Fraction, %[(a,b,c)]	Equivalents, meq
Al	15.9	5.89E-4	4,550	0.0267	0.506
Ba	0.284	2.07E-6	81	9.76	0.0012
Ca	2.82	7.04E-5	807	>6.33	0.0402
Cd	0.183	1.63E-6	52	1.84	0.0009
Co	<0.012	<2.0E-7	<3.3	<0.54	--
Cr	0.452	8.69E-6	129	0.0347	0.0075
Cs[(d)]	46.5	3.50E-4	13,300	87.9	0.100
Fe	0.831	1.49E-5	238	3.44	0.0128
K	1,220	3.12E-2	349,000	0.509	8.92
Mg	[0.057]	[2.3E-6]	[16]	>[0.57]	--
Mn	[0.016]	[2.9E-7]	[4.6]	[1.7]	--
Mo	[0.029]	[3.0E-7]	[8.3]	[0.03]	--
Na	1,770	7.70E-2	506,000	0.174	22.0
Ni	[0.032]	[5.5E-7]	[9.2]	>[1.7]	--
Pb	34.6	1.67E-4	9,900	40.0	0.0955
Sn	<0.46	<3.9E-6	<130	<0.59	--
Ti	[0.013]	[2.7E-7]	[3.7]	>[1.3]	--
Zn	0.148	2.26E-6	42	0.204	0.0013
				meq sum	31.7

Analyte	µg/mL[(a)]	M
carbon[(e)]	[30]	[2.5E-3]
Chloride	[1.7]	[4.8E-5]
Nitrate	21,500[(f)]	3.47E-1
Sulfate	[36]	[3.7E-4]
phosphate	<4.0	<4.2E-5

no data

(a) Bracketed results were greater than the method detection limit (MDL) but less than the EQL. The less-than values (<) indicate that the results were less than the MDL; reported values are the instrument detection limits which in this case equal the MDL since sample dilution was not necessary. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

(b) The percent mass recovery is relative to the analyte mass in the processed feed.

(c) The "greater than" (>) analyte recoveries indicate that the analyte was measured at greater than the MDL in the eluate but was below the detection limit in the feed.

(d) Based on the calculated Cs loading and eluate recovery.

(e) Total carbon (µg C/mL). Carbonate cannot exist in the acidic solution; therefore, all carbon must be from organic sources.

(f) The nitrate concentration was calculated to be 0.347 M; the theoretical eluate nitrate composite concentration (inclusive of the apparatus volume of displaced water) was 0.334 M.

Notes: Results from ASR 7489.

Lead (Pb) recovery in the eluate may be associated with the specific complexant composition in the waste feed. The Pb recovery in the eluate from processing simulant AP-101 was 40%. The Pb recovery from actual AP-101 tank waste testing was ~26% (Fiskum 2006a) and from actual AN-102 testing was ~10% (Fiskum 2006b). The simulant contained oxalate, formate, and acetate; however, the actual waste AP-101 contained ~25% additional organic materials (total organic carbon mass basis) that were not identified (Russell et al. 2003). The AN-102 is a known high-complexant waste. Thus, there appears to be a relationship such that higher complexant concentrations result in lower Pb retention on the resin.

12.0 Wave 3a Testing

The Wave 3a testing was conducted on Microbeads Lot 5E-370/641 spherical RF previously tested in a 16-cycle load and elute process (Adamson et al. 2006).[(a)] The purpose of testing the cycled resin was to compare Cs loading characteristics with those of the fresh resin and thus estimate chemical degradation effects. Embedded in the Wave 3a testing were two additional tests: evaluation of the stop-flow effect on the Cs loading curve, and effectiveness of upflow elution.

A 2-L quantity of the 16-cycle exposed H-form resin, representative of the entire resin bed, was sub-sampled and shipped to PNWD. The resin was received at PNWD on 11/3/05 and given the PNWD ID TI-394-63. The resin sub-sample removed for Wave 3a testing was not pretreated before select physical property testing was conducted. Adamson et al. (2006) calculated that the dissolved oxygen consumption by the original 58.9 kg RF resin bed (assuming a bulk density of 0.30 kg/L) was 0.50 to 0.63 mmoles per g H-form resin.

12.1 Physical Properties

Physical properties were only evaluated on the H-form resin. Micrographs of the H-form resin are shown in Figure 12.1. The resin appeared dark mottled brown, as-received, in the H-form. In contrast, the "virgin" (subjected only to one pre-treatment cycle in the open beaker format) H-form resin appeared burnt orange (see Wave 3 test results). The dark brown color was attributed to the oxygen and chemical exposure during SRNL testing. Micrographs also showed that the 16-cycle resin was darker maroon than the virgin material (shown in Figure 12.2). A small population of small, black resin beads was observed. Otherwise, the particle morphology (shape and size) appeared similar to the virgin material.

(a) A large quantity of the resin was shipped from Norway directly to SRNL for testing. During shipping, the resin was exposed to a maximum temperature of 89.0°F and a minimum temperature of 64.8°F. The resin was received at SRNL on 7/22/05 and stored for 2 months at ~70°F. The resin was removed on 9/16/05 and pretreated by soaking in 0.5 M NaOH solution. The pretreated resin was loaded into a 24-in. ion exchange column on 9/19/05 and then subjected to 16 complete loading and elution cycles. Air was sparged into the simplified AP-101 simulant during each loading cycle. In total, the resin bed had been exposed to approximately 62,000 gallons (1220 BVs) of oxygenated simulant. The resin was removed from the column on 10/27/05 (private communication from D. Adamson, 2006).

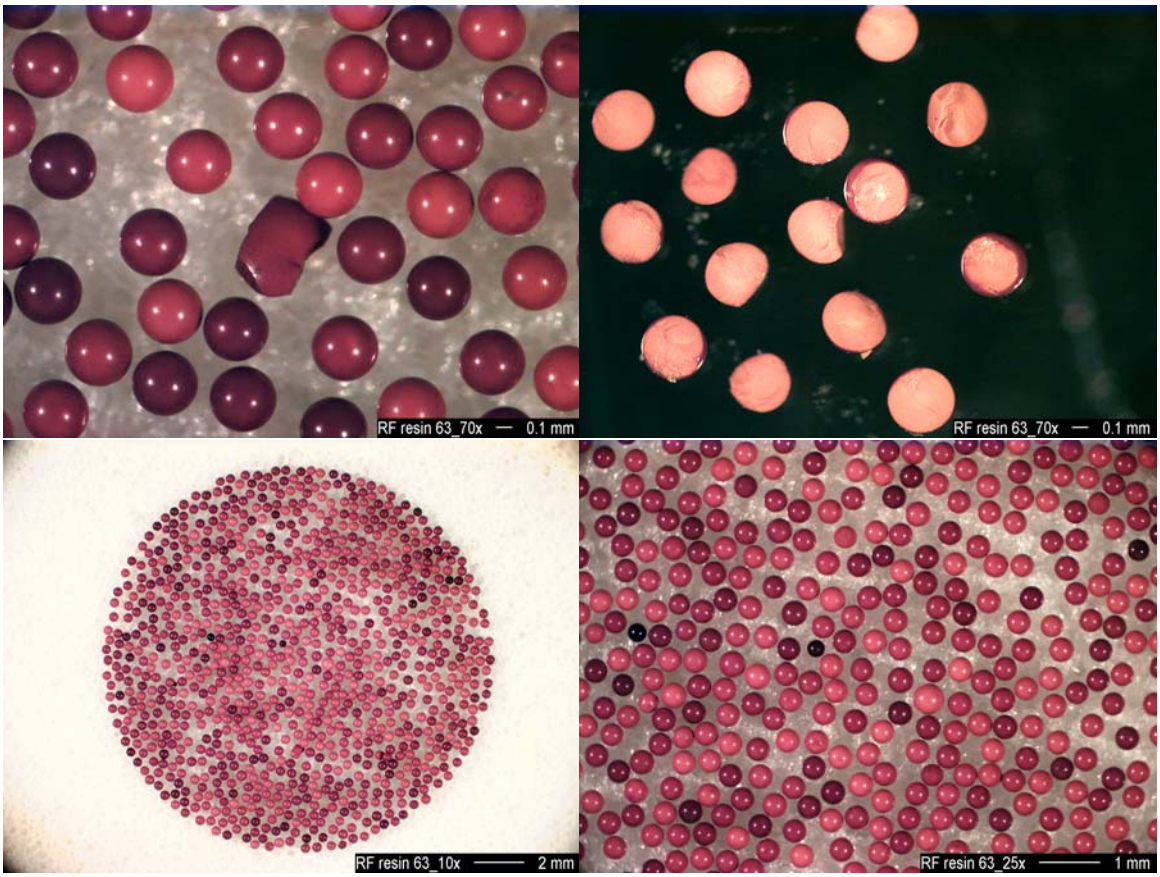


Figure 12.1. Micrographs of TI394-63 (Microbeads 5E-370/641 after 16-cycles at SRNL). Clockwise from top left: 70×, Cross-Section 70×, 25×, and 10×.

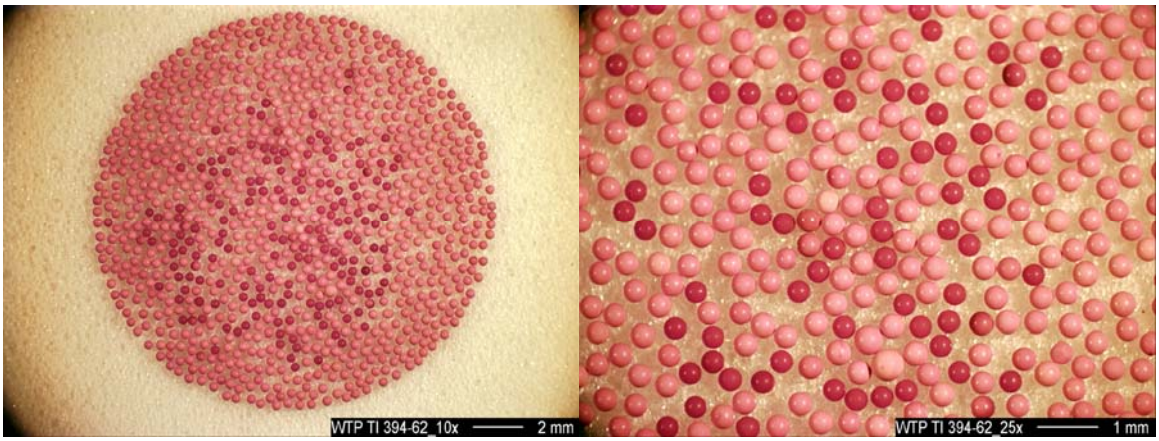


Figure 12.2. Micrographs of TI394-62 (Virgin Microbeads 5E-370/641): 10× and 25×

The H-form resin PSD results are shown in Table 12.1. Both the virgin and 16-cycle resin are compared. The PSDs were in excellent agreement, indicating minimal size change with multiple processing.

Table 12.1. Wave 3a Particle-Size-Distribution Summary

	Volume Distribution (microns)				Number Distribution (microns)				Area Dist. (microns)
Resin ID	m_v	sd	Low 5%[a]	High 90%[b]	m_n	sd	Low 5%[a]	High 90%[b]	m_a
Virgin[c] TI394-62 H-form	421	78	307	538	377	59	288	457	403
Cycled TI394-63 H-form	423	76	312	533	384	61	292	467	408

(a) 5% of the particles are less than the given diameter.
(b) 10% of the particles are greater than the given diameter.
(c) Results from "virgin" material copied from Wave 3 testing.

The calculated H-form dry-bed density determined after the in-column pretreatment shrink-swell testing agreed well with the unconstrained bed density testing of 0.37 g/mL. This value was in agreement with the density of the virgin resin. The H-form skeletal density of the 16-cycled resin (1.476 g/mL ±0.25%) agreed well with the virgin resin (1.477 g/mL ± 0.09%).[a]

12.2 Column Testing

Column testing was conducted similarly to the previous test waves with two variations studied in two separate column tests: upflow elution and stopflow during loading. Out-of-column resin pretreatment was limited to the soak in 10:1 liquid-to-volume ratio of 1 M NaOH. In-column pretreatment was conducted as per the normal process, with a water rinse, a 0.5 M HNO_3 wash, and another water rinse. Because of the dark-brown H-form resin color, the Na-form conversion front was difficult to discern in photographs.

The yellow column test was used to evaluate upflow elution. Column test parameters are summarized in Table 12.2. After the water rinse following feed displacement, the process solution flow was adjusted to the upflow condition simply by switching the quick-disconnects before and after the column. During upflow elution, 0.66 BV (13 mL) were required to reach the bottom of the Na-form bed, 0.78 BV (16 mL) resided in the bed, and 1.3 BV (26 mL) resided between the top of the resin bed and sampling point. The upflow processing caused the ~9-mL air gap in the headspace to close, flushing the air gap through the effluent line. The conversion front from Na-form to H-form started out slightly ragged with a leading-edge variation of about 0.2 cm across the front. As the elution proceeded, the raggedness increased to long finger-like projections with a 1.8-cm length (30% of the resin bed height) along the conversion front. Photographs of the downflow regeneration and upflow elution are provided in Figure 12.3. The contrast was digitally altered to better visualize the conversion fronts. The lighter colored resin is in the H-form.

(a) The uncertainty represents the RPD of duplicate samples.

Table 12.2. Experimental Conditions for TI394-63 (Microbeads Lot 5E-370/641, 17th Cycle, Yellow Column) with Upflow Elution

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (1/5/06)								
Water rinse	DI water	7.51	3.26	153	2.62	0.891	2.87	22
Acid wash	0.5 M HNO_3	7.34	3.19	150	2.72	0.925	2.70	22
Water rinse	DI water	2.97	1.29	60.7	1.25	0.424	2.38	22
Cycle 1 (Start 1/9/06)								
Regeneration	0.5 M NaOH	5.96	2.59	122	2.61	0.889	2.28	21
Loading column	AP-101 Simulant	78.9	NA	1611	1.46	0.497	53.6	21 - 22
Loading column	AP-101 Simulant	56.1	NA	1146	3.13	1.06	18.1	21 - 22
Feed displacement	0.1 M NaOH	2.83	1.23	57.7	3.03	1.03	0.93	22
Rinse	DI water	2.91	1.26	59.3	3.01	1.02	0.97	22
Elution (upflow)	0.5 M HNO_3	15.0	5.3[(c)]	296	1.40	0.477	10.7	22
Rinse (upflow)	DI water	2.89	1.1[(c)]	58.9	1.41	0.479	2.05	22

(a) BV = bed volume (20.4 mL in Na form)
(b) AV = apparatus volume (47 mL)
(c) The apparatus volume (sum of fluid-filled parts) increased ~9 mL because the fluid head above the resin bed increased from upflow processing.
NA = not applicable
All processing was downflow except elution and follow-on water rinse, which were conducted upflow.

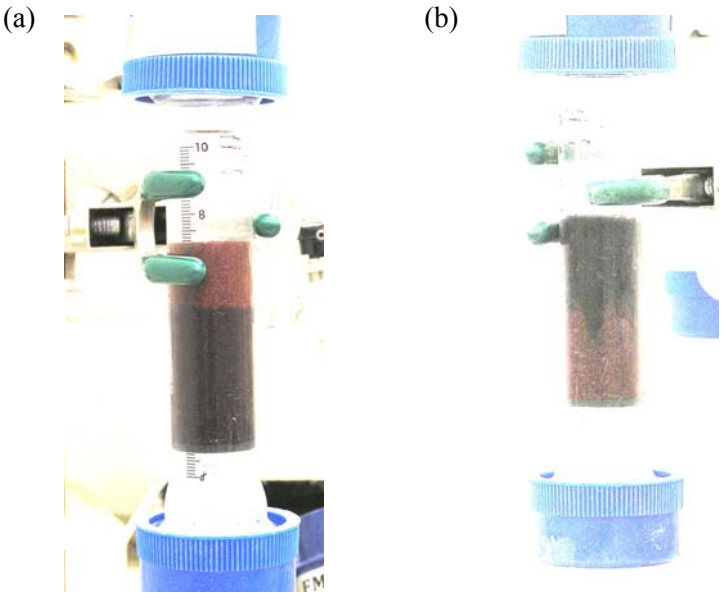


Figure 12.3. TI394-63 (a) Downflow Regeneration (Sharp Conversion Front) and (b) Upflow Elution (Showing Fingering)

The water rinse following elution was also conducted upflow. The fluid above the resin bed (22.7 mL) was removed, and an aliquot was prepared for gamma counting. The ^{137}Cs activity in the fluid volume represented a C/C_o of 2.37E-3. The resin bed was then prepared for gamma counting without the fluid head.

The white column tested the stop-flow condition during the simulant loading step to evaluate particle diffusion; column test parameters are summarized in Table 12.3. The flow was stopped after processing ~79 BVs of AP-101 simulant. The AP-101 simulant remained static in the resin bed for 18 hours. The flow resumed at the same process rate of 1.5 BV/h. Samples were collected in ~0.5-BV increments for the first 3 BVs after stopflow, and then sampling resumed at 5- to 10-BV increments. After the post-loading water rinse, the column was left static over the weekend. No adverse effects on elution characteristics were anticipated by this pause in processing.

Table 12.3. Experimental Conditions for TI394-63 (Microbeads Lot 5E-370/641, 17th Cycle, White Column) with Stop-Flow Loading Condition

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (1/5/06)								
Water rinse	DI water	7.66	3.33	156	2.67	0.910	2.87	22
Acid wash	0.5 M HNO_3	7.36	3.20	150	2.71	0.921	2.72	22
Water rinse	DI water	2.98	1.29	60.8	1.25	0.425	2.38	22
Cycle 1 (Start 1/9/06)								
Regeneration	0.5 M NaOH	5.91	2.57	121	2.59	0.881	2.28	21
Loading column	AP-101 Simulant	79.4	NA	1621	1.46	0.497	54.0	21 - 22
Stop-flow	AP-101 Simulant	--	--	--	--	--	18.0	21 - 22
Loading column	AP-101 Simulant	46.9	NA	959	1.48	0.504	31.7	21 - 22
Feed displacement	0.1 M NaOH	2.8	1.22	57.1	2.89	0.985	0.97	22
Rinse	DI water	2.84	1.23	58.0	2.79	0.951	1.02	22
Stop-flow	DI water	--	--	--	--	--	59.1	21 - 22
Elution	0.5 M HNO_3	15.0	6.51	306	1.40	0.477	10.7	21 - 22
Rinse	DI water	2.85	1.24	58.2	1.40	0.477	2.03	22

(a) BV = bed volume (20.4 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

The Cs loading profile for each resin test is shown in Figure 12.4. The white and yellow column loading profiles duplicated well to 80 BVs. The higher flowrate resulted in a bowing of the loading profile; however, 50% breakthrough was reached at 123 (±1) BVs in both cases, consistent with ion exchange theory (Buckingham 1967). The Cs effluent analysis results[(a)] from

(a) In this case, the Cs analyses were conducted at PNWD using ICP-MS, and all effluent sample results were less than the quantitation limit of 1 μg/L (equivalent to 0.015 % C/C_o where the feed Cs concentration was 6.70 μg/mL), and the relative uncertainties were >15%. Sample results <0.5 μg/mL (7.5E-3 %C/C_o) could not be distinguished from the blank.

the 24-in. column test, 3rd and 13th load cycles, conducted at SRNL (Adamson et al. 2006) are also shown.(a)

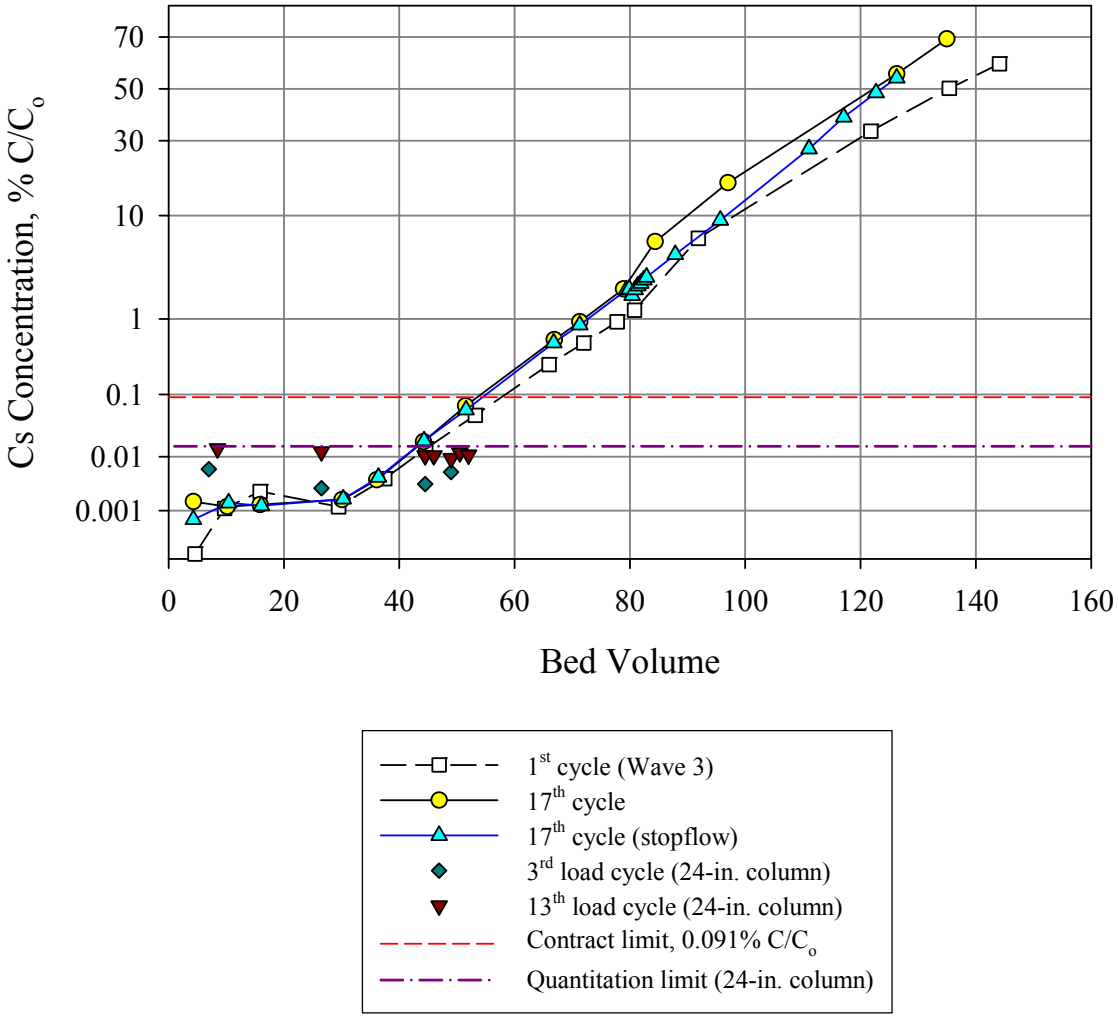


Figure 12.4. 1st and 17th Loading Cycle Profiles of TI394-62 and TI394-63 (Microbeads 5E-370/641) with AP-101 Simulant

The effluent samples, taken from the white column immediately after the stopflow condition, were slightly reduced in Cs concentration, indicating increased Cs removal by the resin. However, the loading profile curve quickly retained its original shape in less than 2 BVs with no evidence of offset or shift. This characteristic supports the concept that Cs exchange onto spherical RF at this superficial velocity (0.16 cm/min) is controlled more heavily by film diffusion rather than particle diffusion (Kurath et al. 1994).

(a) Adamson et al. (2006) defined the third load cycle as "Cycle 1" and the thirteenth load cycle as "Cycle 11." (Two load cycles had been conducted before Cycle 1.)

The resin manifested ~7% reduction in overall capacity after 16 process cycles. Both the yellow and white column loaded a total of 2.3 mg Cs/g dry H-form resin or 0.69 mg Cs/mL Na-form resin. The 1st cycle processing with this resin loaded 2.5 mg Cs/ g resin, equivalent to 0.74 mg Cs/mL Na-form resin. The measured onset of Cs breakthrough at 30 BVs was virtually identical for the 1st and 17th process cycles. The contract limit breakthrough decreased ~8% from 57 BVs to 53 BVs. The 50% breakthrough decreased ~10% from 135 BVs to 123 BVs, respectively. The Cycle 1 Cs loading profile, generated during Wave 3 testing, is shown for direct comparison in Figure 12.4.

The relative reduction in BVs processed to reach contract limit (26%) was significantly greater than the relative reduction (10%) to reach 50% breakthrough following the resin oxidation test (see Section 6.0). In contrast, the Wave 3a testing contract limit and 50% breakthrough limits were essentially equally reduced by ~9%. The relative difference in the contract-limit breakthrough responses was attributed to the distribution of the oxidized or damaged resin within the resin bed. In the former case (Section 6.0), the resin damage was localized at the top of the resin bed; in the current test, the damaged resin was equally mixed throughout the resin bed. The localized resin damage may have had the greatest effect relative to the feed residence time by shortening the active resin bed, which in turn diminished residence time. Additional testing would be required to evaluate the effect of residence time on the contract-limit breakthrough.

The small (2-cm) column data resulted in earlier contract-limit breakthrough (54 to 58 BVs) than that manifested by the 24-in. column test (>52 BVs). Because the 24-in. test process volumes were ≤52 BVs, the process BVs to reach the contract limit and 50% breakthrough could not be assessed. The quantitation limit for Cs breakthrough in the 24-in. column was at 0.015% C/C_o (1 μg/L Cs). This level of breakthrough was not exceeded in the 52 BVs processed through the 24-in. column in either the 3rd or 13th process cycles. The 0.015% C/C_o breakthrough in the 2-cm column averaged ~43 BVs, at least 9 BVs less than the last 24-in. column data point. Therefore, the contract limit breakthrough volume was most likely at least 9 BVs greater in the 24-in. column than was observed in the 2-cm column (i.e., >62 BV).

A direct comparison of selected process system variables for the 2-cm and 24-in. columns is presented in Table 12.4. The most significant difference in process conditions was related to the superficial velocity, where the 24-in column test superficial velocity was over a factor of 12 higher than that of the 2-cm test column. A second large difference is related to the length-to-diameter ratio (L/D). Increasing L/D ratios result in increasing chromatographic resolution. Therefore, under these circumstances, the decreased L/D had no measurable negative effect on the ion exchange performance. The increased superficial velocity most likely reduced the film mass transfer coefficient and, therefore, suggests that the film mass transfer coefficient significantly affects the Cs mass transfer rate at the low flow velocities (0.15 cm/min). The contract-limit breakthrough volumes in the WTP should be higher than that measured in the 2-cm column because of the higher expected superficial velocities (3.99 cm/min at a 15 gallon per minute flowrate and 53-in. column ID).

Table 12.4. Processing Parameter Comparison for the 2-cm and 24-in. Column Tests Using Microbeads 5E-370/641 RF Resin

Parameter	PNWD 2-cm Wave 3	PNWD 2-cm Wave 3a	SRNL 24-in.[(a)]
Resin bed height (Na-form), cm	5.7	6.5	72.5
L/D	2.85	2.85	1.19
Resin volume, mL	17.9	20.4	1.98E+6
Resin H-form mass, g	5.32	5.99	5.89E+4
Process temperature, C	~22	~22	~22
AP-101 Simulant	Full formulation	Full formulation	Simplified[(b)]
Cs concentration, M	4.40E-5	4.40E-5	5.0E-5
Flowrate, BV/h	1.50	1.46	1.5
Superficial velocity, cm/min	0.142	0.158	1.8
Residence time, min	40	41	40

(a) Adamson et al. 2006.
(b) The simplified AP-101 simulant contained Na, Al, K, nitrate, nitrite, carbonate, sulfate, oxalate, acetate, and Cs. Batch-contact testing with the simplified simulant resulted in similar Cs capacity measures as the full simulant formulation (personal communication CA Nash, SRNL, 2006).

The elution profiles are contrasted in Figure 12.5. The upflow elution profile clearly shows the effect of the large mixing volume above the resin bed where the Cs elution peak is spread over a large process volume. The peak Cs concentration was 32 C/C_o. The down-flow elution peak was relatively sharp with peak elution at 63 C/C_o. The downflow eluate Cs concentration quickly tailed, reaching 1% C/C_o at ~11 BVs. The upflow effluent concentration did not reach 1% C/C_o after processing 15 BVs of eluant.

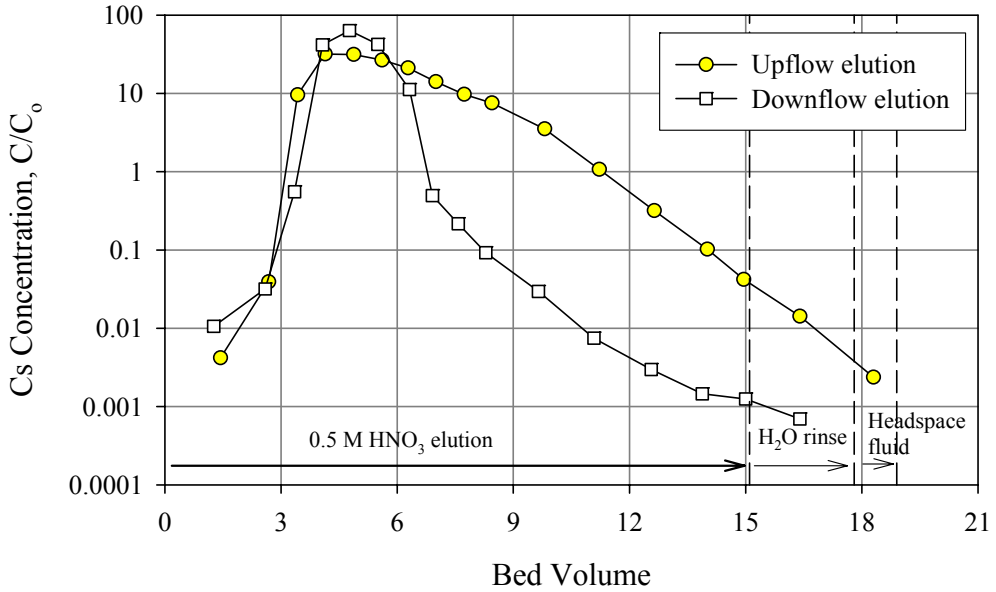


Figure 12.5. Elution of TI394-63 (Microbeads 5E-370/641), 17th-Cycle, Upflow and Downflow

Figure 12.6 shows the modeled[(a)] Cs effluent concentration based on mixing volumes in and above the resin bed. Fluid processing in the transfer lines before and after the ion exchange column is assumed to represent laminar or plug flow. The total mixing volume above the resin bed was 24 mL, and the volume of the H-form resin was 16 mL. The observed Cs elution profile in upflow condition deviated from the model in the tailing region. At 14 BVs, the experimental upflow condition resulted in 0.102 C/C_o, the model projected 0.028 C/C_o, and the downflow experimental condition resulted in 0.00146 C/C_o. Thus, at 14 BVs, the experimental upflow process condition resulted in a factor of 3.6× higher than the model prediction and a factor of 70× higher than then downflow condition.

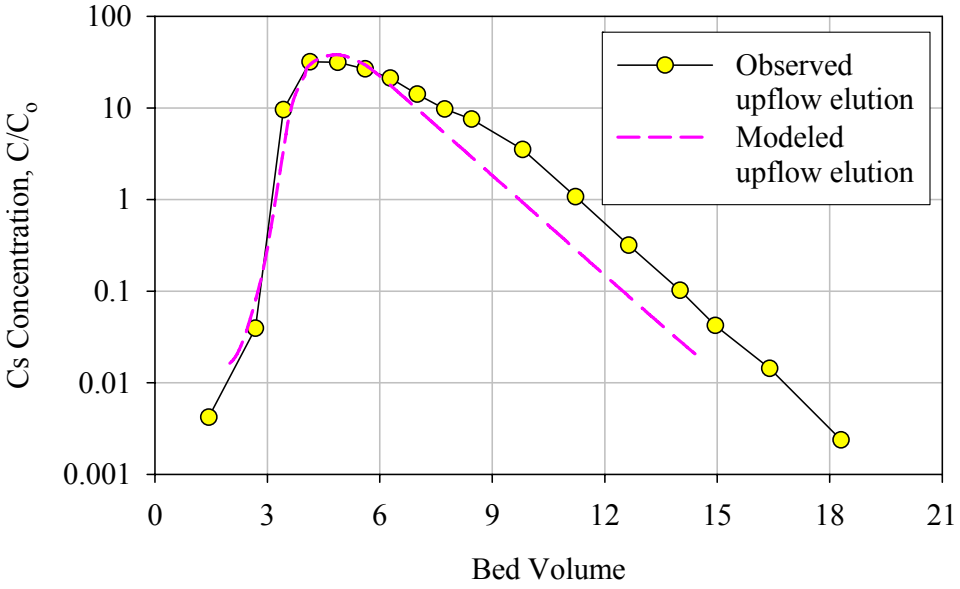


Figure 12.6. Upflow Elution Modeling

The residual resin Cs concentration as a function of eluate BV is summarized in Figure 12.7. Both elution scenarios met the design limit. The final Cs concentration on a dry H-form resin basis was 0.22 µg Cs/g resin for the upflow elution condition and 0.14 µg Cs/g resin for the downflow condition. The downflow elution condition reached 0.27 µg Cs/g resin after processing ~12 BVs eluate compared to the 18 BVs required for the upflow elution condition. The upflow elution condition resulted in 57% more residual ^{137}Cs on the resin/column system than the downflow elution condition. This result may be attributed to the apparent channeling (ragged conversion front) that was occurring during the elution process. Similar responses for upflow and downflow elution conditions were reported in previous testing with ground-gel RF (Fiskum et al. 2004).

(a) The upflow modeling data were provided by the R&T lead, MR Thorson.

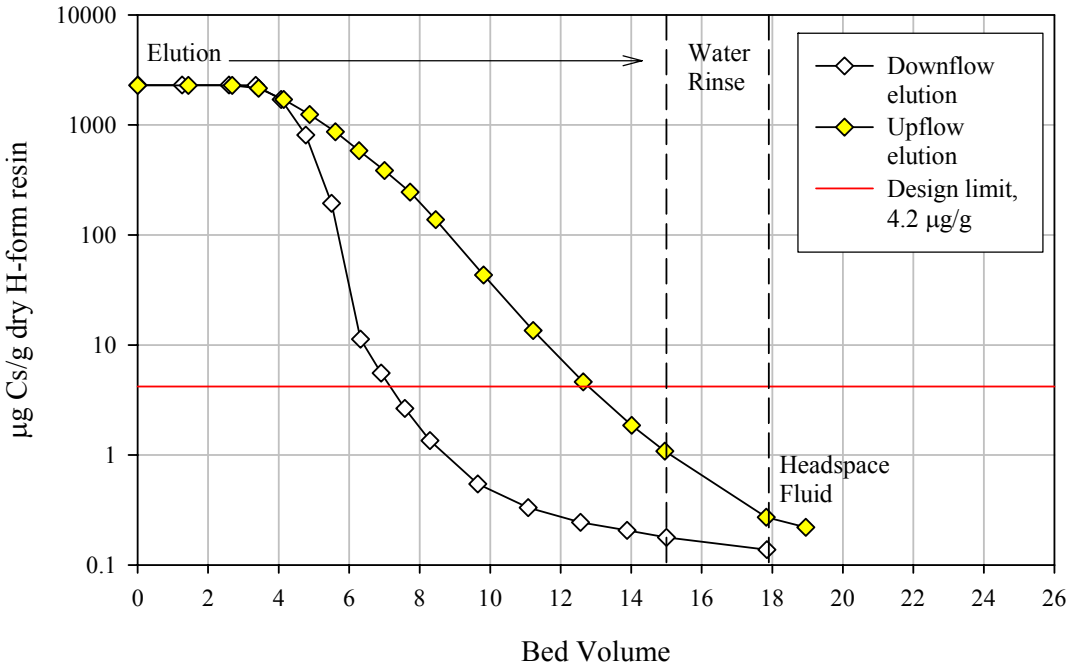


Figure 12.7. Residual Cs on Resin as a Function of Elution Volume TI394-63 (Microbeads 5E-370/641) 17th Process Cycle

The data plotted in the preceding figures are given in Table 12.5 and Table 12.6.

Table 12.5. Cs Effluent Concentration from 24-in. Column Tests, Cycle 3 and 13

Cycle 3			Cycle 13		
Cum. BV	**µg/L**	**% C/C_o**	**Cum. BV**	**µg/L**	**% C/C_o**
7	[0.40]	[0.0060]	8.5	[0.91]	[0.014]
26.5	[0.18]	[0.0027]	26.5	[0.82]	[0.012]
44.5	[0.21]	[0.0032]	44.5	[0.70]	[0.010]
49	[0.35]	[0.0053]	46	[0.70]	[0.010]
			49	[0.62]	[0.009]
			50.5	[0.78]	[0.012]
			52	[0.72]	[0.011]

Bracketed results indicate that the reported value was < EQL (1 µg/L). Sample results from Cycle 3 were <0.5 µg/L (7.5E-3 % C/C_o) and could not be distinguished from the blank. Sample results from Cycle 13 were <1 and >0.5 µg/L (7.5E-3 % C/C_o) and resulted in a signal higher than the instrument blank. Actual results are shown for information-only. Analytical error was >15%.

Table 12.6. Effluent Cs Concentrations During Loading and Elution, TI394-63 (Microbeads 5E-370/641) 17th Process Cycle

Yellow Column (Upflow Elution)					White Column (Stopflow Loading)				
Feed		Elution			Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc. [a]
4.3	<1.50E-3	1.44	4.20E-3	2.29E+3	4.3	<6.66E-4	1.26	1.06E-2	2.30E+3
10.2	<1.20E-3	2.68	3.92E-2	2.29E+3	10.4	<1.41E-3	2.59	3.18E-2	2.30E+3
15.9	<1.31E-3	3.43[b]	9.55E+0	2.15E+3	16.1	<1.25E-3	3.35[b]	5.55E-1	2.29E+3
30.1	<1.63E-3	4.14	3.18E+1	1.70E+3	30.3	<1.67E-3	4.07	4.17E+1	1.70E+3
36.1	3.84E-3	4.88	3.14E+1	1.24E+3	36.4	4.29E-3	4.77	6.35E+1	8.08E+2
44.2	1.80E-2	5.61	2.66E+1	8.65E+2	44.3	1.86E-2	5.50	4.23E+1	1.94E+2
51.5	6.74E-2	6.28	2.11E+1	5.83E+2	51.6	5.85E-2	6.32	1.12E+1	1.13E+1
66.9	5.61E-1	7.00	1.41E+1	3.85E+2	66.8	5.11E-1	6.91	4.95E-1	5.54E+0
71.3	9.26E-1	7.73	9.71E+0	2.45E+2	71.3	8.48E-1	7.58	2.16E-1	2.65E+0
78.9	2.15E+0	8.45	7.54E+0	1.38E+2	79.4	2.10E+0	8.29	9.26E-2	1.35E+0
84.4	6.13E+0	9.81	3.52E+0	4.33E+1	79.9	2.15E+0	9.65	2.96E-2	5.44E-1
97.0	1.71E+1	11.22	1.07E+0	1.35E+1	80.4	1.84E+0	11.08	7.46E-3	3.32E-1
126.3	5.60E+1	12.64	3.17E-1	4.62E+0	80.9	2.11E+0	12.56	2.98E-3	2.44E-1
135.0	6.93E+1	14.01	1.02E-1	1.86E+0	81.4	2.32E+0	13.88	1.46E-3	2.06E-1
		14.95	4.21E-2	1.08E+0	81.9	2.47E+0	15.00	1.25E-3	1.78E-1
		EDI[c]	1.43E-2	2.72E-1	82.4	2.67E+0	EDI[c]	6.97E-4	1.38E-1[e]
		HS[d]	2.37E-3	2.20E-1[e]	82.9	2.84E+0			
					87.9	4.69E+0			
					95.7	9.20E+0			
					111.1	2.72E+1			
					117.1	3.87E+1			
					122.7	4.84E+1			
					126.3	5.44E+1			

(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate observed in eluate.
(c) EDI = effluent water rinse solution composite.
(d) HS = headspace fluid = 22.7 mL.
(e) Final residual Cs on the resin bed after elution and water rinse.

Residual Cs concentrations on the spherical RF resin as a function of elution volume, acid strength, and process cycle are further compared in Figure 12.8. Also included for reference in a 0.5 M HNO_3 elution condition are the results generated for Microbeads resin 5J-370/686 (discussed in Section 13.0). The effect of acid strength is evident—increasing acid concentration (from 0.25 M to 0.5 M) resulted in faster acidification of contact fluid and sooner onset of Cs elution. Once the peak Cs was removed, the Cycle 1 and 2 resins demonstrated tailing, regardless of the acid strength used. The most profound departure in elution condition lies between the 0.5 M HNO_3 eluate in the first cycle 5J-370/686 resin (discussed in Section 13.0) and the 17th process cycle of the 5E-370/641 resin. Resin formulations were similar, and the eluate acid strengths were equivalent. Previous testing with spherical RF indicated that ~20% of the Cs was held by the resin from the first process cycle despite exposure to two additional complete loading

and elution cycles (Fiskum et al. 2006c). Residual stable Cs from processing activities at SRNL may still reside with the resin after the 17th process cycle. This Cs component would not be evident based on the direct detection of the ^{137}Cs tracer by gamma spectrometry. After processing 10-BV of 0.5 M HNO_3, the difference in residual Cs approximates an order of magnitude (i.e., 5.9 versus 0.54 µg Cs/g H-form resin), which exceeds the expected potential 20% contribution from residual Cs. The large discrepancy in residual Cs concentration may be, in part, attributed to continued relaxation of the polymer associated with extensive cycling, allowing for more efficient Cs diffusion from the particle interior to the eluate.

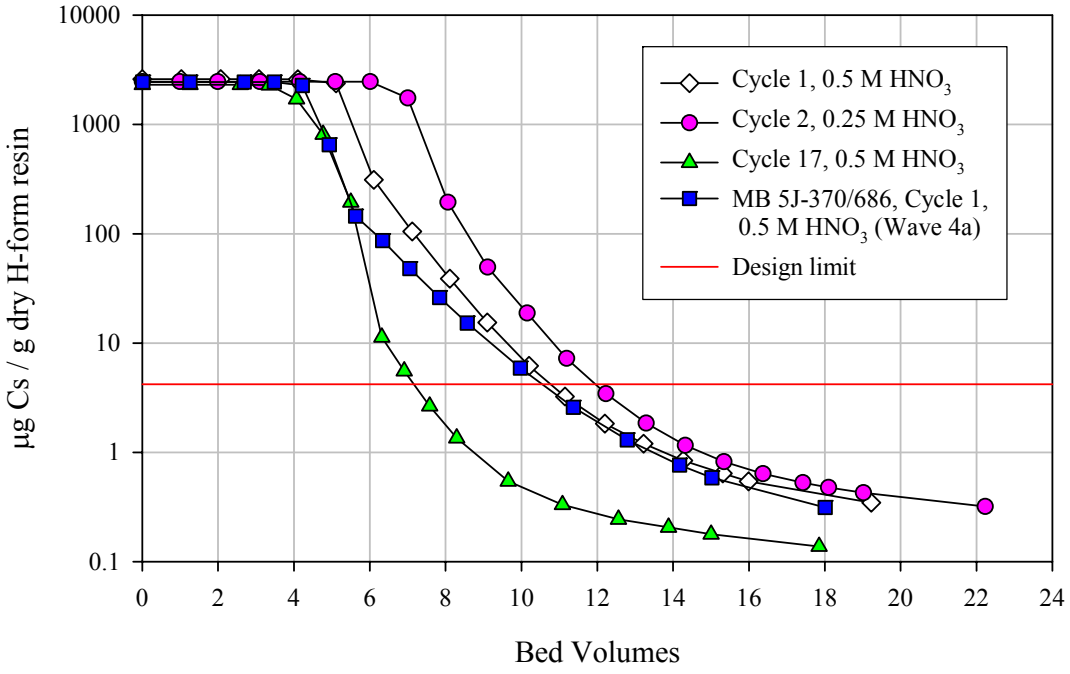
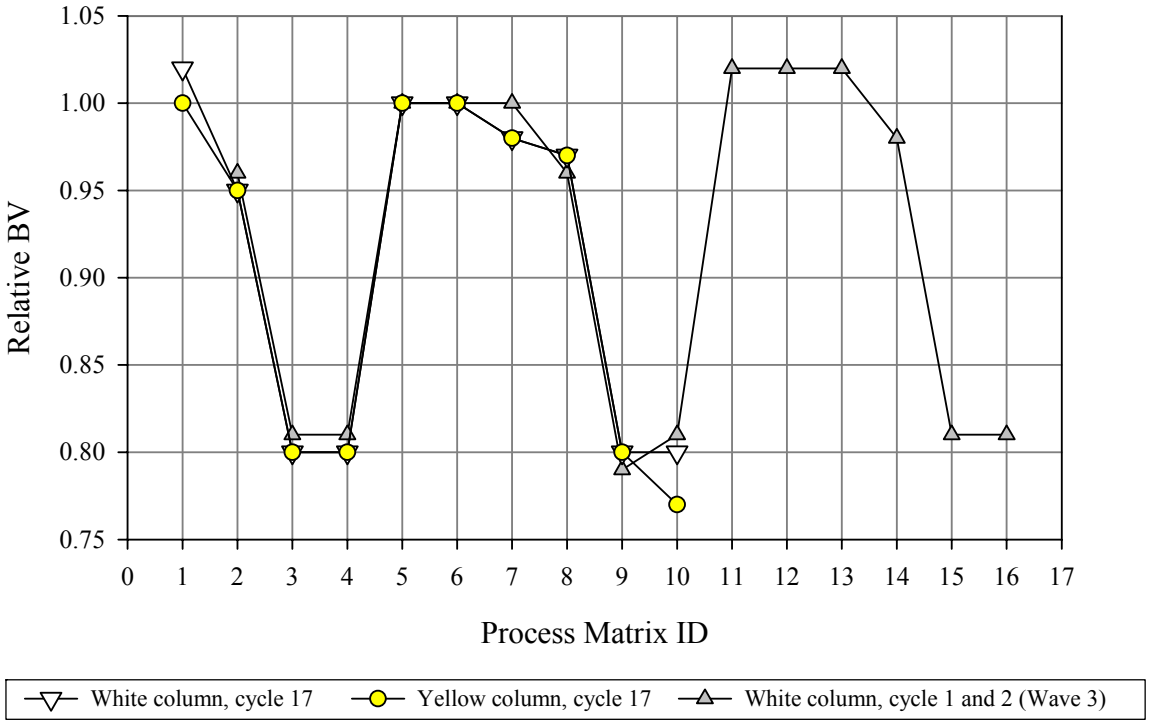


Figure 12.8. Residual Cs Loading on RF Resin as a Function of Elution Volume, Acid Strength, and Process Cycle

Figure 12.9 displays the in-column shrink-swell characteristics of the Wave 3a resin (17th process cycle) with comparison to the Wave 3 (first and second process cycles). The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing. The relative BV shown is the BV observed divided by the BV in 0.5 M NaOH. The seventeenth process cycle shrink-swell characteristics were nearly identical to those of the first and second process cycles. Each displayed ~20% volume contraction from Na-form to H-form. The measured resin volumes are provided in Table 12.7.



Matrix IDs
1: Loaded in column
2: DI water rinse
3: 0.5 M HNO_3
4: DI water rinse
5: 0.5 M NaOH
6: AP-101 simulant
7: 0.1 M NaOH
8: DI water
9: 0.5 M (0.4 M) HNO_3
10: DI water
11: 0.5 M NaOH
12: AP-101 simulant
13: 0.1 M NaOH
14: DI water
15: 0.25 M HNO_3
16: DI water

Figure 12.9. Shrink-Swell Characteristics of Resin 5E-370/641 Cycles 1, 2, and 17

Table 12.7. Actual Resin Volumes as a Function of Feed Matrix, Wave 3a

Feed matrix	Matrix ID	TI394-63 (Yellow column) mL	TI394-63 (White Column) mL
1 M NaOH load-in	1	20.4	20.7
DI rinse	2	19.5	19.5
0.5 M HNO_3	3	16.3	16.3
DI water	4	16.3	16.3
0.5 M NaOH[(a)]	5	20.4	20.4
Feed, AP101 simulant	6	20.4	20.4
0.1 M NaOH	7	20.1	20.1
DI water	8	19.8	19.8
0.5 M HNO_3	9	16.3	16.3
DI water	10	15.7	16.3
Dry resin mass (H-form)	na	5.99 g	6.02 g
(a) Reference volume.			

13.0 Wave 4a Testing

Wave 4a testing was conducted with two resins from ~70-gal[(a)] scaled-up production batches by Microbeads and BSC. Approximately 2-L samples of each batch were received at PNWD. The RF internal PNWD IDs, manufacturer, lot numbers, manufacturing dates and receipt dates are cross-referenced in Table 13.1. Testing was conducted in conjunction with Wave 3a testing.

Table 13.1. Wave 4a Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-64	Microbeads	5J-370/686	72-gal	9/05	10/21/05
TI394-65	BSC	3380-03-0200	66-gal	11/05	12/19/05

13.1 Physical Properties

The pre-treatment resin expansion data are summarized in Table 13.2. The unconstrained resin expansion was ~26% after one swell-shrink cycle pretreatment. The follow-on expansion to Na-form resin was measured at 24%. The overall expansion from the as-received H-form resin to the pretreated Na-form resin was nominally 56%.

Table 13.2. Wave 4a Resins Pretreatment Swell Data Summary

	1st Cycle Expansion			H-form to Na-form Expansion			
PNWD ID	As-received H-form, mL	Pretreated H-form, mL	Expansion Factor	H-form,[(a)] mL	Na-form, mL	Expansion Factor	Total Expansion
TI394-64	59.0	74.0	1.25	29.2	36.0	1.23	1.55
TI394-65	59.5	75.0	1.26	29.0	36.2	1.25	1.57
(a) After first pretreatment cycle.							

Micrographs of the two pretreated RF resins are shown in Figure 13.1 and Figure 13.2. Although some anomalies in resin form or size were observed, both productions were generally uniformly spherical. Broken pieces were evident in the Microbeads product. Some particle aberrations were observed in BSC product. Careful examination of several TI394-64 cross-sectioned particles shows the center section to have slightly less color-density than the outer section. This may indicate that the polymerization was not uniformly complete through the entire particle for the entire batch.

(a) The 70-gal production batch (as-prepared H-form) is equivalent to a ~108-gal volume of Na-form, pretreated resin given an expansion factor of 1.55 for the first expansion to Na-form.

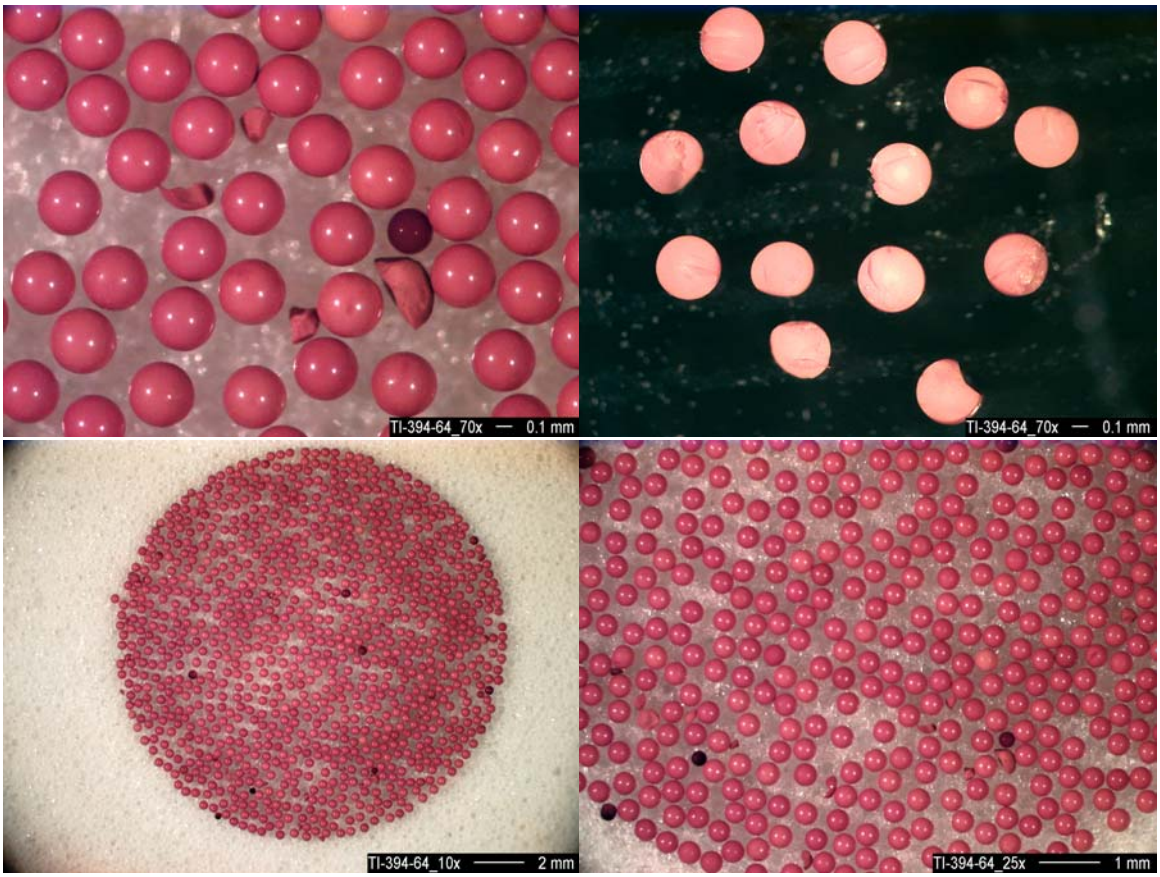


Figure 13.1. Micrographs of TI394-64 (Microbeads 5J-370/686). Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

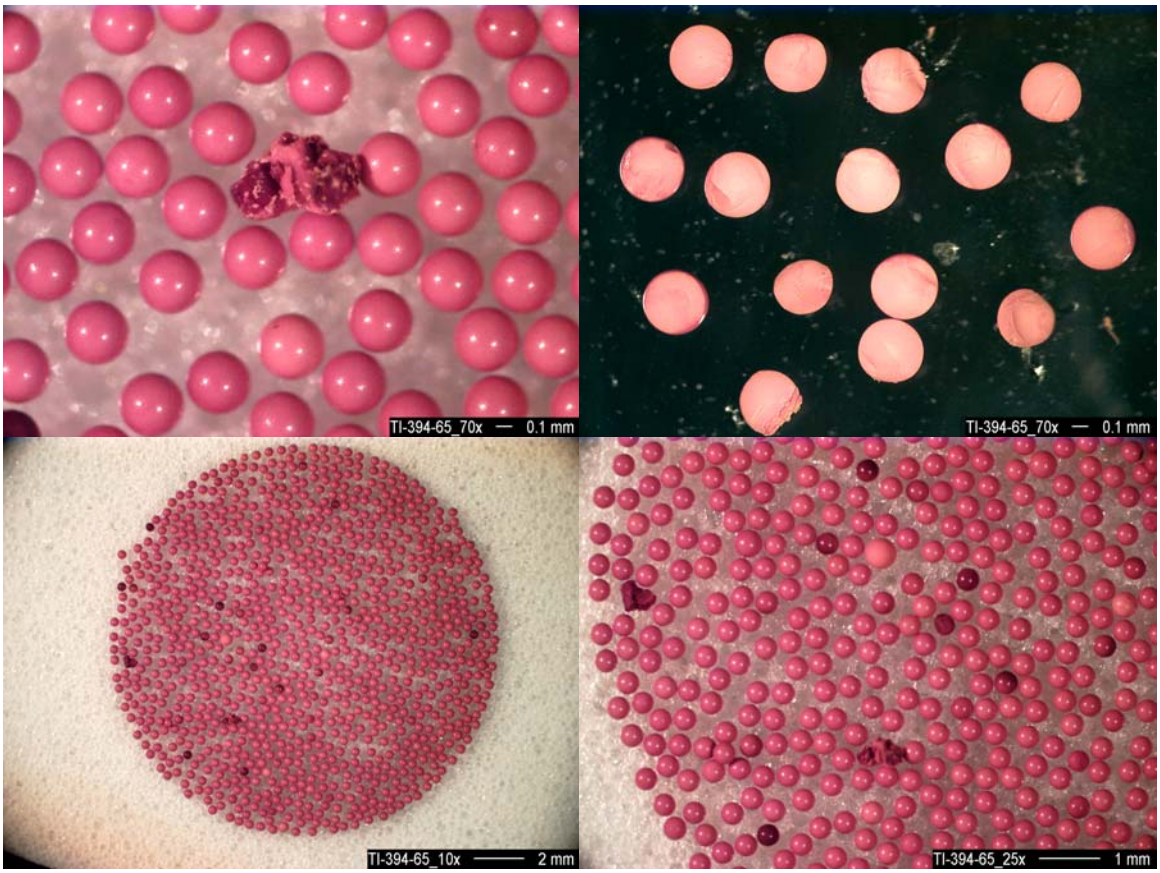


Figure 13.2 Micrographs of TI394-65 (BSC 3380-03-0200).
Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

The PSD results are shown in Figure 13.3 and Table 13.3. Each resin PSD based on volume, number, and area was in good agreement, indicating minimal fines and tight distribution in each sample. The ~27% bead volume difference from the H-form to Na-form was slightly higher than the observed resin volume expansion and contraction in the pretreatment steps and column processing. The detailed PSD results for the pretreated TI394-64 and TI394-65 resins are provided in Appendix D.

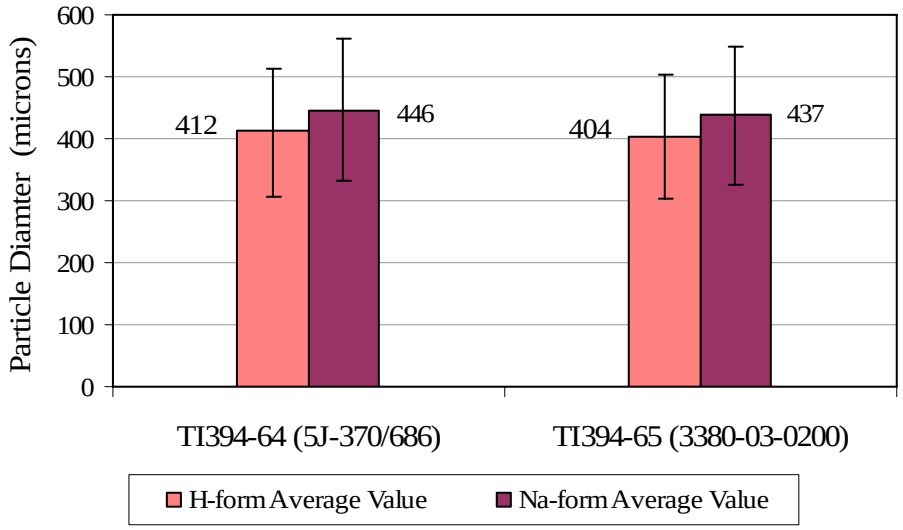


Figure 13.3. Wave 4a Resin PSD Showing Average Values and Low 5% to High 90% Spread (Volume basis)

Table 13.3. Wave 4a Resins Particle-Size-Distribution Summary

Resin ID Lot #	Resin Form	Volume Distribution (microns) m_v	sd	Low 5%[a]	High 90%[b]	Number Distribution (microns) m_n	sd	Low 5%[a]	High 90%[b]	Area Dist. (microns) m_a
TI394-64	H-form	412	72	307	514	376	58	288	454	398
5J-370/686	Na-form	446	80	331	562	405	65	306	492	431
TI394-65	H-form	404	71	302	504	369	56	283	445	391
3380-03-0200	Na-form	437	77	326	547	398	63	302	484	423
Resin ID Lot #	**Resin Form**	**Calculated Average Sphere Volume, mm^3**								
TI394-64	H-form	0.0366				0.0278				0.0331
5J-370/686	Na-form	0.0466				0.0347				0.0418
Expansion factor >		*27%*				*25%*				*26%*
TI394-65	H-form	0.0345				0.0262				0.0312
3380-03-0200	Na-form	0.0438				0.0331				0.0396
Expansion factor >		*27%*				*26%*				*27%*

(a) 5% of the particles were less than the given diameter.
(b) 10% of the particles were greater than the given diameter.

Table 13.4 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed density determined after the in-column pretreatment shrink-swell agreed well with the measured unconstrained bed density. The H-form and Na-form calculated bed densities were similar. The Microbeads product dry bed densities were typical of

previously tested resins. The BSC resin dry-bed densities for both the H-form and Na-form were slightly higher than observed for the Microbeads products.

Table 13.4. Wave 4a Resins Dry Bed Densities

Resin ID Lot #	Resin Form	Settled Vol., mL[(a)]	Dry Mass, g[(a)]	Settled Resin Density, g/mL[(b)]	Column Processing Bed Density, g/mL[(c)]
TI394-64	H-form	16.2	5.9735	0.369	0.37
5J-370/686	Na-form	24.0	8.8124	0.367	na
TI394-65	H-form	16.4	6.6914	0.408	0.41
3380-03-0200	Na-form	24.5	9.9285	0.405	na

(a) Measured for skeletal density.
(b) Dry resin mass per unit wet volume.
(c) Measured during column processing; only the dry H-form mass placed in the column was determined.
na = not applicable, Na-form mass was not determined.

The resin skeletal densities are provided in Table 13.5. Within experimental uncertainty (0.3%), the skeletal densities were consistent between the two manufacturers and with previous measurements.

Table 13.5. Wave 4a Resins Skeletal Densities

		H-form		Na-form	
Resin ID	Lot #	g/mL	RPD	g/mL	RPD
TI394-64	5J-370/686	1.468	0.02%	1.640	0.26%
TI394-65	3380-03-0200	1.471	0.03%	1.637	0.01%

13.2 Column Testing

Column testing was conducted similarly to the previous test waves. The column test parameters are summarized in Table 13.6 and Table 13.7.

Table 13.6. Experimental Conditions for TI394-64 (Microbeads Lot 5J-370/686, Green Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (1/5/06)								
Water rinse	DI water	7.51	3.21	151	2.82	0.944	2.67	22
Acid wash	0.5 M HNO_3	7.77	3.32	156	2.86	0.958	2.72	22
Water rinse	DI water	3.08	1.32	62	1.32	0.443	2.33	22
Cycle 1 (Start 1/9/06)								
Regeneration	0.5 M NaOH	6.28	2.69	126	2.79	0.936	2.25	21
Loading column	AP-101 Simulant	79.5	NA	1598	1.48	0.497	53.1	21 - 22
Loading column	AP-101 Simulant	58.7	NA	1180	2.99	1.00	19.6	21 - 22
Feed displacement	0.1 M NaOH	2.99	1.28	60.1	2.94	0.985	1.02	22
Rinse	DI water	2.90	1.24	58.3	2.95	0.989	0.983	22
Elution	0.5 M HNO_3	15.0	NA	302	1.41	0.474	10.6	22
Rinse	DI water	2.99	1.28	60.1	1.41	0.473	2.12	22

(a) BV = bed volume (20.1 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 13.7. Experimental Conditions for TI394-65 (BSC Lot 3380-03-0200, Pink Column)

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (1/5/06)								
Water rinse	DI water	7.30	3.12	147	2.92	0.978	2.50	22
Acid wash	0.5 M HNO_3	7.85	3.36	158	2.91	0.974	2.70	22
Water rinse	DI water	3.13	1.34	62.9	1.34	0.449	2.33	22
Cycle 1 (Start 1/9/06)								
Regeneration	0.5 M NaOH	6.36	2.72	128	2.83	0.948	2.25	21
Loading column	AP-101 Simulant	78.4	NA	1576	1.47	0.491	53.1	21 - 22
Loading column	AP-101 Simulant	72.0	NA	1448	2.98	1.00	24.1	21 - 22
Feed displacement	0.1 M NaOH	2.92	1.25	58.8	3.03	1.01	0.967	22
Rinse	DI water	3.01	1.29	60.5	3.01	1.01	1.00	22
Elution	0.5 M HNO_3	15.1	NA	303	1.42	0.475	10.6	22
Rinse	DI water	2.94	1.26	59.1	1.39	0.466	2.12	22

(a) BV = bed volume (20.1 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Column processing resulted in generally sharp conversion fronts (H-form to Na-form and vice versa). During preconditioning, the conversion fronts were generally level across the bed. Photographs of the green (TI394-64) and pink (TI394-65) columns during elution processing are shown in Figure 13.4. In this picture, the resin is converting from the Na-form (dark color) to the

H-form (lighter color). The conversion fronts were slightly ragged. The top surface layer on each resin bed was black. The black surface was typical during RF processing and was attributed to be oxidative attack. The final depth of the black layer was about 3 mm.

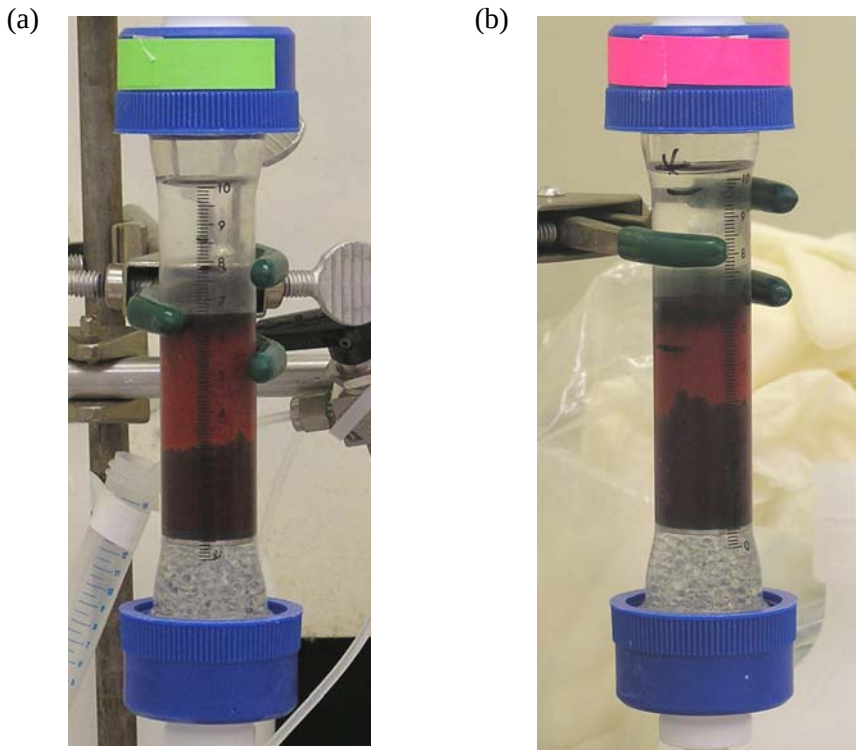


Figure 13.4. TI394-64 (a) and TI394-65 (b) During Elution Step

Specific Cs loading performance results are summarized in Table 13.8. The Cs loading profile for each resin test is shown in Figure 13.5. Based on the loading characteristics, the TI394-65 (BSC) resin appeared to perform slightly better than the TI394-64 (Microbeads) product. The difference may be related to the RF loading density into the resin bead structure. The BSC resin bed density was ~10% higher than the Microbeads resin density.

Table 13.8. Wave 4a Resins Cs Load Parameters

Parameter	TI394-64 5J-370/686	TI394-65 3380-03-0200	Difference %
Cs breakthrough onset, BV	35-40	35-40	0
Cs contract limit breakthrough, BV	58	61	5.2
50% Cs breakthrough, BV	132	146	11
Total Cs load, mg	14.5	15.7	8.3
Cs concentration, mg/g	2.4	2.3	-4.2
Cs concentration, mg/mL	0.72	0.78	8.3

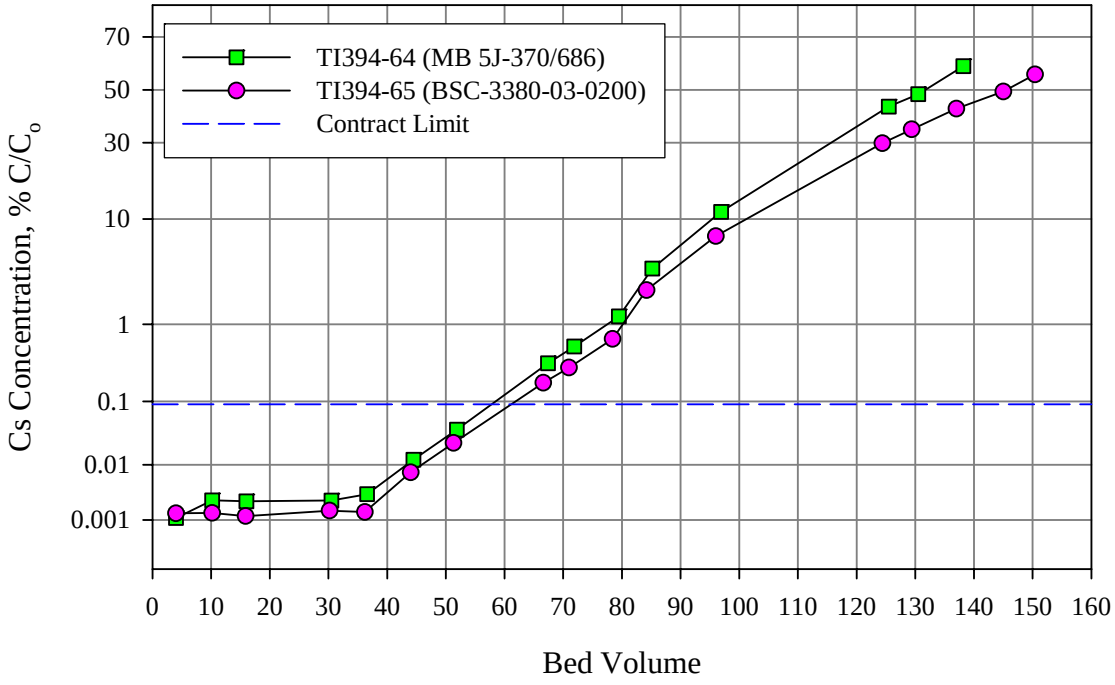


Figure 13.5. TI395-64 (Microbeads 5J-370/686) and TI394-65 (BSC 3380-03-0200) Cs Loading Profiles with AP-101 Simulant

The Cs elution profile for each resin test is shown in Figure 13.6. Both resins eluted similarly with peak C/C_o values >100 at 5 BVs. Despite the higher total Cs load on the TI394-65 resin, the eluate Cs concentration tailed slightly less (i.e., 1 BV offset) than the TI394-64 resin. The TI394-65 resin reached 1% C/C_o after processing 14 BVs eluate.

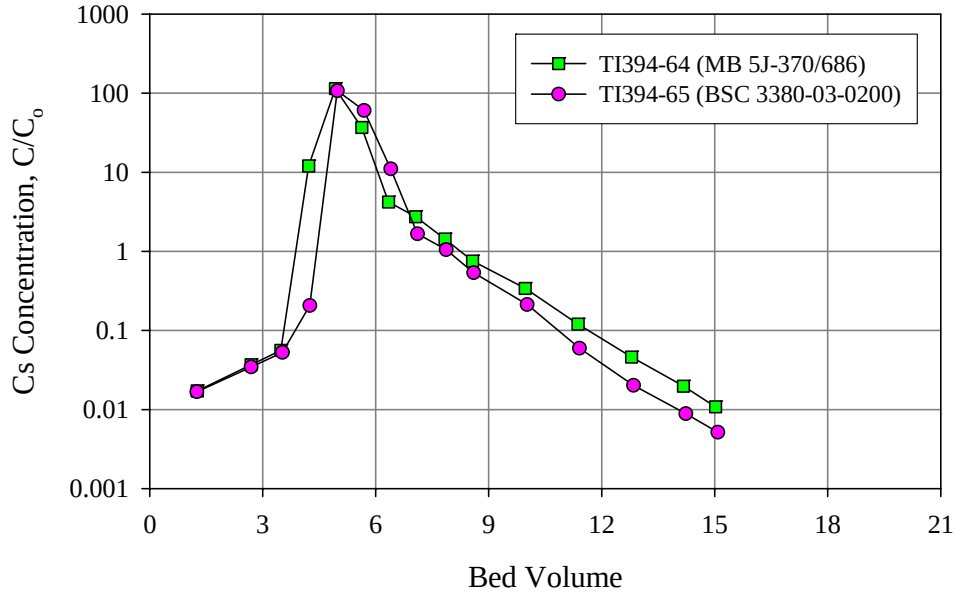


Figure 13.6. TI394-64 (5J-370/686) and TI394-65 (BSC 3380-03-0200) Elution Profiles

Residual resin Cs concentration as a function of the elution bed volume is summarized in Figure 13.7. Both resins released Cs to the design limit (4.2 μg/g) after processing between 10 and 11 BVs. In both cases, the final Cs load on the resin was 0.31 μg/g dry H-form resin.

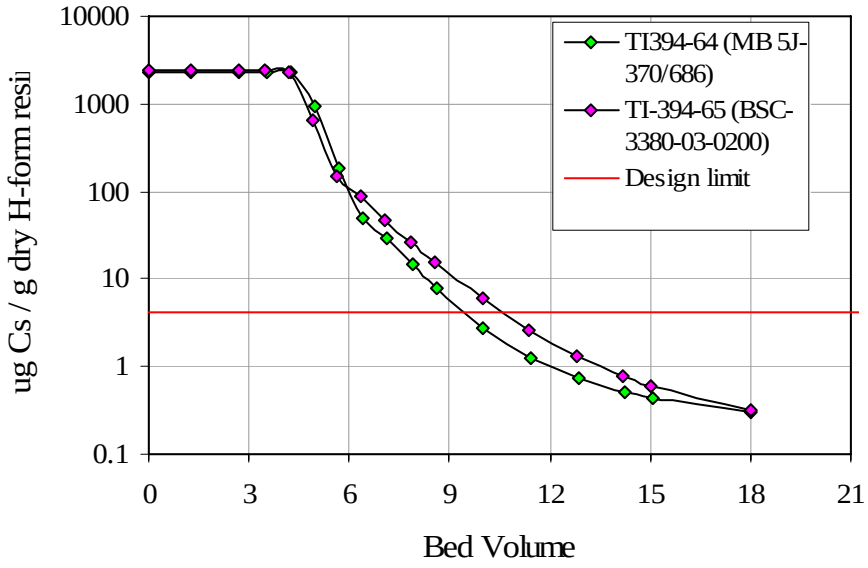


Figure 13.7. Residual Cs on Resin as a Function of Elution Volume

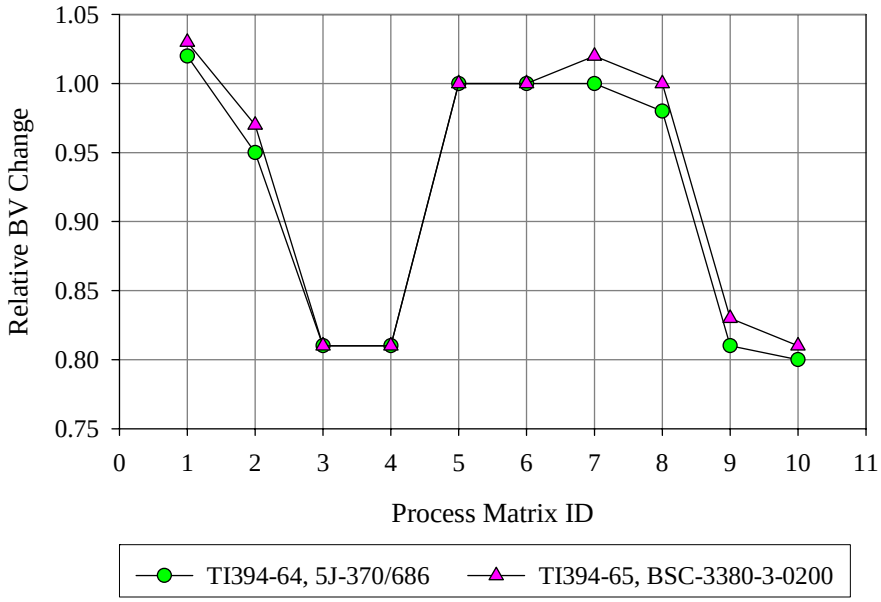
The data plotted in the preceding figures are given in Table 13.9.

Table 13.9. Wave 4a Resins Effluent Cs Concentrations During Load and Elution

TI394-64 (Microbeads 5J-370/686)					TI394-65 (BSC 3380-03-0200)				
Feed		Elution			Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc. [a]
4.0	<1.08E-3	1.26	1.72E-2	2.44E+3	4.0	<1.33E-3	1.25	1.67E-2	2.31E+3
10.2	<2.34E-3	2.69[b]	3.67E-2	2.44E+3	10.2	<1.36E-3	2.69[b]	3.44E-2	2.31E+3
16.0	<2.25E-3	3.48[b]	5.59E-2	2.44E+3	15.9	<1.18E-3	3.52	5.25E-2	2.31E+3
30.5	<2.32E-3	4.22[b]	1.20E+1	2.27E+3	30.2	<1.50E-3	4.25[b]	2.07E-1	2.31E+3
36.6	<3.03E-3	4.93	1.14E+2	6.51E+2	36.2	1.41E-3	4.98	1.07E+2	9.43E+2
44.5	1.22E-2	5.63	3.68E+1	1.45E+2	44.0	7.41E-3	5.69	6.04E+1	1.86E+2
51.9	3.75E-2	6.34	4.20E+0	8.65E+1	51.3	2.31E-2	6.40	1.10E+1	4.94E+1
67.4	3.35E-1	7.06	2.73E+0	4.80E+1	66.6	1.83E-1	7.11	1.66E+0	2.88E+1
71.9	5.46E-1	7.84	1.43E+0	2.61E+1	71.0	2.94E-1	7.87	1.05E+0	1.48E+1
79.5	1.23E+0	8.57	7.55E-1	1.52E+1	78.4	6.75E-1	8.60	5.35E-1	7.96E+0
85.2	3.82E+0	9.97	3.39E-1	5.91E+0	84.2	2.35E+0	10.02	2.12E-1	2.70E+0
96.9	1.13E+1	11.37	1.20E-1	2.59E+0	96.0	7.34E+0	11.41	5.96E-2	1.24E+0
125.5	4.34E+1	12.79	4.60E-2	1.30E+0	124.4	2.98E+1	12.84	2.02E-2	7.38E-1
130.5	4.83E+1	14.17	1.97E-2	7.67E-1	129.4	3.47E+1	14.23	8.85E-3	5.22E-1
138.2	5.93E+1	15.02	1.08E-2	5.86E-1	137.0	4.26E+1	15.08	5.16E-3	4.46E-1
		EDI[c]	4.64E-3	3.13E-1[d]	145.0	4.93E+1	EDI[c]	2.71E-3	3.06E-1[d]
					150.4	5.61E+1			

(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate observed in eluate.
(c) EDI = post-elution water rinse.
(d) Final residual Cs on the resin bed after elution and water rinse.

Figure 13.8 displays the in-column shrink-swell characteristics of the Wave 4a resins. The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing. The relative BV shown is the BV observed divided by the BV in 0.5 M NaOH. Both resins displayed ~20% volume contraction from the Na-form to the H-form. The resin volume remained essentially constant in the caustic matrices despite the large Na concentration variation between simulant, feed displacement, and water rinse process steps. The measured resin volumes are provided in Table 13.10.



Matrix IDs

1: Loaded in column (Na-form)
2: DI water rinse
3: 0.5 M HNO_3
4: DI water rinse
5: 0.5 M NaOH
6: AP101 simulant
7: 0.1 M NaOH feed displacement
8: DI water
9: 0.5 M HNO_3
10: DI water

Figure 13.8. Shrink-Swell Characteristics of Wave 4a Resins

Table 13.10. Actual Resin Volumes as a Function of Feed Matrix, Wave 4a

		TI394-64	TI394-65
	Matrix	5J-370/686	3380-03-0200
Feed matrix	ID	mL	mL
1 M NaOH load-in	1	20.4	20.7
DI rinse	2	19.2	19.5
0.5 M HNO_3	3	16.3	16.3
DI water	4	16.3	16.3
0.5 M NaOH[(a)]	5	20.1	20.1
Feed, AP101 simulant	6	20.1	20.1
0.1 M NaOH	7	20.1	20.4
DI water	8	19.8	20.1
0.5 M HNO_3	9	16.3	16.7
DI water	10	16.0	16.3
Dry resin mass (H-form)	na	5.99 g	6.72 g

(a) Reference volume, defined as 1-BV.

14.0 Wave 4b Testing

Wave 4b tested the spherical RF product performance reproducibility from the scaled-up production process. Testing also incorporated the assessment of a less rigorous resin pretreatment processing and the associated effect on Cs loading performance.

The two test resins were prepared in ~70-gal[(a)] production batches at Microbeads and BSC. The BSC production batch resulted in significant fines production.[(b)] A subsample of the BSC resin was delivered to Microbeads for fines removal using a dispersermixer.[(c)] A 650-mL aliquot of the cleaned BSC resin and 2-L of the Microbeads resin were forwarded to PNWD for testing. The PNWD tracking ID is cross-referenced to the manufacturer, lot number, lot size (as produced in the H-form), preparation date, and receipt date in Table 14.1.

Table 14.1. Wave 4b RF Test Resins

Internal PNWD ID	Manufacturer	Lot Number	Production Lot Size	Preparation Date	Receipt Date
TI394-72	Microbeads	6C-370/745	74-gal	4/06	4/25/06
TI394-73	BSC	3380-3-0201	66-gal	4/06	5/12/06

14.1 Parametric RF Pretreatment Testing

The resin conversion process to the Na-form was tested by varying the 0.5 M NaOH:resin volume:volume ratio and evaluating the associated Na consumption. The results derived from these scoping tests were used to help guide the minimal pretreatment processing steps to apply to the column testing.

Resin from TI394-64 (MB 5J-370/686) was selected for the parametric testing. A 61.5-mL aliquot was washed with 3 BVs water. A series of 10-mL aliquots of settled resin volumes were transferred to beakers. The estimated mass was for each test aliquot was ~4.6 g H-form resin. The resin contact fluid was removed, and a specific volume of 0.5 M NaOH (40, 50, 60, 80, and 100 mL) was added to a sample. Another sample was contacted with 48 mL 0.6 M NaOH. The resin samples were swirled approximately once every 5 min. The resin aliquots were transferred to 25-mL graduated cylinders for volume measurements and then transferred back to the beaker. The volume measurements were repeated several times over a ~70-min interval and then again after soaking overnight to determine volume change as a function of time. Samples of the 17-h equilibrated contact fluid were removed for characterization. Hydroxide concentrations were determined by titration with a standard HCl solution according to ASR 7734; Na concentrations were determined for indication-only by ICP-AES.

(a) The 70-gal as-received H-form resin typically expands about 55% after pretreatment, resulting in a nominal 100-gal Na-form volume.
(b) Personal communication, MR Thorson.
(c) Product insert description—details of the cleanup process were not provided.

The resin volume change as a function of contact time for each test is shown in Figure 14.1. The time axis was broken so that the initial volume change for the first 120 min can be compared to the final expanded volume obtained at ~1000 min. This figure shows that the expansion rate increased as the available Na ions increased from 20 mmoles to 50 mmoles. Also, the initial concentration of Na influenced the initial expansion rate. This is shown from the equivalent expansion rates of the 28.8 mmoles Na supplied by the 0.6 M Na contact solution and the 50 mmoles Na supplied by the 0.5 M Na contact solution. The final achieved volume from the 28.8 mmoles (0.6 M Na addition) contact solution was more consistent with the volumes achieved from the 25 and 30 mmoles Na supplied from the 0.5 M Na contact solutions. The expansion rates were generally linear over the first 60 minutes of contact time followed by a significantly slower expansion rate to the equilibrated volume at 1000 min. This implies that the resin will need at least a 1-h contact time with the regeneration fluid to achieve most of the swell volume.

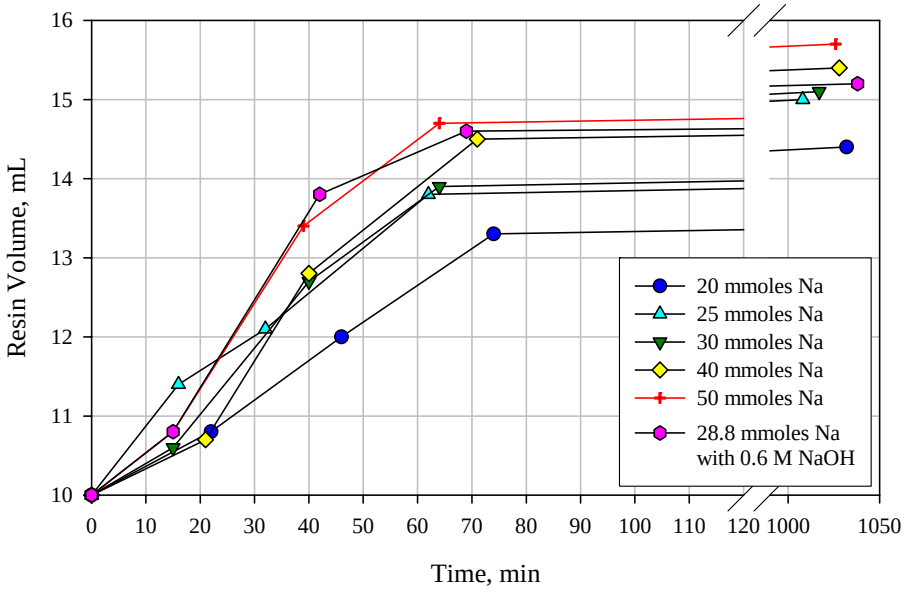


Figure 14.1. Resin Volume Expansion as a Function of Time

The mmoles of Na consumed by the resin upon conversion to the Na-form for each contact test was evaluated based on the input NaOH and equilibrated Na and hydroxide solution concentrations according to Equation 14.1.

$$C_{Na} = (M_I \times V_I) - M_F \times (V_S - [0.224 \times V_R]) \qquad (14.1)$$

where:
C_{Na} = mmoles of Na consumed
M_I = molarity (Na or OH^-) of initial contact solution
V_I = volume of initial contact solution
M_F = molarity (Na or OH^-) of equilibrated contact solution
$V_S - [0.224 \times V_R]$ = volume of equilibrated contact solution, including fluid in the resin bead structure
V_S = volume of resin plus fluid (slurry)
V_R = settled resin bed volume of equilibrated resin

0.224 = fraction of resin structure [a]

The Na consumption was calculated directly from the Na analysis and indirectly from the hydroxide analysis. The mmoles of added NaOH, equilibrated Na and hydroxide concentrations, and Na consumption determined from both analyses are provided in Table 14.2. Results from the two analytical techniques were in good agreement.

Table 14.2. Equilibrated Resin, Sodium and Hydroxide Concentrations, and Na Consumption

Added NaOH[a]	Equilibrated Resin and Solution			Consumed Na, mmoles		
mmoles	Volume, mL	Na, M[c]	OH^-, M	Na basis[c]	OH^- basis[d]	Average
20.0	14.4	0.0572	0.0490	17.3	17.7	17.5
25.0	15.0	0.0893	0.0852	19.9	20.2	20.1
30.1	15.1	0.123	0.109	21.9	22.8	22.4
40.0	15.4	0.195	0.179	23.2	24.5	23.8
50.0	15.7	0.236	0.231	24.9	25.4	25.1
28.8[b]	15.2	0.138	0.125	21.2	22.0	21.6

(a) The NaOH was added as 0.5 M NaOH in various volumes to 10-mL settled H-form (as-received, i.e., not relaxed and expanded) resin.
(b) The initial NaOH concentration for this test was 0.6 M.
(c) Na was measured for indication-only.
(d) The OH^- was measured according to ASR 7734, and overall uncertainty was estimated to be ±10%.

Figure 14.2 graphically shows the equilibrium resin volume percent expansion as a function of the input mmoles of Na (points are plotted relative to the right y-axis). In this case, the maximum volume expansion was determined to be 15.5 mL based on a 1.55× expansion factor from the as-received H-form condition to the fully-expanded Na-form (see Section 13). Also shown is the average Na consumed by, or exchanged onto, the resin (points are plotted relative to the left y-axis). Because the same H-form resin volume was used in each case, the left y-axis data may be normalized relative to H-form volume or mass.

The low-Na contacted resin (2 mmole Na per mL as-received resin) expanded to 93% of the maximum; expansion was virtually complete after contact with 4 mmoles Na per mL resin. This indicated that under plant operating pretreatment conditions, an accidental low Na variation during pretreatment will still provide well-swollen beads.

Also evident is the direct relationship of Na consumption as a function of available Na ions. The initial NaOH concentration (0.6 M vs 0.5 M) did not have a discernable effect on the mmoles of Na consumed by conversion.

(a) The resin structural fraction, 0.224, was obtained by dividing the bed density (0.367 g/mL, Table 13.4) by the skeletal density (1.64 g/mL. Table 13.5).

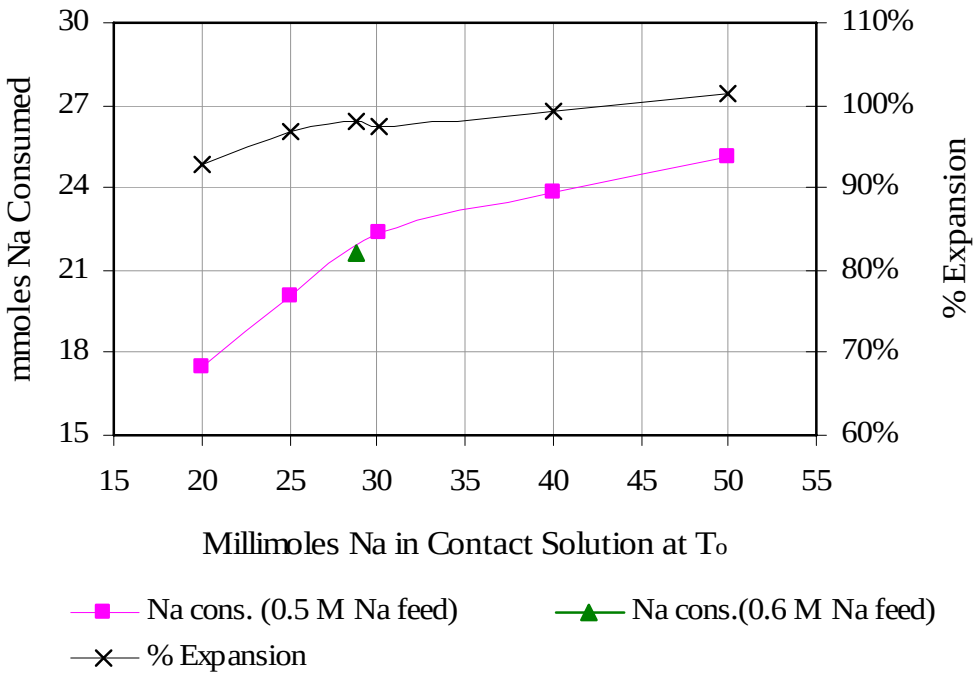


Figure 14.2. Resin Volume Change and Millimoles Na Consumed as a Function of Initial Available Na

Figure 14.3 shows the equilibrium curve of mmoles of Na consumed per mL of equilibrated Na-form resin volume as a function of the average equilibrated NaOH molarity. Also provided is the best-fit curve[(a)] for the combined data. The Na consumption (or resin conversion) increased with increasing equilibrium Na molarity. The maximum value reached in this test matrix was 1.60 mmoles Na/mL Na-form resin. Judging from the curve shape, this may not represent the maximum Na loading.

The Na consumption results for Microbeads lot 5J-370/686 were higher than previously reported for Microbeads resin lot 5E-370/641. Arm et al. (2005) reported an RF resin Na consumption of 1.55 mmoles/mL at an equilibrium NaOH concentration of 0.12 M (based on effluent conductivity measurement during in-column regeneration testing). Adamson, et al. (2006) reported a Na consumption of 1.04 mmoles/mL at 0.15 M free NaOH and 1.54 mmoles/mL at 0.5 M free NaOH. The meq of Na consumption were compared to the meq recovered during elution. Section 11.3 of this report indicated 1.77 meq per mL of Na-form resin were eluted from the resin. Eluate analysis from actual waste testing of AP-101 and AN-102 resulted in a calculated loading capacity of 1.3 to 1.4 meq/mL (Fiskum et al. 2006b).[(b)]

(a) The logarithmic function was generated with the Microsoft Excel curve-fitting function.

(b) In these cases, 25× dilutions were required to allow for safe handling of the samples at the workstation, which in turn could have contributed to a low bias within the overall reported ±15 % uncertainty.

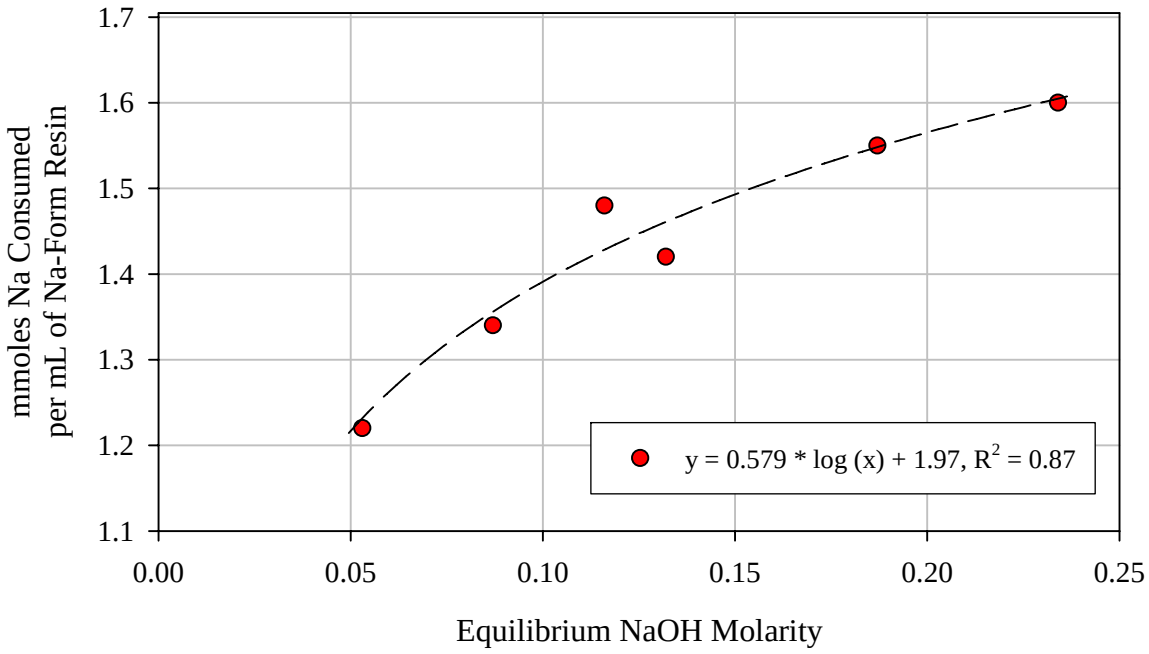


Figure 14.3. Na Consumption per Volume of Resin as a Function of Equilibrated Solution NaOH Molarity
(Note: Microbeads resin lot 5J-370/686 density is 0.30 g H-form resin / mL Na-form resin)

14.2 Resin Pretreatment

The standard Protocol P1-RF pretreatment was applied to the MB and BSC resins for follow-on physical property testing (micrographs, PSD, skeletal density) and ion exchange processing. Two additional pretreatments (Mod-1 and Mod-2) were evaluated using the MB resin; each employed minimal processing.[(a)] The Mod-1 pretreatment employed a simple water wash followed by a soak in dilute NaOH solution. Modification 2 employed a simple water wash followed by a soak in dilute NaOH solution, a soak in dilute nitric acid, and another NaOH solution soak. The efficacy of the pretreatment modifications was determined from Cs ion exchange loading characteristics in side-by-side loading tests with the P1-RF pretreated resins. The specific steps of the modified pretreatments are summarized in Table 14.3. For comparison, the nominal P1-RF pretreatment processing is also shown.

(a) All pretreatment processing was conducted according to TI-RPP-WTP-449, *Resin Pretreatment and Physical Properties Evaluation of Spherical RF Resins Wave 4b*. SK Fiskum, May 2006.

Table 14.3. Pretreatment Comparisons for TI394-72 (MB 6C-370/745) Resin

Protocol P1-RF			Mod-1			Mod-2		
Reagent	**Volume, mL (BV)[a]**	**Agitation/ Soak time, min**	**Reagent**	**Volume, mL (BV)[a]**	**Agitation/ Soak time, min**	**Reagent**	**Volume, mL (BV)[a]**	**Agitation/ Soak time, min**
Resin	61.0 (0.67)[b]	na	Resin	13.6 (0.75)[b]	na	Resin	13.6 (0.75)[b]	na
Water rinse	305 (3.1)	30	Water rinse	25.8 (1.2)	60	Water rinse	25.5 (1.2)	60
1 M NaOH	305 (3.1)	30/947	Water	29.7 (1.4)	120 (mix) /	Water	32.7 (1.5)	120 (mix) /
Water rinse	187 (1.9)	30	+		869 (soak) /	+		869 (soak) /
Water rinse	184 (1.9)	30	2 M NaOH	19.4 (0.89)	10 (mix)	2 M NaOH	17.0 (0.77)	10 (mix)
Water rinse	188 (1.9)	30	Load in column			0.5 M HNO_3	41.5 (1.9)	42
0.5 M HNO_3	600 (6.1)	30/110				0.5 M HNO_3	22.7 (1.0)	69
Water rinse	183 (1.9)	30				Water	26.1 (1.2)	125 (mix) /
Water rinse	186 (1.9)	30				+		984 (soak) /
Water rinse	185 (1.9)	30				2 M NaOH	20.0 (0.91)	10 (mix)
Subsample for column processing	17.0	na				Load in column		
1 M NaOH[c]	85 (5.0)	86						
Water rinse[d]	161 (7.7)	160						
0.5 M HNO_3[d]	144 (6.9)	143						
Water rinse[d]	62 (2.9)	125						
0.5 M NaOH[d]	118 (5.6)	120						

(a) The BV shown is relative to the Na-form resin volume instead of the as-received form volume.
(b) The as-received resin volume was measured in the H-form and is shown in mL. The relative measure in terms of BV was corrected for the expansion factor of 1.60 (as-received H-form to cycled Na-form volume).
(c) The expanded resin was loaded into the ion exchange column in preparation for in-column pretreatment.
(d) One shrink-swell cycle was conducted in-column before actual waste loading. (See section 14.4 for more discussion on in-column pretreatment processing.)
na = not applicable

In preparation for the modified pretreatment processing, 13.6-mL aliquots of settled resin volume, as-received form, were measured in a graduated cylinder; this volume was calculated to result in ~20-mL Na-form resin volume. Each resin aliquot was transferred to 60-mL glass bottles (ID ~3.5 cm) with polyethylene-lined screw caps. Each resin bed was individually processed according to the modified pretreatment operation. Contact fluid removal was controlled such that a fluid height of ~2 to 4 mm above the resin was maintained minimizing the chance the resin could be directly exposed to air. This minimal cover-fluid volume was approximately equal to 2 to 4 mL. All transfers in and out of the pretreatment bottle were determined by mass difference.

The Mod-1 pretreatment entailed the initial fluid removal and the addition of 25.8 mL DI water. The headspace was flushed with nitrogen gas and then the bottle was capped. The bottle was mounted sideways in a reciprocal shaker to generate effective mixing, and agitated for 1 h. The bottle was removed, and the resin was allowed to settle. Fluid above the resin bed was removed, and 29.7 mL DI water were added followed by 19.4 mL 2 M NaOH. The headspace was flushed with nitrogen, the bottle was sealed, and the mixture was agitated on the reciprocal shaker for 2 h. The resin bottle was removed and allowed to stand overnight. The slurry was agitated again for 10 min then the resin was allowed to settle. The solution was sampled for hydroxide analysis, and the resin was transferred directly into the ion exchange column (Blue column). No further in-column pretreatment was conducted.

The Mod-2 pretreatment entailed the initial fluid removal and the addition of 25.5 mL DI water. The headspace was flushed with nitrogen gas and then the bottle was capped. The bottle was placed in the reciprocal shaker and agitated for 1 h. The bottle was removed, and the resin was allowed to settle. The fluid above the resin bed was removed, and 32.7 mL DI water were added followed by 17 mL 2 M NaOH. The headspace was flushed with nitrogen, the bottle was sealed, and the mixture was agitated on the reciprocal shaker for 2 h. The resin bottle was removed and allowed to stand overnight. The slurry was agitated again for 10 min then the resin was allowed to settle. The solution was sampled for hydroxide analysis. The solution was removed, and 41.5 mL 0.5 M nitric acid was directly added to the resin slurry. The headspace was flushed with nitrogen, and the slurry was agitated for 42 min on the shaker. The solution was removed and pH-tested with narrow range test strips (EM Science, Gibbstown, NJ). The solution pH was ~5.5. An additional 22.7-mL volume of 0.5 M nitric acid was added to the resin, and shaking commenced for 69 min. The fluid (pH was confirmed to be <0.5) was removed, and 26.1 mL DI water were added followed by 20.0 mL 2 M sodium hydroxide. The headspace was flushed with nitrogen, and the slurry was shaken for 2 h. The bottle was removed from the shaker, and the resin was allowed to stand overnight. After agitating again for 10 min, the resin was allowed to settle and the solution was sampled for hydroxide analysis. The resin was transferred directly into the ion exchange column (Pink column). No further in-column pretreatment was conducted.

Two additional 13.6 mL as-received MB resin samples were dried to constant mass to assess the ion exchange column H-form resin mass. The duplicate dry masses averaged 6.143 g ± 0.006 g.

The equilibrated NaOH solutions were analyzed for hydroxide concentration by titration with standard HCl solution according to ASR 7734; results are shown in Table 14.4 and Figure 14.4. The Na consumed by conversion was determined by applying Equation 14.1 where V_R was calculated based on the measured resin bed height as loaded in the ion exchange column. The Mod-1 conversion was conducted in duplicate, and each contact solution was submitted for analysis; the duplicate results showed good agreement. The Mod-2 pretreatment incorporated two swell cycles. The first swell cycle did not

incorporate the measure of Na-form resin volume; in this case, 20.4 mL (full resin volume) was ascribed to V_R. The Mod-1 and Mod-2 Na consumptions agreed fairly well with the predictive curve established from the scoping tests.

Table 14.4. Mod-1 and Mod-2 Pretreatment Na Consumption

Sample ID	Measured OH^-, M	Na-form Resin Volume, mL[(a)]	Consumed Na, mmoles
Mod-1 A	0.141	20.4	30.3
Mod-1 B	0.135	20.4	30.6
Mod-2 (cycle 1)	0.0948	20.4[(b)]	28.1
Mod-2 (cycle 2)	0.129	20.4	32.6

(a) The resin bed volumes were measured to two significant figures.
(b) Volume was not measured; the estimated volume is provided.

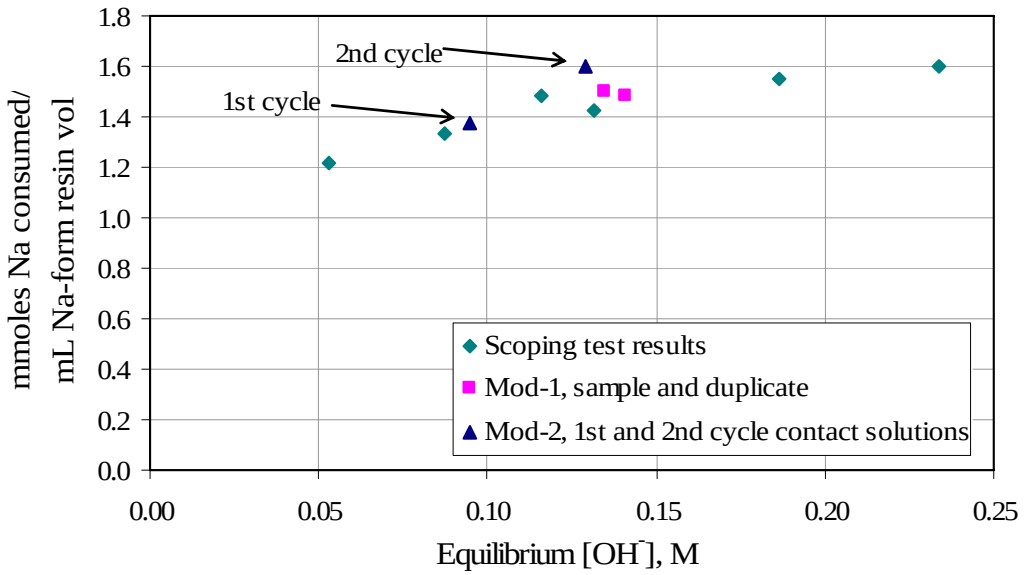


Figure 14.4. Na Consumption for Mod-1 and Mod-2 Pretreatments

14.3 Physical Properties

The P1-RF (standard protocol) pre-treatment shrink-swell data are summarized in Table 14.5. The unconstrained resin expansion was ~26% after one cycle pretreatment. The follow-on expansions to Na-form resin were measured at 29% and 26%. The overall expansions from the as-received H-form resin to the pretreated Na-form resin were nominally 58 and 61%, slightly higher than the 55% previously observed.

Table 14.5. Summary of Pretreatment Resin Swell Data, P1-RF Pretreatment

PNWD ID	1st Cycle Expansion			H-form to Na-form Expansion			Total Expansion
	As-received H-form, mL	Pretreated H-form, mL	Expansion Factor	H-form,[(a)] mL	Na-form, mL	Expansion Factor	
TI394-72	61.0	76.5	1.25	28.0	36.0	1.29	1.61
TI394-73	62.3	78.5	1.26	14.5	18.2	1.26	1.58

(a) After first pretreatment cycle.

The Mod-1 and Mod-2 pretreated Microbead resin volumes were not measured in a graduated cylinder. The slurries were transferred directly into the ion exchange columns. The Na-form volumes, measured immediately after loading in the column, were 20 mL (both Mod-1 and Mod-2). This volume corresponded to an expansion factor of 50%. In this case, the simplified pretreatments resulted in ~93% expansion and thus were ~93% effective in relaxing the resin bead structure relative to the 61% expansion determined from the P1-RF pretreatment.

Micrographs of the two RF resins, pretreated per P1-RF, are shown in Figure 14.5 and Figure 14.6. Broken pieces and small dark spheres were evident in the Microbead product. A higher incidence of particle aberrations was observed in the BSC product. In both products, the cross-sectioned particle color appears to be uniform throughout the interior.

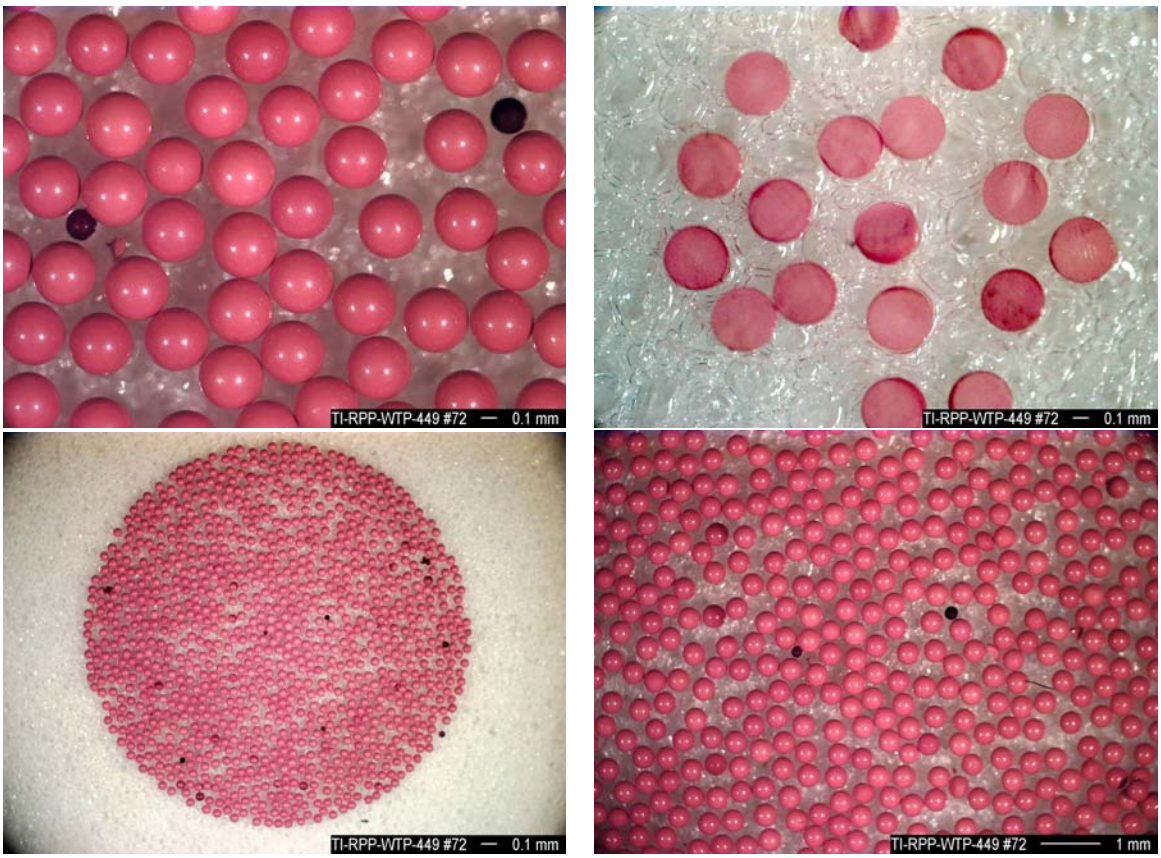


Figure 14.5. Micrographs of TI394-72 (Microbeads 6C-370/745). Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

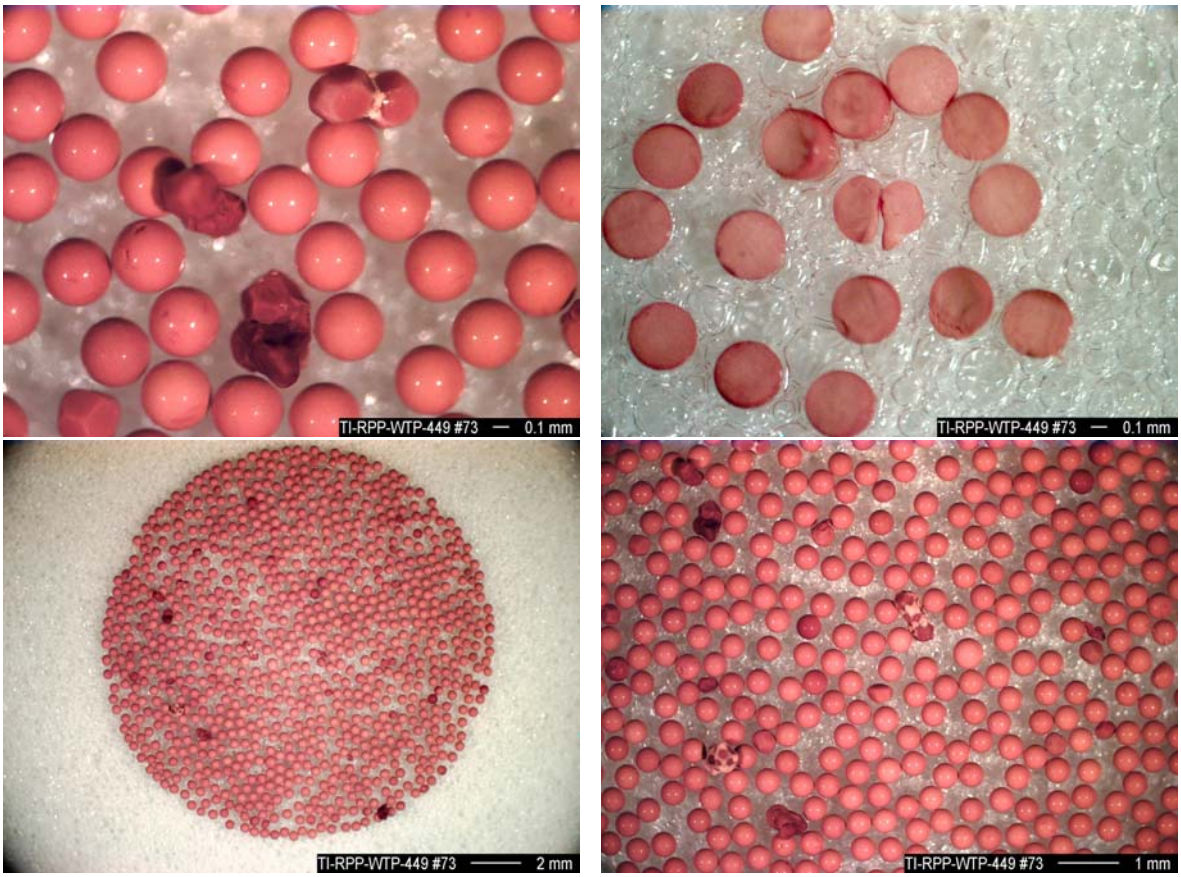


Figure 14.6. Micrographs of TI394-73 (BSC-3380-3-0201). Clockwise from top left: 70×, Cross-section 70×, 25×, and 10×.

The PSD results are shown in Figure 14.7 and Table 14.6. Unlike previously tested resins, the average particle diameters determined on a number, volume, and area basis were significantly different. This was attributed to a fraction of resin beads in the large-diameter range, which has the largest effect on the m_v PSD measure. In an effort to accommodate the m_v values, the evaluated PSD range was increased from 1 to 1086 microns to 1 to 1408 microns. The extended region still did not appear to capture the entire high-end region. Evaluation of the micrographs did not reveal high-diameter beads in the TI394-72 product; deformations and merged resin particles were observed in the TI394-73 product, which could have contributed to the larger spread in m_v PSD data. The detailed PSD results for the pretreated TI394-72 and TI394-73 resins are provided in Appendix D.

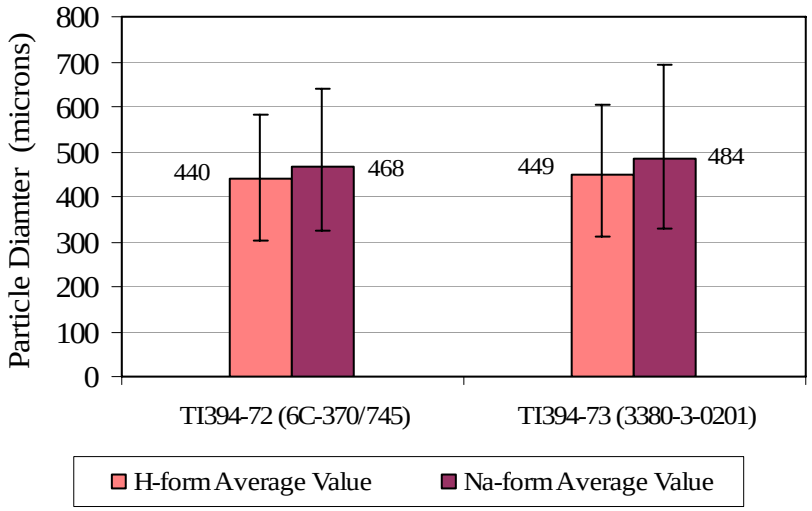


Figure 14.7. Wave 4b Resins PSD Showing Average Values and Low 5% to High 90% Spread (Volume basis)

Table 14.6. Wave 4b Resins Particle-Size-Distribution Summary

Resin ID Lot #	Resin Form	Volume Distribution (microns)[a]				Number Distribution (microns)				Area Dist. (microns)
		m_v	sd	Low 5%[b]	High 90%[c]	m_n	sd	Low 5%[b]	High 90%[c]	m_a
TI394-72	H-form	440	81	304	581	368	52	287	436	395
6C-370/745	Na-form	468	103	324	640	398	63	301	487	433
TI394-73	H-form	449	82	310	603	375	53	293	443	401
3380-3-0201	Na-form	484	118	329	691	404	65	304	496	443
Resin ID Lot #	**Resin Form**	**Calculated Average Sphere Volume, mm^3**								
TI394-72	H-form	0.0446				0.0262				0.0323
6C-370/745	Na-form	0.0535				0.0330				0.0426
Expansion factor >		*20%*				*26%*				*32%*
TI394-73	H-form	0.0475				0.0276				0.0339
3380-3-0201	Na-form	0.0595				0.0345				0.0457
Expansion factor >		*25%*				*25%*				*35%*

(a) Volume size range was extended from 1 to 1086 microns (as reported for Waves 1-4a) to 1 to 1408 microns to better accommodate a small fraction of particles at the high end. Evaluation of PSD spectrum from 1 to 704 microns resulted in H-form resin of 398 micron diameter (TI397-72) and 404 micron diameter (TI397-73).
(b) 5% of the particles are less than the given diameter.
(c) 10% of the particles are greater than the given diameter.

Table 14.7 summarizes the resin bed densities determined from pretreatment testing and column testing. The calculated dry-bed densities determined after the first in-column pretreatment shrink-swell cycle agreed well with those obtained from unconstrained bed density testing. The H-form and Na-form calculated densities were similar. The Microbead product dry bed densities were typical of previously

tested resins. As observed with Wave 4a testing, the BSC resin dry-bed densities for both the H-form and Na-form were slightly higher than those observed for the Microbeads products.

Table 14.7. Wave 4b Resins Dry Bed Densities

Resin ID Lot #	Resin Form	Settled Vol., mL[(a)]	Dry Mass, g[(a)]	Settled Resin Density, g/mL[(b)]	Column Processing Bed Density, g/mL[(c)]
TI394-72	H-form	17.0	6.1591	0.362	0.36
6C-370/745	Na-form	14.1	5.2954	0.376	na
TI394-73	H-form	17.0	7.1892	0.423	0.42 - 0.43
3380-3-0201	Na-form	10.8	4.4623	0.413	na

(a) Aliquot measured for skeletal density.
(b) Dry resin mass per unit wet volume
(c) Measured during column processing; only the dry H-form mass placed in the column was determined.
na = not applicable, Na-form mass was not determined.

The Mod-1 and Mod-2 pretreated Microbeads resin densities were calculated from the untreated resin mass (6.143g) and the eluted (H-form) resin volume (17.3 mL). The H-form resin bed densities were 0.36 g/mL, in agreement with the P1-RF pretreated resin results.

The resin skeletal densities are provided in Table 14.8. The skeletal densities between the two products were similar. However, the Na-form TI394-73 (BSC) resin skeletal density was slightly higher (beyond the uncertainty range) than those found for the Wave 4a test resin and the TI394-72 (MB) resin.

Table 14.8. Wave 4b Resins Skeletal Densities

		H-form		Na-form	
Resin ID	Lot #	g/mL	RPD	g/mL	RPD
TI394-72	6C-370/745	1.475	0.18%	1.626	0.090%
TI394-73	3380-3-0201	1.484	0.12%	1.661	0.058%

14.4 Column Testing

Two of the column tests were conducted similarly to the previous test waves; columns White (TI394-72 resin) and Yellow (TI394-73 resin) tested the nominal pretreatment processing (P1-RF) with AP-101 simulant loading. Column Green (TI394-73 resin) tested the nominal pretreatment processing with AZ-102 simulant loading. Column Blue (TI394-72 resin) tested the Mod-1 pretreatment consisting of a resin water rinse followed by swelling with a NaOH solution in the batch contact mode. Column Pink (TI394-72 resin) tested the Mod-2 pretreatment consisting of the resin water rinse, swell-shrink-swell in batch contact mode. The specific column processing test parameters are summarized in Table 14.9 through Table 14.13.

Table 14.9. Experimental Conditions for TI394-73 (BSC Lot BSC-3380-3-0201, Green Column) with AZ-102 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (5/18/06)								
Water rinse	DI water	7.66	3.22	152	2.93	0.965	2.62	22
Acid wash	0.5 M HNO_3	8.19	3.45	162	3.00	0.988	2.73	24
Water rinse	DI water	3.04	1.28	60	1.52	0.501	2.00	25
Cycle 1 (Start 5/19/06)								
Regeneration	0.5 M NaOH	4.53	1.91	89.6	3.02	0.995	1.50	21
Loading column	AZ-102 Simulant	222	NA	4393	1.50	0.495	147	19 – 24
Feed displacement	0.1 M NaOH	2.96	1.25	58.7	3.12	1.03	0.950	23
Rinse	DI water	2.88	1.21	56.9	3.03	0.999	0.950	23
Elution	0.5 M HNO_3	29.1	NA	576	1.41	0.467	20.6	22 – 23
Rinse	DI water	2.86	1.20	56.5	1.42	0.467	2.02	22

(a) BV = bed volume (19.8 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 14.10. Experimental Conditions for TI394-73 (BSC Lot BSC-3380-3-0201, Yellow Column) with AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (5/19/06)								
Water rinse	DI water	7.78	3.43	161	2.96	1.02	2.63	22
Acid wash	0.5 M HNO_3	6.95	3.07	144	2.96	1.02	2.35	22
Water rinse	DI water	3.01	1.33	62.4	1.47	0.507	2.05	23
Cycle 1 (Start 5/22/06)								
Regeneration	0.5 M NaOH	5.71	2.52	118	2.86	0.987	2.00	22
Loading column	AP-101 Simulant	79.1	NA	1641	1.46	0.503	54.5	22 – 24
Loading column	AP-101 Simulant	55.4	NA	1149	2.94	1.01	18.8	22 – 24
Feed displacement	0.1 M NaOH	2.94	1.30	61.0	2.89	1.00	1.02	22
Rinse	DI water	2.84	1.25	58.9	2.89	0.998	0.983	22
Elution	0.5 M HNO_3	12.1	NA	251	1.40	0.482	8.70	22
Rinse	DI water	2.77	1.22	57.5	1.40	0.483	1.98	22

(a) BV = bed volume (20.7 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 14.11. Experimental Conditions for TI394-72 (MB 6C-370/745, White Column) with AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
In-situ Preconditioning (5/19/06)								
Water rinse	DI water	7.74	3.47	163	2.90	1.02	2.67	22
Acid wash	0.5 M HNO_3	6.90	3.09	145	2.89	1.01	2.38	22
Water rinse	DI water	2.89	1.30	60.9	1.39	0.487	2.08	23
Cycle 1 (Start 5/22/06)								
Regeneration	0.5 M NaOH	5.60	2.51	118	2.80	0.982	2.00	22
Loading column	AP-101 Simulant	80.7	NA	1699	1.48	0.519	54.5	22 – 24
Loading column	AP-101 Simulant	54.4	NA	1145	2.88	1.01	18.8	22 – 24
Feed displacement	0.1 M NaOH	2.89	1.29	60.9	2.84	0.998	1.02	22
Rinse	DI water	2.81	1.26	59.1	2.86	1.00	0.983	22
Elution	0.5 M HNO_3	12.3	NA	258	1.41	0.496	8.67	22
Rinse	DI water	2.78	1.25	58.6	1.40	0.492	1.98	22

(a) BV = bed volume (21.0 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 14.12. Experimental Conditions for Mod-1 Pretreated TI394-72 (MB 6C-370/745, Blue Column) with AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
Cycle 1 (Start 5/22/06)								
Loading column	AP-101 Simulant	80.7	NA	1648	1.54	0.524	52.4	22 - 24
Loading column	AP-101 Simulant	56.4	NA	1151	3.00	1.02	18.7	22 – 24
Feed displacement	0.1 M NaOH	2.92	1.27	59.7	2.97	1.01	0.983	22
Rinse	DI water	2.85	1.24	58.2	3.06	1.04	0.933	22
Elution	0.5 M HNO_3	15.0	NA	307	1.41	0.480	10.7	22
Rinse	DI water	2.87	1.25	58.6	1.40	0.476	2.05	22

(a) BV = bed volume (20.4 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

Table 14.13. Experimental Conditions for Mod-2 Pretreated TI394-72 (MB 6C-370/745, Pink Column) with AP-101 Simulant

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[(a)]	AV[(b)]	mL	BV/h	mL/min	h	
Cycle 1 (Start 5/22/06)								
Loading column	AP-101 Simulant	84.8	NA	1732	1.54	0.524	55.1	22 - 24
Loading column	AP-101 Simulant	57.1	NA	1165	3.05	1.04	18.7	22 – 24
Feed displacement	0.1 M NaOH	3.08	1.34	63.0	2.89	0.984	1.07	22
Rinse	DI water	2.91	1.26	59.4	3.06	1.04	0.950	22
Elution	0.5 M HNO_3	15.5	NA	316	1.46	0.498	10.6	22
Rinse	DI water	2.86	1.24	58.5	1.46	0.496	1.97	22

(a) BV = bed volume (20.4 mL in Na form)
(b) AV = apparatus volume (47 mL)
NA = not applicable
All processing was downflow.

In-column resin pretreatment processing resulted in generally sharp Na-form to H-form conversion fronts; the opposite (H-form to Na-form) conversion fronts were slightly more diffuse. In all cases, the conversion fronts were generally level across the bed. Photographs of the white column (resin TI394-72) pretreatment and elution processing are shown in Figure 14.8. The Na-form resin is the dark-colored material.

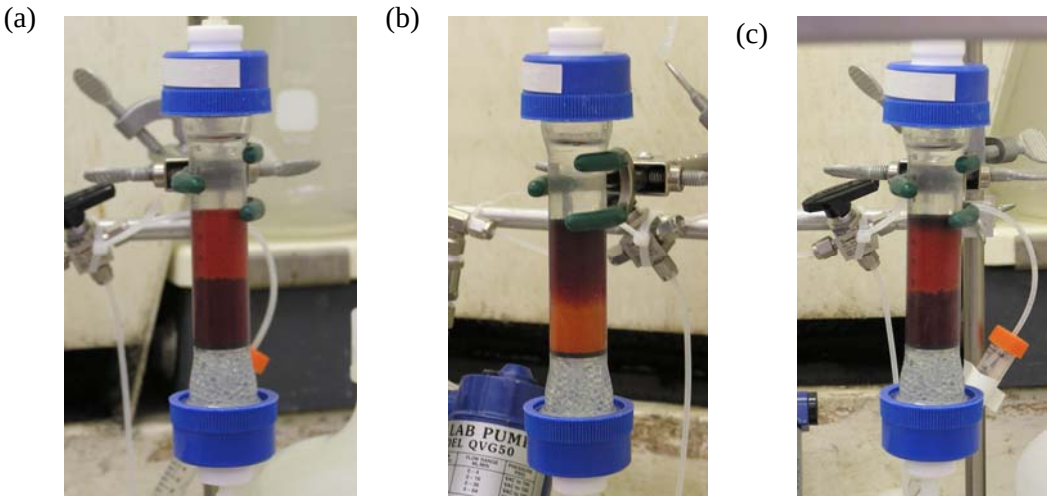


Figure 14.8. Column Processing of TI394-72 Showing Conversion Fronts During Pretreatment to Acid Form (a) and to Na-Form (b), and During Elution (c)

The conversion front during elution was generally sharp. The TI394-73 BSC resin (Yellow column) demonstrated the highest ragged nature. Selected resin elution profiles (post simulant loading) are shown in Figure 14.9.

(a) (b) (c) (d)

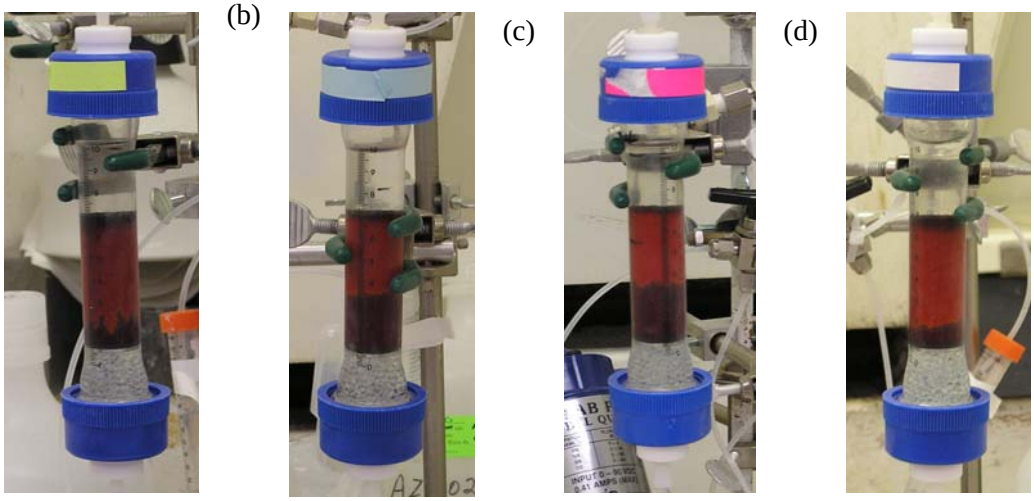


Figure 14.9. Elution Conversion Front Comparison (a) Yellow Column, (b) Blue Column, (c) Pink Column, (d) White Column

The top surface layer on each resin bed (from the P1-RF pretreatment) was initially the same as the rest of the resin bed. During processing, the top layer eventually turned black. The black surface was typical during RF processing and was attributed to be oxidative attack. The final depth of the black layer was about 3 mm.

The black bands at the surfaces of the Mod-1 and Mod-2 pretreated resins were noticeably smaller. The Mod-1 band was ~2 mm deep, and the Mod-2 band was ~1 mm deep. This indicated that the oxidative attack associated with dissolved oxygen in the various feeds was less pronounced for the minimally pretreated resin beds. Photographs of the Mod-1 and Mod-2 pretreated resin beds, post elution, are provided in Figure 14.10; also shown is a P1-RF pretreated resin for comparison.

a) b) c)

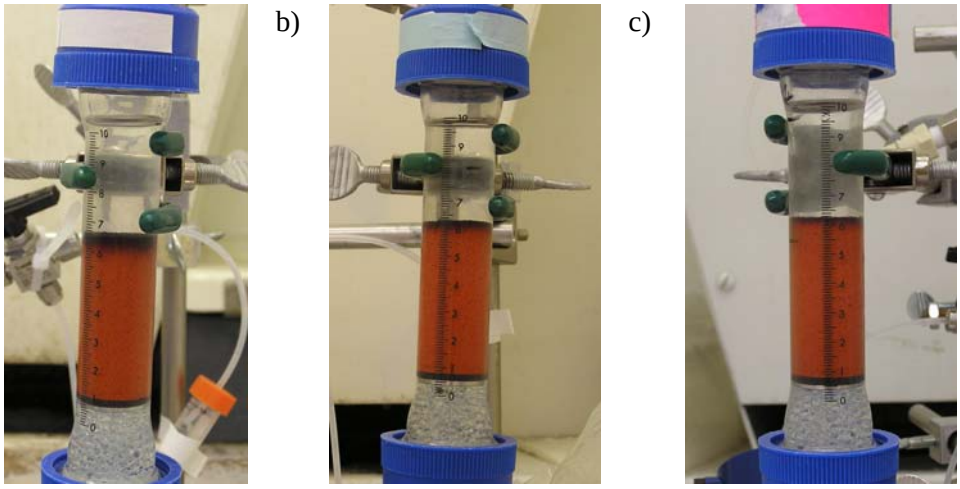


Figure 14.10. Comparison of Resin Oxidation (Black Band) Following Elution Processing; Pretreatment Conditions: a) P1-RF, b) Mod-1, c) Mod-2

Specific Cs loading performance results are summarized in Table 14.14. Processing with AP-101 resulted in similar Cs concentrations on the resin beds for the various tests. The AZ-102 process test loaded 14× more Cs onto the resin than the AP-101 process tests. The high K concentration (0.68 M) in the AP-101 simulant reduced the Cs exchange capacity onto the RF resin (note that the AZ-102 contained 0.145 M K). The Wave 4b resin Cs loading capacity increased ~10% relative to Wave 1 BRF-14 resin formulation.[(a)]

Table 14.14. Cs Loading Concentrations onto RF Resins, Wave 4b

	AP-101				AZ-102
	TI394-72			TI394-73	
	MB 6C-370/745			BSC 3380-3-0201	
Parameter	**P1-RF**	**Mod-1**	**Mod-2**	**P1-RF**	
Cs breakthrough onset, BV	37	32	48	30	67-85
Cs contract limit breakthrough, BV	61	54	66	52	95
50% Cs breakthrough, BV	131	132	135	148	200
Total Cs load, mg	14.9	14.4	14.9	15.2	203
Cs concentration, mg/g	2.42	2.35	2.43	2.11	28.3
Cs concentration, mg/mL	0.71	0.71	0.73	0.73	10.3

The AP-101 simulant Cs loading profiles for the Wave 4b resins (P1-RF pretreatment) are shown in Figure 14.11. Also shown for direct comparison are the Wave 4a 100-gal production batch resin results. As shown with previous test waves, the change in flowrate from 1.5 BV/h to 3.0 BV/h was associated with a slight change in the linearity of the load profile on the probability plot (~80 BVs).

(a) Wave 1 cycle 1 BRF-14 resin loaded 2.0 mg Cs/g resin and 0.66 mg Cs/mL resin bed (AP-101 simulant), and 25 mg Cs/g resin and 8.3 mg Cs/mL resin bed (AZ-102 processing).

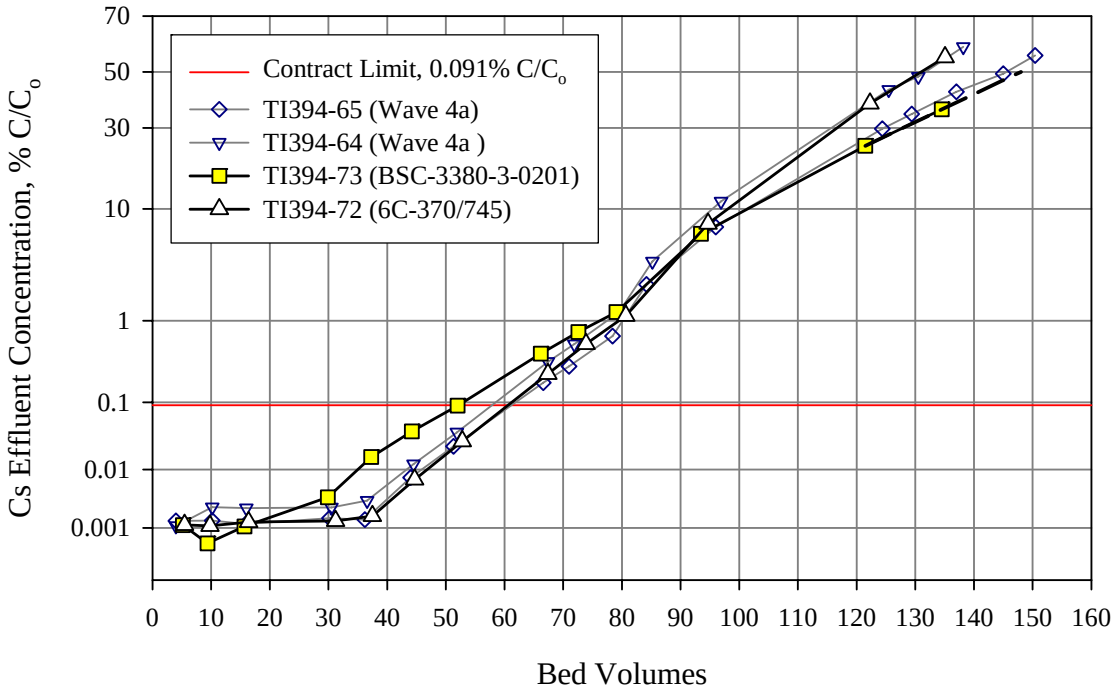


Figure 14.11. AP-101 Simulant Loading Profiles for TI394-72 (6C-370/745), TI394-73 (BSC-3380-3-0201), and Wave 4a Resins (Pretreated per P1-RF)

The Microbeads resins (TI395-64 and TI394-72) tracked nearly identically with respect to the various Cs breakthrough points. This indicted that spherical RF resin can be reproducibly manufactured in large quantity (100 gal when converted to the Na-form) with predictable and targeted Cs load characteristics.

The BSC resins (TI394-65 and TI394-73) showed small but significant variation through the first ~70 BVs processed; the Wave 4b production batch resulted in earlier Cs breakthrough relative to the Wave 4a production batch. The Wave 4b production batch had significant fines adhering to the beads before it was cleaned at Microbeads and forwarded to PNWD for testing. It is possible that the phenomenon that led to the fines production also resulted in the early Cs leakage, perhaps through entrainment of RF microstructures from the resin pores after swelling to the Na-form. The observed effect would diminish relative to the normal Cs breakthrough as progression is made along the breakthrough curve. The projected 50% Cs breakthrough point for TI394-73 (148 BVs) was virtually identical to the measured value for TI394-65 (146 BVs).

The Cs loading profile for the AZ-102 process test is shown in Figure 14.12. Also shown for comparison are the AZ-102 breakthrough profiles for previously-tested resins: SL-644 and Resin #3 RF (Fiskum et al. 2004), and BRF-14 (from Wave 1 testing). The Resin #3 and BRF-14 resins were made according to the same manufacturing conditions with the exception of particle size; the BRF-14 had a larger average particle size distribution than Resin #3. The TI394-73 particle size was smaller than both the BRF-14 and Resin #3.

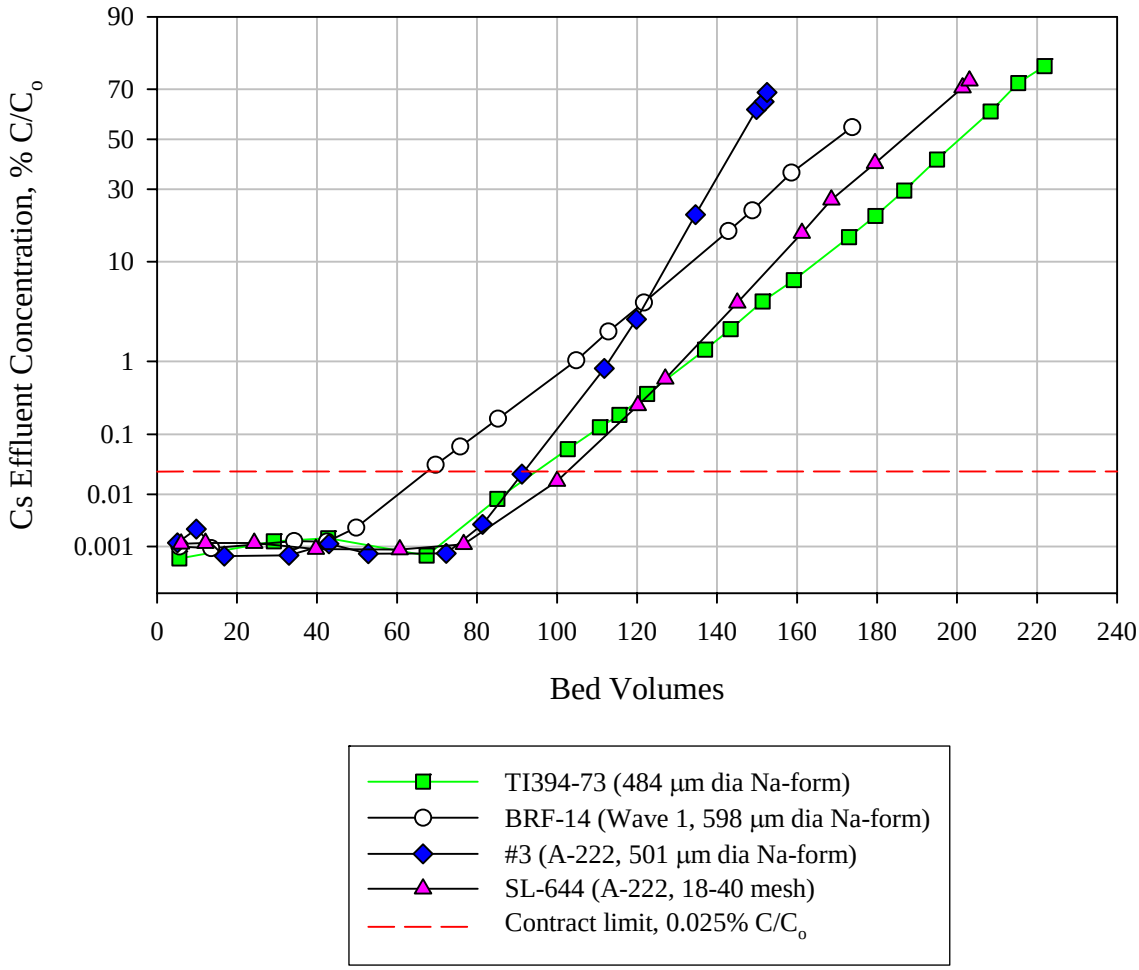


Figure 14.12. AZ-102 Simulant Loading Profiles of TI394-73 (BSC 3380-3-0201), BRF-14 (Wave 1), Resin #3 (Fiskum et al. 2004), and SL-644 (Fiskum et al. 2004)

The onset of Cs breakthrough observed for TI394-73 (~68 BVs) was ~10 BVs sooner than found in Resin #3 and SL-644 (75 and 80 BVs, respectively). The first 67 BVs processed through TI394-78 resulted in a Cs DF of ~150,000 (C/C_o = 6.7E-4%). The contract limit (at 0.025% C/C_o) Cs breakthrough volume was similar to that of Resin #3 resin; however, the 50% Cs breakthrough demonstrated a 57-BV improvement, indicating higher Cs capacity in the resin. The TI394-73 RF resin contract limit breakthrough was reached ~8 BVs sooner than found for SL-644, but the 50% breakthrough capacity was ~15 BVs higher. In all test cases, the spherical RF demonstrated more than enough capacity and selectivity to process the Envelope B wastes where only a 20 to 40 BV load is anticipated in the WTP.[(a)]

The effect of different spherical RF resin pretreatments on the Cs loading characteristics is shown in Figure 14.13. The 50% Cs breakthrough points for all three tests were virtually identical (131 to 135 BVs). The Cs onset and contract limit breakthroughs varied according to the pretreatment process.

(a) The small-volume load constraint is driven by the total curie loading on the resin and its associated safety-basis.

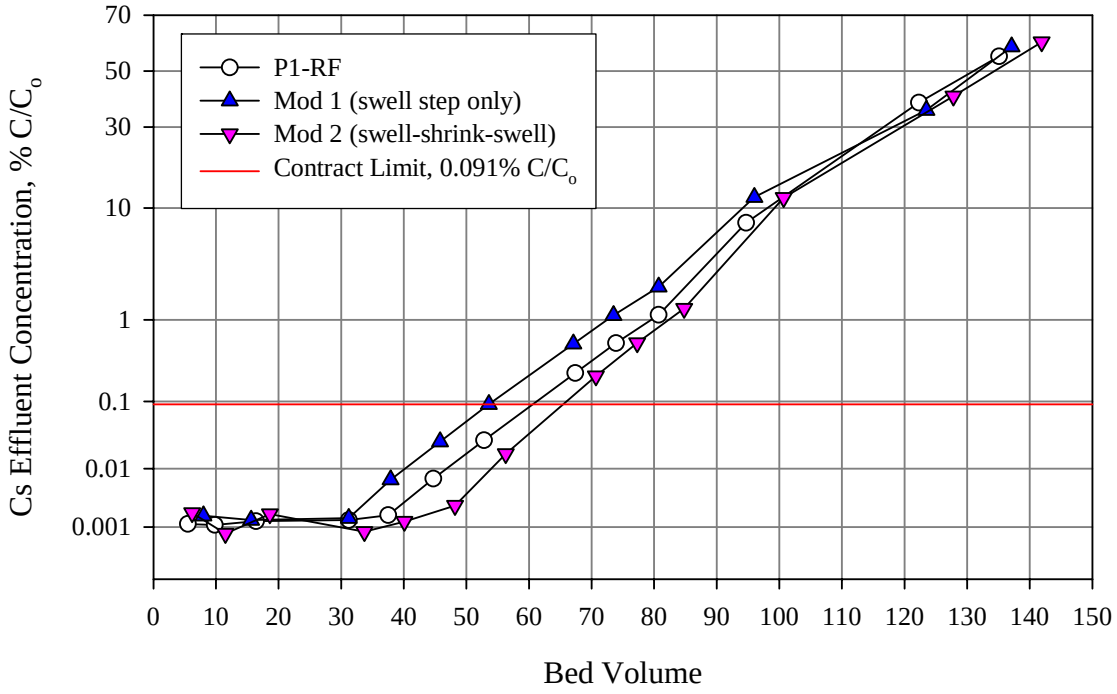


Figure 14.13. Cs Loading Profile Variation as a Function of Resin Pretreatment (TI394-72 Resin) with AP-101 Simulant

The Mod-1 (swell only) pretreated resin performance resulted in a relatively early onset of Cs break through at ~32 BVs and the contract limit breakthrough at ~54 BVs. Since the Mod-1 pretreated resin had not undergone a full shrink-swell cycle, some active sites in the resin may have been too tightly constrained structurally and thus not have been available. It is conjectured that after elution (resin shrinkage step), the next load cycle (resin expansion step) would be similar to that of the P1-RF or Mod-2 pretreatment Cs load results.[(a)]

The Mod-2 (swell-shrink-swell) pretreated resin resulted in superior load performance relative to Mod-1. The onset of Cs breakthrough was delayed an additional ~15 BVs, and the contract limit breakthrough was delayed an additional ~10 BVs relative to the Mod-1. This effect was attributed to a better opening of the bead macroporous structure, allowing for more efficient use/access to active sites in the resin structure.

The Mod-2 contract limit breakthrough (66 BVs) was 8% higher than that of the P1-RF pretreated resin. The slightly poorer performance of the P1-RF resin relative to the Mod-2 pretreated resin may be

(a) Wave 1 testing pretreatment was similar to Mod-1 in that only a swell step was applied to the resin (see Figure 5.3 for pretreatment sequence). In Wave 1 testing, the second process cycle demonstrated slightly improved Cs exchange performance relative to the first process cycle. Table 5.13 compares the contract limit breakthrough for Cycle 1 and Cycle 2. The BV processed to reach the contract limit breakthrough in the second cycle was ~10 to 25% greater than that of the first process cycle. Similarly, the Mod-1 contract limit breakthrough (54 BVs) was 13% less than the P1-RF pretreated resin (61 BV).

attributed to oxidative attack during the pretreatment process. The RF resin has been shown to effectively "consume" dissolved oxygen, and resin performance was reduced after oxygen exposure (see Section 6). The P1-RF pretreatment processing used relatively large liquid volumes in an open-beaker format for relatively long time periods, increasing the resin exposure to dissolved oxygen.

The Cs elution profiles following AP-101 simulant load tests are shown in Figure 14.14. Only two resins (TI394-72, Mod-2, and TI394-64 P-RF) achieved 1% C/C_o eluate Cs concentration at 15 BVs of processed eluate. Two resin elutions were ceased at 12 BVs before reaching 1% C/C_o. Tailing was similar for all tests with the Mod-1 pretreated resin showing a slightly higher eluate Cs concentration. The broader peak for TI394-73 resin may be an artifact of the effluent sampling process where the high-level peak sample was split between the fifth and sixth sample collections.

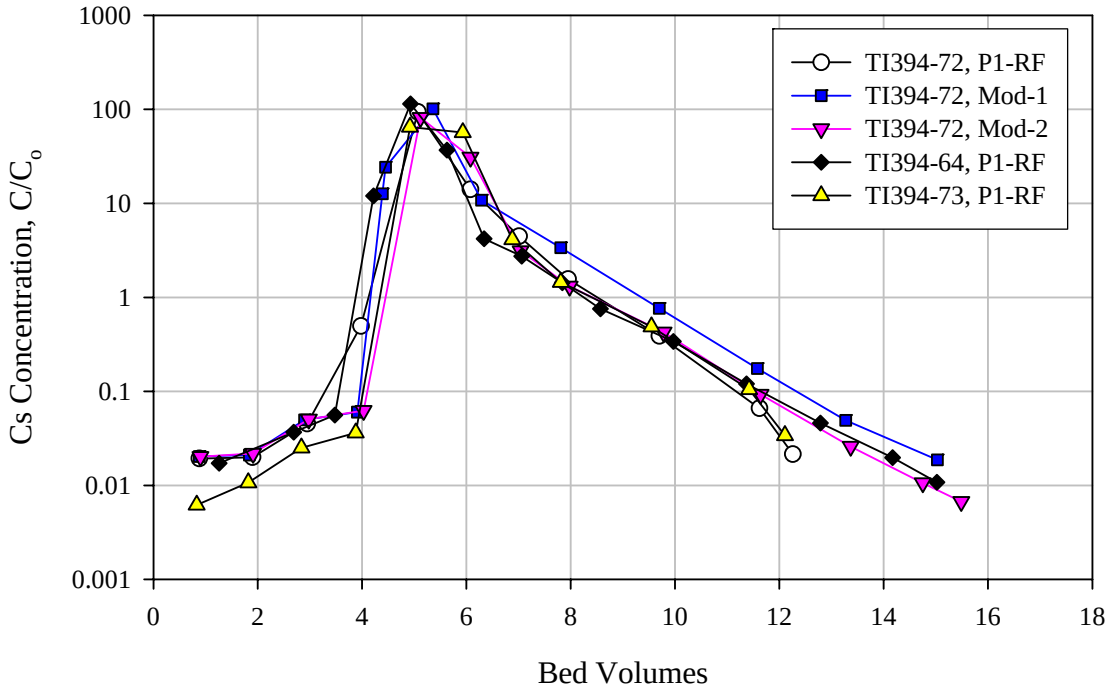


Figure 14.14. Elution Profiles after Processing AP-101 Simulant for TI394-72 (6C-370/745) from Various Pretreatment Operations Compared to TI394-64 (5J-370/686)

The elution profile following AZ-102 processing on TI394-73 is shown in Figure 14.15. The AP-101 process eluate and the BRF-14 AZ-102 process eluate are also shown for comparison. The elution characteristics, peak shape, height, width, and tailing are virtually equivalent. The only apparent difference is the lead-in Cs C/C_o, which started about a factor of 5× lower for the AP-101 process feed than for the AZ-102 process feed. The initial Cs concentrations on the resin differed by an order of magnitude: 28 mg Cs/ g resin from the AZ-102 processing and 2.1 mg Cs/g resin from the AP-101 processing. The 1% C/C_o eluate concentration was reached within 15 BVs.

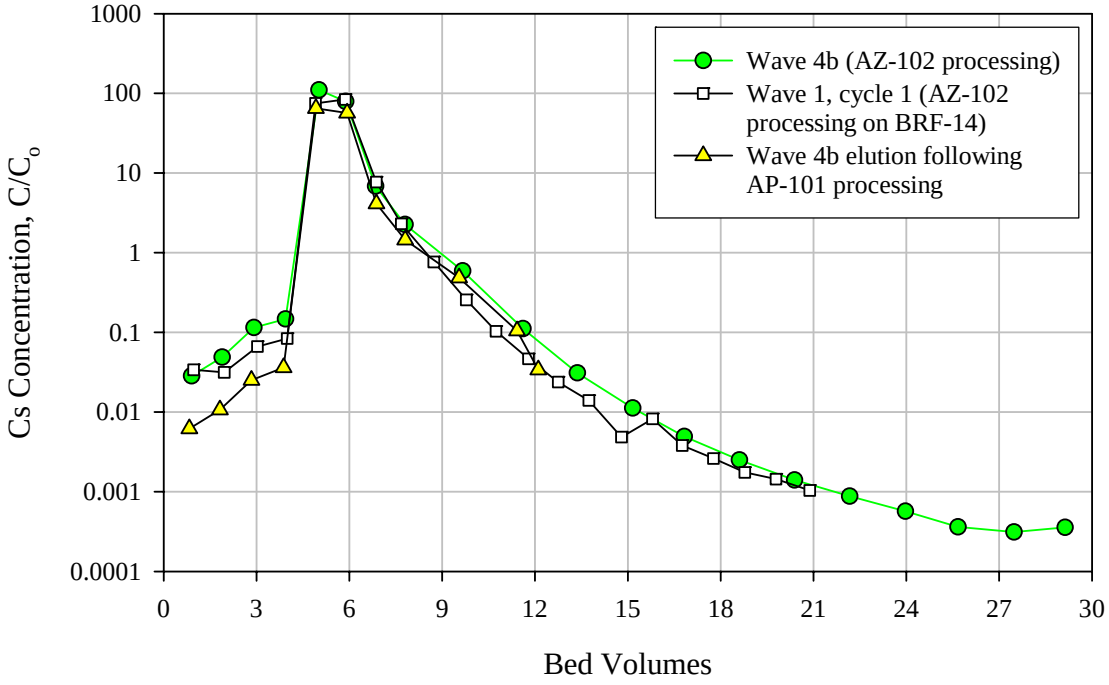


Figure 14.15. Elution Profiles after Processing AZ-102 and AP-101 Simulants for TI394-73 (BSC 3380-3-0201) Compared to BRF-14 Resin after Processing AZ-102 Simulant

Residual resin Cs concentration as a function of the elution BV is summarized in Figure 14.16. The design-limit spent-resin Cs concentration of 4.2 μg/g resin associated with Envelope A waste processing corresponds to 3.2 μg/g for Envelope B waste processing. Both scale-up manufactured test resins released Cs to these design limits. The AP-101 (Envelope A) elution design limit was reached after processing between 10 and 12 BVs; the AZ-102 elution design limit was reached at 16 BVs. The Envelope B test was conducted to >50% Cs breakthrough (220 BVs load); the WTP plans to operate at significantly less Cs loading, ~20 BVs. In this case, the elution design limit may be reached sooner.

The AZ-102 loaded resin resulted in twice the residual Cs concentration as the AP-101 Cs loaded resins. The residual Cs concentrations (post-elution) were about an order of magnitude less than the design limit following AP-101 processing. The buffer between residual Cs and the design limit narrowed to a factor of five following the AZ-102 processing.

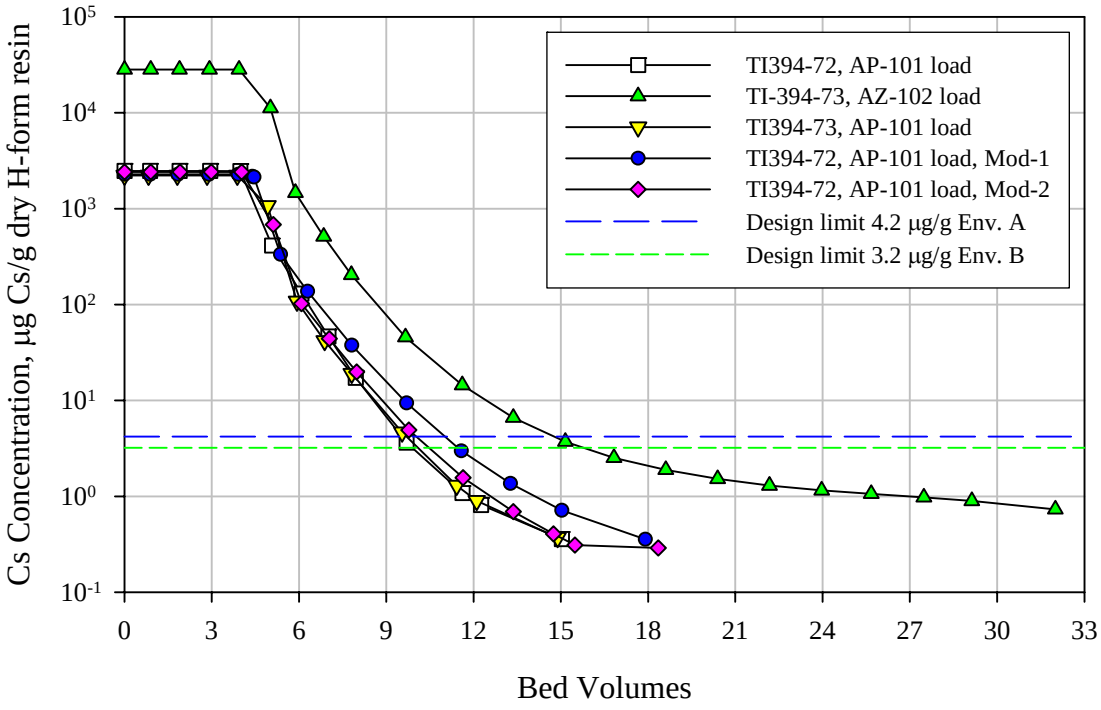


Figure 14.16. Residual Cs on Resin as a Function of Elution Volume

The data plotted in the preceding load and elution figures are given in Table 14.15 through Table 14.17. Trace quantities of precipitates were observed in several eluate samples as identified in the data tables. In most cases, the precipitates were observed after several days (i.e., not immediately) and were orange and flocculent, with the general appearance of $Fe(OH)_3$. In most cases, the precipitate appeared in the fourth BV processed sample, consistent with the pH transition through the neutral range. The precipitates generated from the Blue column process test resulted in orange flocculent material at 4 BVs and white material at 4.4 and 4.5 BVs. The different precipitate characteristics of this test may be associated with the fractionation process where small subsamples were collected in the 4- to 5-BV interval instead of the larger 1-BV increments for the other concurrent tests. The white precipitate was probably associated with aluminum and lead hydroxides. Precipitates were not observed in the Green column eluate samples where AZ-102 simulant was processed.

The WTP will need to address whether precipitate formation will be an issue, especially in regard to the piping configuration following the resin bed. In the current test suite, eluate was collected in discreet increments. As analytes were removed from the ion exchanger during the elution process, they were (somewhat randomly) collected in an increment where pH was near neutral, that is, where iron and aluminum will precipitate as hydroxides. The observed quantities were small. The total precipitate quantity is expected to scale up with the resin bed and processing. High flowrates may be sufficient to entrain the precipitates and carry them to the eluate collection tank. Continued elution will drop the fluid pH in the lines, which should dissolve any precipitated matter.

Table 14.15. Effluent Cs Concentrations During Loading and Elution, Wave 4b P1-RF Pretreated Resins

TI394-72 (MB 6C-370/745) White Column					TI394-73 (BSC-3380-3-0201) Yellow Column				
Feed		Elution			Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]
5.5	<1.13E-3	0.88	1.93E-2	2.47E+3	5.2	<1.12E-3	0.83	6.18E-3	2.21E+3
9.8	<1.09E-3	1.90[b]	1.99E-2	2.47E+3	9.4	<5.19E-4	1.82[b]	1.07E-2	2.21E+3
16.4	<1.27E-3	2.95[b]	4.53E-2	2.47E+3	15.7	<1.06E-3	2.84[b]	2.51E-2	2.21E+3
31.2	<1.33E-3	3.98[b]	4.95E-1	2.46E+3	29.9	3.46E-3	3.88[b]	3.62E-2	2.21E+3
37.5	1.63E-3	5.07	9.37E+1	4.13E+2	37.3	1.58E-2	4.92	6.46E+1	1.07E+3
44.7	6.87E-3	6.08	1.40E+1	1.30E+2	44.2	3.87E-2	5.93	5.66E+1	1.08E+2
52.8	2.76E-2	7.01	4.46E+0	4.64E+1	52.0	8.98E-2	6.88	4.12E+0	4.17E+1
67.4	2.37E-1	7.95	1.56E+0	1.72E+1	66.2	4.21E-1	7.81	1.45E+0	1.90E+1
73.9	5.48E-1	9.70	3.88E-1	3.62E+0	72.6	7.52E-1	9.55	4.86E-1	4.64E+0
80.7	1.13E+0	11.62	6.59E-2	1.08E+0	79.1	1.24E+0	11.42	1.05E-1	1.29E+0
94.7	7.81E+0	12.26	2.14E-2	8.11E-1	93.5	6.50E+0	12.11	3.39E-2	8.98E-1
122.3	3.84E+1	EDI[3]	8.14E-3	3.57E-1[f]	121.5	2.45E+1	EDI[d]	1.13E-2	3.69E-1[f]
135.1	5.55E+1				134.5	3.63E+1			
FD[c]	3.96E+1				FD[c]	2.43E+1			
FDI[d]	2.15E+0				FDI[d]	1.24E+0			

(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate (orange flocculent) observed in eluate.
(c) FD = feed displacement (0.1 M NaOH).
(d) FDI = water rinse following feed displacement.
(e) EDI = post-elution water rinse.
(f) Final residual Cs on the resin bed after elution and water rinse.

Table 14.16. Effluent Cs Concentrations During Loading and Elution, Wave 4b Modified Pretreated Resins

TI394-72 (MB 6C-370/745) Blue Column					TI394-72 (MB 6C-370/745) Pink Column				
Feed		Elution			Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]	Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]
8.0	<1.59E-3	0.88	2.03E-2	2.28E+3	6.2	<1.79E-3	0.90	2.02E-2	2.41E+3
15.6	<1.33E-3	1.84	2.10E-2	2.28E+3	11.5	<7.55E-4	1.91	2.18E-2	2.41E+3
31.2	1.44E-3	2.90	4.98E-2	2.28E+3	18.6	<1.71E-3	2.98	5.04E-2	2.41E+3
37.9	6.63E-3	3.91[b]	6.00E-2	2.28E+3	33.7	<8.19E-4	4.03[b]	6.24E-2	2.41E+3
45.8	2.64E-2	4.39[b]	1.26E+1	2.16E+3	40.1	<1.25E-3	5.12	8.15E+1	6.82E+2
53.6	9.18E-2	4.45[b]	2.41E+1	2.13E+3	48.2	<2.40E-3	6.08	3.08E+1	1.02E+2
67.1	5.36E-1	5.36	1.01E+2	3.33E+2	56.3	1.71E-2	7.04	3.11E+0	4.39E+1
73.5	1.12E+0	6.29	1.08E+1	1.37E+2	70.7	2.17E-1	7.98	1.31E+0	1.98E+1
80.7	2.18E+0	7.81	3.36E+0	3.75E+1	77.3	5.51E-1	9.78	4.26E-1	4.92E+0
96.0	1.19E+1	9.70	7.63E-1	9.38E+0	84.8	1.32E+0	11.64	9.28E-2	1.56E+0
123.5	3.57E+1	11.58	1.75E-1	2.97E+0	100.7	1.18E+1	13.37	2.59E-2	6.91E-1
137.1	5.90E+1	13.27	4.91E-2	1.36E+0	127.8	4.07E+1	14.75	1.06E-2	4.05E-1
FD[c]	4.01E+1	15.03	1.87E-2	7.13E-1	141.9	6.07E+1	15.49	6.71E-3	3.10E-1
FDI[d]	2.03E+0	EDI[e]	6.39E-3	3.56E-1[f]	FD[c]	4.15E+1	EDI[e]	3.67E-4	2.89E-1[f]
					FDI[d]	1.99E+0			

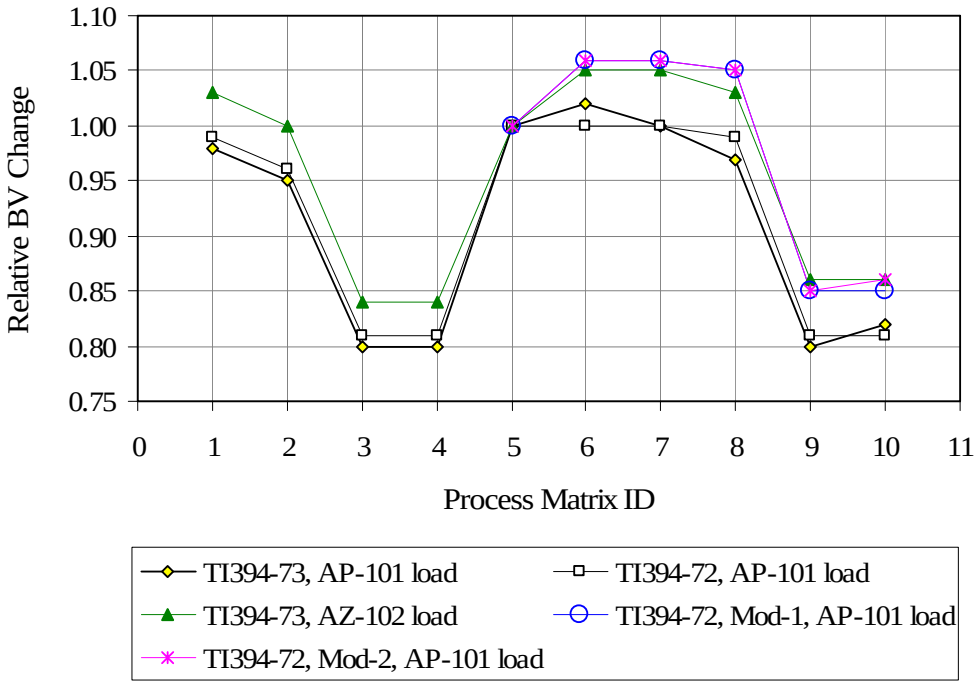
(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) Precipitate observed in eluate.
(c) FD = feed displacement (0.1 M NaOH).
(d) FDI = water rinse following feed displacement.
(e) EDI = post-elution water rinse.
(f) Final residual Cs on the resin bed after elution and water rinse.

Table 14.17. Effluent Cs Concentrations During AZ-102 Simulant Loading and Elution, P1-RF Pretreated Resin TI394-73

TI394-73 (BSC 3380-3-0201)				
Green Column				
Feed		Elution		
Cum. BV	% C/C_o	Cum. BV	C/C_o	Resin Cs Conc.[a]
5.6	<5.65E-4	0.90	2.84E-2	2.83E+4
29.2	<1.27E-3	1.89	4.89E-2	2.83E+4
42.8	<1.46E-3	2.92	1.15E-1	2.83E+4
67.4	<6.50E-4	3.94	1.47E-1	2.83E+4
85.1	8.31E-3	5.02	1.10E+2	1.12E+4
102.7	5.85E-2	5.88	7.89E+1	1.46E+3
110.7	1.28E-1	6.85	6.84E+0	5.11E+2
115.6	1.94E-1	7.80	2.25E+0	2.04E+2
122.5	3.81E-1	9.66	5.90E-1	4.55E+1
137.0	1.38E+0	11.61	1.11E-1	1.44E+1
143.4	2.34E+0	13.37	3.10E-2	6.58E+0
151.4	4.47E+0	15.16	1.12E-2	3.70E+0
159.2	7.00E+0	16.83	4.92E-3	2.52E+0
173.0	1.53E+1	18.61	2.51E-3	1.88E+0
179.6	2.11E+1	20.39	1.40E-3	1.52E+0
186.8	2.95E+1	22.17	8.78E-4	1.29E+0
195.0	4.16E+1	23.97	5.69E-4	1.15E+0
208.4	6.15E+1	25.67	3.63E-4	1.06E+0
215.3	7.22E+1	27.48	3.12E-4	9.77E-1
221.9	7.78E+1	29.13	3.57E-4	8.93E-1
FD[b]	5.78E+1	EDI[d]	3.96E-4	7.30E-1[e]
FDI[c]	4.63E+0			

(a) Concentration of Cs on resin as micrograms Cs/g dry H-form resin.
(b) FD = feed displacement (0.1 M NaOH).
(c) FDI = water rinse following feed displacement.
(d) EDI = post-elution water rinse
(e) Final residual Cs on the resin bed after elution and water rinse.

Figure 14.17 displays the in-column shrink-swell characteristics of the Wave 4b resins. The reference volume was defined as the volume of resin in the first regeneration condition following 0.5 M NaOH processing for the P1-RF pretreated resins. For the Mod-1 and Mod-2 pretreatments, the reference volumes were determined from the as-loaded condition just before the simulant feed processing. The relative BV shown is the BV observed divided by the reference volume. The Mod-1 and Mod-2 resins continued to expand 5% during simulant feed processing and remained static during feed displacement and water rinse. All resin tests resulted in ~20% volume contraction from the Na-form to the H-form. The measured volumes are provided in Table 14.18.



Matrix IDs
1: Loaded in column (Na-form)
2: DI water rinse
3: 0.5 M HNO_3
4: DI water rinse
5: 0.5 M NaOH or loaded in column (Na-form—Mod-1 and Mod-2)
6: AP101 or AZ-102 simulant
7: 0.1 M NaOH feed displacement
8: DI water
9: 0.5 M HNO_3
10: DI water

Figure 14.17. Shrink-Swell Characteristics of Wave 4b Resins

Table 14.18. Actual Resin Volume as a Function of Feed Matrix, Wave 4b

Feed matrix	Matrix ID	TI394-72 6C-370/745 White, mL	TI394-73 3380-3-0201 Yellow, mL	TI394-73 3380-3-0201 Green, mL	TI394-72 6C-370/745 Blue, mL	TI394-72 6C-370/745 Pink, mL
1 M NaOH load-in	1	20.7	20.4	20.4	--	--
DI rinse	2	20.1	19.8	19.8	--	--
0.5 M HNO_3	3	17.0	16.7	16.7	--	--
DI water	4	17.0	16.7	16.7	--	--
0.5 M NaOH[(a)]	5	21.0	20.7	19.8	20.4[(b)]	20.4[(b)]
Feed, AP101 simulant	6	21.0	21.0	20.7	21.7	21.7
0.1 M NaOH	7	21.0	20.7	20.7	21.7	21.7
DI water	8	20.7	20.1	20.4	21.4	21.4
0.5 M HNO_3	9	17.0	16.7	17.0	17.3	17.3
DI water	10	17.0	17.0	17.0	17.3	17.6
Dry resin mass (H-form)	na	6.16 g	7.19 g	7.19 g	6.14 g	6.14 g

(a) Reference volume, defined as 1-BV.
(b) Volumes were the NaOH-swelled volume as loaded into the ion exchange columns.

15.0 Resin Neutralization Test

Sodium exchange into H-form RF resin (and reverse reaction) requires time to accommodate the exchange processes associated with surface film and particle diffusion. The total time required for the equilibrium exchange had not been quantified. The speed of resin neutralization with 0.5 M NaOH was of interest with respect to understanding flowrate requirements through the resin bed in both hydraulic- and full-scale testing and operations.

The spherical RF resin titration curve (from Na-form to acid form) was evaluated. The acid consumption during resin conversion was calculated in another attempt to determine optimal volumes of 0.5 M NaOH and 0.5 M nitric acid required for bulk-contact resin conversion. The resin conversion rate was evaluated from bulk contact of a resin sample with the appropriate contact solution as a function of pH change with time.

15.1 Experimental

The TI394-62 (Microbeads 5E-370/641) resin from Wave 3 testing was selected for study. Two 8.0-mL aliquots of pretreated H-form resin were soaked in 5 BVs (40-mL) 0.5 M NaOH overnight. The Na-form resin had a settled BV of 10.1 mL. The contact solution was replaced with fresh 6 BVs (60-mL) 0.5 M NaOH for an additional 30-min soak. Most of the 0.5 M NaOH was removed and 6 BVs (60 mL) DI water were added to each sample.

A 40-mL fraction of water was removed from one sample leaving a contact-solution volume of ~24 mL (including the 40% interstitial void space of the packed resin bed) with the resin.[(a)] The sample was transferred to a titration workstation with a magnetic stirrer, recording pH meter, and burette. One-mL increments of 0.5 M HNO_3 were added to the slurry with continuous stirring. The pH was monitored with an Orion 3-STAR pH benchtop meter (Thermo Electron Corporation, Beverly, MA) equipped with an Orion ROSS Ultra™ pH electrode (81-02BNUWP). The pH meter had a logging capability allowing pH to be recorded as a function of time. Acid was added incrementally, allowing up to a nominal 30-min equilibration time. The titration process was spread over a 2-day period because the pH equilibration was slow. The titration parameters are provided in Table 15.1.

(a) The test plan indicated that a pH indicator should be added to the solution. A scoping study was conducted with an indicator. The resin color in the agitated solution masked the indicator color. A pH probe provided a superior evaluation of solution pH conditions.

Table 15.1. Titration Parameters

Volume Added,[(a)] mL	Cumulative Sum, mL	Date and Time	Volume Added,[(a)] mL	Cumulative Sum, mL	Date and Time
2.00	2.00	7/12/05 11:00		(continued)	
2.00	4.00	7/12/05 11:00	1.05	22.00	7/12/05 16:50
1.00	5.00	7/12/05 11:34	1.00	23.00	7/13/05 8:37
1.00	6.00	7/12/05 11:49	1.00	24.00	7/13/05 8:53
1.00	7.00	7/12/05 12:09	1.00	25.00	7/13/05 9:15
2.00	9.00	7/12/05 13:13	1.00	26.00	7/13/05 9:32
1.00	10.00	7/12/05 13:27	1.00	27.00	7/13/05 9:46
1.10	11.10	7/12/05 13:39	1.00	28.00	7/13/05 10:07
1.35	12.45	7/12/05 13:49	1.00	29.00	7/13/05 10:35
0.55	13.00	7/12/05 14:00	1.00	30.00	7/13/05 10:57
1.00	14.00	7/12/05 14:11	1.00	31.00	7/13/05 11:11
1.00	15.00	7/12/05 14:25	1.00	32.00	7/13/05 11:24
1.00	16.00	7/12/05 14:38	1.00	33.00	7/13/05 11:35
1.00	17.00	7/12/05 14:48	0.50	33.50	7/13/05 11:41
0.95	17.95	7/12/05 14:59	0.50	34.00	7/13/05 11:43
1.05	19.00	7/12/05 15:54	0.50	34.50	7/13/05 11:47
1.00	20.00	7/12/05 16:07	0.50	35.00	7/13/05 11:49
0.95	20.95	7/12/05 16:24	0.90	35.90	7/13/05 11:52

Titrant was 0.5 M HNO_3.

Two follow-on tests evaluated the speed of bulk resin conversion. One test was conducted to investigate the Na-form resin conversion rate. The contact solution (pH 12.7) was removed from the 10-mL aliquot of Na-form resin. The resin was rapidly placed into 18-mL 0.5 M HNO_3 using minimal contact solution volume to transfer the resin. Concurrently, the slurry was vigorously mixed, and the pH was monitored as a function of time. The reverse process was tested with the titrated acid form resin. In this test, 7 mL of 0.5 M NaOH were placed in a beaker, and the H-form resin was added at once. Because the conversion processes were rather slow, the comparison test with reagents only, as specified in the test plan, was deleted from the test scope.[(a)]

The Na-form resin conversion testing was conducted in the presence of the BNI R&T lead and was conducted according to his specifications. It was decided after this test that the experimental design did not capture the test objectives. Further resin neutralization results were obtained during hydraulic test operations.[(b)]

(a) TP-RPP-WTP-368, Section 5.6.8, Step 12 was deleted per direction of the R&T lead.

(b) ST Arm, DL Blanchard, Jr., KP Brooks, BJ Cook, JM Cuta, SK Fiskum, Z. Hontz, C Isackson, AA Schepmoes, and DE Wallace. *Progress Report on Laboratory Scale Hydraulic Testing of Spherical Resorcinol Formaldehyde Ion Exchange Resins*. WTP-RPT-138, August 2005. Battelle—Pacific Northwest Division, Richland, WA.

15.2 RF Resin Titration

The RF resin was shown to equilibrate fairly slowly through the pH region 11.5 to 2. The neutralization process results, titrating from Na-form resin to H-form with incremental addition of 0.5 M HNO_3, are provided in Figure 15.1. The resin was characteristic of a slow buffer. Adding acid resulted in initial low solution pH; however, the resin slowly exchanged the Na^+ for H^+ causing the pH to drift up. This process and rate of exchange can be followed from evaluating the initial pH drop and the incremental pH change to the next drop in pH that corresponds to the next acid addition. Although a 15-min equilibration time was allowed between acid additions, the pH equilibration may not have been reached. The pH continued to change during the overnight break from pH 8 to pH 9. No clear inflection points were observed for this titration curve. As the neutralization progressed, the time to equilibrium increased from 1-min (see T = 4:00) to >30 min (see T = 8:30). The increasing time for establishing equilibrium was probably associated with the location of Na ions in the RF bead. Surface Na ions would be expected to exchange quickly whereas Na exchange in the particle may be limited by particle diffusion.

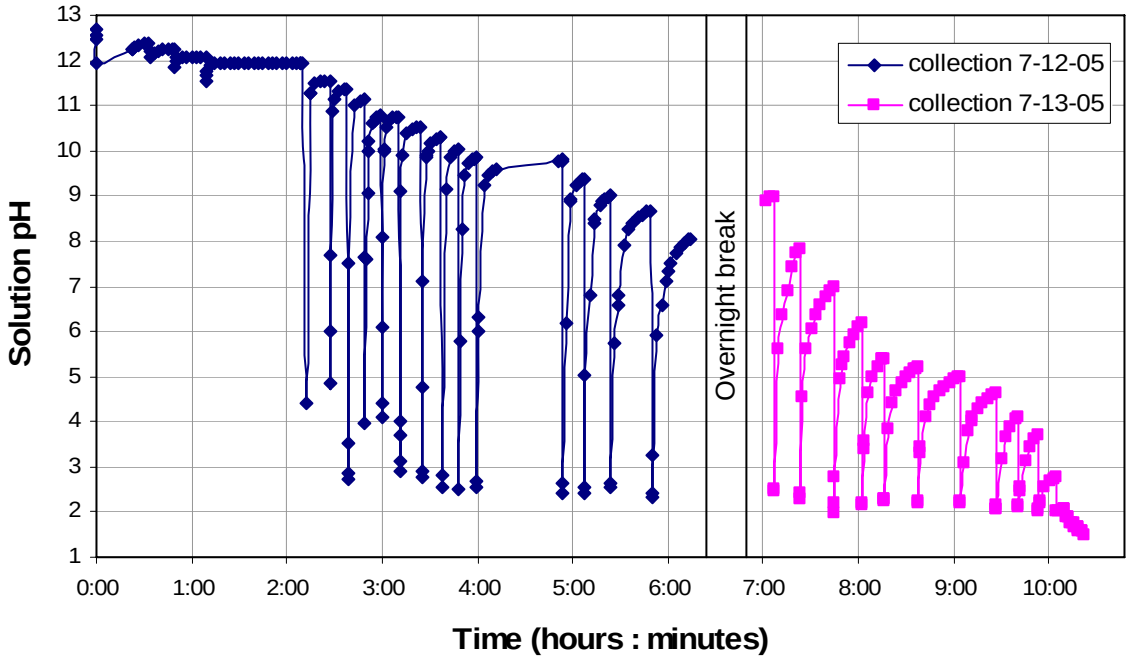


Figure 15.1. Titration of Na-form RF Resin as a Function of Time

The same data are shown in Figure 15.2 where the pH is given as a function of acid titration volume. The 10-mL Na-form resin required 33 mL of 0.5 M HNO_3 to reach the point of H^+ saturation of available exchange sites. After accounting for the neutralization of the starting interstitial solution, 15.4 meq of H^+ were required to react with the 10 mL Na-form resin (equivalent to 2.87 g dry H-form resin). Therefore, the Na-form resin capacity was calculated to be 1.54 meq/mL based on the titration results. This value was 15% lower than the 1.77 meq/mL Na-form resin determined from the eluate analysis during Wave 3 testing (see Section 11), and 7% lower than the 1.65 meq/mL Na-form resin determined from the Na consumption testing (Section 14.0). It was in good agreement with the 1.55 moles/L capacity previously reported by Arm et al. (2005). The observed 15% variation in RF resin capacity is within the uncertainty of the analytical methods.[(a)]

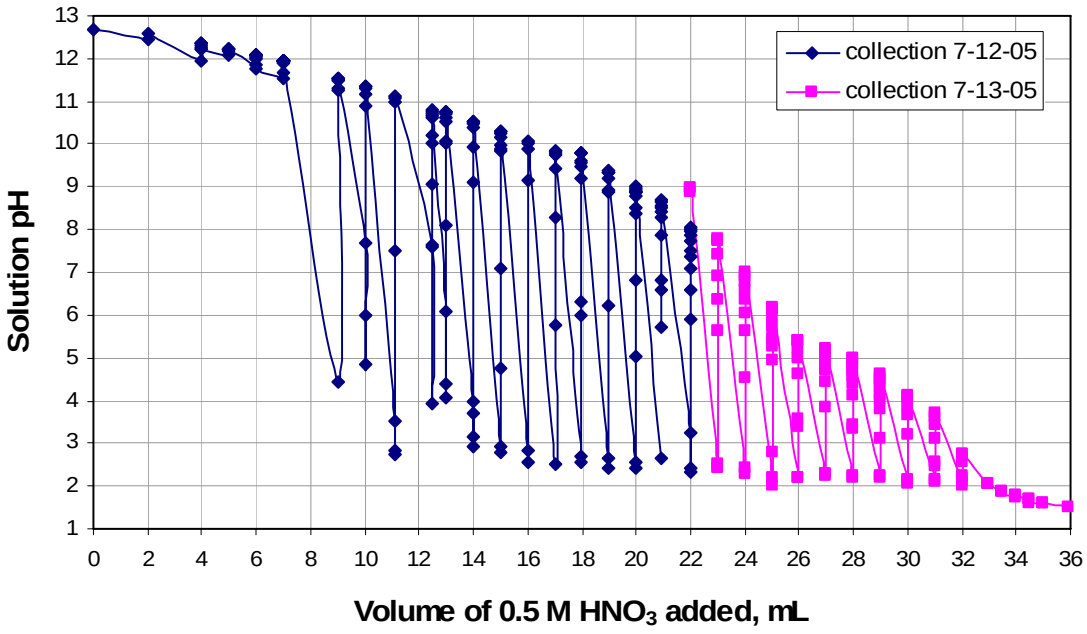


Figure 15.2. Titration of Na-Form RF Resin as a Function of Acid Volume

(a) Analytical uncertainties (associated with Na and hydroxide analyses) were generally reported to be within ±15%.

The timed resin neutralization test results, both H-form and Na-form conversions, are shown in Figure 15.3 (linear scales) and Figure 15.4 (log time scale). The H-form resin converted to Na-form resin more slowly and less completely than the opposite conversion of Na-form to H-form resin.

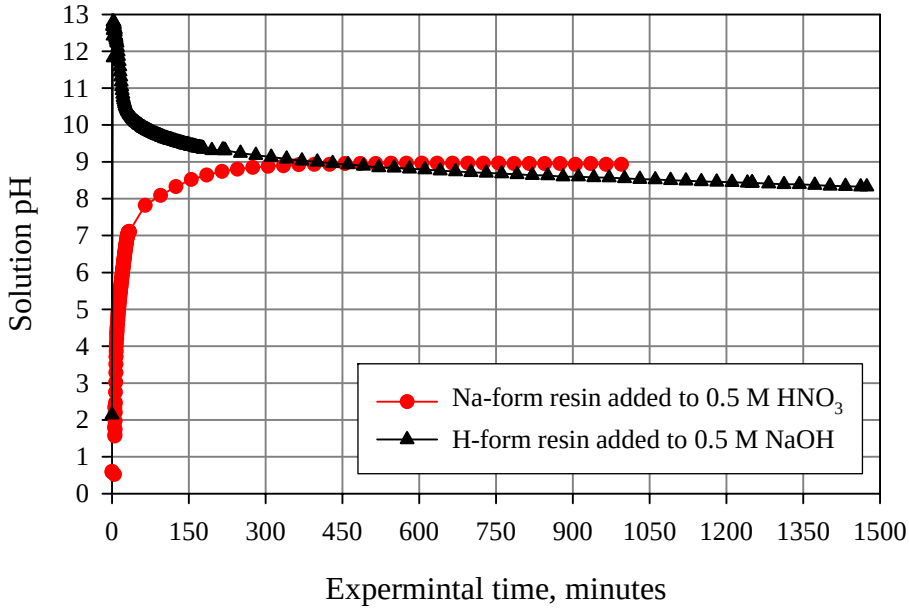


Figure 15.3. Resin Neutralization with Bulk Solution Contact (Linear Scales)

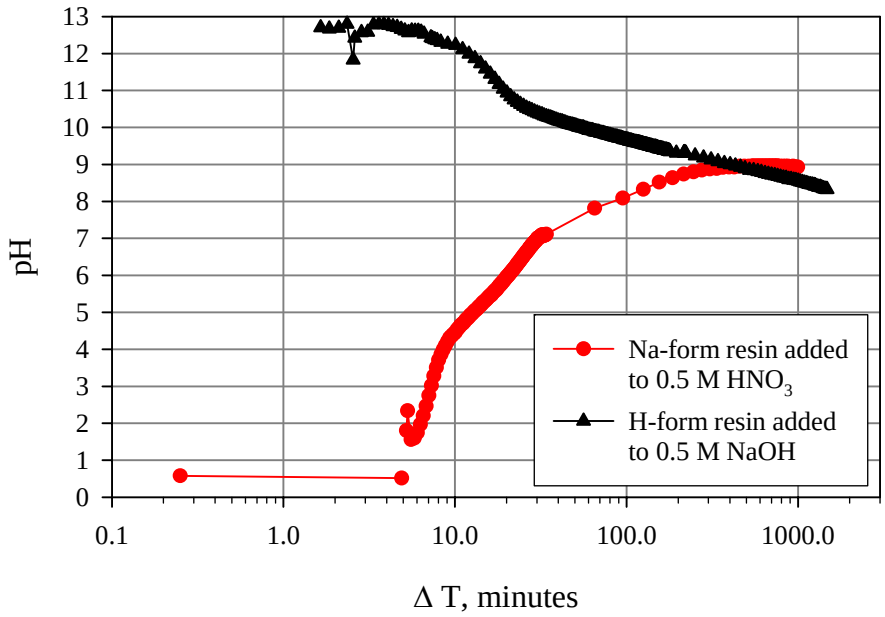


Figure 15.4. Resin Neutralization with Bulk Solution Contact (Log Time Scale)

In the case of the Na-form resin addition to 0.5 M HNO_3, a final solution reached the equilibrium pH 9 after 300 min equilibration time. As the Na-form resin exchanged Na^+ for H^+, the contact solution pH rapidly increased from acid to base, slowing after the first 10 minutes. Even after the pH 7 was reached, the exchange reaction still continued. The decreasing rate may be associated with the decreasing acid strength (through exchange or consumption). The Na-form resin (representing 15 meq Na) appeared to exchange all of the available 9 meq of acid H^+ in solution plus additional protons donated from water.

In the case of the H-form resin addition to 0.5 M NaOH, the final solution pH did not drop below pH 8; after the initial 300-min contact time, the solution continued a slow, constant drift downward from pH 9 to pH 8. The H-form resin (representing 15 meq H^+) did not exchange all available 3.5 meq of Na (pH remained basic). This indicated that a larger excess of NaOH was required to drive the exchange of Na ions in solution for H ions on the resin at a reasonable reaction rate. The slower H+ to Na+ exchange rate may also be limited by the intra-particle diffusion rate; the H-form resin bead is smaller than the swollen Na-form resin bead, and thus the intra-particle void space in the H-form resin is smaller.

16.0 Test Resin Summary Results and Comparisons

This section summarizes and compares the physical-property and ion exchange results from the various test waves. Waves 1 through 2b represent resin products developed under changing production conditions in an effort toward optimization. Resins BRF-14, TI394-11 (BSC 3380-2P-0100), and TI394-15 (Microbeads 5C-370/522) were synthesized under similar conditions. Resins TI394-16 (Microbeads 5C-370/523) and TI394-61 (Microbeads 5E-370/639) were synthesized under similar conditions. Waves 3 (specifically TI394-62) through 4b represent resins prepared under one set of manufacturing conditions to evaluate resin production scalability and reproducibility. Resin TI394-73 (BSC 3380-3-0201), however, was prepared using slightly altered curing steps and underwent an additional cleaning step to remove fines.[(a)] Considerations are suggested for a spherical RF resin purchase specification.

16.1 Physical Properties

Resin colorations were similar (maroon or brick red) on receipt with the notable exception of BRF-16, which was tan colored. Particle morphologies were uniformly spherical with few broken or abnormal pieces and very little fines. All unconstrained resin expansions from the as-received H-form to the relaxed H-form were ~30% (one swell-shrink cycle of the as-received resin). Overall unconstrained resin expansions, from the as-received H-form to the pretreated Na-form, were typically 55% to 60%. Therefore, a 65-gal production batch of spherical RF resin will provide a 100-gal Na-form resin bed. The pretreated resins demonstrated a 20 to 25% volume contraction from the Na-form to the H-form during ion exchange processing. Table 16.1 summarizes the selected physical properties for all tested resins.

Most resins had similar H-form skeletal densities and similar Na-form skeletal densities. Settled bed densities varied slightly; the BSC resins tended to exhibit higher bed densities than the Microbeads resins. The PSD analysis results demonstrated the resins achieved a narrow size distribution.

Two different vendors successfully manufactured six large-volume production batches[(b)] of the spherical RF resin.[(c)] The large-volume resin preparations (Waves 3, 4a, and 4b) exhibited generally reproducible physical properties. Two exceptions in consistency were observed. The Wave 4b resins contained a slightly higher fraction of large particles than were observed in previously analyzed samples; overall average particle size variation from batch to batch was within 11% (m_v basis, worst-case). As already noted, the BSC resins exhibited a higher bed density than the Microbeads resins.

(a) The manufacturing conditions were communicated directly from Microbeads to the R&T lead.
(b) The 65-gal production batch (H-form resin) represents ~100-gal pretreated Na-form resin using an expansion factor of ~55%.
(c) The Wave 4b BSC resin production experienced difficulties that were (mostly) resolved by additional resin cleanup. See Section 14.0 for discussion.

Table 16.1. Selected Physical Properties Summary

Test Wave	PNWD Resin ID	Manufacturer	Resin Lot Number	Production Lot Size	Average Na-form Resin Bead Diameter, μm			Skeletal Density, g/mL		Bed Density, g/mL	
					m_v	m_n	m_a	H-form	Na-form	H-form[a]	Na-form[b]
Wave 1	BRF-14	SINTEF	BRF-14[c]	<1 L	598	538	575	1.442	1.577	nm	0.329
	BRF-15	SINTEF	BRF-15[c]	<1 L	601	545	580	1.441	1.632	nm	0.342
	BRF-16	SINTEF	BRF-16[c]	<1 L	777	708	754	1.440	1.632	nm	0.190
	BRF-17	SINTEF	BRF-17[c]	<1 L	619	555	595	1.440	1.608	nm	0.332
Wave 1b	420	Microbeads	PS-420	<1 L	nm	nm	nm	nm	nm	0.391	0.301
	424	Microbeads	PS-424	<1 L	nm	nm	nm	nm	nm	0.393	0.297
Wave 2	TI394-11	BSC	3380-2P-0100	<1 L	457	408	437	1.497	na	0.428	0.347
	TI394-12	BSC	3380-2P-0101	<1 L	427	382	409	1.511	1.605	0.426	0.330
	TI394-13	BSC	3380-2P-0102	<1 L	nm	nm	nm	1.467	1.610	0.331	0.250
	TI394-14	BSC	3380-2P-0103	<1 L	nm	nm	nm	1.479	1.618	0.378	0.289
	TI394-5	Microbeads	PS-493	<1 L	nm	nm	nm	1.463	nm	0.364	0.284
	TI394-8	Microbeads	PS-501	<1 L	nm	nm	nm	1.472	1.593	0.301	0.235
Wave 2a	TI394-15	Microbeads	5C-370/522	<1 L	nm	nm	nm	nm	nm	0.346	0.278
	TI394-16	Microbeads	5C-370/523	<1 L	nm	nm	nm	nm	nm	0.319	0.260
Wave 2b	TI394-9	Microbeads	PS-502	<1 L	nm	nm	nm	nm	nm	0.326	0.257
	TI394-21	Microbeads	PS-518	<1 L	nm	nm	nm	nm	nm	0.339	0.261
	TI394-17	Microbeads	PS-513	<1 L	nm	nm	nm	nm	nm	0.373	0.285
	TI394-20	Microbeads	PS-517	<1 L	nm	nm	nm	nm	nm	0.373	0.288
	TI394-18	Microbeads	PS-514	<1 L	nm	nm	nm	nm	nm	0.336	0.261
	TI394-19	Microbeads	PS-515	<1 L	nm	nm	nm	nm	nm	0.338	0.266
Wave 3	TI394-61	Microbeads	5E-370-639	50 gal	439	394	421	1.477	1.614	0.356	0.293
	TI394-62	Microbeads	5E-370-641	75 gal	452	406	434	1.477	1.628	0.368	0.297
Wave 4a	TI394-64	Microbeads	5J-370/686	73 gal	446	405	431	1.468	1.640	0.369	0.298
	TI394-65	BSC	3380-03-0200	66 gal	437	398	423	1.471	1.637	0.408	0.334
Wave 4b	TI394-72	Microbeads	6C-370/745	74 gal	468	398	433	1.475	1.626	0.362	0.293
	TI394-73	BSC	3380-3-0201	66 gal	484	404	443	1.484	1.661	0.423	0.347

(a) Dry H-form resin mass in settled wet H-form resin bed; H-form resin volume was measured in a graduated cylinder.
(b) Dry H-form resin mass in a wet Na-form resin bed; Na-form resin volume was measured from resin height in the ion exchange column with an uncertainty of ~6% (i.e., the third significant figure is for information only).
(c) Resins exhibited a separate and distinct core structure.
Notes: nm = not measured

16.2 Column Performance

Various column-performance parameters of all resins tested with AZ-102 simulant and/or AP-101 simulant are summarized in Table 16.2. The SL-644 ion exchange performance parameters are also included for comparison.

Table 16.2. Selected Column Performance Results Summary

Test Wave	Resin ID	Resin Lot #	Onset[d] Cs BT, BV	Contract Limit Cs BT, BV	50% Cs BT, BV	Cs Loading, mg Cs/g H-Form Resin	Residual Cs, μg Cs/g H-Form Resin
AZ-102 Simulant							
A-222[a]	SL-644[e]	C-01-11-05-02-35-60	80	103	185	39.6	3.5
A-222[a]	#3	#3	75	93	143	25.8	0.34
Wave 1	BRF-14	BRF-14	45	68	170	25.4	0.64
Wave 4b	TI394-73	3380-3-0201	68	95	200	28.3	0.73
AP-101 Simulant							
Wave 1	SL-644[e]	C-01-11-05-02-35-60	30	73	220	5.4	0.96
Cycle 2	BRF-14	BRF-14	15	31	121	2.1	0.14
	BRF-15	BRF-15	5	30	120	2.0	0.13
	BRF-16	BRF-16	0	<5	~55	1.6	0.0049
	BRF-17	BRF-17	15	27	122	2.0	0.16
Wave 1b	420	PS-420	20	45	110	nm	nm
	424	PS-424	24	42	100	nm	nm
Wave 2	TI394-11	BSC-3380-2P-0100	15	36	113	1.88	0.16
Cycle 1	TI394-12	BSC-3380-2P-0101	30	50	121	2.05	0.17
	TI394-13	BSC-3380-2P-0102	20	37	83	2.04	0.15
	TI394-14	BSC-3380-2P-0103	20	39	92	1.87	0.15
	TI394-5	PS-493	15	33	102	2.11	0.15
	TI394-8	PS-501	30	41	100	2.41	0.12
Wave 2a	TI394-15	5C-370/522	15	40	98	2.06	0.14
	TI394-16	5C-370/523	30	48	106	2.40	0.16
Wave 2b	TI394-9	PS-502	30	51	109	2.46	0.21
	TI394-21	PS-518	30	53	105	2.38	0.18
	TI394-17	PS-513	30	48	113	2.25	0.16
	TI394-20	PS-517	30	52	113	2.27	0.14
	TI394-18	PS-514	32	54	107	2.41	0.19
	TI394-19	PS-515	30	54	110	2.42	0.16
Wave 3	TI394-61	5E-370-639	35	67	139	2.57	0.40
Cycle 1	TI394-62	5E-370-641	30	57	135	2.50	0.35
Wave	TI394-63	5E-370-641	30	54	122	2.34	0.22[c]
3a[b]	TI394-63	5E-370-641	30	55	124	2.31	0.14
Wave 4a	TI394-64	5J-370/686	38	58	132	2.41	0.31
	TI394-65	3380-3-0200	38	61	146	2.33	0.31
Wave 4b	TI394-72	6C-370/745	37	61	131	2.42	0.37
	TI394-73	3380-3-0201	30	52	148	2.11	0.36

(a) Fiskum et al. (2004).
(b) Processing (using the 16-cycle [at SRNL] post-tested RF resin) represented the 17th process cycle.
(c) Upflow elution.
(d) The onset of breakthrough was defined as the BVs processed before the C/C_o starts to increase.
(e) The SL-644 particle size distribution was ~18 to 40 mesh (425 to 1000 microns) wet Na-form (Fowley et al. 2003).
Notes: nm = not measured

Figure 16.1 graphically summarizes breakthrough (onset, contract limit, and 50%) performance with the RF resins in the AZ-102 simulant feed matrix. The SL-644 is also shown as a benchmark reference. The BRF-14 contract-limit breakthrough occurred sooner than that of RF Resin #3 and was attributed to its larger particle size (598 μm vs 501 μm, respectively). The scaled-up resin product (TI394-73) performed similarly to the baseline resin, SL-644. The baseline RPP-WTP Cs ion exchange operations[(a)] indicate that 20 to 40 BVs of Envelope B wastes will be processed in one load cycle (at 1.1 BV/h). The TI394-73 resin approached the contract limit at 95 BVs (1.5 BV/h), indicating that the resin will easily accommodate the Envelope B wastes.

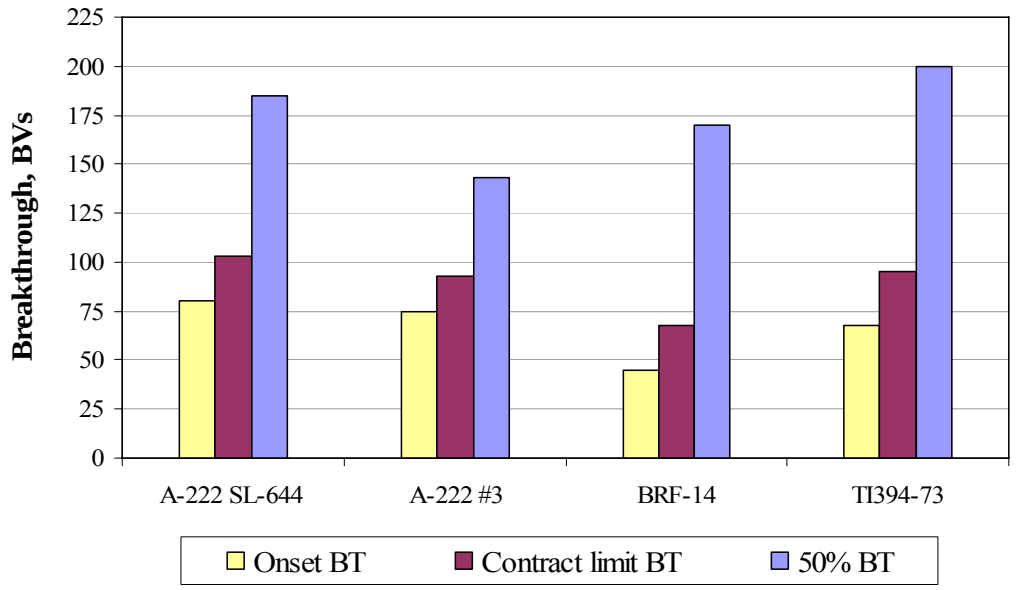


Figure 16.1. BVs to Onset, Contract Limit, and 50% Cs Breakthrough (BT) with AZ-102 Simulant (1.5 BV/h Flowrate)

The AP-101 tank waste matrix contains the highest K concentration of the Hanford tank waste suite and thus presents what is anticipated to be the worst-case scenario for Cs ion exchange processing on RF resin. Figure 16.2 graphically shows the breakthrough performances of the various RF test formulations in the AP-101 simulant feed matrix. The SL-644 is also shown as a benchmark reference. The scale-up production-lot resins resulted in generally consistent Cs ion exchange load qualities. The spherical RF Cs breakthrough performances with AP-101 simulant were not as high as that of SL-644.

(a) JW Olson. 2001. *System Description for Cesium Removal Using Ion Exchange – System CXP*, 24950-PTF-3YD-CXP-0001, Rev. A, Bechtel, Richland, WA.

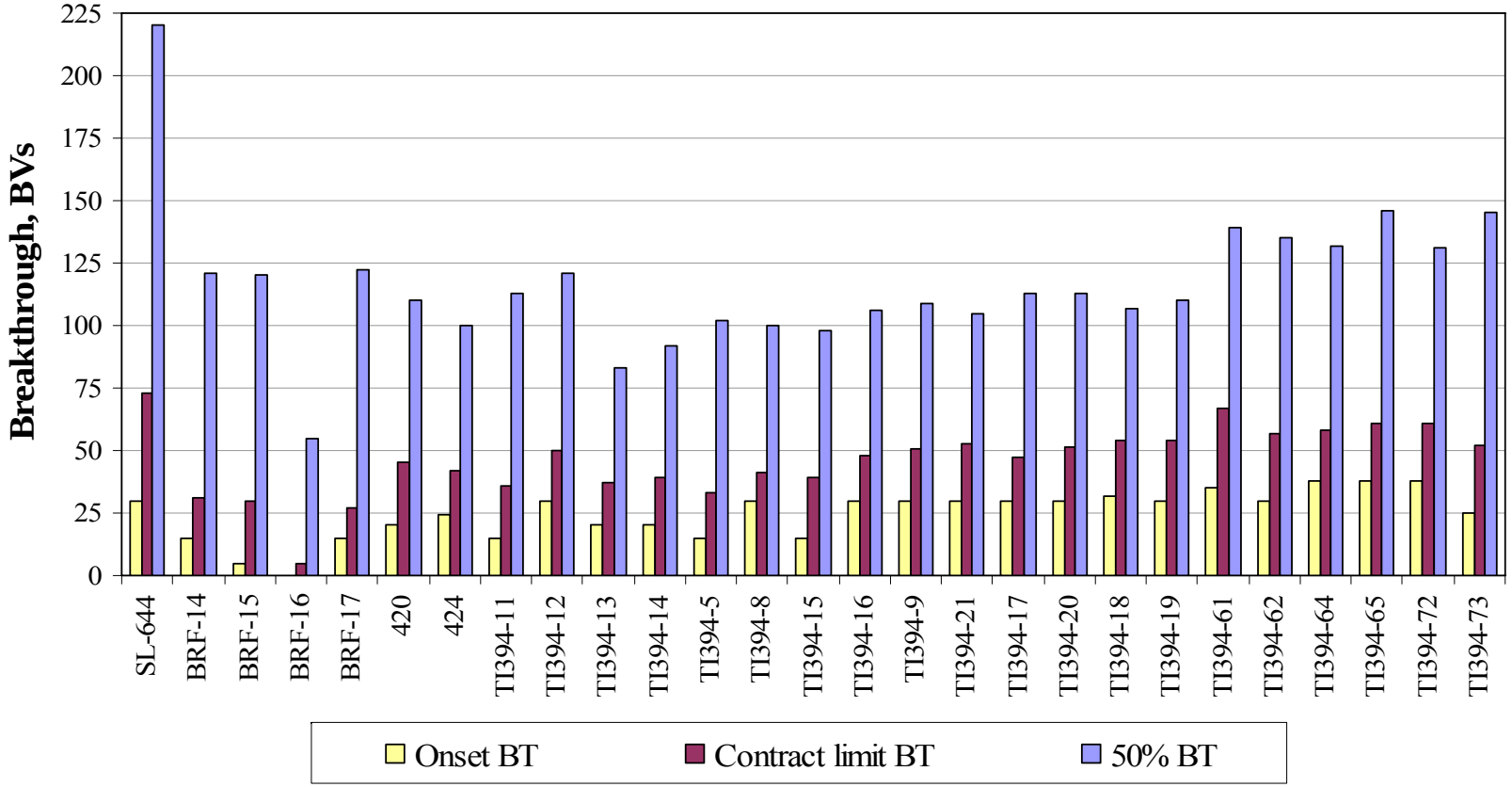


Figure 16.2. BVs to Onset, Contract Limit, and 50% Breakthrough (BT) with AP-101 Simulant, All Test Resins

The various RF resin formulations and curing conditions appeared to have the greatest effect on the Cs onset and contract-limit breakthroughs. The onset and contract limit breakthroughs varied by factors of 7 and 2, respectively (excludes BRF-16 from comparison). The improvement in the 50% Cs breakthrough (as a function of RF formulation and curing) was not as profound; the incremental improvement between TI394-13 (83 BVs), and TI394-73 (148 BVs) was 78%. The highest RF contract limit breakthrough represented by TI394-61 (67 BVs) came close to that of SL-644 (73 BVs) for the AP-101 matrix.

The total Cs load concentrations and post-elution residual Cs concentrations for AP-101 processing are shown in Figure 16.3. The resin mass basis is the dry H-form RF resin. The Cs load concentration increased modestly with manufacturing modifications/improvements. The higher trend in residual Cs concentration (starting with TI394-61) indicated that the elution was not as extensive and coincided with altered, less rigorous elution conditions. The TI394-61 and TI394-62 resin samples were eluted with 0.25 M HNO_3 and 0.4 M HNO_3, respectively, whereas the previous tests used 0.5 M HNO_3 eluant. The TI394-64 and TI394-65 resin samples were eluted with 15 BVs 0.5 M HNO_3, whereas the previous tests used ~21 BVs. The combination of higher eluant acidity and volumes resulted in more complete Cs removal from the resin. However, the results indicate that the use of lower eluant acidity and volumes was adequate to meet the process requirements with respect to design limits for residual Cs concentration on the resin. The effect of the elution volumes on the post-elution residual Cs concentrations is further shown in Figure 16.4

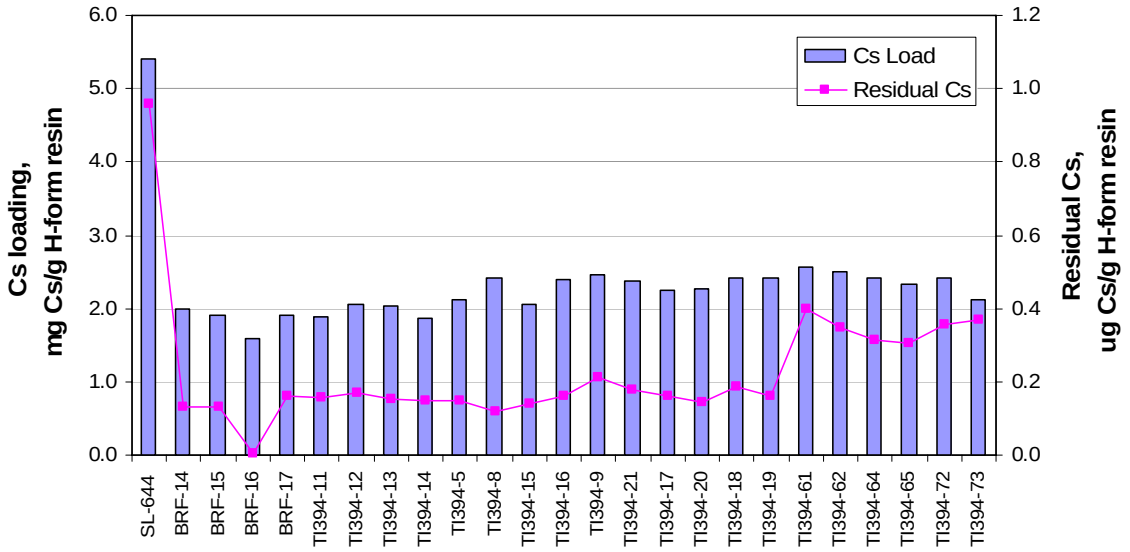


Figure 16.3. Loaded and Post-Elution Cs Concentration in Resin (AP-101 Simulant Load Matrix)

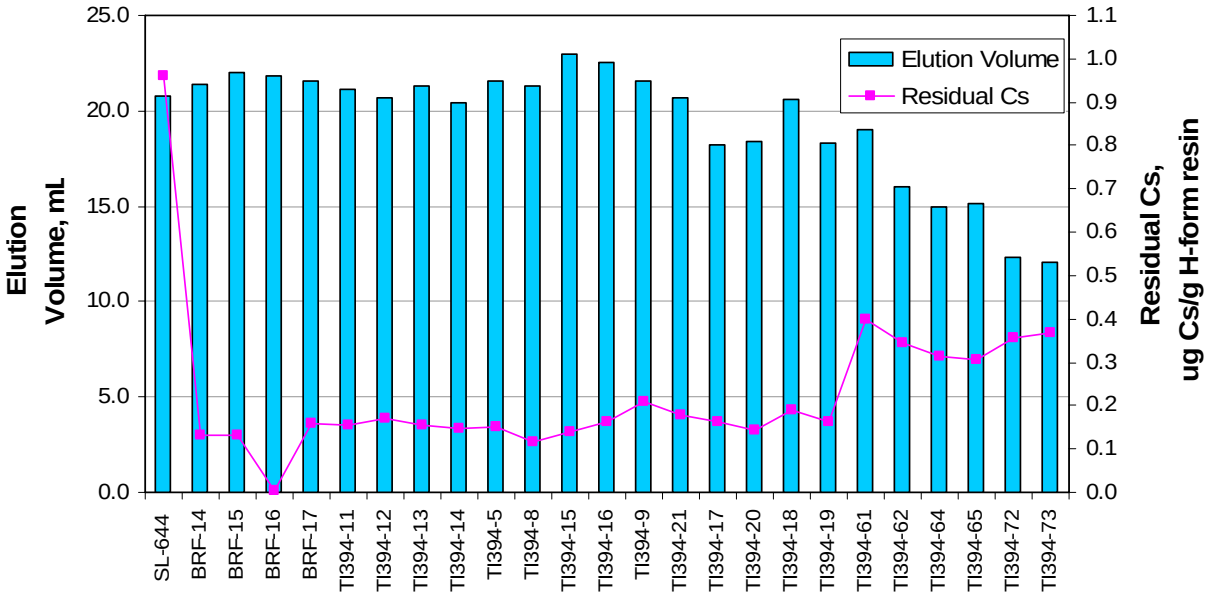


Figure 16.4. Elution Volumes and Post-Elution Cs Concentration in Resin (AP-101 Simulant Load Matrix)

The total Cs load concentrations and post-elution residual Cs concentrations for AZ-102 processing are shown in Figure 16.5. The resin mass basis is the dry H-form RF resin. The BRF-14 resin Cs loading achieved a concentration similar to that of the previously manufactured A-222 spherical RF test Resin #3. The TI394-73 resin demonstrated increased capacity resulting from product manufacturing refinements. Residual Cs (post-elution) was significantly reduced relative to the SL-644 resin. The elution volume and associated residual Cs is shown in Figure 16.6.

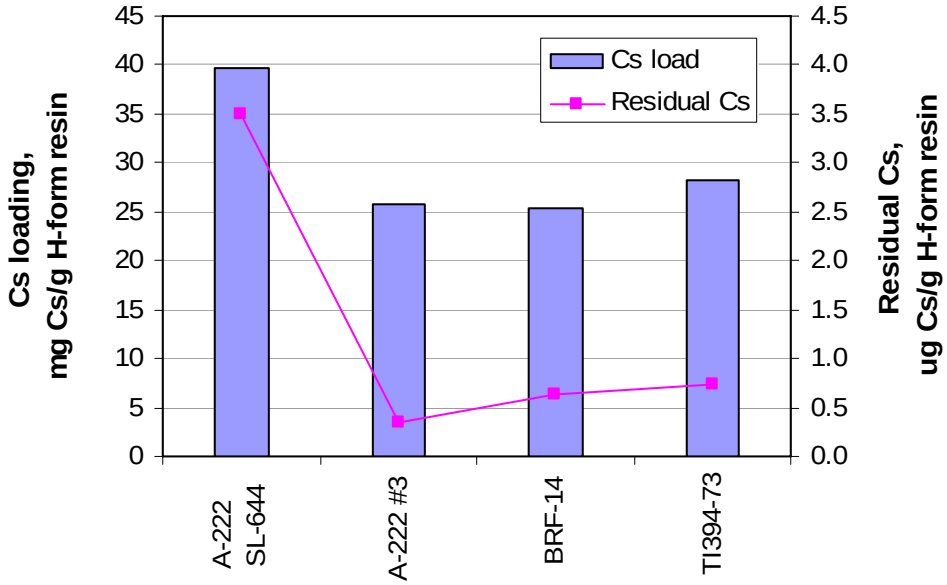


Figure 16.5. Loaded and Post-Elution Cs Concentration in Resin (AZ-102 Simulant Load Matrix)

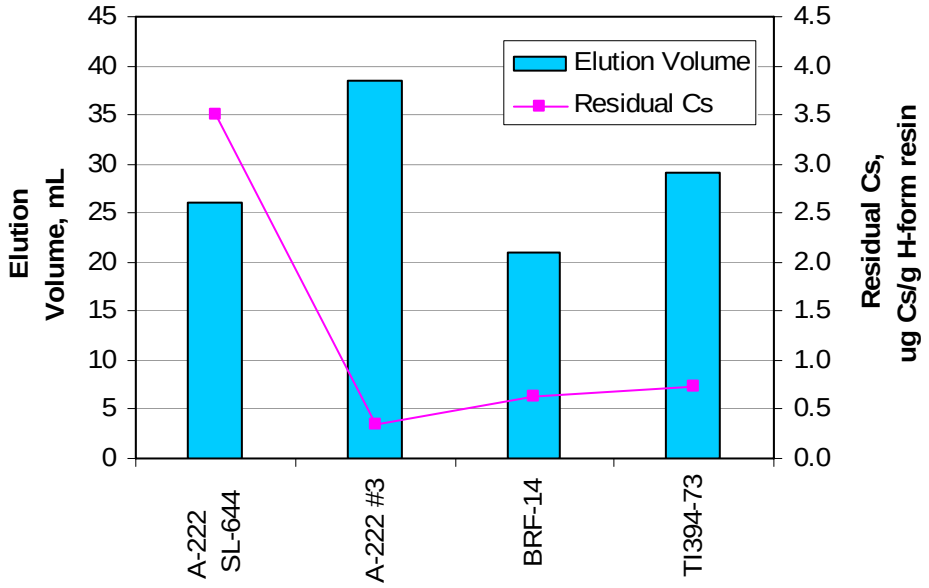


Figure 16.6. Elution Volumes and Post-Elution Cs Concentration in Resin (AZ-102 Simulant Load Matrix)

16.3 Post-Elution Cs Leakage

The Cs leakage into the final product was estimated from second-cycle processing associated with Wave 2 and Wave 3. The nominal Cs concentrations in the effluent samples collected from the second process cycle were compared to those of the first process cycle over the first ~12 BVs (Wave 2) and ~30 BVs (Wave 3) processed. These were compared to the residual Cs loading in the resin bed. Results are summarized in Table 16.3.

Table 16.3. Post-Elution Cs Leakage Effects

Test Wave Resin Lot #	Resin ID	Cycle 1 Avg. [Cs], µg/mL	Cycle 1 Residual Cs µg/g Resin	Cycle 2 Avg. [Cs], µg/mL	Effluent Cs Increase Factor[(a)]	Effluent [Cs] Increase / Residual [Cs][(b)]
Wave 2						
PS-493	TI394-5	<7.3E-5	0.15	4.3E-4	>5.9	>2.4E-3
PS-501	TI394-8	<5.3E-5	0.12	3.9E-4	>7.5	>2.9E-3
BSC-3380-2P-0100	TI394-11	<8.8E-5	0.16	5.9E-4	>6.7	>3.2E-3
BSC-3380-2P-0101	TI394-12	<6.4E-5	0.17	4.3E-4	>6.7	>2.1E-3
BSC-3380-2P-0102	TI394-13	<9.0E-5	0.15	5.0E-4	>5.5	>2.6E-3
BSC-3380-2P-0103	TI394-14	<8.6E-5	0.15	3.2E-4	>3.7	>1.6E-3
Wave 3						
5E-370-639	TI394-61	4.6E-5	0.40	8.6E-4	19	2.1E-3
5E-370-641	TI394-62	7.0E-5	0.35	6.3E-4	9.0	1.6E-3

(a) Effluent increase factor = Cycle 2 [Cs] / Cycle 1 [Cs].
(b) Net effluent Cs concentration increase (µg/mL) divided by residual Cs on the resin (µg/g).

The effluent Cs concentrations increased a factor of 4 to 19 between Cycle 1 and Cycle 2. The net increase in Cs effluent concentrations were normalized to the residual Cs on the RF resins; they averaged 2.3 E-3 ± 26 % μg/mL Cs in the effluent per μg/g Cs residual on the resin. In order to reach the contract limit of 0.091% C/C_o (~0.0055 μg/mL) for AP-101 processing, the eluted resin would need to contain no more than 2.4 μg residual Cs per g H-form resin. Actual leakage of Cs into the follow-on process cycle may be a function of resin particle diameter and RF cross-linking.

16.4 Application to the RPP-WTP

Conservatisms were built into the small-column test design to bound the Cs ion exchange performance through the second column in series in the RPP-WTP. The RPP-WTP nominal design condition provided in the system description[(a)] includes a three-column sequence with feed processing rates at 2.2 BV/h (15 gpm for 415-gal resin beds) for Envelope A and C wastes. The flowrate was bounded at 3 BV/h (conservatively faster than design) and the column sequence bounded at two columns (conservatively fewer than design). The effluent Cs concentration from the second column may be predicted from the Cs effluent concentration from a single column operated at half the flowrate, 1.5 BV/h (which in turn provides a lower superficial velocity). The test matrix contained the highest K concentration (0.7 M) expected in the various feeds (bounding the K ion competitor effect).

The current test processing with AP-101 simulant was conducted in a single-column format at a flowrate of 1.5 BV/h (0.16 cm/min) through the first ~80 BVs. The Cs breakthrough profiles simulated a two-column system processing at 3 BV/h. The higher residence time at the expected WTP flow rate (2.2 BV/h) is expected to provide sharper breakthrough curves with greater volumes processed before reaching initial cesium breakthrough. Lower K concentrations will result in a smaller competitor ion effect, increasing BVs processed to the contract limit. In addition, the higher superficial flow rates caused by the greater column height in the in the WTP are expected to provide sharper breakthrough curves even when the residence time is the same as in the test columns.

The design basis for Envelope B processing indicates it will process at a 1.1 BV/h flowrate corresponding to 0.55 BV/h relative to a combined two-column system. Current testing with simulant AZ-102 was conducted similarly to the AP-101 at the bounding flowrate of 1.5 BV/h. As with the Envelope A simulant, the slower flowrate and faster superficial velocity are expected to improve the Cs ion exchange performance.

16.5 Considerations for Resin Purchase Specification

Key factors of RF resin related to physical properties and ion exchange performance should be considered in a purchase specification (or acceptance testing). These are summarized and discussed in Table 16.4. Note, these values are related to measured performance, not WTP needs. The WTP personnel will need to evaluate purchase specification parameters relative to the plant process requirements.

(a) JW Olson. 2001. *System Description for Cesium Removal Using Ion Exchange – System CXP*, 24950-PTF-3YD-CXP-0001, Rev. A, Bechtel, Richland, WA.

Table 16.4. Suggestions for a Spherical RF Purchase Specification

Parameter	Criteria	Method	Discussion
Narrow particle size distribution	35 to 50 mesh	Microtrac or equivalent	Current tests show that 95% of H-form resin particles can be prepared in the "relaxed" 302-µm to 504-µm range (nominally 35 to 50 mesh) (TI394-65).
Bed density	>0.35 g dry H-form resin/mL H-form settled resin volume	Gravimetric	Modeling and current tests show that low bed density decreases the total Cs loading and reduces the process volume to 50% breakthrough (compare Wave 4a resin results). Scale-up resin manufacturing shows that 0.35-g/mL resin bed densities can be achieved.
Morphology	Uniformly spherical allowing up to 2% damage or misshapen	Microscopy	Current tests show that the resin can be prepared with only a small number of broken pieces or deformities (TI394-65 two deformities per 900 beads, TI394-64 12 broken pieces per 900 beads).
Color bleed	Minimal coloration of 1 M NaOH after overnight soak	Solution contact	Polymer should be cured and not show tendency to leach from bead. This test can be conducted in conjunction with pretreatment supporting the column test.
Cs Exchange Performance	50 BVs processed before contract limit BT; 120 BVs processed before 50% BT with AP-101 simulant	Column test	Current tests show that ≥ 57 BVs of AP-101 simulant can be processed before reaching contract limit breakthrough at 1.5 BV/h; >130 BVs can be processed before 50% Cs breakthrough. Column tests should be conducted according to a reproducible protocol.

Absent from the proposed purchase specification is the skeletal density measure. Skeletal density measured with water pycnometry did not vary much through the varying formulations tested; no extra value is anticipated from its measure.

17.0 Summary and Conclusions

A 2-year program of resin performance testing was conducted to support the alternate ion exchange resin (spherical RF) test program. Initial resin testing was conducted with prompt feedback on Cs ion exchange performance to the BNI R&T lead, which supported an iterative process to better define manufacturing conditions that would provide optimal product performance. After ion exchange performance evaluation of 20 test samples, a formulation was promulgated by the R&T lead and Microbeads for scale-up production testing. Testing was continued to demonstrate resin performance from six scale-up production batches. Additional Cs ion exchange loading and/or elution testing activities were included to evaluate oxygen attack on resin performance, elution flow direction on residual Cs content, and minimal resin pretreatment on subsequent Cs ion exchange performance. Ion exchange performances for the spherical RF and SL-644 were compared.

The efficacy of spherical RF resin production in 65-gal lot sizes (equivalent to 100-gal pretreated Na-form resin volume) has been demonstrated based on physical property measurements and Cs loading and elution results from small-scale (20-mL resin beds) column testing. The key conclusions from this work are as follows:

- Six ≥50-gal spherical RF production lots have been reproducibly prepared by two vendors. Four lots were prepared by Microbeads (Skedsmorkorset, Norway) and two lots were prepared by Boulder Scientific Corporation (Mead, CO).
- Morphology, determined in micrographs, showed that resin lots contained a highly spherical product with few deformations and fines.
- Tight PSDs were measured demonstrating good control and reproducibility in bead diameter.
    - The Na-form PSD range (low 5% to high 90%) was typically 300 μm to 500 μm (number basis).
    - The average Na-form (pretreated) particle diameters on a volume, number, and area basis from the six production lots were 454 μm (±18), 401 μm (±5), and 431 μm (±8), respectively.
- A ~20% volume shrinkage was observed upon converting pretreated resin from Na-form to H-form.
- H-form resin bed densities varied from 0.36 g/mL to 0.43 g/mL. Resins equilibrated in 0.5 M NaOH solution contained 0.29 to 0.35 g H-form resin per mL of resin bed. The BSC product densities were characteristically higher than the Microbeads product densities.
- Loading and elution testing with AP-101 simulant demonstrated that the spherical RF will meet the RPP-WTP plant Cs ion exchange processing requirements for the high-K waste feed (anticipated to pose the highest chemical performance challenge for RF application to Cs ion exchange).
    - The contract limit (0.091%) Cs breakthrough was reached after processing 59 BVs (±8%).
    - The 50% Cs breakthrough was reached after processing 139 BVs (±5%).
    - Cs was loaded onto the resin bed to a concentration of 2.4 mg Cs per gram H-form resin at ~50% breakthrough.

- Cs was eluted to the design-basis limit (4.2 μg Cs/g dry H-form resin mass) after processing typically ≤11 BVs 0.5 M HNO_3.

- Loading and elution testing with AZ-102 simulant demonstrated that the spherical RF will meet RPP-WTP plant Cs ion exchange requirements for Envelope B waste processing.
  - The contract limit (0.025%) Cs breakthrough was reached after processing 95 BVs.
  - The 50% Cs breakthrough was reached after processing 200 BVs. The current RPP-WTP flowsheet indicates 20 to 40 BVs of AZ-102 waste types will be processed in one cycle.
  - Cs was loaded onto the resin bed to a concentration of 28 mg Cs per gram H-form resin at ~50% breakthrough.
  - Cs was eluted to the design-basis limit (3.2 μg Cs/g dry H-form resin mass) after processing typically 16 BVs 0.5 M HNO_3.
- The equilibrium Na consumption of the RF resins, in meq/mL Na-form resin, was determined to range from 1.22 (at 0.05 M NaOH) to 1.60 (at 0.23 M NaOH). Elution analysis indicated the resin achieved 1.77 meq/mL Na-form resin (combined K and Na).
- Resin pretreatment variations showed that minimal processing (i.e., swell step only) will provide effective Cs ion exchange loading performance. No in-column cycling was required.
- Resin conversion testing showed that the Na-form resin readily releases Na in exchange for H ions; initial exchange occurs rapidly (within 30-min), but the exchange rate decreases with equilibrium reached in 5 h. The reverse exchange, H-form to Na-form, requires a longer equilibration time and a higher ionic Na^+ gradient to drive the exchange reaction.
- In-column and downflow processing of the spherical RF regeneration step was refined to the use of 6 BVs of 0.5 M NaOH at 3 BV/h. A contact time of at least 1 h with the regenerating solution was required to reach 90% resin volume expansion.
- Spherical RF resin oxygen consumption (indication-only measures) from oxygen-saturated 0.5 M NaOH was typically >80% (processing at ~3 BV/hr with a superficial velocity of ~0.3 cm/min). Oxidative degradation occurred from the top of the resin bed and migrated down with continued processing. Despite oxidative attack, the resin bed did not plug or otherwise fail. The average reduction in 50% Cs breakthrough was ~16% per mmole O_2 exposure per g H-form resin.
- Seventeen loading and elution cycles and associated chemical exposure resulted in 8 to 10% degradation in Cs breakthrough performance, corresponding to 14-18% reduction per mmole O_2 per gram resin.
- Upflow elution resulted in the Cs elution peak broadening (attributed to uneven flow through the resin bed and to mixing with the solution above the resin bed). The residual Cs concentration in the resin bed was slightly higher than the Cs concentration associated with downflow processing.
- The spherical RF ion exchange performance was compared to SL-644.
  - Cs ion exchange performance indicators for two tank waste simulants are shown in Table 17.1.

Table 17.1. Performance Comparison for Spherical RF and SL-644

Simulant	Flowrate, BV/h	Contract Limit Breakthrough, BVs		50% Breakthrough, BVs	
		RF	SL-644[(b)]	RF	SL-644[(a)]
AZ-102	1.5	95[(b)]	103	200[(b)]	185
AP-101	1.5	59[(c)]	73	139[(c)]	220

(a) SL-644 lot number C-01-11-05-02-35-60, 250-gal production batch, 18-40 mesh (425 – 1000 microns) wet screened fraction.
(b) RF lot number BSC 3380-3-0201, 400 ± 100 microns (Na-form, number basis).
(c) Average results from 4 scale-up RF lots, 400 ± 100 microns (Na-form, number basis).

Cs ion exchange performance was similar for the low-K AZ-102 simulant. The total Cs capacity of RF in the high-K AP-101 simulant was reduced relative to that of SL-644.

- Both spherical RF and SL-644 exhibited effects of extensive oxygen exposure. The SL-644 resin expanded with increasing oxygen exposure culminating in a 39% Na-form volume expansion followed by a net 24% volume contraction (27.6 mL to 21.0 mL) with a corresponding pressure drop of ≥10 psi and a permeability of <5E-14 m^2. The RF resin maintained its physical structure and fluid flow in the column; the top of the resin bed slowly darkened with increasing oxygen exposure.

Appendix A

Temperature Fluctuation Profiles During Ion Exchange Test Operations

Appendix A: Temperature Fluctuation Profiles During Ion Exchange Test Operations

Figures A.1 through A.11 show the test temperature fluctuations as a function of BVs processed for all test waves and cycles. Only one column from each process wave is shown except in the few cases where process times are extensively different. This was the case, for example, when AP-101 and AZ-102 simulants were processed; AZ-102 simulant processing was about 70 h longer than that of AP-101.

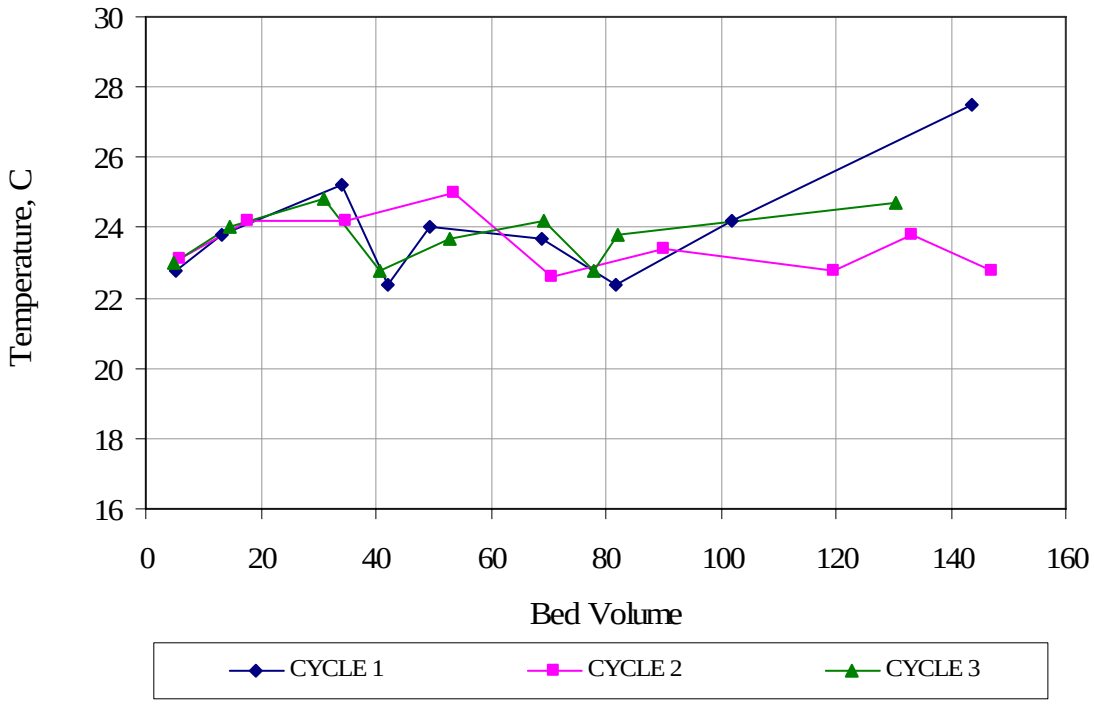


Figure A.1. Temperature Fluctuation During Wave 1 Processing with AP-101 Simulant Cycles 1-3

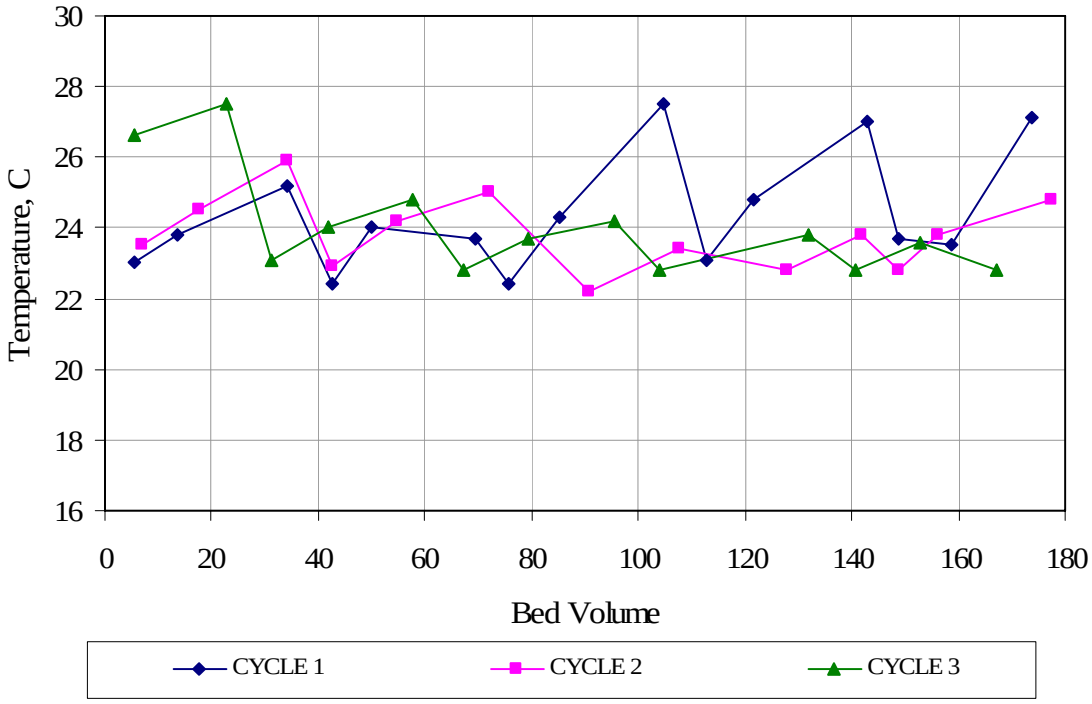


Figure A.2. Temperature Fluctuations During Wave 1 Processing with AZ-102 Simulant Cycles 1-3

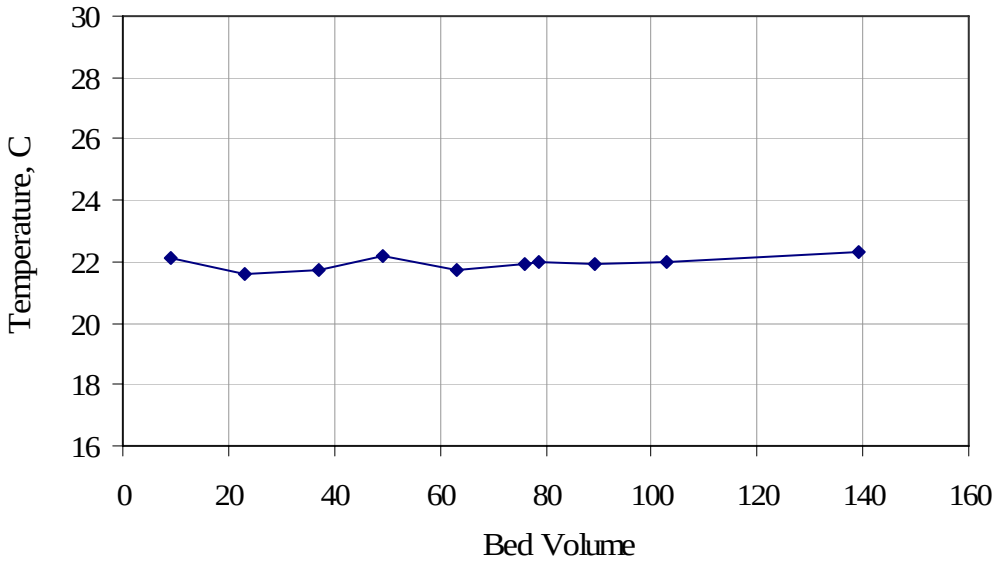


Figure A.3. Temperature Fluctuation During Wave 1b Processing with AP-101 Simulant

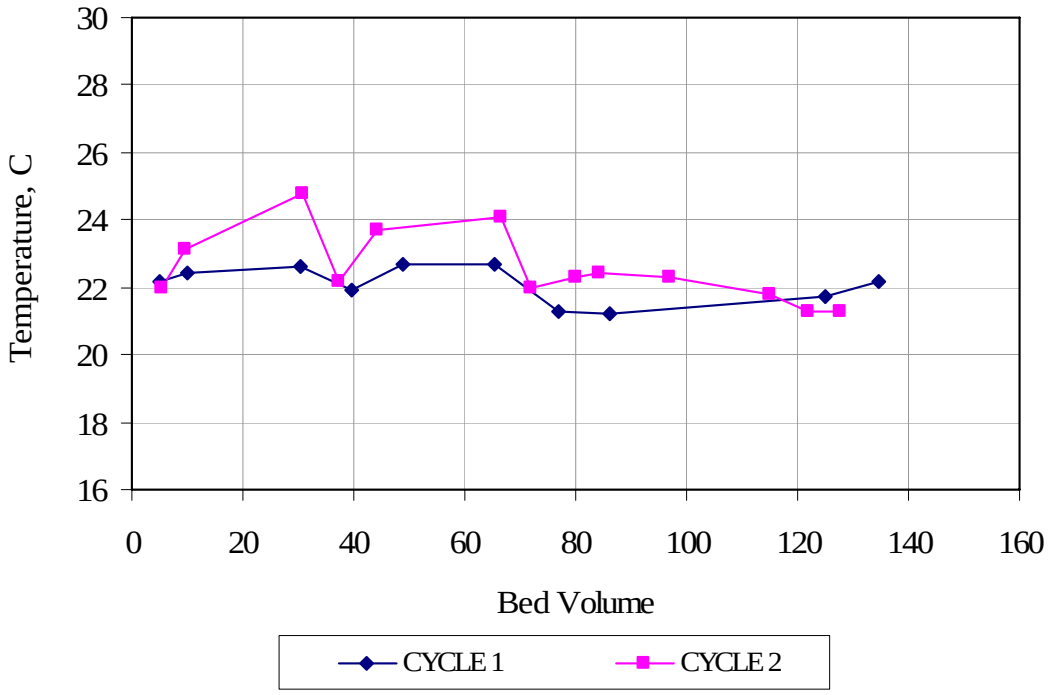


Figure A.4. Temperature Fluctuation During Wave 2 Processing with AP-101 Simulant Cycles 1 and 2

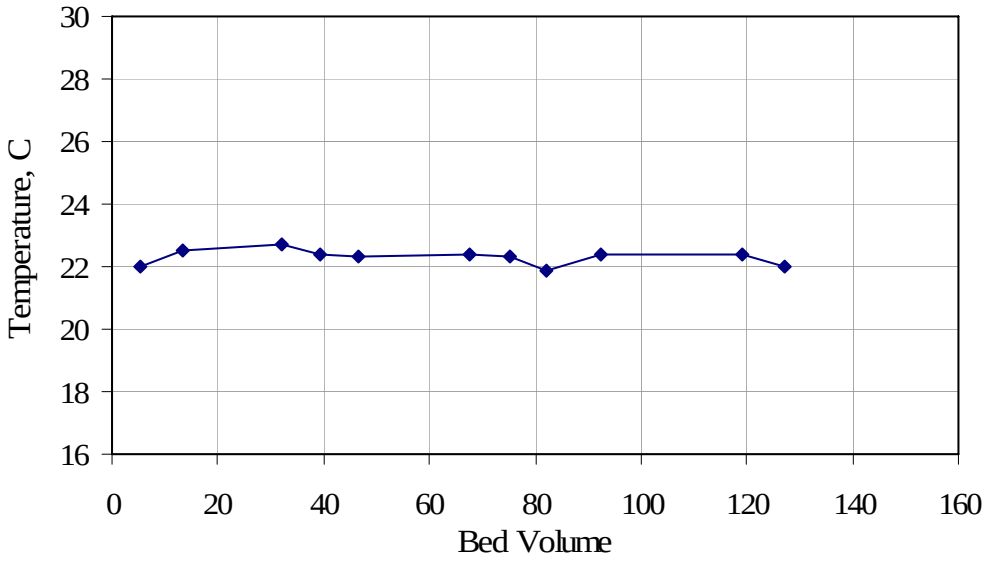


Figure A.5. Temperature Fluctuation During Wave 2a Processing with AP-101 Simulant

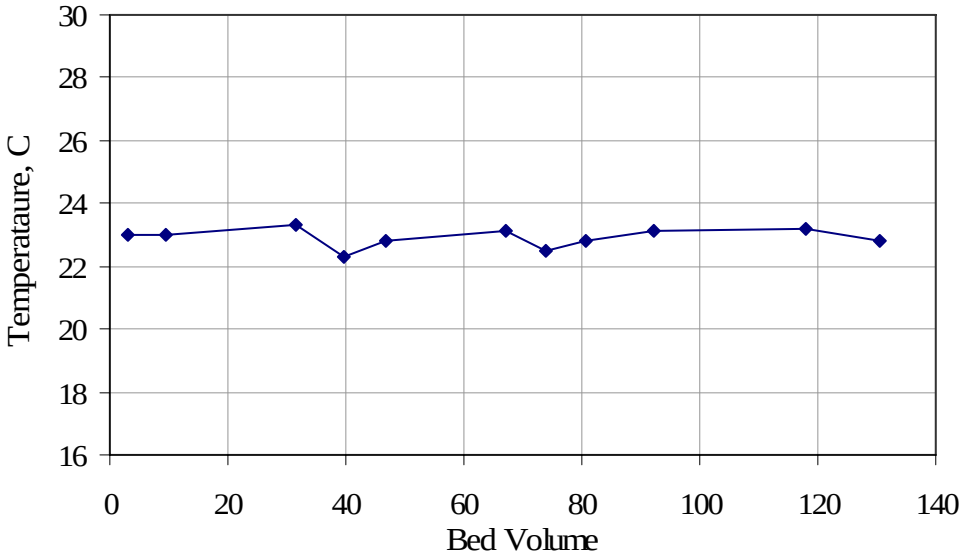


Figure A.6. Temperature Fluctuation During Wave 2b Processing with AP-101 Simulant

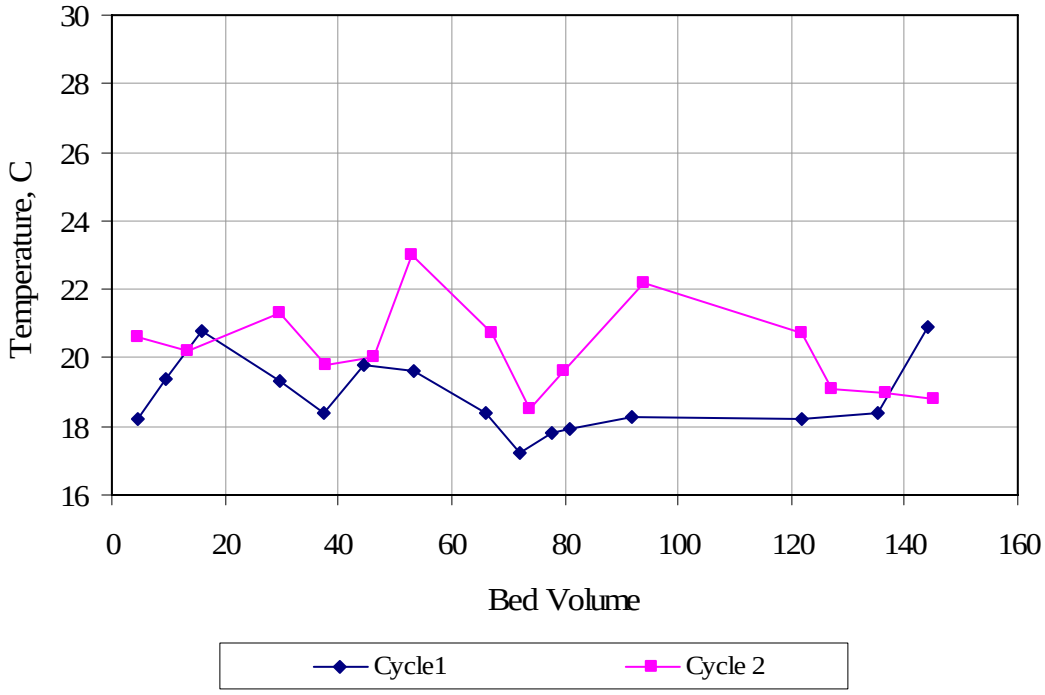


Figure A.7. Temperature Fluctuation During Wave 3 Processing with AP-101 Simulant Cycles 1 and 2

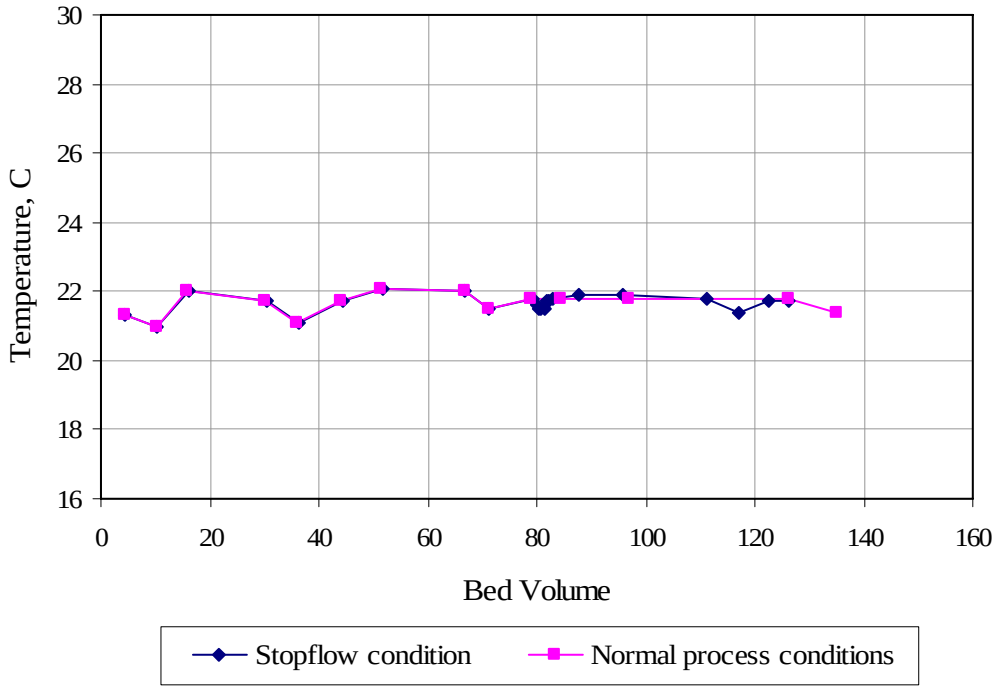


Figure A.8. Temperature Fluctuation During Wave 3a Processing with AP-101 Simulant

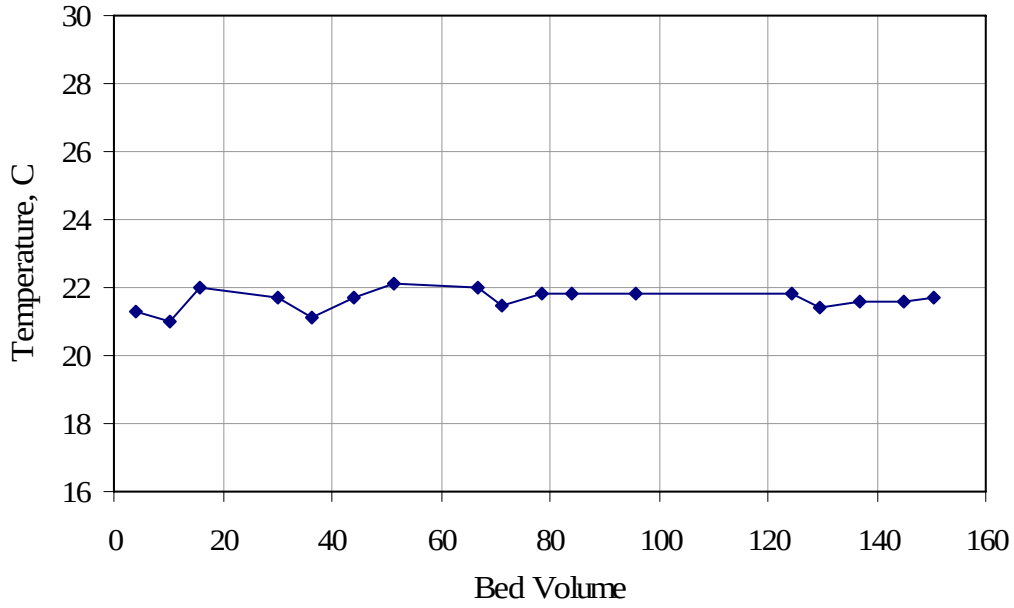


Figure A.9. Temperature Fluctuation During Wave 4a Processing with AP-101 Simulant

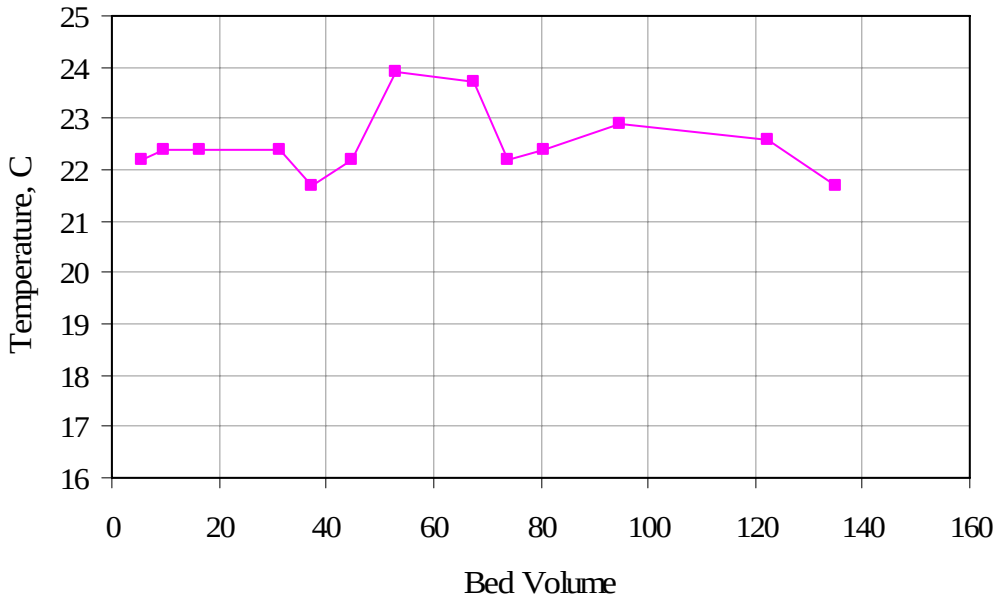


Figure A.10. Temperature Fluctuation During Wave 4b Processing with AP-101 Simulant

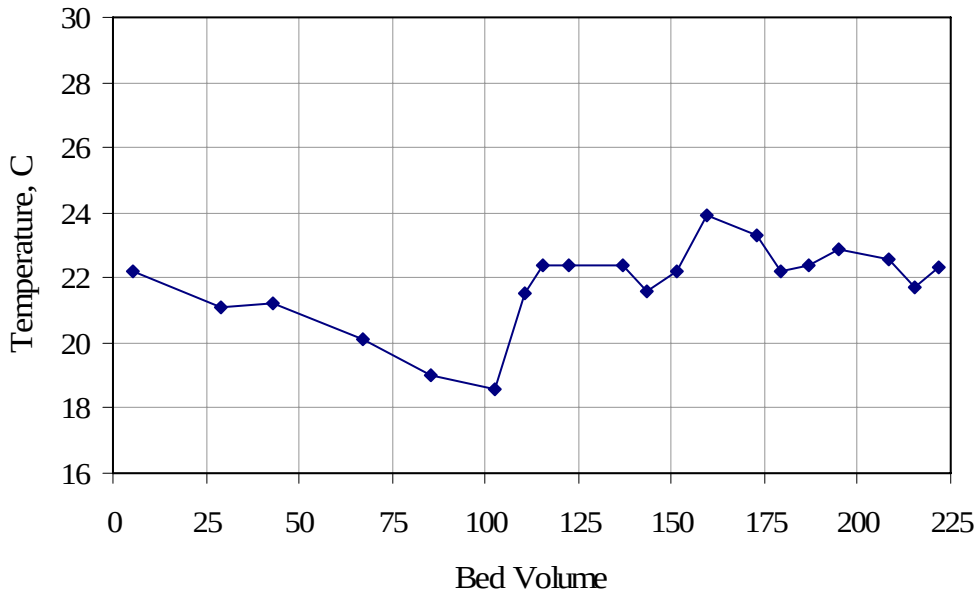


Figure A.11. Temperature Fluctuation During Wave 4b Processing with AZ-102 Simulant

Appendix B

Contract Limit Calculation for AP-101 and AZ-102

Appendix B: Contract Limit Calculation

AP-101

The ^{137}Cs contract limit in AP-101 (Envelope A) vitrification feed is discussed below.

Assumptions

1) Concentration of Na_2O in Env. A glass = 18.5% (=18.5 g Na_2O/100 g glass)
2) Fraction of Na in glass from tank waste = 0.96.
3) Glass density = 2.69 MT/m^3 (=2.69 g/mL)
4) Maximum ^{137}Cs in glass = 0.3 Ci/m^3 (= 0.3 Ci/1E+6 mL = 3E-7 Ci/mL)
5) AP-101 actual waste Na concentration = 5.64 M[(a)]
6) AP-101 actual waste ^{137}Cs concentration = 121 μCi/mL (decayed to 1/1/08)[(a)]

Na Loading in Glass

(18.5 g Na_2O/100g glass) * (1 mole Na_2O/62 g Na_2O) * (2 mole Na/mole Na_2O)* (23 g Na/mole Na) * (2.69 g glass/mL glass) * 0.96 = 0.354 g Na/mL glass

Maximum ^{137}Cs:Na in Glass

(3.0E-7 Ci ^{137}Cs/mL glass)/(0.354 g Na/mL glass) = 8.47 E-7 Ci ^{137}Cs/g Na

(8.47 E-7 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 1.95 E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in Feed

(1.95 E-5 Ci ^{137}Cs/mole Na) * (5.64 mole Na/L feed) = 1.10 E-4 Ci ^{137}Cs/L
= 110 μCi ^{137}Cs/L
= 0.110 μCi ^{137}Cs/mL

AP-101 Actual Waste Cs Fraction Remaining (C/C_o) Contractual Limit

(0.110 μCi ^{137}Cs/ mL) / (121 μCi ^{137}Cs / mL) = 9.09 E-4 C/C_o
= 0.091 % C/C_o

Decontamination Factor (DF) Contract Limit

1/9.09 E-4 C/C_o = 1100 C_o/C

(a) SK Fiskum, PR Bredt JA Campbell, OT Farmer, LR Greenwood, EW Hoppe, FV Hoopes, GJ Lumetta, GM Mong RT Ratner, CZ Soderquist, MJ Steele, RG Swoboda, MW Urie, and JJ Wagner. 2000. *Inorganic, Radioisotopic, and Organic Analysis of 241-AP-101 Tank Waste*, PNNL-13354, Pacific Northwest National Laboratory, Richland, WA.

AZ-102

The ^{137}Cs contract limit in AZ-102 (Envelope B) vitrification feed is discussed below.

Assumptions

1) Concentration of Na_2O in Env. B glass = 5% (=5 g Na_2O/100 g glass)[a]

2) For maximum ^{137}Cs concentration in glass, assume that all Na comes from the feed. If some Na is added to the vitrification feed, multiply the maximum ^{137}Cs value determined below by ratio of total Na:feed Na.

3) Glass density = 2.75 MT/m^3 (=2.75 g/mL)

4) Maximum ^{137}Cs in glass = 0.3 Ci/m^3 (= 0.3 Ci/1E+6 mL = 3E-7 Ci/mL)

5) AZ-102 actual waste Na concentration = 2.80 M[(b)]

6) AZ-102 actual waste ^{137}Cs concentration = 755 μCi/mL (decayed to 12/4/2011)[(b)]

Na Loading in Glass

(5 g Na_2O/100g glass) * (1 mole Na_2O/62 g Na_2O) * (2 mole Na/mole Na_2O)*
(23 g Na/mole Na) * (2.75 g glass/mL glass) = 0.102 g Na/mL glass

Maximum ^{137}Cs:Na in Glass

(3.0E-7 Ci ^{137}Cs/mL glass)/(0.102 g Na/mL glass) = 2.9 E-6 Ci ^{137}Cs/g Na

(2.9 E-6 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 6.8 E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in Feed

(6.8 E-5 Ci ^{137}Cs/mole Na) * (2.80 mole Na/L feed) = 1.9 E-4 Ci ^{137}Cs/L
= 0.190 μCi ^{137}Cs/mL

AZ-102 Actual Waste Cs Fraction Remaining (C/C_o) Contractual Limit

(0.19 μCi ^{137}Cs/ mL) / (755 μCi ^{137}Cs / mL) = 2.5 E-4 C/C_o
= 0.025 % C/C_o

Decontamination Factor (DF) Contract Limit

$1/(2.5\text{ E-}4\ C/C_o) = 4000\ C_o/C$

(a) This value is higher than recently communicated Na_2O glass loading requirement of 2.85%. Using the adjusted value and a waste Na loading factor of 0.96, the contract limit will adjust up to 0.046% C/C_o. Equivalent calculations for AZ-101 tank wastes result in similar Cs removal requirements.

(b) SK Fiskum, OT Farmer, LR Greenwood, ED Jenson, BM Oliver, RL Russell, CZ Soderquist, MJ Steele, RG Swoboda, MW Urie, and JJ Wagner. 2003. *Hanford Tank 241-AZ-102 Waste Concentration and Composition*. PNWD-3235, Battelle—Pacific Northwest Division, Richland, WA.

Appendix C

Simulant Feed Formulation

Appendix C: Simulant Feed Formulation

Table C.1 describes the input reagents to the AP-101 simulant. The full simulant preparation was described by Russell et al. (2003).

Table C.1. Reagent Masses for 1-L of AP-101 Simulant Solution

Compound Name	Formula	Formula Weight, g/mole	Amount Added (g) per 1-L Volume	Molarity
Sodium acetate	$NaCH_3CO_2$	82.03	2.029	2.47E-2
Sodium oxalate	$Na_2C_2O_4$	134	2.385	1.78E-2
Aluminum nitrate nonahydrate (60% solution)	$Al(NO_3)_3$-$9H_2O$	375.13	161.75	4.31E-1
Barium nitrate	$Ba(NO_3)_2$	261.35	0.000552	2.11E-6
Beryllium oxide	BeO	25.01	0.003247	1.30E-4
Cadmium nitrate tetrahydrate	$Cd(NO_3)_2$-$4H_2O$	308.47	0.004885	1.58E-5
Calcium nitrate tetrahydrate	$Ca(NO_3)_2$-$4H_2O$	236.16	0.04036	1.71E-4
Cesium nitrate	$CsNO_3$	194.92	0.008784	4.51E-5
Rubidium nitrate	$RbNO_3$	147.47	0.006091	4.13E-5
Copper nitrate trihydrate	$Cu(NO_3)_2$-$3H_2O$	241.6	0.005399	2.23E-5
Iron nitrate nonahydrate	$Fe(NO_3)_3$-$9H_2O$	404	0.01606	3.98E-5
Lead nitrate	$Pb(NO_3)_2$	331.226	0.02132	6.44E-5
Lithium nitrate	$LiNO_3$	69	0.002982	4.32E-5
Nickel nitrate hexahydrate	$Ni(NO_3)_2$-$6H_2O$	290.81	0.03488	1.20E-4
Zinc nitrate hexahydrate	$Zn(NO_3)_2$-$6H_2O$	297.47	0.02266	7.62E-5
Boric acid	H_3BO_3	61.83	0.08164	1.32E-3
Molybdenum oxide	MoO_3	143.95	0.01930	1.34E-4
Sodium chloride	NaCl	58.44	2.390	4.09E-2
Sodium fluoride	NaF	41.99	0.1180	2.81E-3
Sodium dihydrogen phosphate	$Na_2H_2PO_4$	119.98	1.492	1.24E-2
Sodium sulfate	Na_2SO_4	142.04	5.298	3.73E-2
Sodium nitrate	$NaNO_3$	84.99	60.00	7.06E-1
Potassium nitrate	KNO_3	101.11	20.02	1.98E-1
Sodium hydroxide (50% solution)	NaOH	40	238.4	5.96E+0
Tungstic acid	H_2WO_4	249.86	0.0344	1.38E-4
Sodium meta-silicate	Na_2SiO_3-$9H_2O$	284.2	1.234	4.34E-3
Sodium chromate	Na_2CrO_4	161.97	0.4735	2.92E-3
Sodium formate	HCOONa	68.01	1.614	2.37E-2
Sodium nitrite	$NaNO_2$	69	48.78	7.07E-1
Sodium carbonate	Na_2CO_3	105.99	20.03	1.89E-1
Potassium carbonate	K_2CO_3	138.21	35.52	2.57E-1

Table C.2 describes the input reagents to the AZ-102 simulant and preparation.

Table C.2. Reagent Masses for 1-L of AZ-102 Simulant Solution

Compound Name	Formula	Formula Weight, g/mole	Amount Added (g) per 1-L Volume	Molarity
Dissolve the following in 200 mL DI water and mix thoroughly.				
Aluminum nitrate nonahydrate	$Al(NO_3)_3$-$9H_2O$	375.13	18.82	5.02E-2
Boric acid	H_3BO_3	61.83	0.04956	8.02E-4
Calcium nitrate tetrahydrate	$Ca(NO_3)_2$-$4H_2O$	236.16	0.3594	1.52E-3
Cesium nitrate	$CsNO_3$	194.92	0.07412	3.80E-4
Potassium molybdenum oxide	K_2MoO_4	238.14	0.2626	1.10E-3
Potassium nitrate	KNO_3	101.1	14.50	1.43E-1
Strontium nitrate	$Sr(NO_3)_2$	211.63	0.000879	4E-6
Sodium acetate	CH_3COONa-$3H_2O$	136.08	2.756	2.03E-2
Ethylenediaminetetraacetatic acid, disodium salt dihydrate	$(HOOCCH_2)_2NCH_2CH_2N(CH_2COONa)_2$-$2H_2O$	372.24	0.5149	1.38E-3
n-(2-Hydroxyethyl)-ethylenediaminetriacetic acid	$HOCH_2CH_2CH_2N(CH_2CO_2H)CH_2CH_2N(CH_2CO_2H)_2$	278.26	0.1535	5.52E-4
Iminodiacetic acid	HN(CH2CO2H)2	133.10	0.428	3.22E-3
Citric acid monohydrate	$HOCOCH_2C(OH)(COOH)CH_2OOH$-$H_2O$	210.14	8.6116	4.10E-2
Sodium fluoride	NaF	41.99	4.050	9.64E-2
Sodium sulfate	Na_2SO_4	142.04	44.09	3.10E-1
Add the following slowly with good mixing.				
Sodium hydroxide	NaOH	40.00	21.37	5.34E-1
Sodium formate	HCOONa	68.01	12.51	1.84E-1
Sodium glycolate	$HOCH_2CO_2Na$	98.03	20.18	2.06E-1
Sodium oxalate	$Na_2C_2O_4$	134	7.777	5.80E-2
Sodium phosphate	Na_3PO_4-$12H_2O$	380.12	3.685	9.69E-3
Add nominally 300-mL DI water and mix thoroughly. Then add the following.				
Sodium chromate	Na_2CrO_4	161.97	4.330	2.67E-2
Sodium carbonate	Na_2CO_3	105.99	92.85	8.76E-1
Mix the solution thoroughly and add the following and mix.				
Sodium nitrate	$NaNO_3$	84.99	16.60	1.95E-1
Sodium nitrite	$NaNO_2$	69.00	82.09	1.19E+0
Add water to 1-L final volume and density of 1.24 g/mL.[(a)]				

(a) This AZ-102 simulant recipe was provided by SRNL staff via personal communication on 1/31/03.

Appendix D

Particle Size Distribution Printouts from the Microtrac S3000

Appendix D: Particle Size Distribution Printouts from the Microtrac S3000

The following pages are printouts from the Microtrac S3000 showing the PSD characteristics of selected scale-up production resins. The resins were pretreated through the P1-RF process so the particle sizes represent the "relaxed" bead size (as opposed to the "as-received" bead size). Table D.1 provides a cross-reference for test wave, lot number, and resin form to the Appendix D page.

Table D.1. Cross-Reference to Resin Identification and Form to PSD Printout Page

Wave number	PNWD Internal ID	Vendor	Lot number	H-form	Na-form
Wave 3	TI394-62	Microbeads	5E-370/641	m_v, page D.2 m_n, page D.3 m_a, page D.4	m_v, page D.5 m_n, page D.6 m_a, page D.7
Wave 4a	TI394-64	Microbeads	5J-370/686	m_v, page D.8 m_n, page D.9 m_a, page D.10	m_v, page D.11 m_n, page D.12 m_a, page D.13
	TI394-65	BSC	3380-3-0200	m_v, page D.14 m_n, page D.15 m_a, page D.16	m_v, page D.17 m_n, page D.18 m_a, page D.19
Wave 4b	TI394-72	Microbeads	6C-370/745	m_v, page D.20 m_n, page D.21 m_a, page D.22	m_v, page D.23 m_n, page D.24 m_a, page D.25
	TI394-73	BSC	3380-3-0201	m_v, page D.26 m_n, page D.27 m_a, page D.28	m_v, page D.29 m_n, page D.30 m_a, page D.31

Samples were analyzed in duplicate in both the H-form and Na-form. Only one each of the duplicate sample printouts is provided.

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
Meas : Recalc

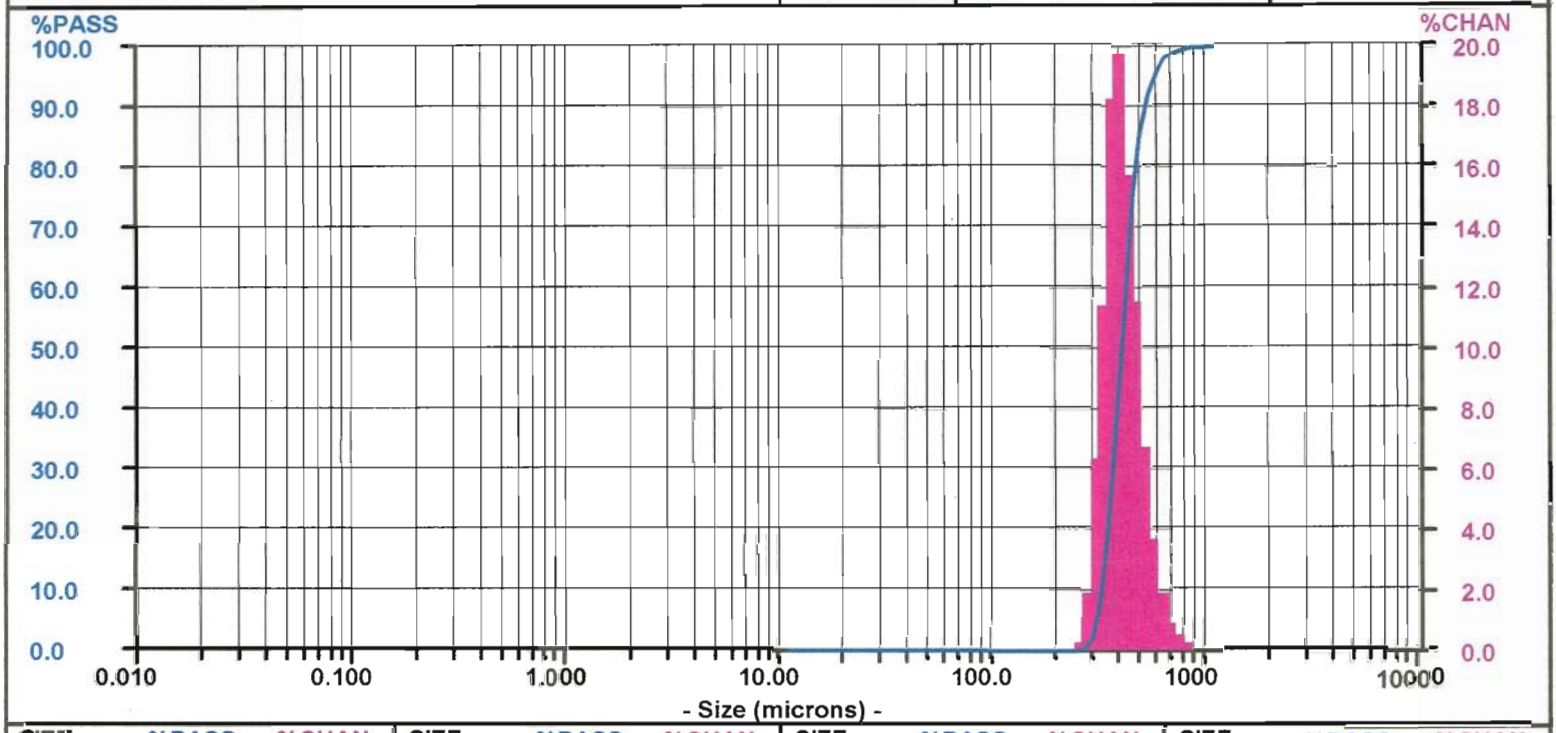
Wave 3 Resin PSD Analysis

061405WCB28

Date: 06/14/05 Meas #: 82
Time: 16:21 Pres #: 1

RF Resin #62
H-Form
Fiskum 06/07/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Vol%	Width
mv	= 418.2	5% = 309.0	60% = 422.6		403.6	100%	149.6
mn	= 379.3	20% = 351.7	70% = 446.2				
ma	= 403.6	30% = 369.8	80% = 476.0				
cs	= 0.015	40% = 386.5	90% = 525.2				
sd	= 74.79	50% = 403.5	95% = 576.3				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00			
995.6	100.00	0.02	114.1	0.00	0.00	13.08	0.00	0.00			
913.0	99.98	0.09	104.7	0.00	0.00	12.00	0.00	0.00			
837.2	99.89	0.29	95.96	0.00	0.00	11.00	0.00	0.00			
767.7	99.60	0.57	88.00	0.00	0.00						
704.0	99.03	1.01	80.70	0.00	0.00						
645.6	98.02	2.01	74.00	0.00	0.00						
592.0	96.01	3.77	67.86	0.00	0.00						
542.9	92.24	6.81	62.23	0.00	0.00						
497.8	85.43	11.52	57.06	0.00	0.00						
456.5	73.91	15.70	52.33	0.00	0.00						
418.6	58.21	19.77	47.98	0.00	0.00						
383.9	38.44	18.31	44.00	0.00	0.00						
352.0	20.13	11.43	40.35	0.00	0.00						
322.8	8.70	6.33	37.00	0.00	0.00						
296.0	2.37	2.00	33.93	0.00	0.00						
271.4	0.37	0.37	31.11	0.00	0.00						
248.9	0.00	0.00	28.53	0.00	0.00						
228.2	0.00	0.00	26.16	0.00	0.00						
209.3	0.00	0.00	23.99	0.00	0.00						
191.9	0.00	0.00	22.00	0.00	0.00						
176.0	0.00	0.00	20.17	0.00	0.00						
161.4	0.00	0.00	18.50	0.00	0.00						
148.0	0.00	0.00	16.96	0.00	0.00						
135.7	0.00	0.00	15.56	0.00	0.00						

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 54

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.9394
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/19/05

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
Meas : Recalc

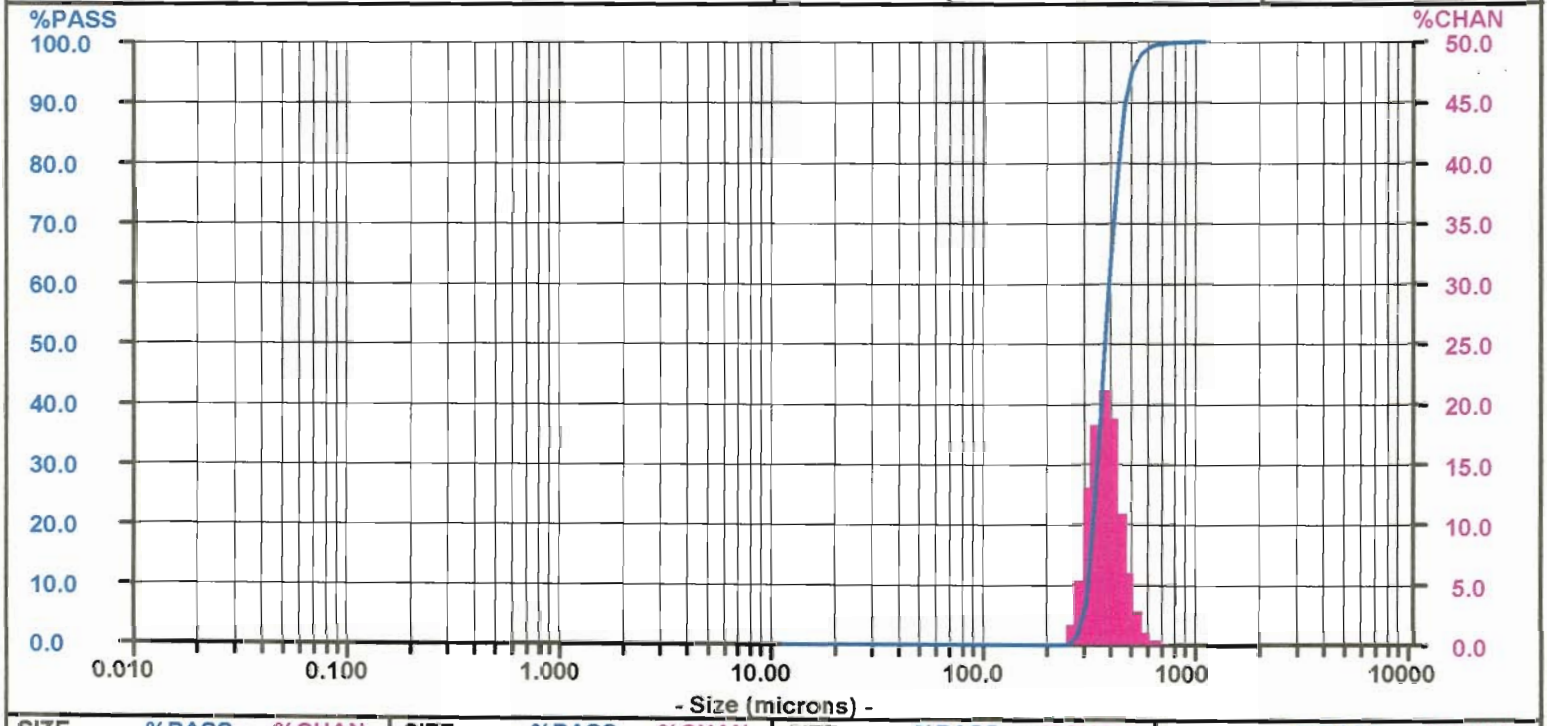
Wave 3 Resin PSD Analysis

061405WCB28

Date: 06/14/05 Meas #: 82
Time: 16:20 Pres #: 1

RF Resin #62
H-Form
Fiskum 06/07/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Num%	Width
mv = 418.2		5% = 288.3	60% = 384.7		369.4	100%	120.3
mn = 379.3		20% = 322.6	70% = 401.7				
ma = 403.6		30% = 339.0	80% = 423.6				
cs = 0.015		40% = 354.4	90% = 460.7				
sd = 60.16		50% = 369.4	95% = 496.4				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00			
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00			
913.0	100.00	0.01	104.7	0.00	0.00	12.00	0.00	0.00			
837.2	99.99	0.04	95.96	0.00	0.00	11.00	0.00	0.00			
767.7	99.95	0.08	88.00	0.00	0.00						
704.0	99.87	0.19	80.70	0.00	0.00						
645.6	99.68	0.48	74.00	0.00	0.00						
592.0	99.20	1.11	67.86	0.00	0.00						
542.9	98.09	2.81	62.23	0.00	0.00						
497.8	95.28	6.01	57.06	0.00	0.00						
456.5	89.27	10.97	52.33	0.00	0.00						
418.6	78.30	18.78	47.98	0.00	0.00						
383.9	59.52	21.15	44.00	0.00	0.00						
352.0	38.37	18.24	40.35	0.00	0.00						
322.8	20.13	13.09	37.00	0.00	0.00						
296.0	7.04	5.34	33.93	0.00	0.00						
271.4	1.70	1.70	31.11	0.00	0.00						
248.9	0.00	0.00	28.53	0.00	0.00						
228.2	0.00	0.00	26.16	0.00	0.00						
209.3	0.00	0.00	23.99	0.00	0.00						
191.9	0.00	0.00	22.00	0.00	0.00						
176.0	0.00	0.00	20.17	0.00	0.00						
161.4	0.00	0.00	18.50	0.00	0.00						
148.0	0.00	0.00	16.96	0.00	0.00						
135.7	0.00	0.00	15.56	0.00	0.00						

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 54

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.9394
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/19/05

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

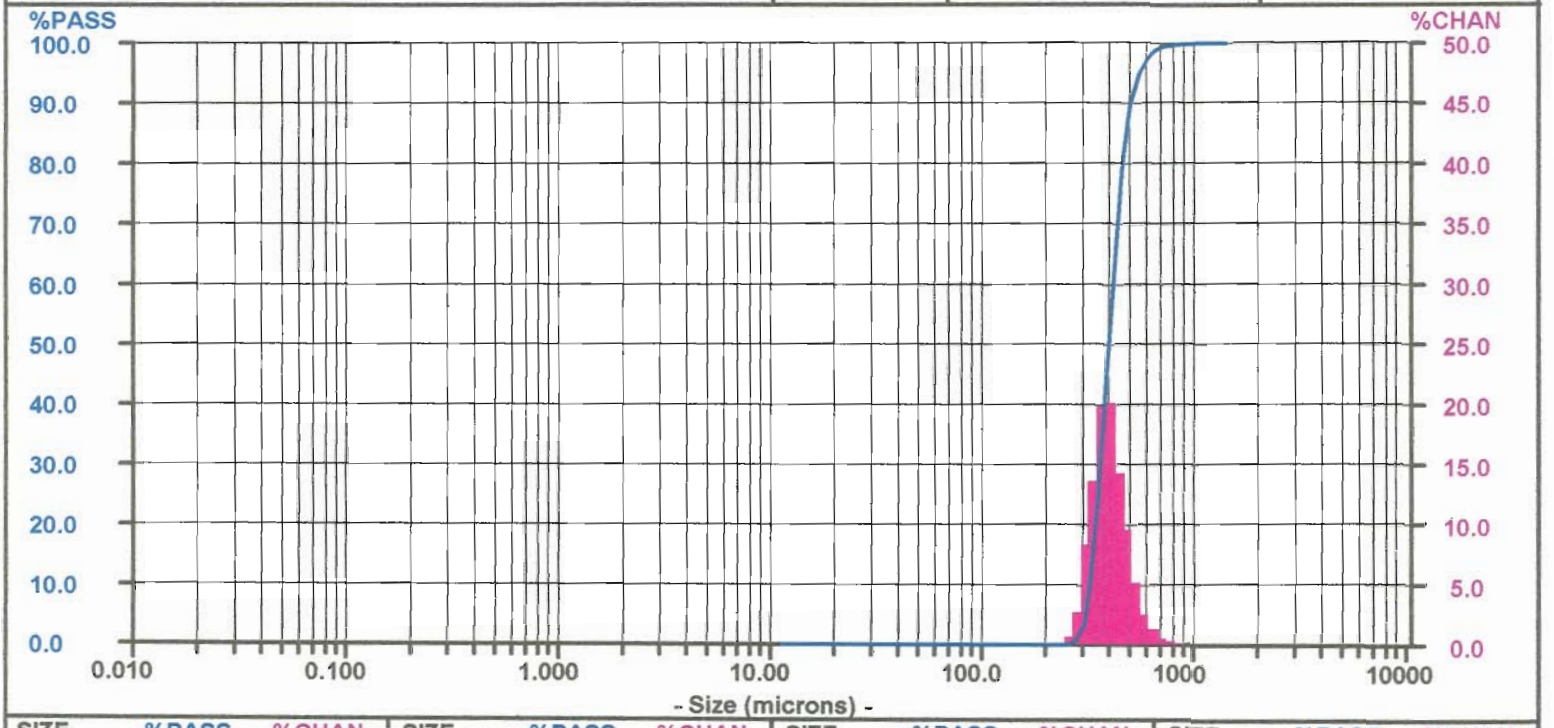
Wave 3 Resin PSD Analysis

061405WCB28

Date: 07/10/06 Meas #: 82
Time: 15:56 Pres #: 1

RF Resin #62
H-Form
Fiskum 06/07/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Area%	Width
mv	= 418.2	5% =	301.4	60% = 408.1	391.1	100%	139.0
mn	= 379.3	20% =	340.8	70% = 428.7			
ma	= 403.6	30% =	359.1	80% = 456.2			
cs	= 0.015	40% =	375.2	90% = 499.6			
sd	= 69.48	50% =	391.1	95% = 542.9			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	0.00	0.00	18.50	0.00	0.00			
1291	100.00	0.00	148.0	0.00	0.00	16.96	0.00	0.00			
1184	100.00	0.00	135.7	0.00	0.00	15.56	0.00	0.00			
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00			
995.6	100.00	0.01	114.1	0.00	0.00	13.08	0.00	0.00			
913.0	99.99	0.04	104.7	0.00	0.00	12.00	0.00	0.00			
837.2	99.95	0.15	95.96	0.00	0.00	11.00	0.00	0.00			
767.7	99.80	0.30	88.00	0.00	0.00						
704.0	99.50	0.69	80.70	0.00	0.00						
645.6	98.91	1.29	74.00	0.00	0.00						
592.0	97.62	2.61	67.86	0.00	0.00						
542.9	95.01	5.25	62.23	0.00	0.00						
497.8	89.76	9.65	57.06	0.00	0.00						
456.5	80.11	14.40	52.33	0.00	0.00						
418.6	65.71	20.13	47.98	0.00	0.00						
383.9	45.58	19.88	44.00	0.00	0.00						
352.0	25.70	13.79	40.35	0.00	0.00						
322.8	11.91	8.39	37.00	0.00	0.00						
296.0	3.52	2.87	33.93	0.00	0.00						
271.4	0.65	0.65	31.11	0.00	0.00						
248.9	0.00	0.00	28.53	0.00	0.00						
228.2	0.00	0.00	26.16	0.00	0.00						
209.3	0.00	0.00	23.99	0.00	0.00						
191.9	0.00	0.00	22.00	0.00	0.00						
176.0	0.00	0.00	20.17	0.00	0.00						

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 57

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.9394
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 7/11/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.16
Meas : Original

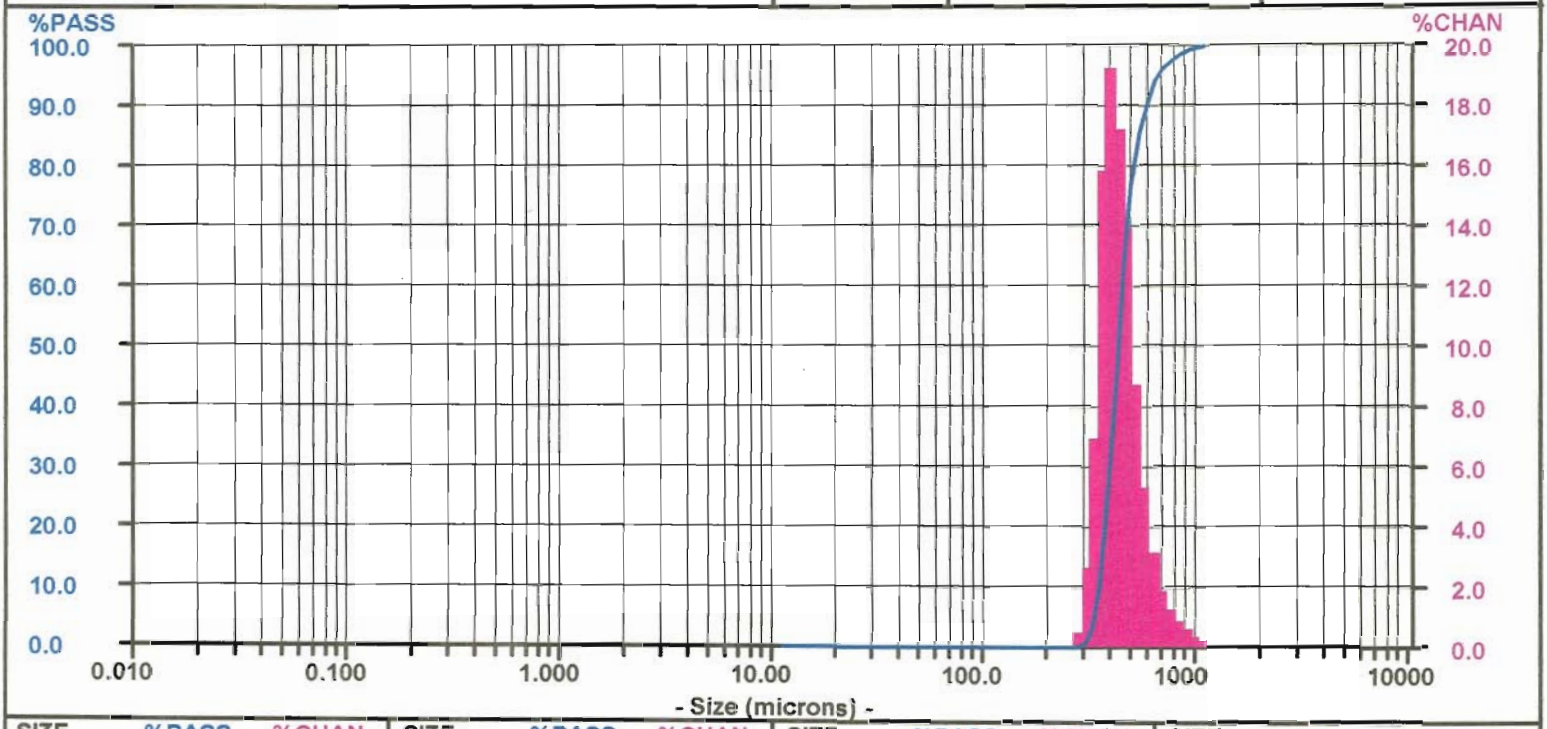
Wave 3 Resin PSD Analysis

061505WCB08

Date: 06/15/05 Meas #: 91
Time: 11:34 Pres #: 1

TI 394-62
Na-Form
Fiskum 06/10/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Vol%	Width
mv	452.1	5%	331.1	60% = 449.7	428.3	100%	171.3
mn	403.2	20%	372.1	70% = 476.6			
ma	432.1	30%	390.4	80% = 511.8			
cs	0.014	40%	408.3	90% = 580.6			
sd	84.36	50%	427.7	95% = 662.5			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.28	124.5	0.00	0.00	14.27	0.00	0.00			
995.6	99.72	0.44	114.1	0.00	0.00	13.08	0.00	0.00			
913.0	99.28	0.64	104.7	0.00	0.00	12.00	0.00	0.00			
837.2	98.64	0.96	95.96	0.00	0.00	11.00	0.00	0.00			
767.7	97.69	1.37	88.00	0.00	0.00						
704.0	96.32	1.97	80.70	0.00	0.00						
645.6	94.35	3.27	74.00	0.00	0.00						
592.0	91.08	5.39	67.86	0.00	0.00						
542.9	85.69	8.83	62.23	0.00	0.00						
497.8	76.86	13.94	57.06	0.00	0.00						
456.5	62.92	17.30	52.33	0.00	0.00						
418.6	45.62	19.30	47.98	0.00	0.00						
383.9	26.32	15.86	44.00	0.00	0.00						
352.0	10.46	7.07	40.35	0.00	0.00						
322.8	3.39	2.72	37.00	0.00	0.00						
296.0	0.67	0.61	33.93	0.00	0.00						
271.4	0.06	0.06	31.11	0.00	0.00						
248.9	0.00	0.00	28.53	0.00	0.00						
228.2	0.00	0.00	26.16	0.00	0.00						
209.3	0.00	0.00	23.99	0.00	0.00						
191.9	0.00	0.00	22.00	0.00	0.00						
176.0	0.00	0.00	20.17	0.00	0.00						
161.4	0.00	0.00	18.50	0.00	0.00						
148.0	0.00	0.00	16.96	0.00	0.00						
135.7	0.00	0.00	15.56	0.00	0.00						

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 54

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.8325
Transmission: 0.94
Above Residual: 0.55
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/19/05

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
Meas.: Recalc

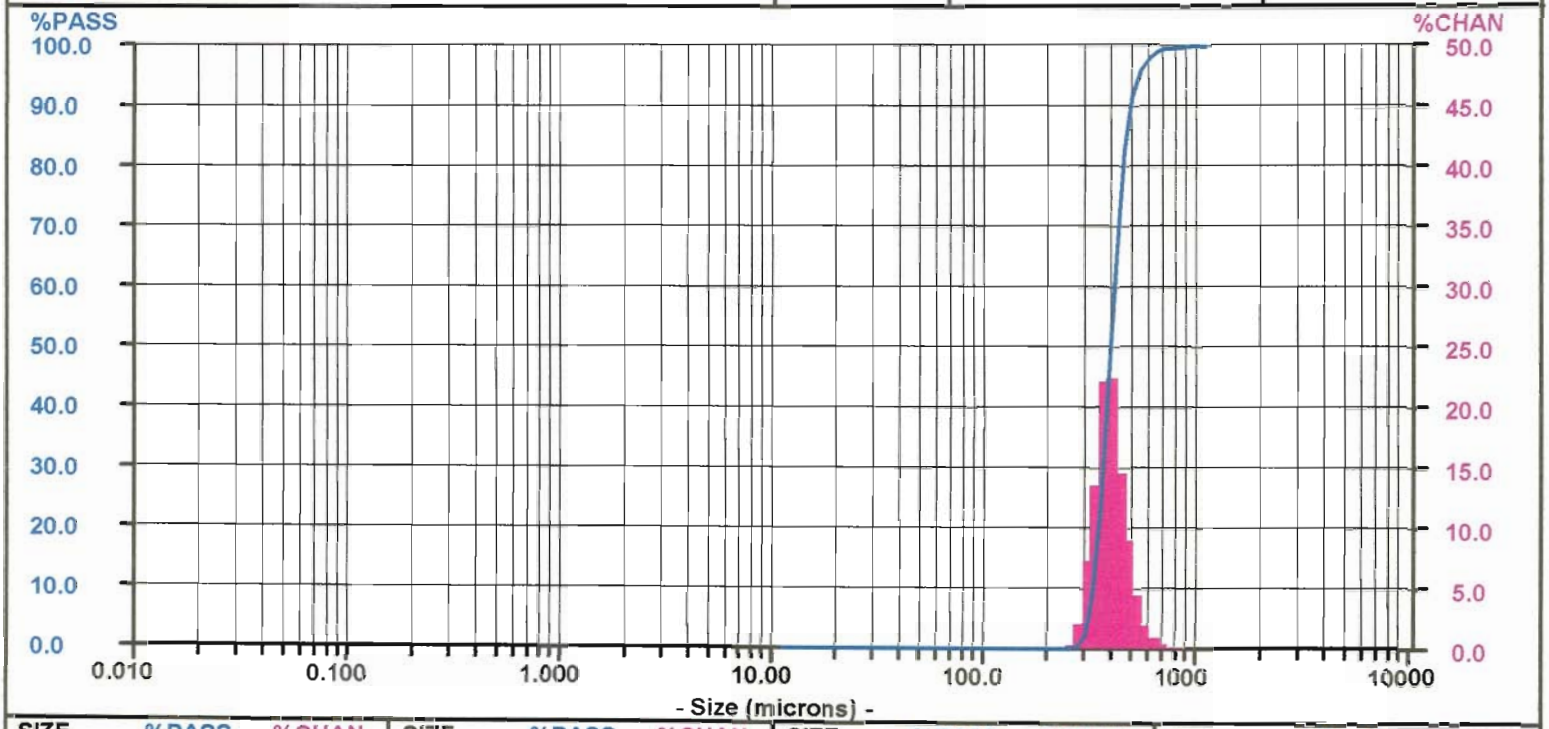
Wave 3 Resin PSD Analysis

061505WCB08

Date: 06/15/05 Meas #: 91
Time: 11:34 Pres #: 1

TI 394-62
Na-Form
Fiskum 06/10/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Num%	Width
mv	452.1	5%	307.0	60% = 405.2	390.3	100%	124.0
mn	403.2	20%	345.4	70% = 423.0			
ma	432.1	30%	361.9	80% = 448.4			
cs	0.014	40%	376.2	90% = 488.6			
sd	61.98	50%	390.3	95% = 527.9			



SIZE	%PASS	%CHAN
1086	100.00	0.02
995.6	99.98	0.04
913.0	99.94	0.07
837.2	99.87	0.14
767.7	99.73	0.23
704.0	99.50	0.44
645.6	99.06	0.96
592.0	98.10	1.95
542.9	96.15	4.45
497.8	91.70	8.99
456.5	82.71	14.64
418.6	68.07	22.52
383.9	45.55	22.16
352.0	23.39	13.56
322.8	9.83	7.40
296.0	2.43	2.12
271.4	0.31	0.31
248.9	0.00	0.00
228.2	0.00	0.00
209.3	0.00	0.00
191.9	0.00	0.00
176.0	0.00	0.00
161.4	0.00	0.00
148.0	0.00	0.00
135.7	0.00	0.00

SIZE	%PASS	%CHAN
124.5	0.00	0.00
114.1	0.00	0.00
104.7	0.00	0.00
95.96	0.00	0.00
88.00	0.00	0.00
80.70	0.00	0.00
74.00	0.00	0.00
67.86	0.00	0.00
62.23	0.00	0.00
57.06	0.00	0.00
52.33	0.00	0.00
47.98	0.00	0.00
44.00	0.00	0.00
40.35	0.00	0.00
37.00	0.00	0.00
33.93	0.00	0.00
31.11	0.00	0.00
28.53	0.00	0.00
26.16	0.00	0.00
23.99	0.00	0.00
22.00	0.00	0.00
20.17	0.00	0.00
18.50	0.00	0.00
16.96	0.00	0.00
15.56	0.00	0.00

SIZE	%PASS	%CHAN
14.27	0.00	0.00
13.08	0.00	0.00
12.00	0.00	0.00
11.00	0.00	0.00

SIZE	%PASS	%CHAN

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 54

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.8325
Transmission: 0.94
Above Residual: 0.02
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 6/19/05

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

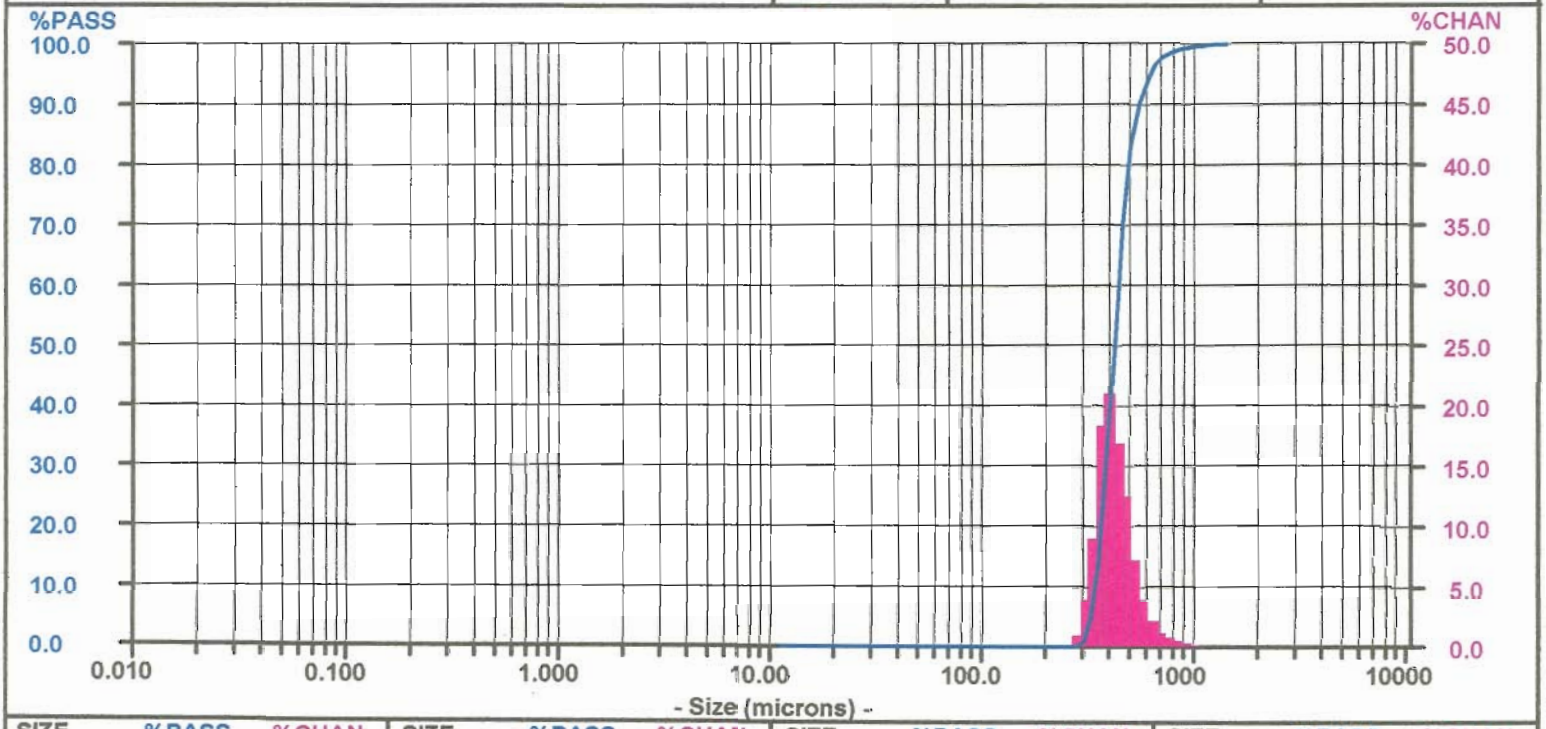
Wave 3 Resin PSD Analysis

061505WCB08

Date: 07/10/06 Meas #: 91
Time: 16:04 Pres #: 1

TI 394-62
Na-Form
Fiskum 06/10/05
Aliquot#: 1
Re-run (as necessary)#: NA

Summary		Percentiles			Dia	Area%	Width
mv	= 456.3	5% = 322.5	60% = 431.8		412.4	100%	147.1
mn	= 403.4	20% = 362.9	70% = 455.2				
ma	= 433.6	30% = 379.8	80% = 486.4				
cs	= 0.014	40% = 395.7	90% = 540.1				
sd	= 73.67	50% = 412.4	95% = 604.5				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.04	161.4	0.00	0.00	18.50	0.00	0.00			
1291	99.96	0.07	148.0	0.00	0.00	16.96	0.00	0.00			
1184	99.89	0.08	135.7	0.00	0.00	15.56	0.00	0.00			
1086	99.81	0.12	124.5	0.00	0.00	14.27	0.00	0.00			
995.6	99.69	0.20	114.1	0.00	0.00	13.08	0.00	0.00			
913.0	99.49	0.31	104.7	0.00	0.00	12.00	0.00	0.00			
837.2	99.18	0.51	95.96	0.00	0.00	11.00	0.00	0.00			
767.7	98.67	0.79	88.00	0.00	0.00						
704.0	97.88	1.24	80.70	0.00	0.00						
645.6	96.64	2.26	74.00	0.00	0.00						
592.0	94.38	4.01	67.86	0.00	0.00						
542.9	90.37	7.29	62.23	0.00	0.00						
497.8	83.08	12.55	57.06	0.00	0.00						
456.5	70.53	16.95	52.33	0.00	0.00						
418.6	53.58	21.03	47.98	0.00	0.00						
383.9	32.55	18.40	44.00	0.00	0.00						
352.0	14.15	9.10	40.35	0.00	0.00						
322.8	5.05	3.98	37.00	0.00	0.00						
296.0	1.07	0.97	33.93	0.00	0.00						
271.4	0.10	0.10	31.11	0.00	0.00						
248.9	0.00	0.00	28.53	0.00	0.00						
228.2	0.00	0.00	26.16	0.00	0.00						
209.3	0.00	0.00	23.99	0.00	0.00						
191.9	0.00	0.00	22.00	0.00	0.00						
176.0	0.00	0.00	20.17	0.00	0.00						

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 10.09
Residuals: Disabled
Number Of Channels: 57

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.8326
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 7/11/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

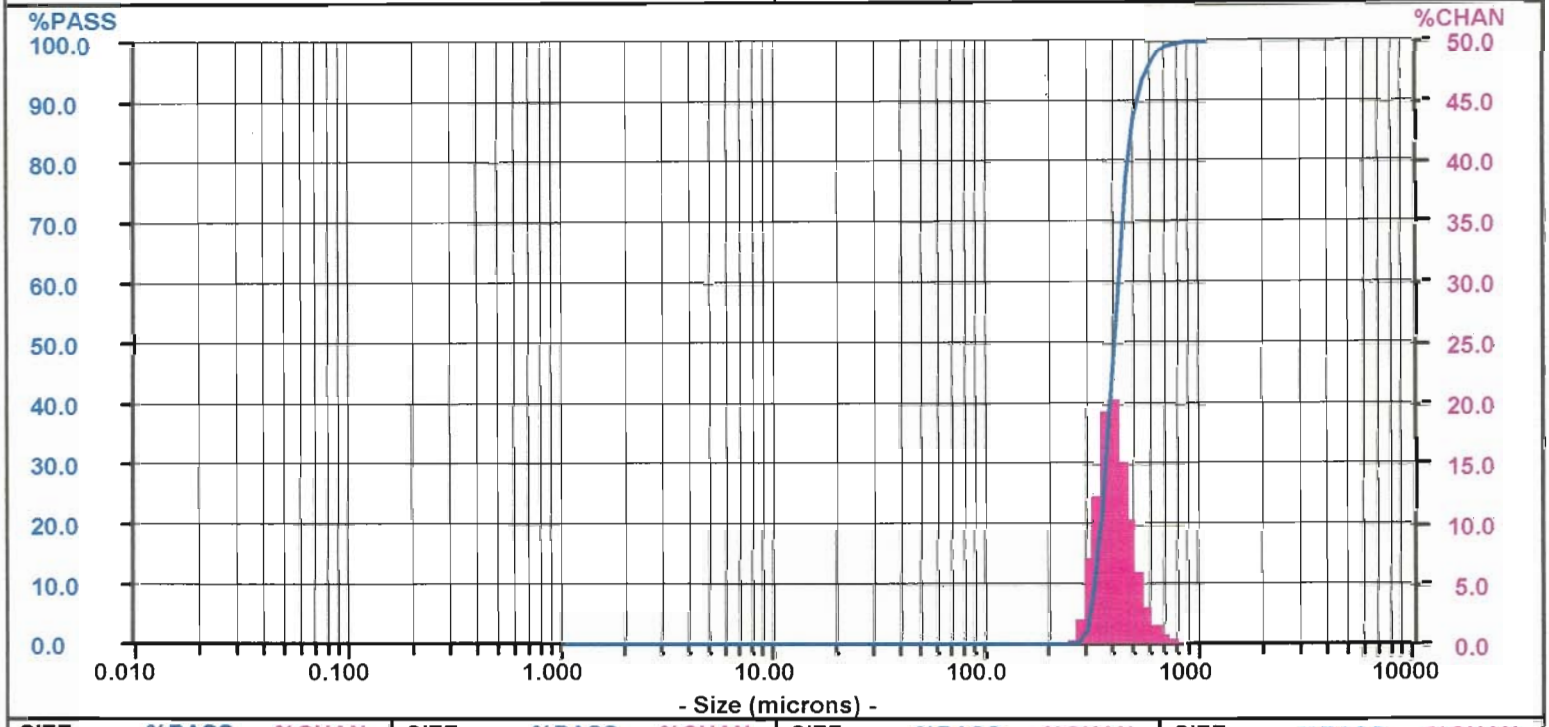
Resin Scale-up Testing

021006WCB06

Date: 02/10/06 Meas #: 128
Time: 20:46 Pres #: 1

TI-394-64, H-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/6/06

Summary		Percentiles			Dia	Vol%	Width
mv = 410.8		5% = 307.2	60% = 415.1		397.5	100%	142.0
mn = 375.4		20% = 347.6	70% = 436.7				
ma = 397.5		30% = 365.5	80% = 466.3				
cs = 0.015		40% = 381.4	90% = 512.0				
sd = 71.00		50% = 397.5	95% = 559.2				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.22	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.78	0.45	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.33	0.79	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	98.54	1.63	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	96.91	3.14	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	93.77	6.00	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	87.77	10.59	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	77.18	15.21	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	61.97	20.41	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	41.56	19.47	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	22.09	12.53	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	9.56	7.10	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	2.46	2.11	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.35	0.35	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5568
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCBuhmiller
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

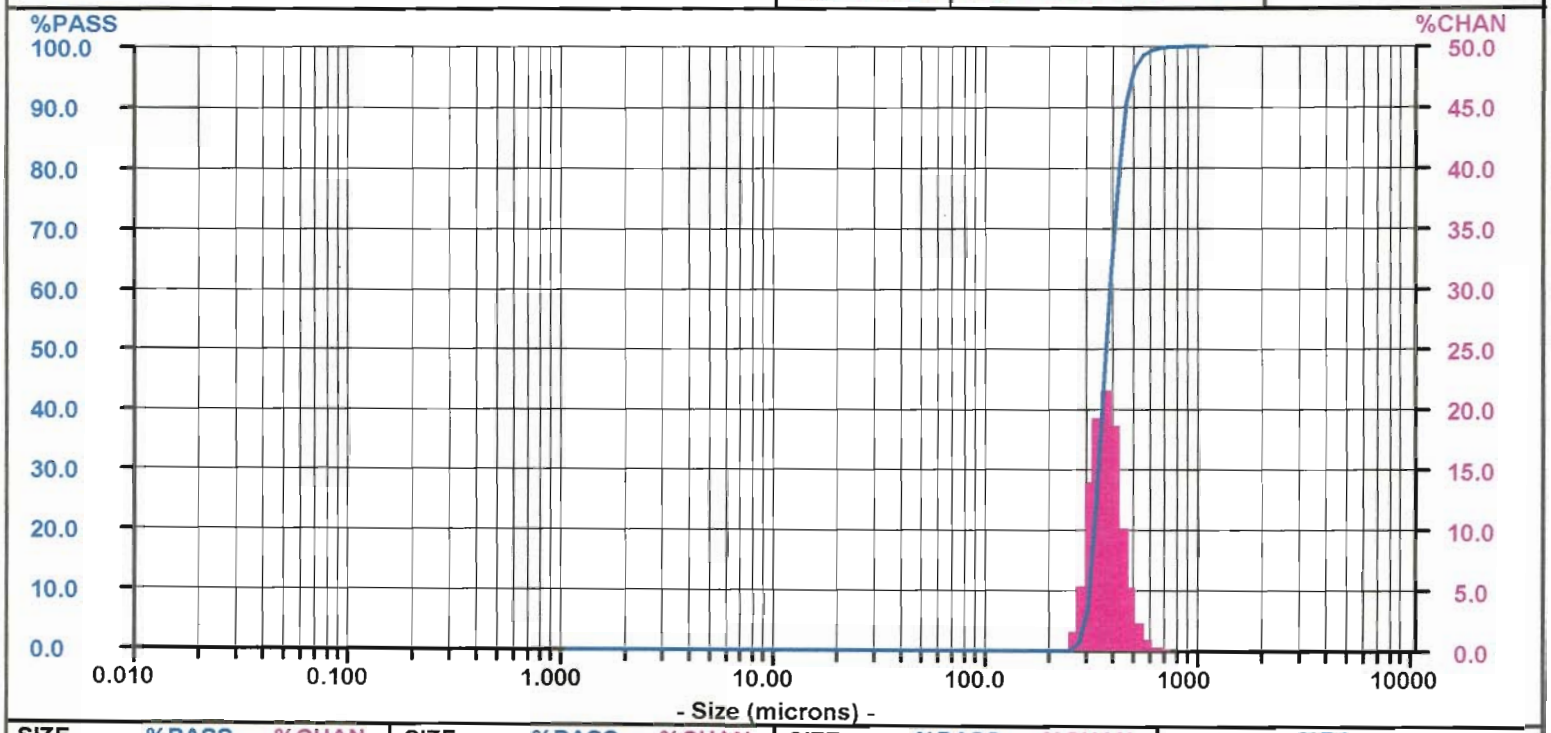
Resin Scale-up Testing

021006WCB05

Date: 02/11/06 Meas #: 128
Time: 02:44 Pres #: 2

TI-394-64, H-form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/6/06

Summary		Percentiles			Dia	Num%	Width
mv	= 410.8	5% = 288.5	60% = 380.9		365.9	100%	115.1
mn	= 375.4	20% = 320.9	70% = 397.4				
ma	= 397.5	30% = 336.6	80% = 417.3				
cs	= 0.015	40% = 351.4	90% = 452.5				
sd	= 57.54	50% = 365.9	95% = 486.9				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.03	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.97	0.06	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.91	0.15	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.76	0.37	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	99.39	0.89	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	98.50	2.38	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	96.12	5.25	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	90.87	10.24	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	80.63	18.65	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	61.98	21.60	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	40.38	19.31	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	21.07	14.04	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	7.03	5.42	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	1.61	1.61	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5668
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCBuchanlle
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

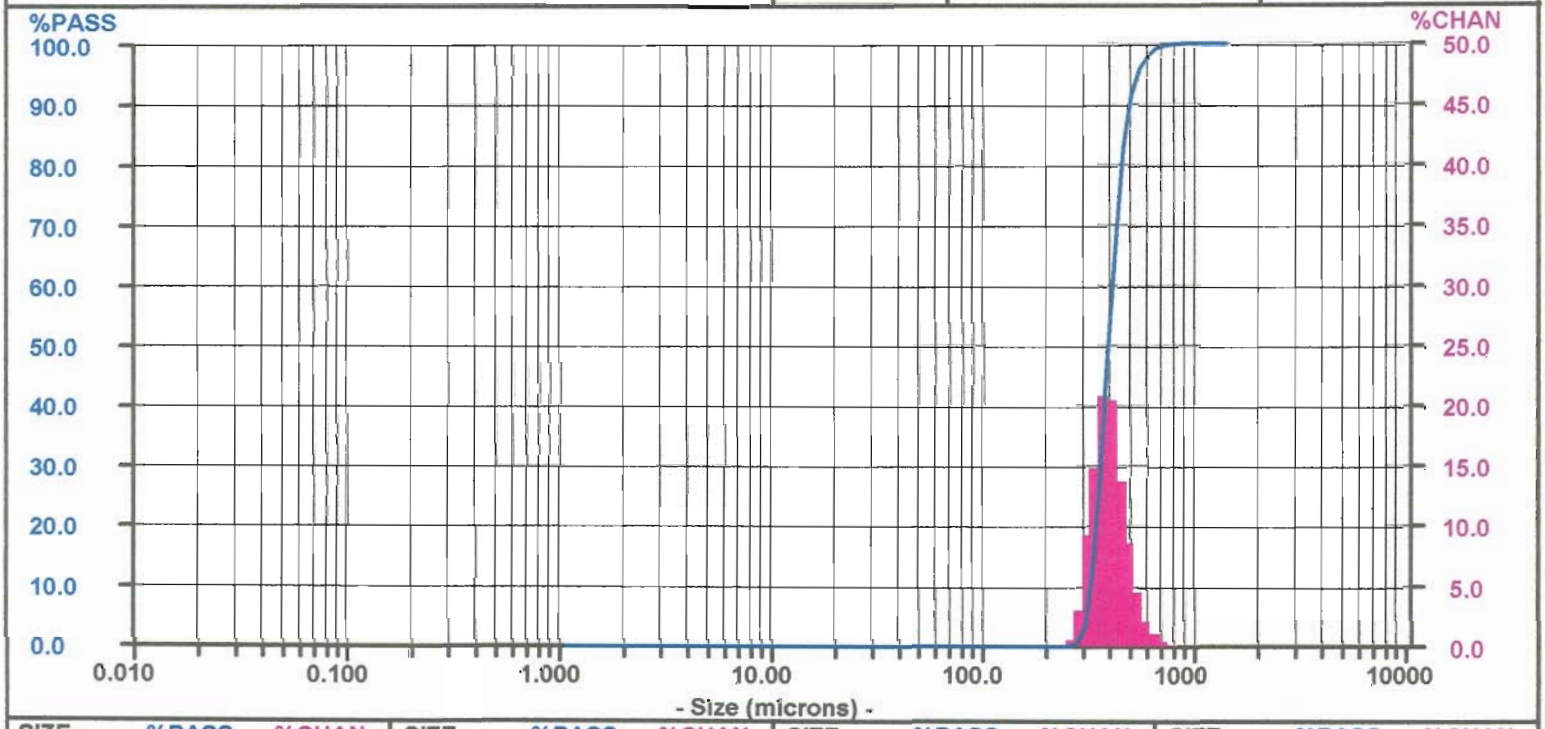
Resin Scale-up Testing

021006WCB05

Date: 07/10/06 Meas #: 128
Time: 14:25 Pres #: 1

TI-394-64, H-form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/6/06

Summary		Percentiles			Dia	Area%	Width
mv = 410.8		5% = 300.7	60% = 402.1		386.2	100%	132.1
mn = 375.4		20% = 337.8	70% = 421.0				
ma = 397.5		30% = 355.5	80% = 447.8				
cs = 0.015		40% = 371.0	90% = 489.7				
sd = 66.06		50% = 386.2	95% = 530.2				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	100.00	0.00	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	100.00	0.00	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.11	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.89	0.23	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.66	0.46	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.20	1.03	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	98.17	2.14	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	96.03	4.55	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	91.48	8.71	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	82.77	13.74	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	69.03	20.46	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	48.57	20.80	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	27.77	14.91	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	12.86	9.25	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	3.61	2.99	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.62	0.62	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5568
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
Meas : Original

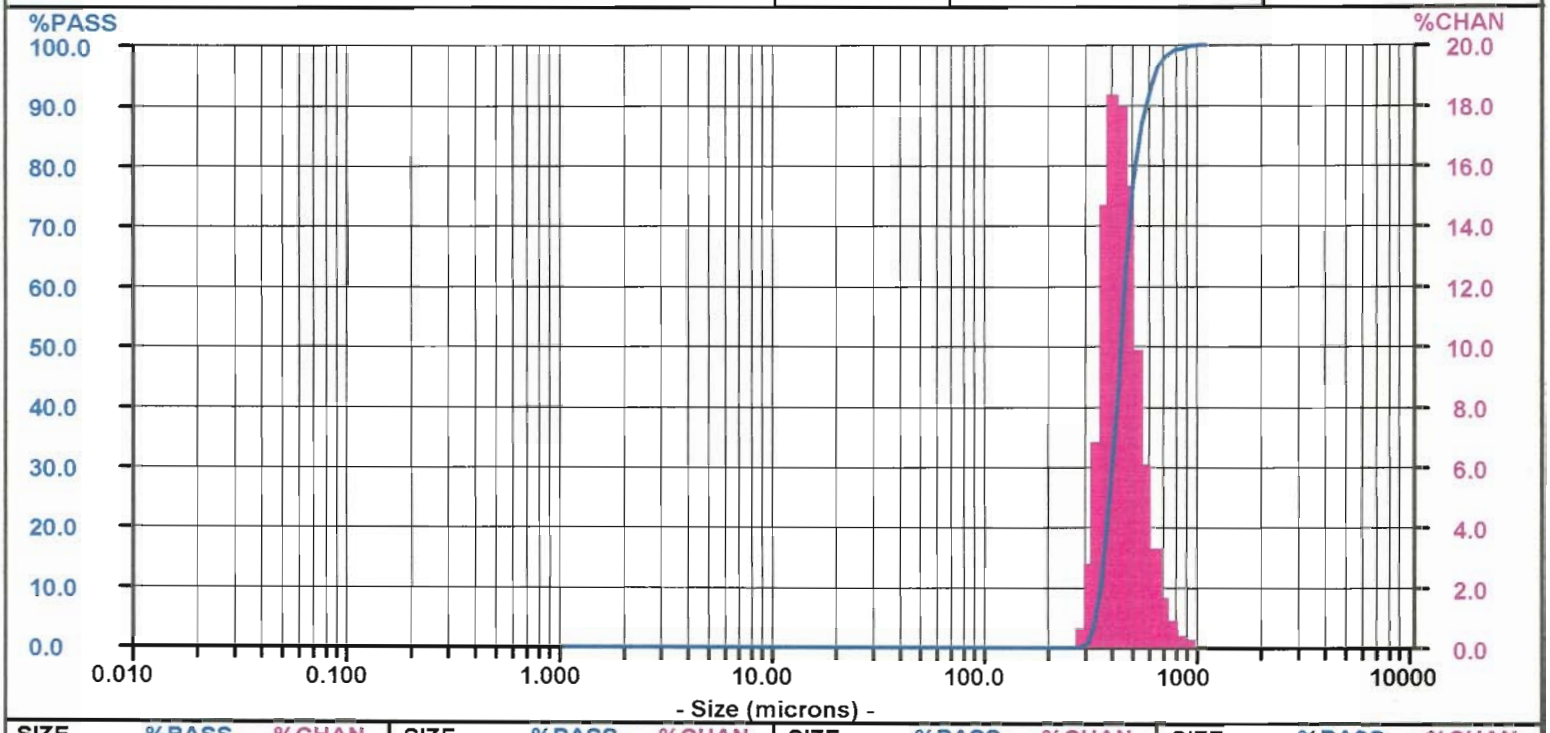
Resin Scale-up Testing

021006WCB07

Date: 02/11/06 Meas #: 130
Time: 00:54 Pres #: 1

#64 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles		Dia	Vol%	Width
mv = 447.1		5% = 330.0	60% = 452.8	431.4	100%	162.1
mn = 404.2		20% = 373.4	70% = 477.0			
ma = 430.9		30% = 392.9	80% = 509.5			
cs = 0.014		40% = 411.7	90% = 563.7			
sd = 81.03		50% = 431.4	95% = 617.1			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.12	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.88	0.26	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.62	0.49	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.13	0.93	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	98.20	1.71	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	96.49	3.36	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	93.13	6.11	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	87.02	9.97	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	77.05	15.33	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	61.72	18.09	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	43.63	18.43	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	25.20	14.70	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	10.50	6.88	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	3.62	2.79	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	0.83	0.74	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.09	0.09	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5189
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCBuchholz
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

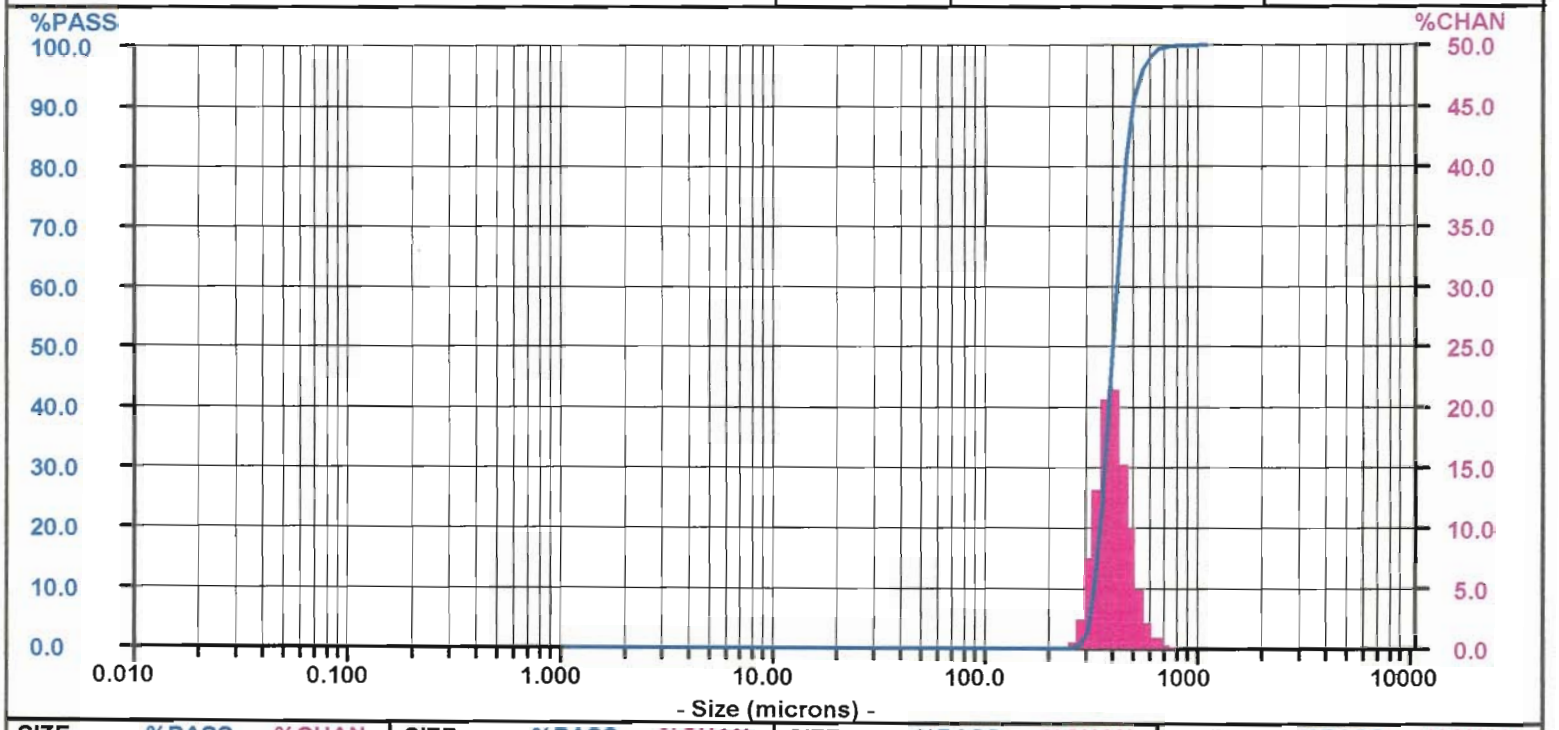
Resin Scale-up Testing

021006WCB07

Date: 02/11/06 Meas #: 130
Time: 01:54 Pres #: 1

#64 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles			Dia	Num%	Width
mv = 447.1		5% = 304.4	60% = 408.6		392.4	100%	130.6
mn = 404.2		20% = 344.6	70% = 427.8				
ma = 430.9		30% = 362.1	80% = 453.4				
cs = 0.014		40% = 377.3	90% = 492.3				
sd = 65.26		50% = 392.4	95% = 529.7				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.01	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.99	0.02	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.97	0.07	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.90	0.15	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.75	0.37	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.38	0.98	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	98.40	2.22	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	96.18	5.01	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	91.17	10.04	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	81.13	15.26	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	65.87	21.45	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	44.42	20.73	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	23.69	13.20	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	10.49	7.60	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	2.99	2.48	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.51	0.51	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5189
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCBuchanan
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

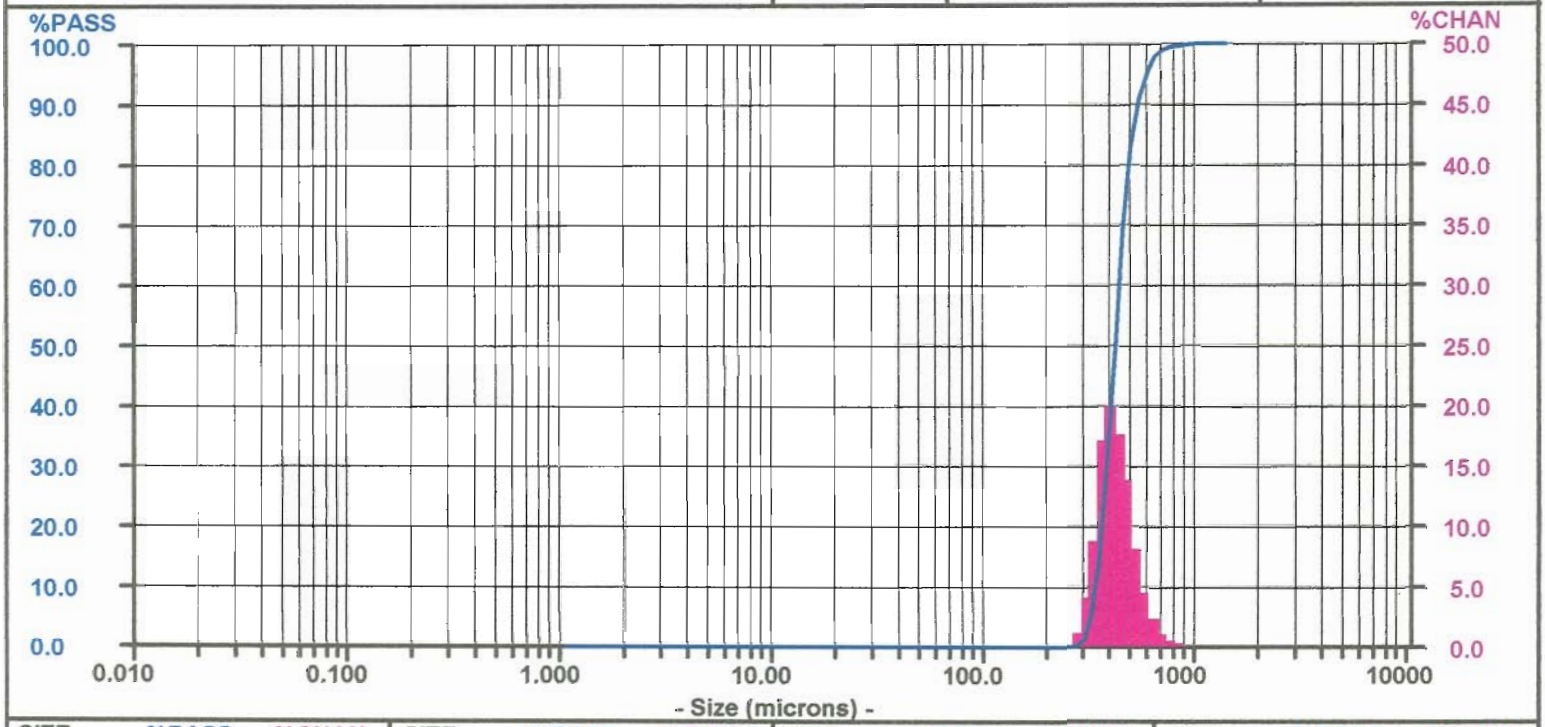
Resin Scale-up Testing

021006WCB07

Date: 07/10/06 Meas #: 130
Time: 14:27 Pres #: 1

#64 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles			Dia	Area%	Width
mv	= 447.1	5% = 320.7	60% = 436.0		416.2	100%	147.4
mn	= 404.2	20% = 363.6	70% = 459.1				
ma	= 430.9	30% = 381.6	80% = 488.2				
cs	= 0.014	40% = 398.6	90% = 535.7				
sd	= 73.72	50% = 416.2	95% = 583.7				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	100.00	0.00	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	100.00	0.00	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.06	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.94	0.12	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.82	0.26	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.56	0.54	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.02	1.07	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	97.95	2.31	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	95.64	4.54	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	91.10	8.21	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	82.89	13.84	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	69.05	17.68	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	51.37	20.06	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	31.32	17.10	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	14.22	8.84	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	5.38	4.06	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	1.32	1.16	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.16	0.16	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5189
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
Meas : Original

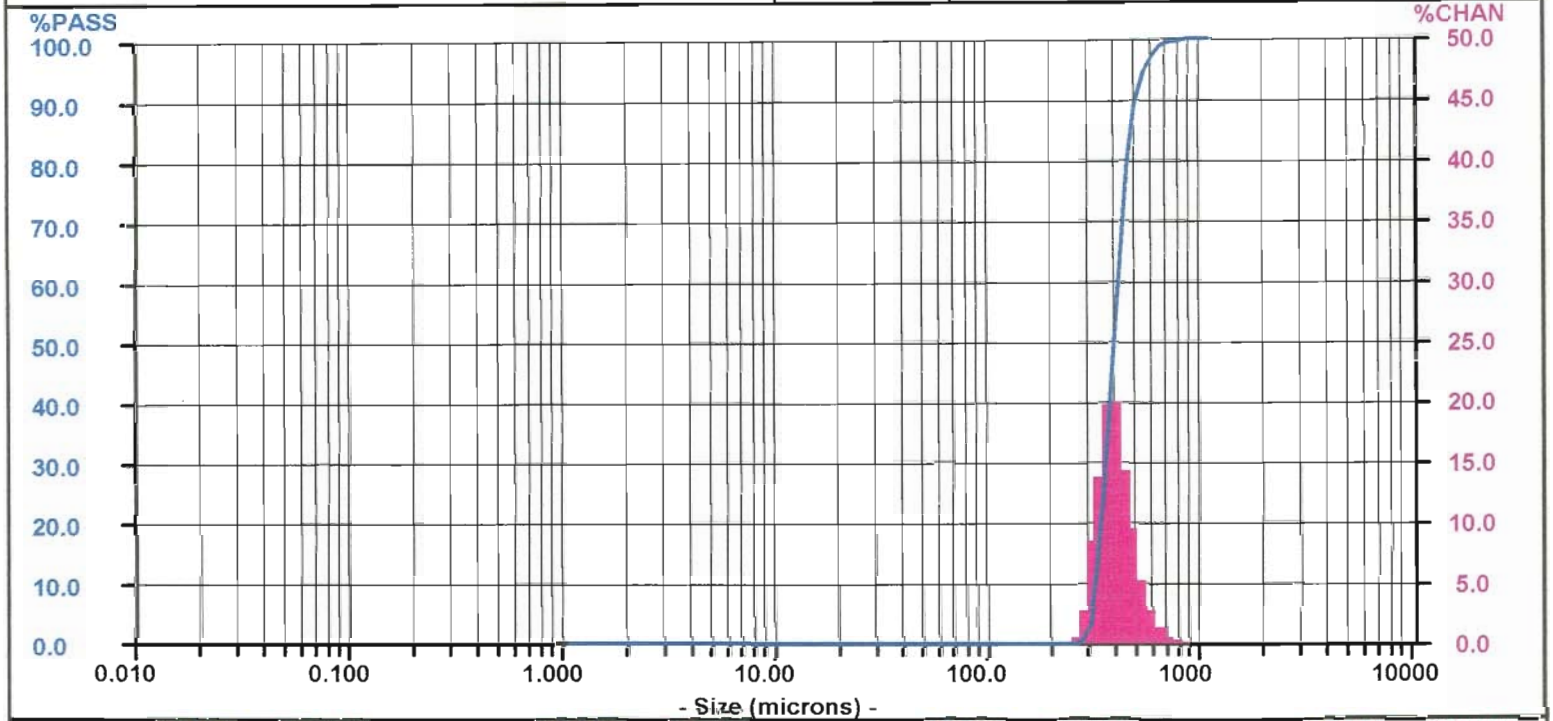
Resin Scale-up Testing

021006WCB06

Date: 02/10/06 Meas #: 129
Time: 20:42 Pres #: 1

TI-394-65, H-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/5/06

Summary		Percentiles			Dia	Vol%	Width
mv	= 403.1	5% = 302.1	60% = 408.3		391.1	100%	139.7
mn	= 368.7	20% = 340.8	70% = 429.1				
ma	= 390.3	30% = 359.0	80% = 456.8				
cs	= 0.015	40% = 376.1	90% = 501.1				
sd	= 69.83	50% = 391.1	95% = 545.3				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.16	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.84	0.33	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.51	0.63	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	98.88	1.35	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	97.53	2.69	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	94.84	5.31	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	89.53	9.61	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	79.92	14.32	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	65.60	20.01	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	45.59	19.84	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	25.75	13.92	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	11.83	8.50	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	3.33	2.76	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.57	0.57	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5993
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WC Buchmiller
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

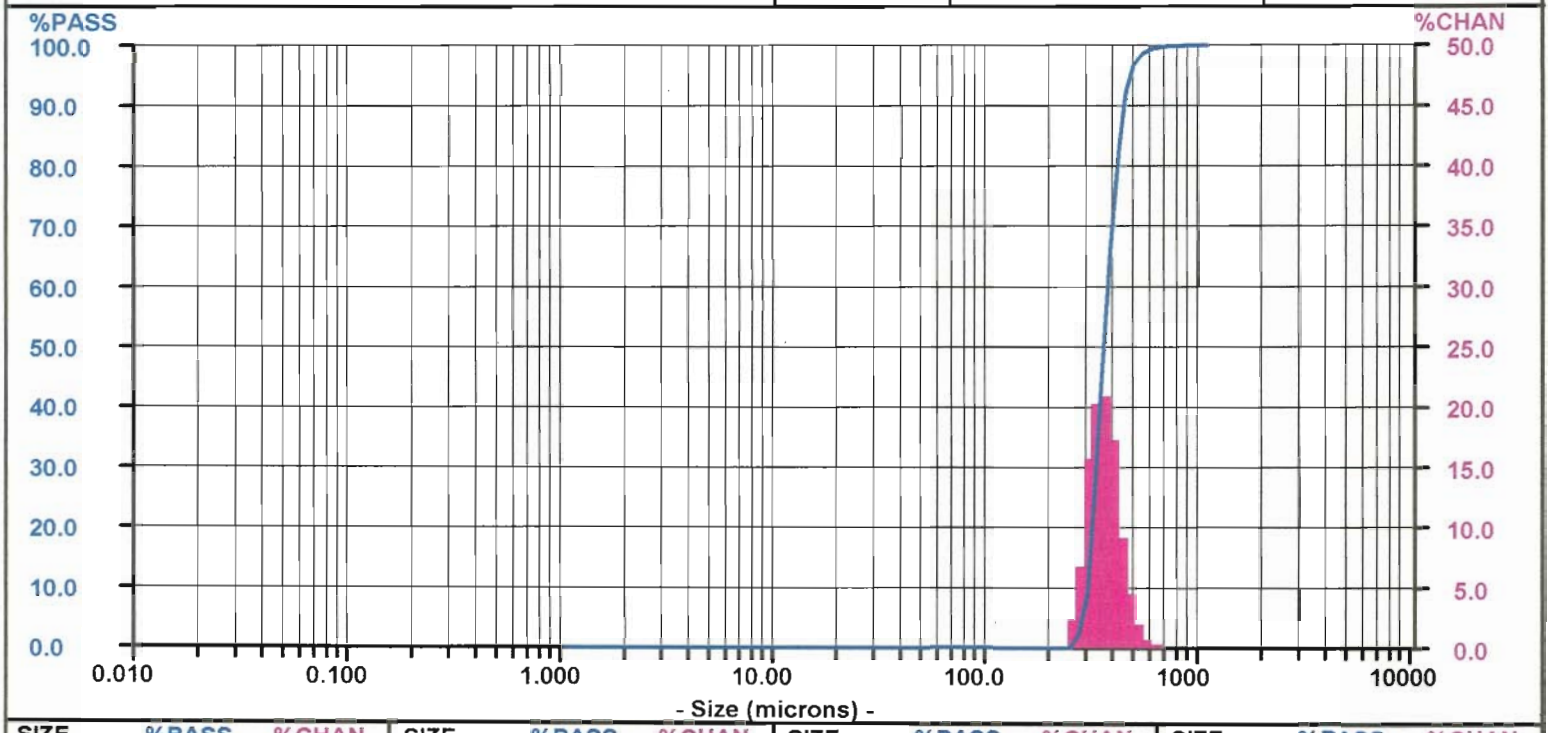
Resin Scale-up Testing

021006WCB06

Date: 02/11/06 Meas #: 129
Time: 01:53 Pres #: 1

TI-394-65, H-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/5/06

Summary		Percentiles			Dia	Num%	Width
mv = 403.1		5% = 283.7	60% = 374.4		359.2	100%	112.2
mn = 368.7		20% = 315.4	70% = 391.2				
ma = 390.3		30% = 330.4	80% = 411.3				
cs = 0.015		40% = 344.7	90% = 444.6				
sd = 56.09		50% = 359.2	95% = 477.0				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.02	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.98	0.04	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.94	0.11	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.83	0.29	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	99.64	0.72	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	98.82	1.99	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	96.83	4.48	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	92.35	9.13	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	83.22	17.22	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	66.00	20.90	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	45.10	20.37	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	24.73	15.71	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	9.02	6.71	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	2.31	2.31	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5993
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WC Buchwalter
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

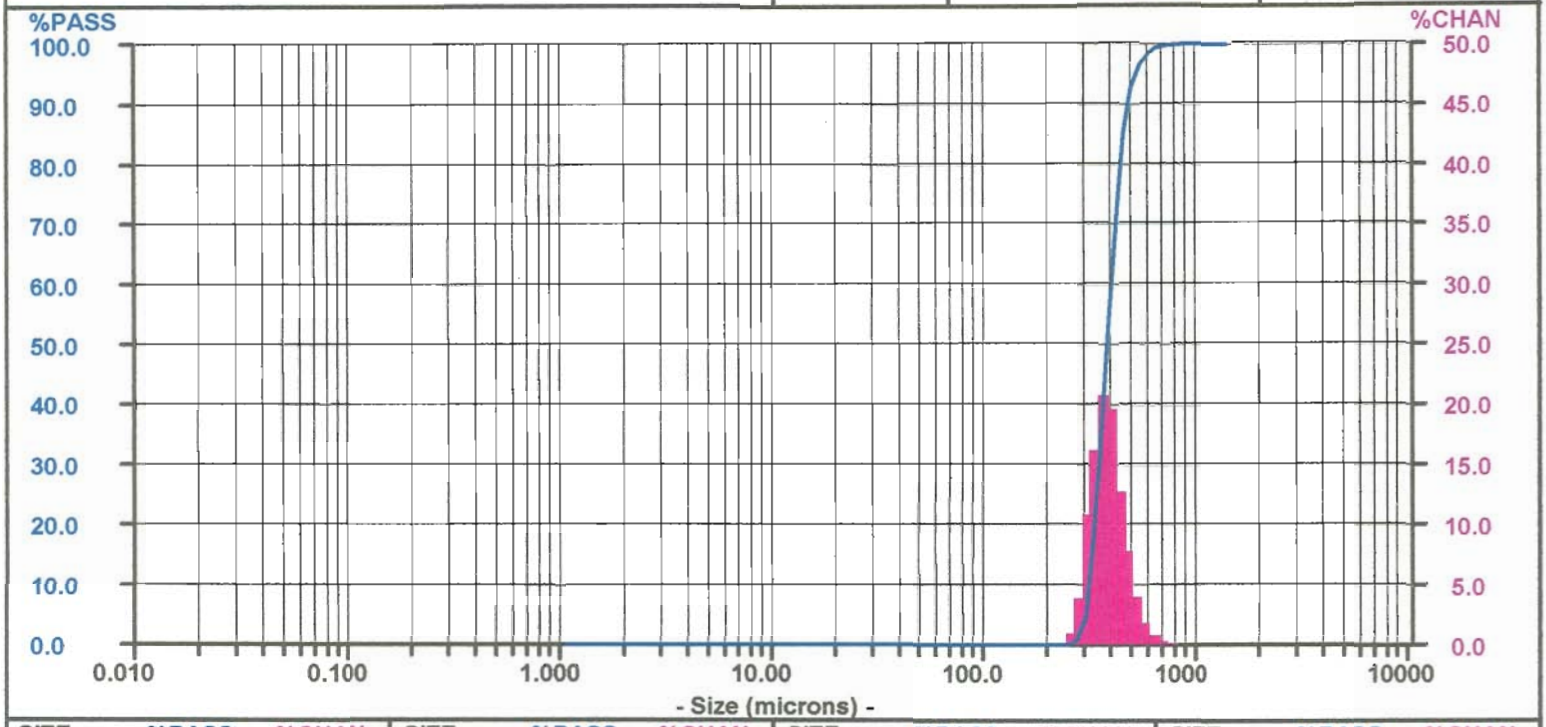
Resin Scale-up Testing

021006WCB06

Date: 07/10/06 Meas #: 129
Time: 14:26 Pres #: 1

TI-394-65, H-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/5/06

Summary		Percentiles			Dia	Area%	Width
mv = 403.1		5% = 296.6	60% = 395.8		379.8	100%	129.3
mn = 368.7		20% = 331.1	70% = 414.1				
ma = 390.3		30% = 348.8	80% = 439.0				
cs = 0.015		40% = 364.5	90% = 480.3				
sd = 64.63		50% = 379.8	95% = 518.4				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	100.00	0.00	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	100.00	0.00	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.08	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.92	0.17	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.75	0.36	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.39	0.84	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	98.55	1.79	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	96.76	3.95	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	92.81	7.74	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	85.07	12.70	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	72.37	19.67	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	52.70	20.82	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	31.88	16.28	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	15.60	10.81	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	4.79	3.83	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.96	0.96	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5993
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 µm

MICROTRAC - S3000

V:9.1.15
Meas : Original

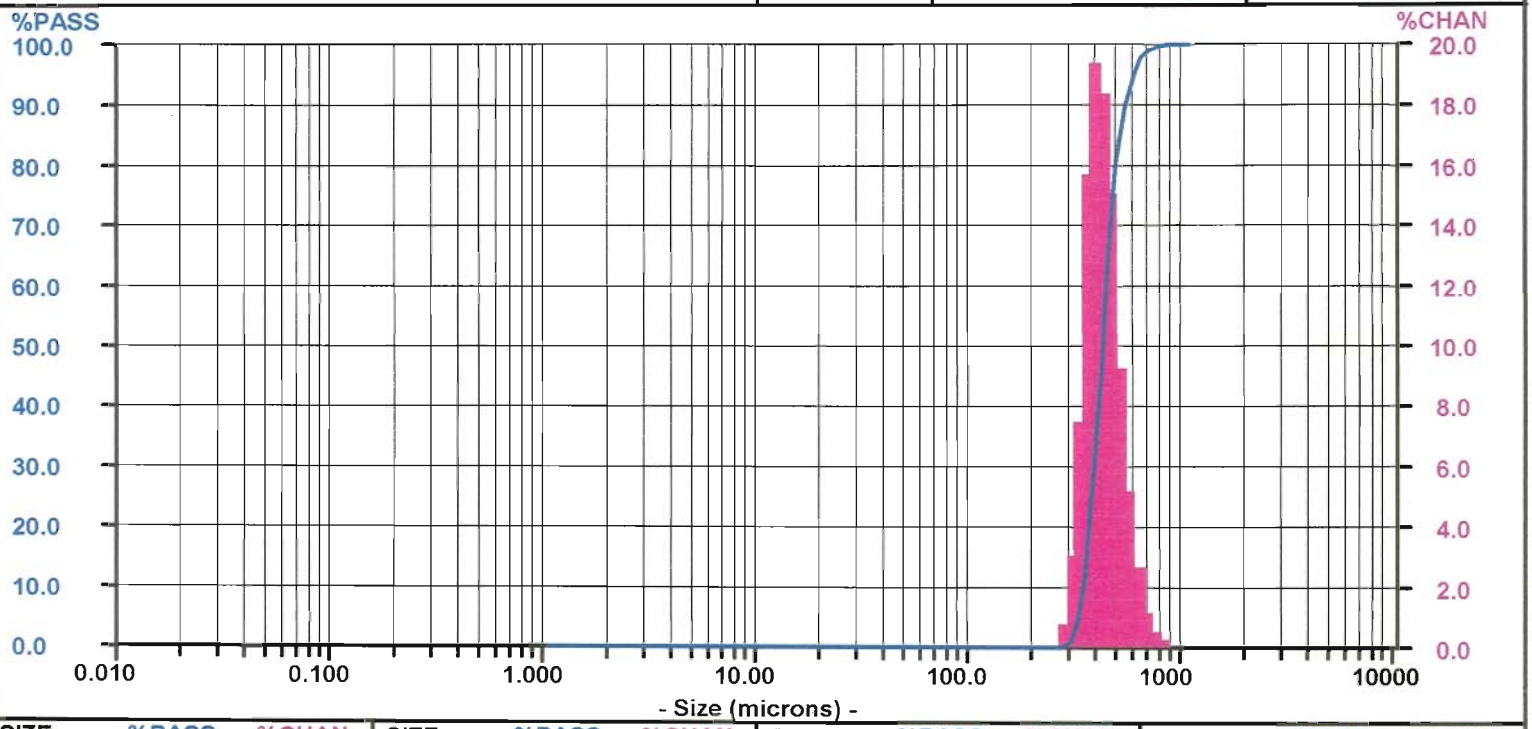
Resin Scale-up Testing

021006WCB08

Date: 02/11/06 Meas #: 131
Time: 01:06 Pres #: 1

#65 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles			Dia	Vol%	Width
mv	= 437.1	5% = 327.1	60% = 444.9		424.6	100%	150.6
mn	= 399.5	20% = 369.9	70% = 467.8				
ma	= 423.3	30% = 388.4	80% = 496.1				
cs	= 0.014	40% = 406.1	90% = 543.9				
sd	= 75.29	50% = 424.6	95% = 590.0				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.27	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.73	0.63	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.10	1.23	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	97.87	2.69	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	96.18	5.31	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	89.87	9.29	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	80.58	15.17	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	65.41	18.48	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	46.93	19.46	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	27.47	15.82	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	11.65	7.56	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	4.09	3.15	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	0.94	0.83	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.11	0.11	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5697
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCBuchanan
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB ; Recalc

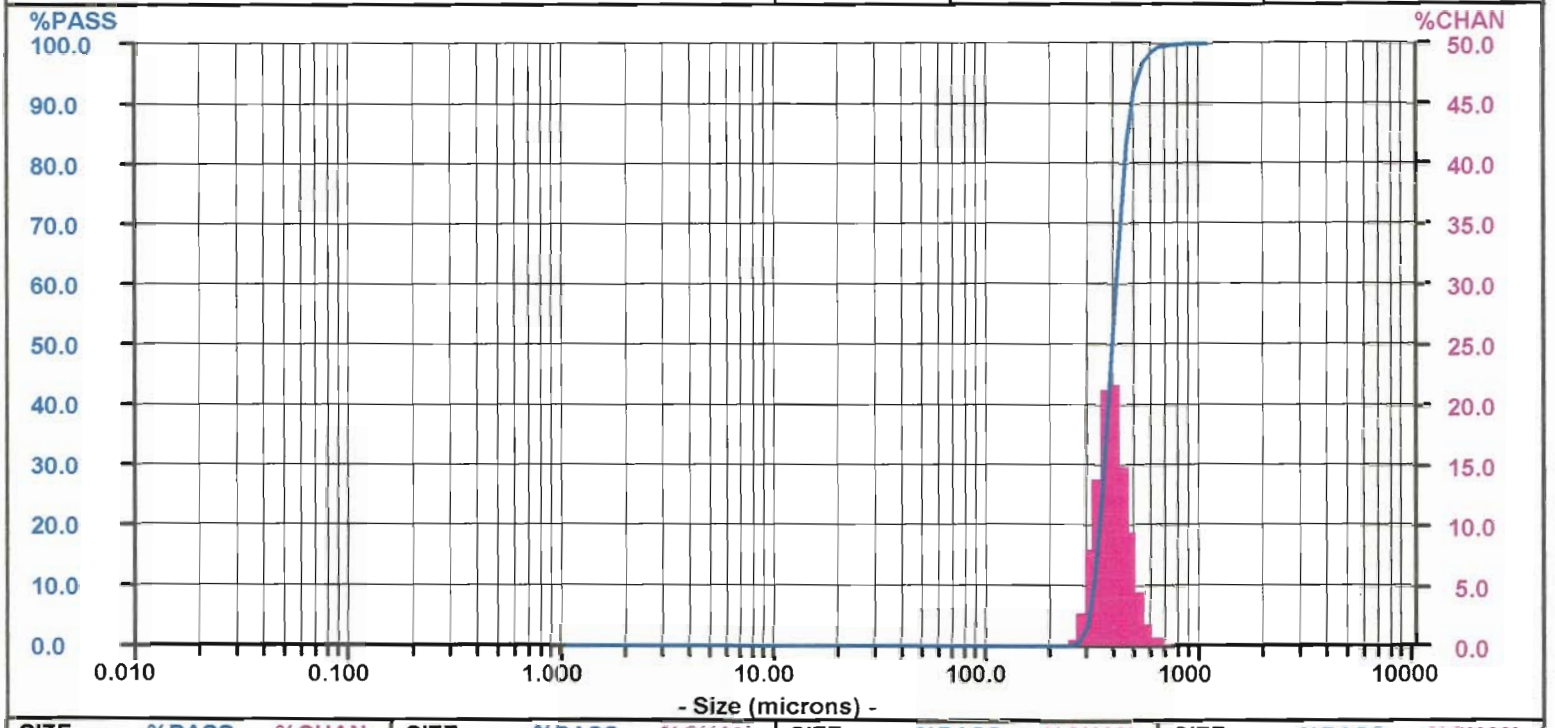
Resin Scale-up Testing

021006WCB08

Date: 02/11/06 Meas #: 131
Time: 01:54 Pres #: 1

#65 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles			Dia	Num%	Width
mv = 437.1		5% = 302.8	60% = 404.6		389.1	100%	126.2
mn = 399.5		20% = 342.0	70% = 422.7				
ma = 423.3		30% = 359.4	80% = 447.6				
cs = 0.014		40% = 374.5	90% = 484.7				
sd = 63.09		50% = 389.1	95% = 517.6				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.04	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.96	0.10	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.86	0.25	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.61	0.75	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	98.86	1.82	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	97.04	4.47	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	92.57	9.47	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	83.10	14.92	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	68.18	21.67	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	46.51	21.31	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	25.20	13.89	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	11.31	8.06	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	3.25	2.68	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.57	0.57	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5697
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WC Buchmiller
3/24/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

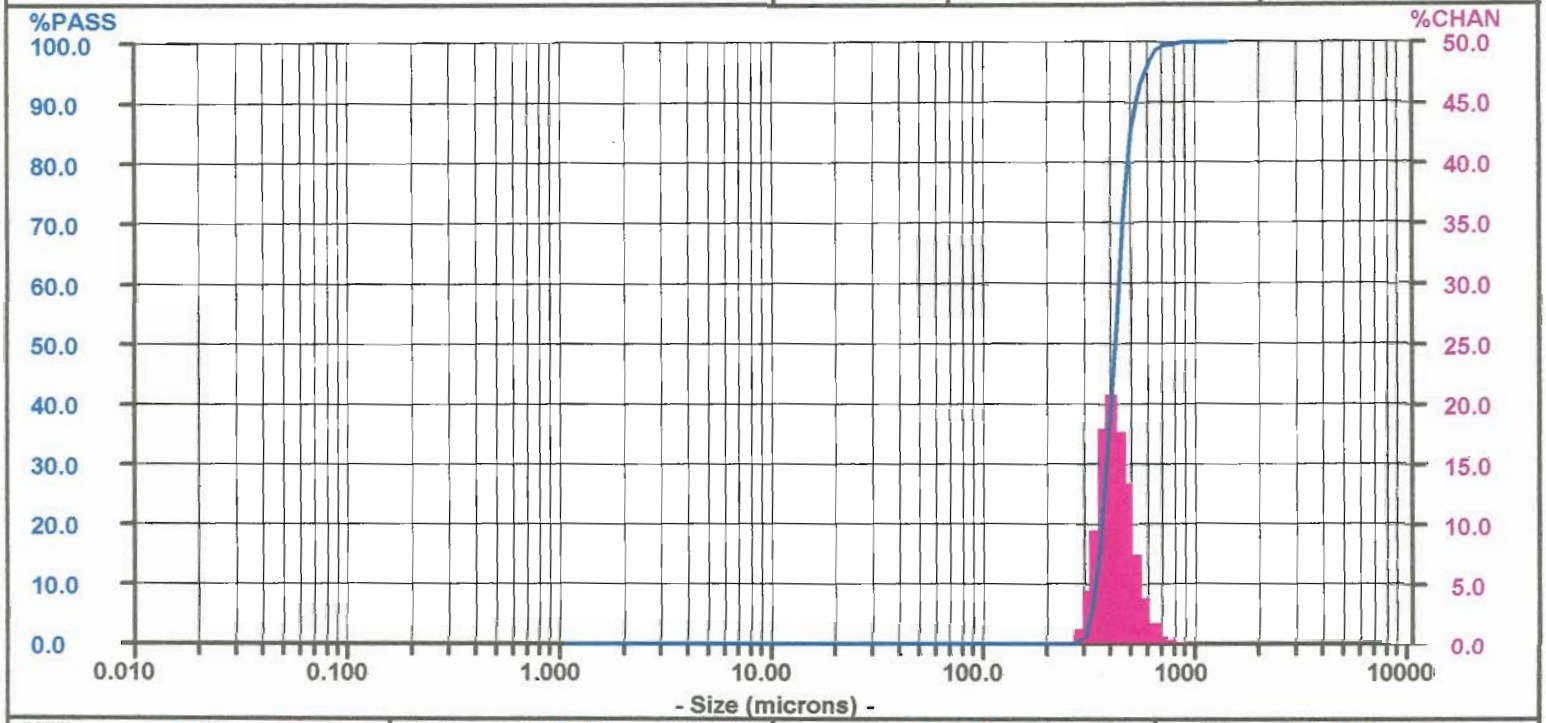
Resin Scale-up Testing

021006WCB08

Date: 07/10/06 Meas #: 131
Time: 14:27 Pres #: 1

#65 Na-Form
RF Resin, Fiskum
Aliquot: 1
Re-run# (if applicable): NA
1/10/06

Summary		Percentiles			Dia	Area%	Width
mv = 437.1		5% = 317.9	60% = 429.8		411.1	100%	139.5
mn = 399.5		20% = 360.5	70% = 451.6				
ma = 423.3		30% = 378.0	80% = 478.5				
cs = 0.014		40% = 394.3	90% = 520.5				
sd = 69.73		50% = 411.1	95% = 563.5				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	100.00	0.00	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	100.00	0.00	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	100.00	0.00	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	100.00	0.00	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	100.00	0.00	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	100.00	0.14	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.86	0.35	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.51	0.75	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	98.76	1.82	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	96.94	3.86	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	93.08	7.51	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	85.57	13.44	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	72.13	17.74	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	54.39	20.83	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	33.56	18.06	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	15.50	9.54	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	5.96	4.49	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	1.47	1.29	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.18	0.18	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5697
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Original

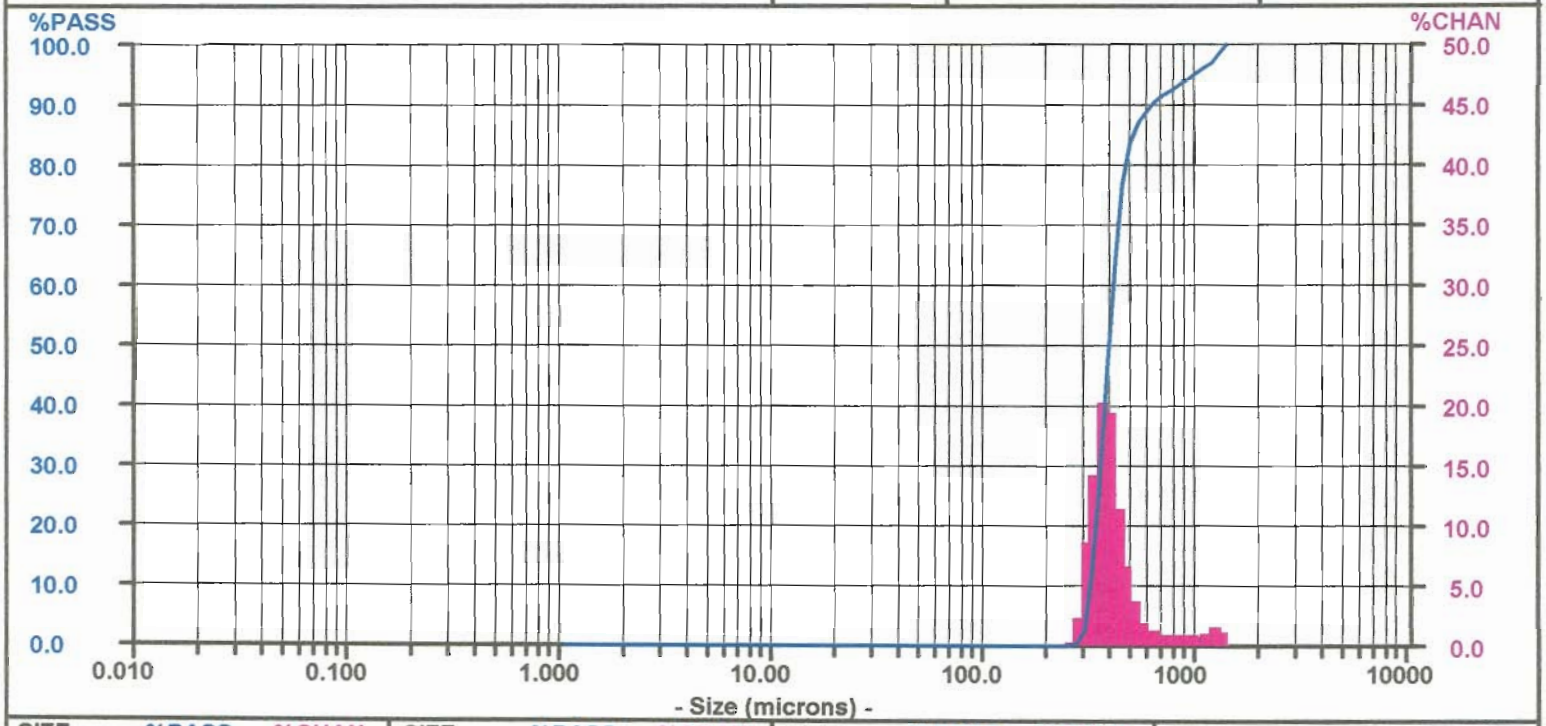
061206 Resin Anal - SKF

061206WCB06

Date: 06/26/06 Meas #: 147
Time: 14:13 Pres #: 1

MB #72 RF Resin H-Form,
SK Fiskum, 5/17/06
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Vol%	Width
mv = 460.5		5% = 304.1	60% = 408.0		936.3	11%	590.7
mn = 368.3		20% = 341.3	70% = 431.5		381.8	89%	119.8
ma = 406.3		30% = 359.0	80% = 473.2				
cs = 0.015		40% = 374.7	90% = 617.7				
sd = 84.64		50% = 390.5	95% = 967.0				



SIZE	%PASS	%CHAN
1408	100.00	1.25
1291	98.75	1.58
1184	97.17	1.02
1086	96.15	0.84
995.6	95.31	0.92
913.0	94.39	0.94
837.2	93.45	0.94
767.7	92.51	0.94
704.0	91.57	0.97
645.6	90.60	1.29
592.0	89.31	2.01
542.9	87.30	3.75
497.8	83.55	6.63
456.5	76.92	11.46
418.6	65.46	19.46
383.9	46.00	20.36
352.0	25.64	14.27
322.8	11.37	8.63
296.0	2.74	2.40
271.4	0.34	0.34
248.9	0.00	0.00
228.2	0.00	0.00
209.3	0.00	0.00
191.9	0.00	0.00
176.0	0.00	0.00

SIZE	%PASS	%CHAN
161.4	0.00	0.00
148.0	0.00	0.00
135.7	0.00	0.00
124.5	0.00	0.00
114.1	0.00	0.00
104.7	0.00	0.00
95.96	0.00	0.00
88.00	0.00	0.00
80.70	0.00	0.00
74.00	0.00	0.00
67.86	0.00	0.00
62.23	0.00	0.00
57.06	0.00	0.00
52.33	0.00	0.00
47.98	0.00	0.00
44.00	0.00	0.00
40.35	0.00	0.00
37.00	0.00	0.00
33.93	0.00	0.00
31.11	0.00	0.00
28.53	0.00	0.00
26.16	0.00	0.00
23.99	0.00	0.00
22.00	0.00	0.00
20.17	0.00	0.00

SIZE	%PASS	%CHAN
18.50	0.00	0.00
16.96	0.00	0.00
15.56	0.00	0.00
14.27	0.00	0.00
13.08	0.00	0.00
12.00	0.00	0.00
11.00	0.00	0.00
10.09	0.00	0.00
9.250	0.00	0.00
8.482	0.00	0.00
7.778	0.00	0.00
7.133	0.00	0.00
6.541	0.00	0.00
5.998	0.00	0.00
5.500	0.00	0.00
5.044	0.00	0.00
4.625	0.00	0.00
4.241	0.00	0.00
3.889	0.00	0.00
3.566	0.00	0.00
3.270	0.00	0.00
2.999	0.00	0.00
2.750	0.00	0.00
2.522	0.00	0.00
2.312	0.00	0.00

SIZE	%PASS	%CHAN
2.121	0.00	0.00
1.945	0.00	0.00
1.783	0.00	0.00
1.635	0.00	0.00
1.499	0.00	0.00
1.375	0.00	0.00
1.261	0.00	0.00
1.156	0.00	0.00
1.060	0.00	0.00

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.6103
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/29/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB ; Recalc

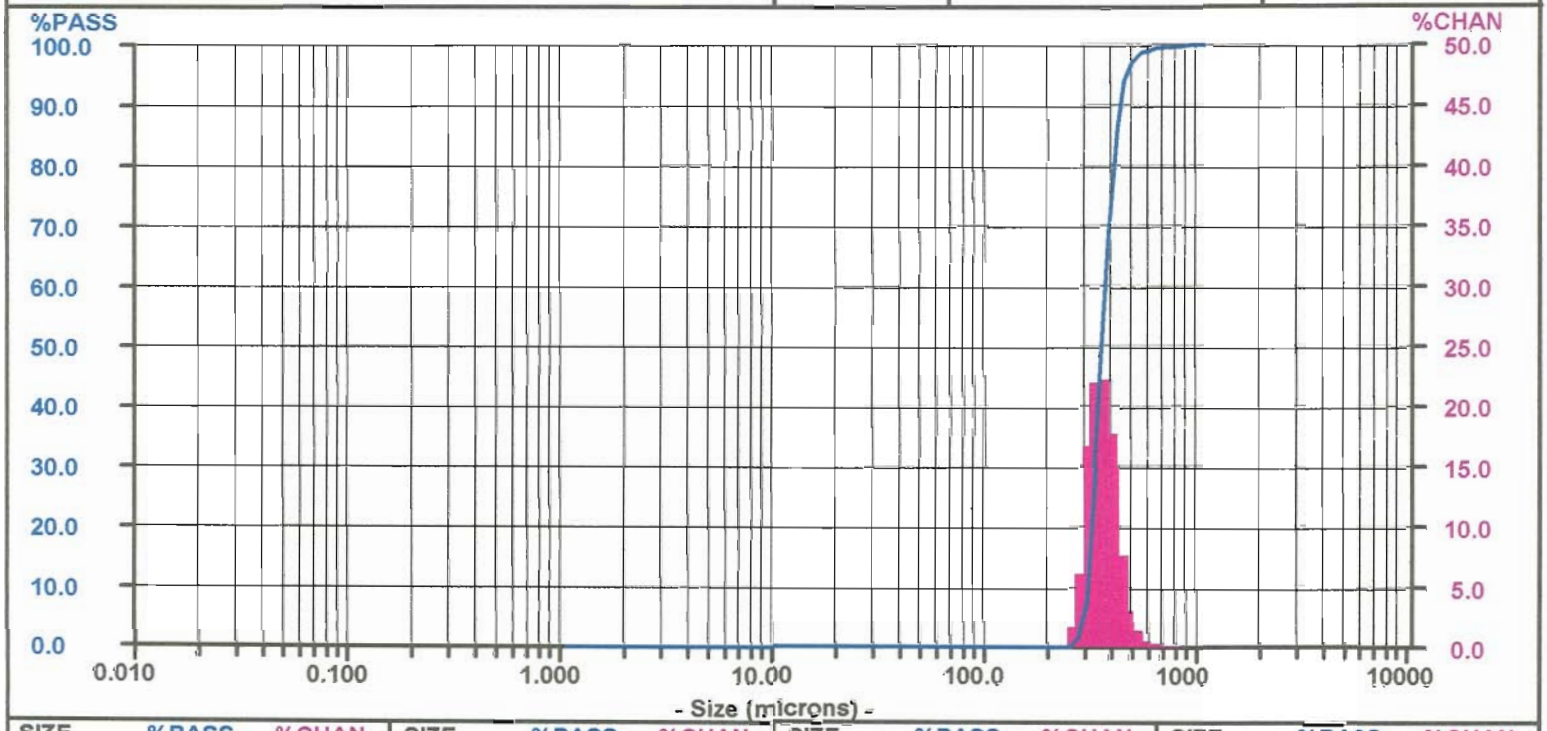
061206 Resin Anal - SKF

061206WCB06

Date: 06/16/06 Meas #: 147
Time: 13:26 Pres #: 1

MB #72 RF Resin H-Form,
SK Fiskum, 5/17/06
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Num%	Width
mv	= 418.7	5% = 287.0	60% = 371.0		357.1	100%	103.2
mn	= 367.3	20% = 316.3	70% = 386.3				
ma	= 394.7	30% = 330.2	80% = 404.5				
cs	= 0.015	40% = 343.5	90% = 433.2				
sd	= 51.52	50% = 357.0	95% = 467.5				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.04	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.96	0.06	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.90	0.08	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.82	0.11	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.71	0.13	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.58	0.19	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.39	0.29	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	99.10	0.59	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	98.51	1.45	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	97.06	3.06	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	94.00	7.72	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	86.28	17.64	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	68.64	22.27	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	46.37	21.93	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	24.44	16.67	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	7.77	6.09	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	1.68	1.68	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5103
Transmission: 0.96
Above Residual: 0.11
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

6/19/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

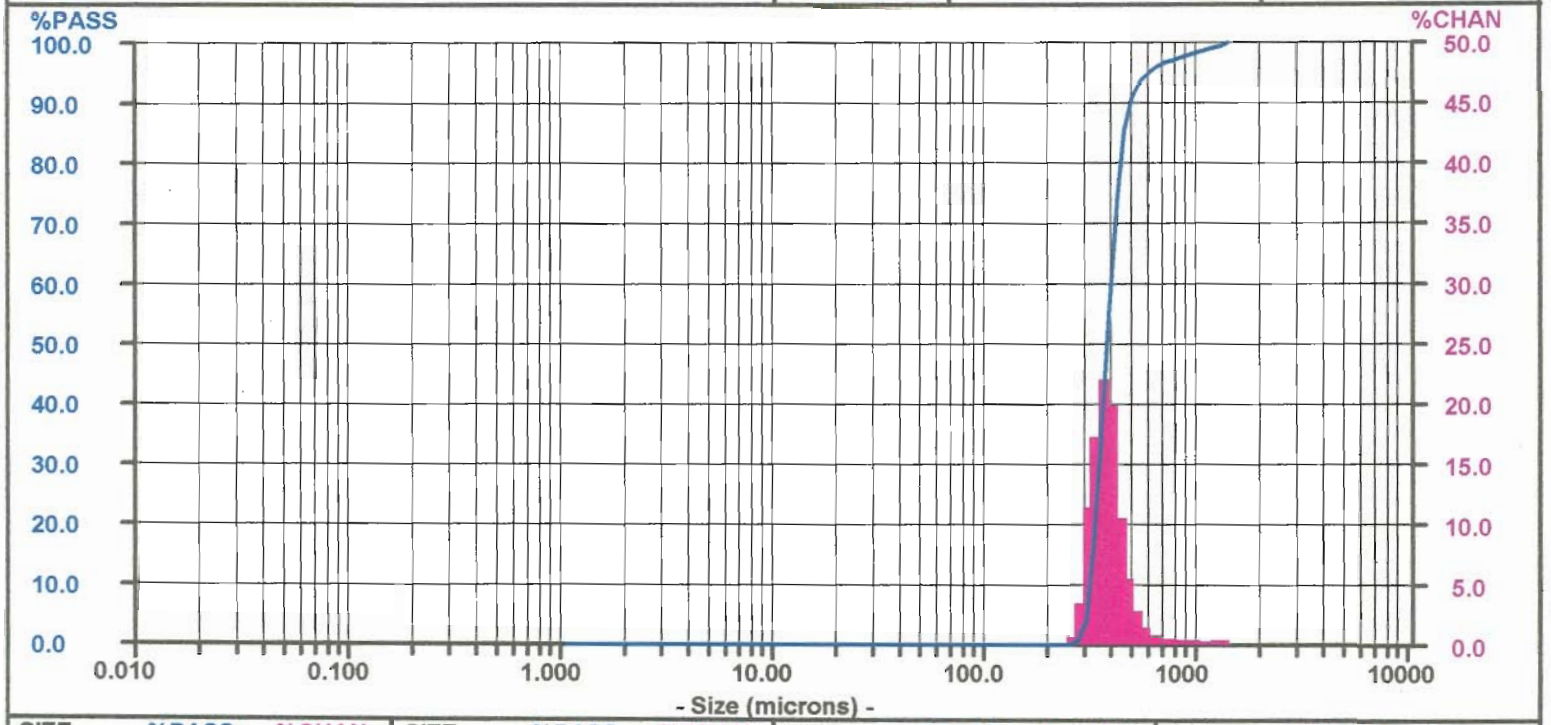
061206 Resin Anal - SKF

061206WCB06

Date: 07/10/06 Meas #: 147
Time: 15:58 Pres #: 1

MB #72 RF Resin H-Form,
SK Fiskum, 5/17/06
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Area%	Width
mv = 450.5		5% = 298.4	60% = 391.9		376.8	100%	126.6
mn = 368.3		20% = 330.8	70% = 409.2				
ma = 405.3		30% = 347.5	80% = 434.1				
cs = 0.015		40% = 362.4	90% = 489.8				
sd = 63.28		50% = 376.8	95% = 584.1				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.38	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.62	0.50	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.12	0.36	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	98.76	0.33	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	98.43	0.39	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	98.04	0.43	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	97.61	0.48	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	97.13	0.51	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	96.62	0.58	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	96.04	0.83	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	95.21	1.40	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	93.81	2.89	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	90.92	5.46	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	85.46	10.59	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	74.87	19.91	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	54.96	22.13	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	32.83	17.34	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	15.49	11.40	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	4.09	3.45	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.64	0.64	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.5103
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Original

061206 Resin Anal - SKF

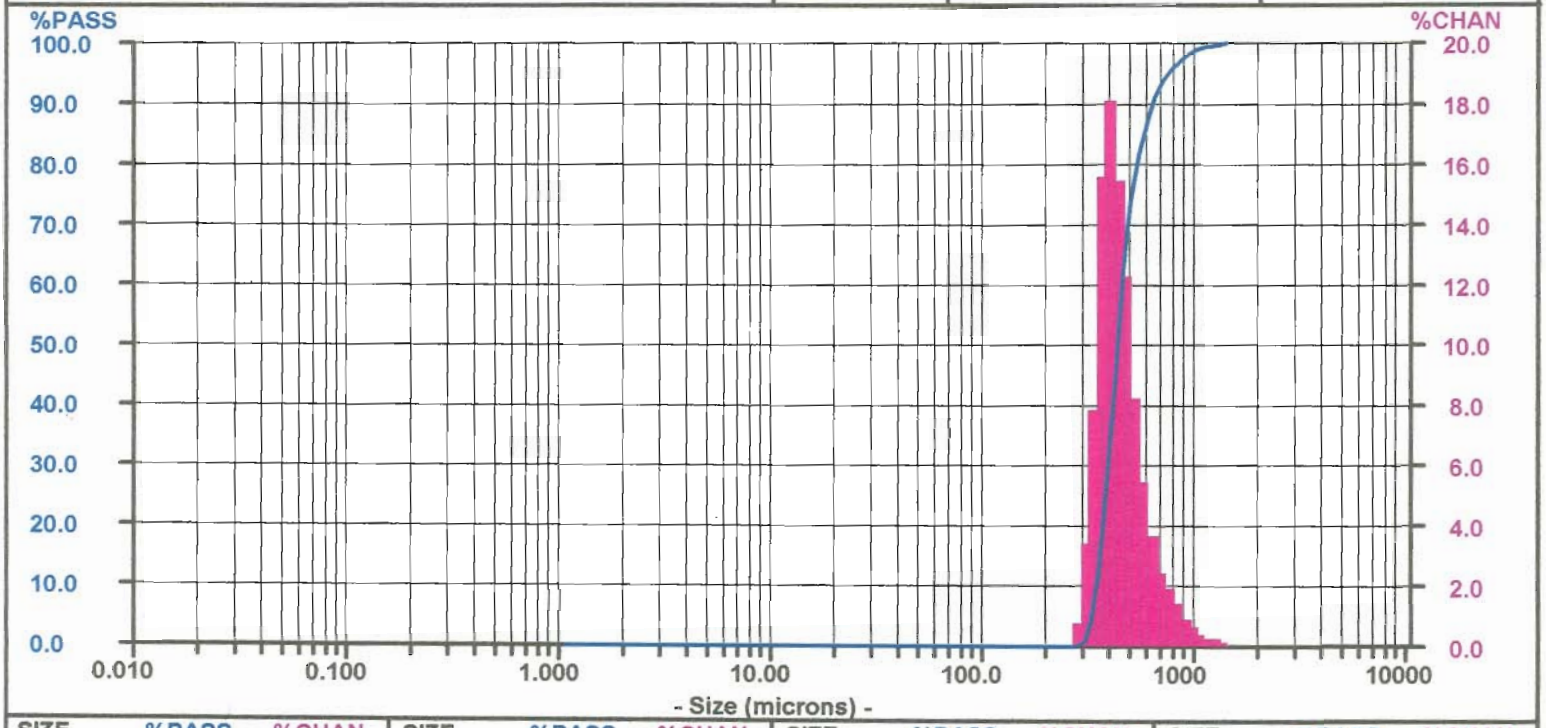
061506WCB05

Date: 06/15/06 Meas #: 167
Time: 17:39 Pres #: 2

MB #72 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Vol%	Width
mv = 464.5		5% = 325.4	60% = 452.7		427.8	100%	197.9
mn = 399.9		20% = 368.8	70% = 483.9				
ma = 435.9		30% = 387.9	80% = 530.0				
cs = 0.014		40% = 406.8	90% = 624.0				
sd = 98.94		50% = 427.8	95% = 739.9				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.19	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.81	0.28	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.53	0.28	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	99.25	0.41	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	98.84	0.68	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	98.16	0.99	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	97.17	1.41	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	95.76	1.91	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	93.85	2.53	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	91.32	3.72	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	87.60	5.51	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	82.09	8.33	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	73.76	12.31	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	61.45	15.45	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	46.00	18.14	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	27.86	15.58	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	12.28	7.87	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	4.41	3.51	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	0.90	0.82	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.08	0.08	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7606
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/29/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

061206 Resin Anal - SKF

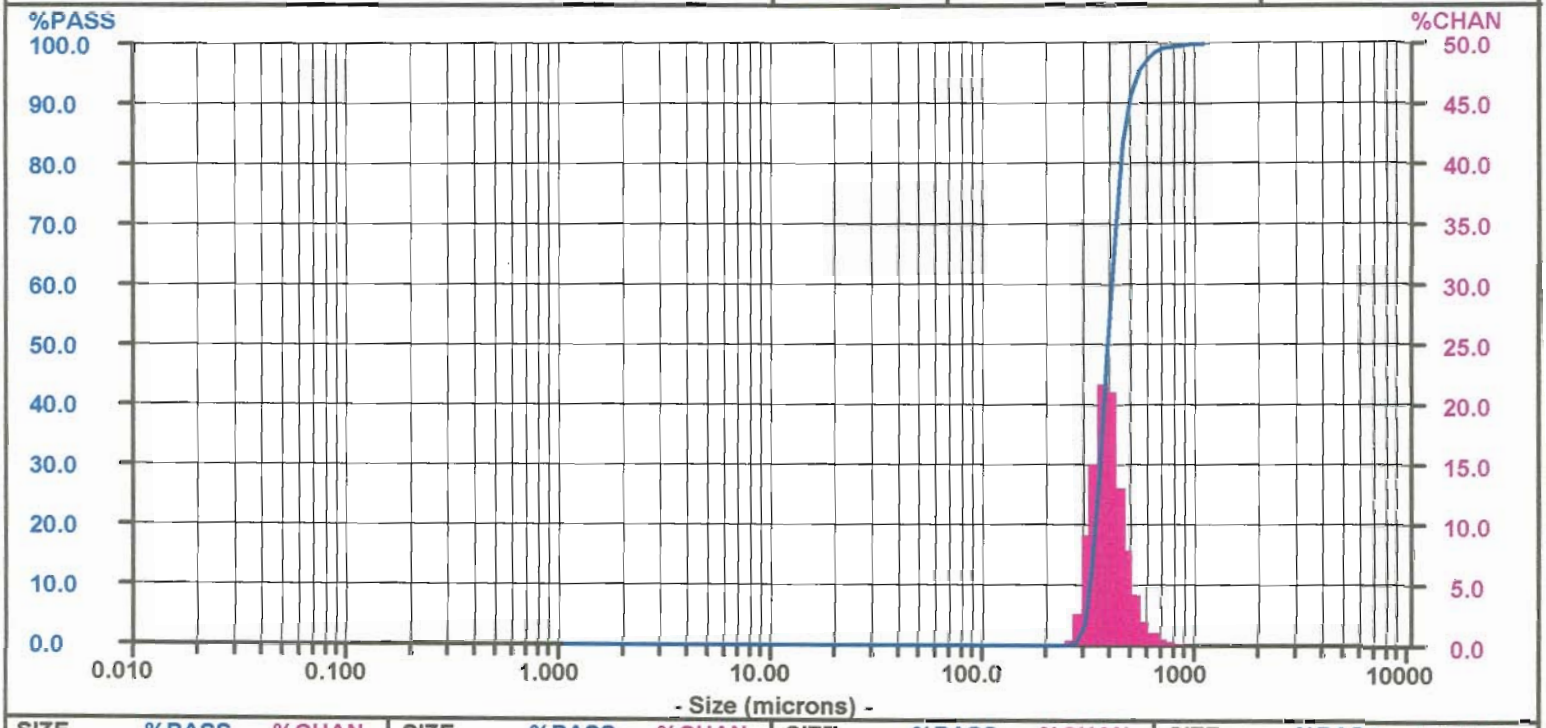
061506WCB05

Date: 06/16/06 Meas #: 167
Time: 13:34 Pres #: 1

MB #72 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Num%	Width
mv	= 458.8	5% = 302.0	60% = 400.1		385.0	100%	128.4
mn	= 399.6	20% = 338.5	70% = 417.9				
ma	= 433.8	30% = 356.7	80% = 444.6				
cs	= 0.014	40% = 370.5	90% = 489.2				
sd	= 64.14	50% = 384.9	95% = 536.0				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.03	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.97	0.06	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.91	0.10	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.81	0.20	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.61	0.33	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.28	0.58	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	98.70	1.09	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	97.61	2.03	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	95.58	4.19	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	91.39	7.86	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	83.53	13.12	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	70.41	21.11	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	49.30	21.73	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	27.57	15.15	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	12.42	9.18	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	3.24	2.76	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.48	0.48	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7606
Transmission: 0.92
Above Residual: 0.03
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/19/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

061206 Resin Anal - SKF

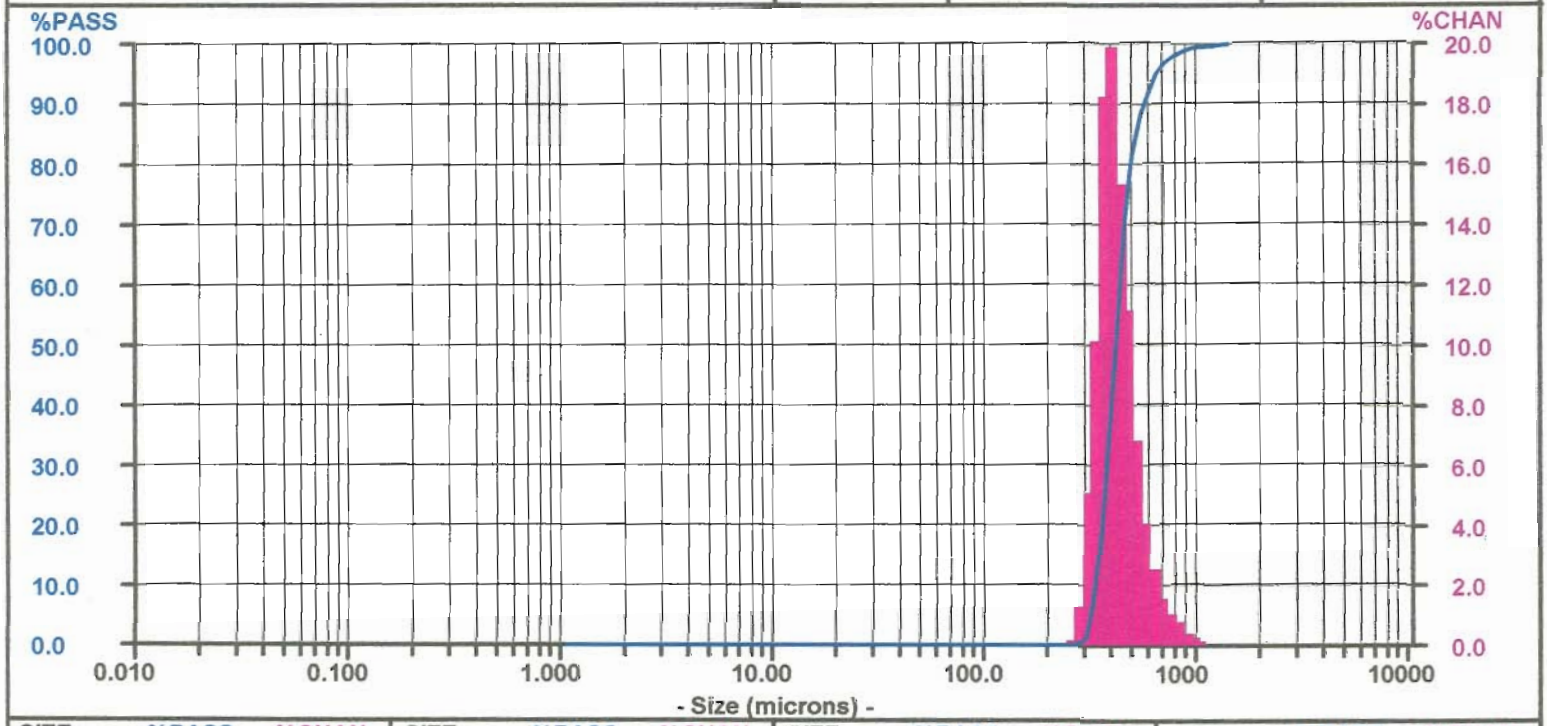
061506WCB05

Date: 07/10/06 Meas #: 167
Time: 15:59 Pres #: 1

MB #72 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Area%	Width
mv = 464.5		5% = 315.8	60% = 429.7		409.4	100%	161.5
mn = 399.9		20% = 357.9	70% = 455.4				
ma = 435.9		30% = 375.5	80% = 491.3				
cs = 0.014		40% = 392.1	90% = 558.3				
sd = 80.76		50% = 409.4	95% = 639.2				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.06	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.94	0.10	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.84	0.11	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	99.73	0.17	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.56	0.31	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.25	0.49	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	98.76	0.77	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	97.99	1.12	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	96.87	1.62	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	95.25	2.60	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	92.65	4.16	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	88.49	6.95	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	81.54	11.17	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	70.37	16.33	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	55.04	19.97	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	35.07	18.26	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	16.81	10.25	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	6.56	5.11	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	1.45	1.30	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.15	0.15	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7606
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Original

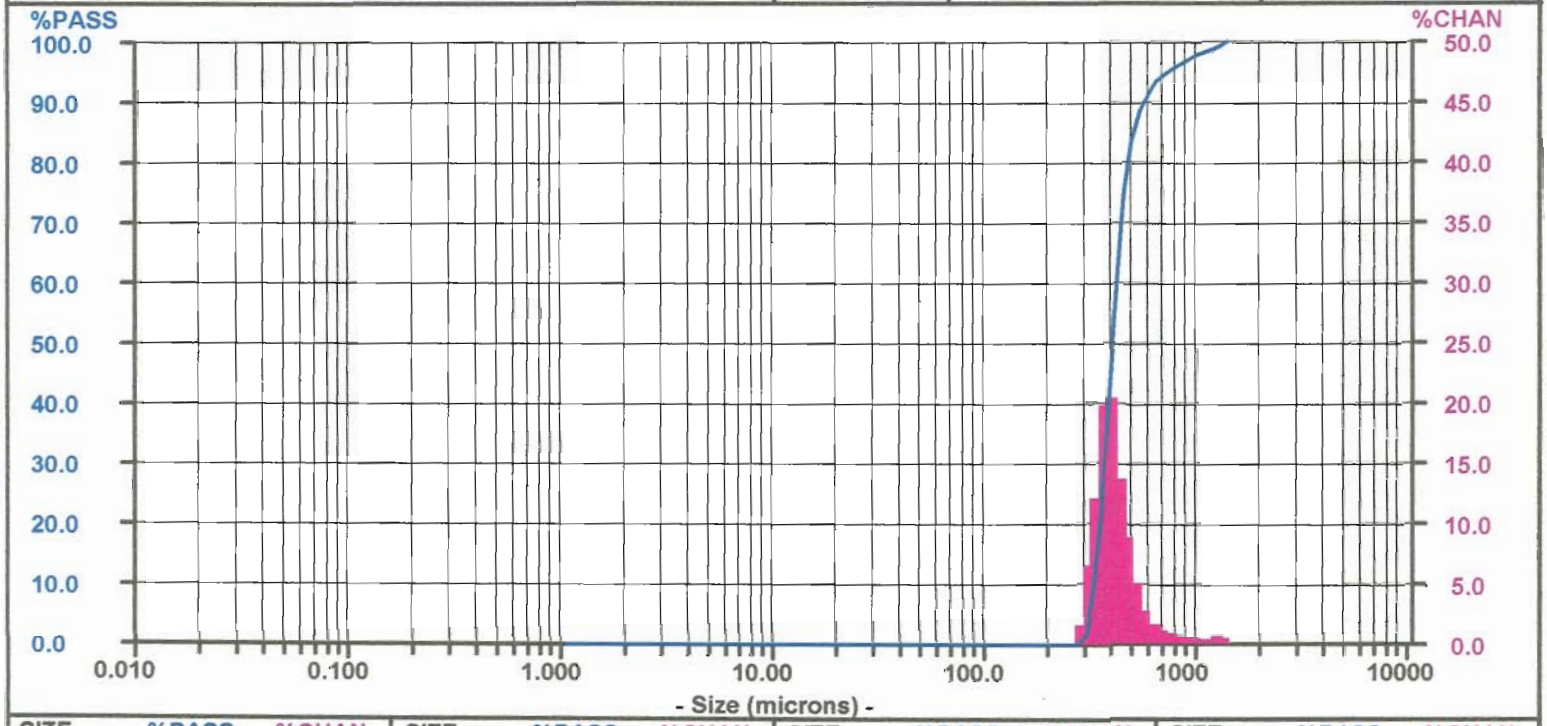
061206 Resin Anal - SKF

061206WCB10

Date: 06/12/06 Meas #: 151
Time: 21:01 Pres #: 2

BSC #73 H-Form,
SK Fiskum, 5/17/06
TI-RPP-WTP-449
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Vol%	Width
mv = 439.5		5% = 310.3	60% = 417.1		399.1	100%	159.0
mn = 377.6		20% = 360.6	70% = 442.1				
ma = 408.9		30% = 367.7	80% = 478.9				
cs = 0.015		40% = 383.2	90% = 561.7				
sd = 79.62		50% = 399.1	95% = 732.4				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.55	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.45	0.71	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	98.74	0.50	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	98.24	0.49	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	97.75	0.62	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	97.13	0.73	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	96.40	0.87	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	95.53	1.00	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	94.53	1.19	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	93.34	1.77	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	91.57	2.83	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	88.74	5.16	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	83.58	8.97	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	74.61	13.76	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	60.85	20.38	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	40.47	19.81	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	20.66	12.14	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	8.52	6.58	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	1.94	1.73	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.21	0.21	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7010
Transmission: 0.96
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/29/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

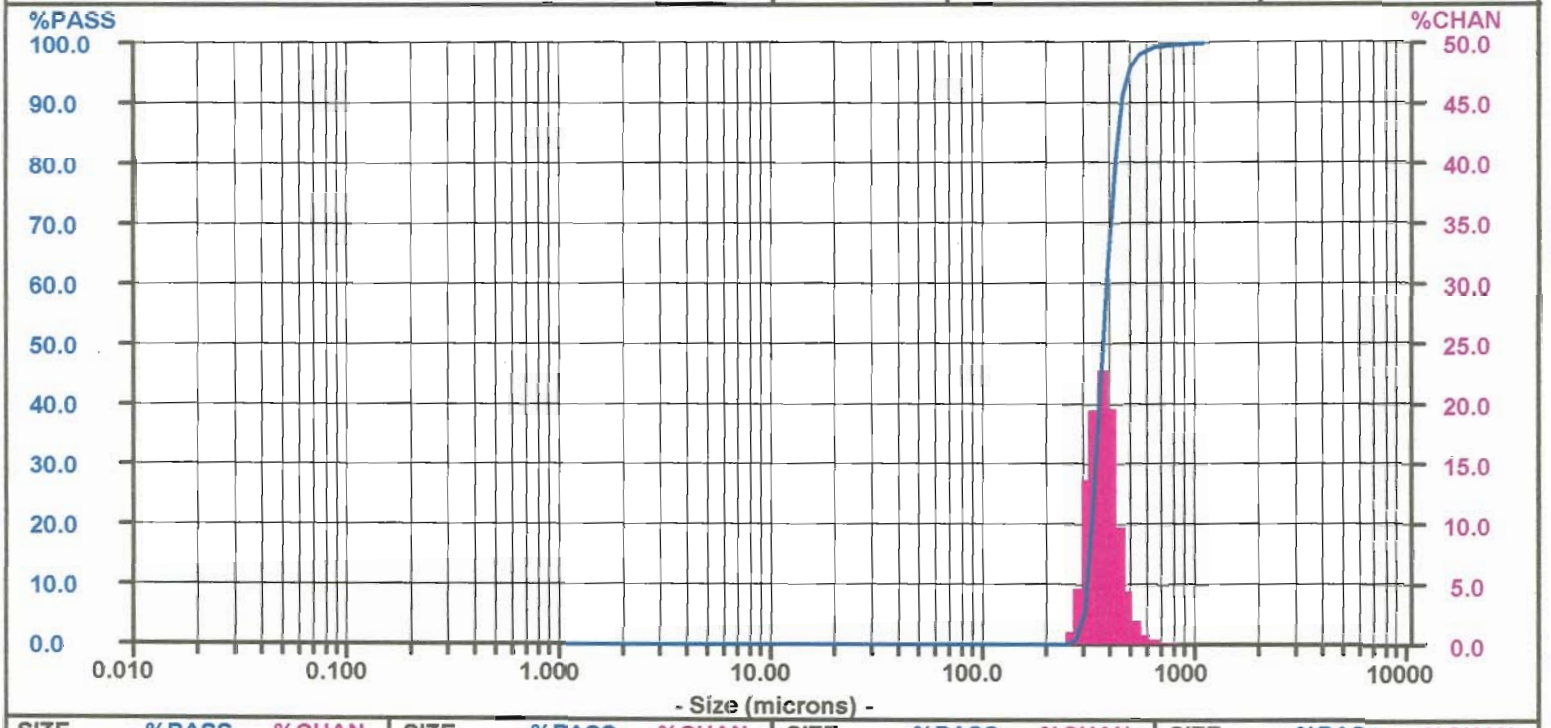
061206 Resin Anal - SKF

061206WCB10

Date: 06/16/06 Meas #: 151
Time: 13:30 Pres #: 1

BSC #73 H-Form,
SK Fiskum, 5/17/06
TI-RPP-WTP-449
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles		Dia	Num%	Width
mv = 425.1	5% = 292.6	60% = 381.1		367.0	100%	109.7
mn = 377.1	20% = 323.5	70% = 396.6				
ma = 404.0	30% = 338.8	80% = 415.3				
cs = 0.015	40% = 353.2	90% = 449.6				
sd = 54.77	50% = 367.0	95% = 486.9				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.03	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.97	0.04	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.93	0.06	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.87	0.11	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.76	0.14	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.62	0.24	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	99.38	0.42	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	98.96	0.85	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	98.11	2.14	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	95.97	4.55	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	91.42	9.76	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	81.66	19.66	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	62.00	22.86	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	39.14	19.59	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	19.55	13.74	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	5.81	4.69	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	1.12	1.12	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7010
Transmission: 0.95
Above Residual: 0.05
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

Filter: On

Database Path: C:\MTWIN9~1\WCBRESIN.DB

WCB 6/19/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

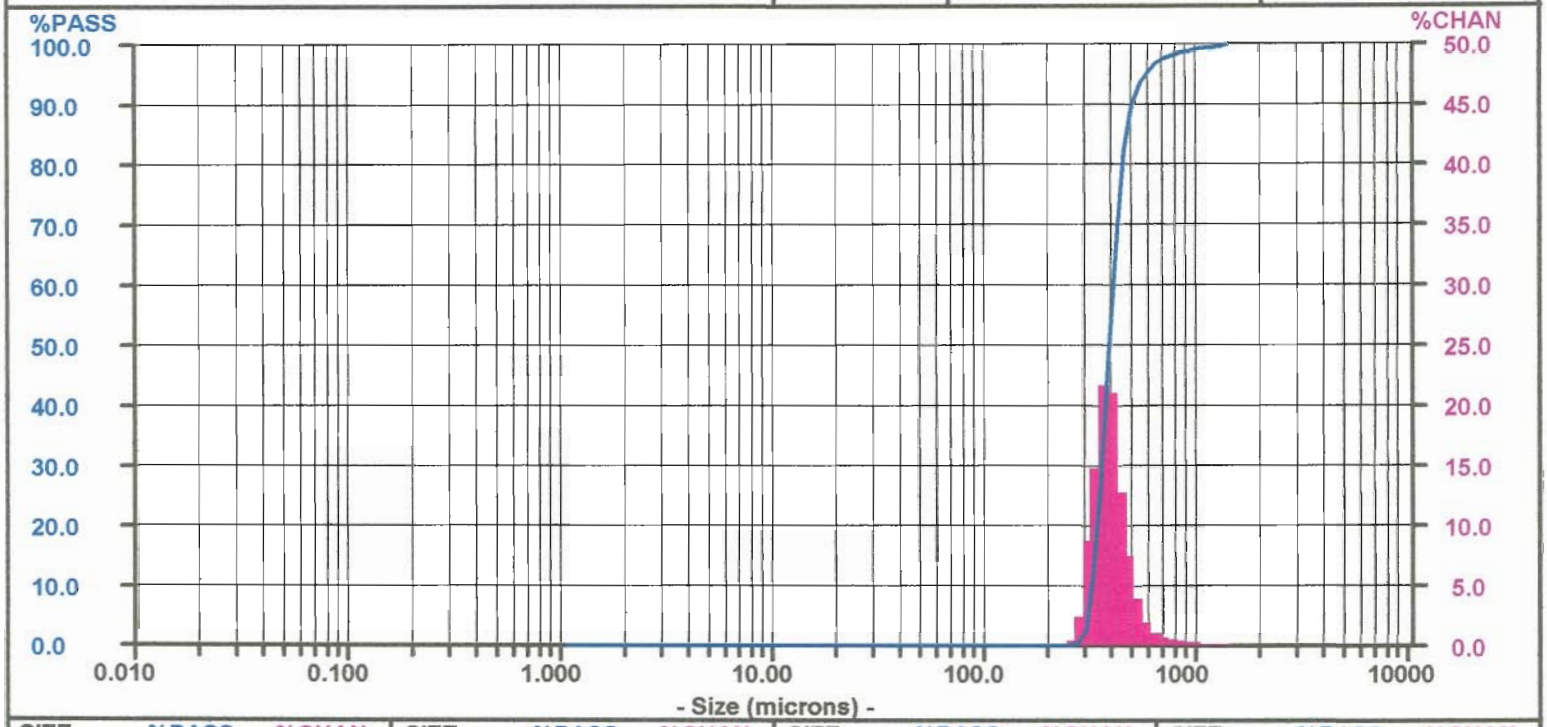
061206 Resin Anal - SKF

061206WCB10

Date: 07/10/06 Meas #: 151
Time: 15:58 Pres #: 1

BSC #73 H-Form,
SK Fiskum, 5/17/06
TI-RPP-WTP-449
Aliquot: 1
Re-run# (if applicable): NA

Summary		Percentiles			Dia	Area%	Width
mv	= 439.5	5% = 303.3	60% = 401.8		386.4	100%	133.1
mn	= 377.6	20% = 340.0	70% = 420.1				
ma	= 408.9	30% = 357.1	80% = 448.9				
cs	= 0.015	40% = 371.9	90% = 500.2				
sd	= 66.57	50% = 386.4	95% = 569.2				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.17	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.83	0.23	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.60	0.18	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	99.42	0.19	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.23	0.27	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	98.96	0.34	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	98.62	0.45	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	98.17	0.55	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	97.62	0.72	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	96.90	1.15	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	95.75	1.99	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	93.76	4.03	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	89.73	7.54	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	82.19	12.81	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	69.38	21.07	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	48.31	21.69	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	26.62	14.85	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	11.77	8.85	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	2.92	2.53	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.39	0.39	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.7010
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 7/11/12

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Original

061206 Resin Anal - SKF

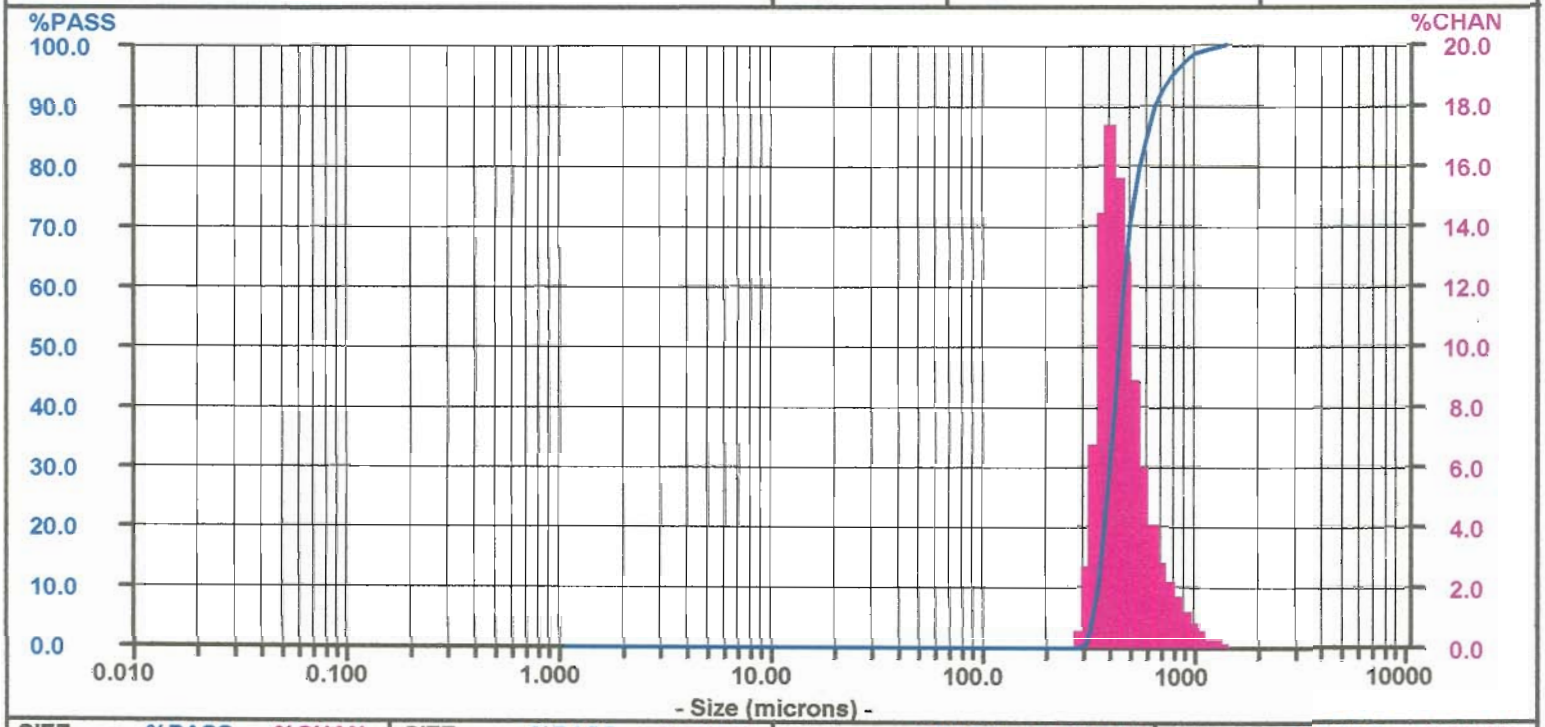
061506WCB01

Date: 06/15/06 Meas #: 163
Time: 16:54 Pres #: 2

BSC #73 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable):

Summary		Percentiles			Dia	Vol%	Width
mv = 476.6		5% = 330.9	60% = 463.3		436.7	100%	211.7
mn = 406.8		20% = 374.3	70% = 495.7				
ma = 445.5		30% = 394.3	80% = 546.3				
cs = 0.013		40% = 414.3	90% = 650.0				
sd = 105.8		50% = 436.7	95% = 772.8				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.23	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.77	0.35	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.42	0.35	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	99.07	0.51	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	98.56	0.83	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	97.73	1.20	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	96.53	1.66	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	94.87	2.22	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	92.65	2.89	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	89.76	4.16	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	85.60	6.07	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	79.53	8.93	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	70.60	12.89	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	57.71	15.61	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	42.10	17.41	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	24.69	14.44	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	10.25	6.78	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	3.47	2.78	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	0.69	0.63	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.06	0.06	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.6980
Transmission: 0.93
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 6/29/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

061206 Resin Anal - SKF

061506WCB01

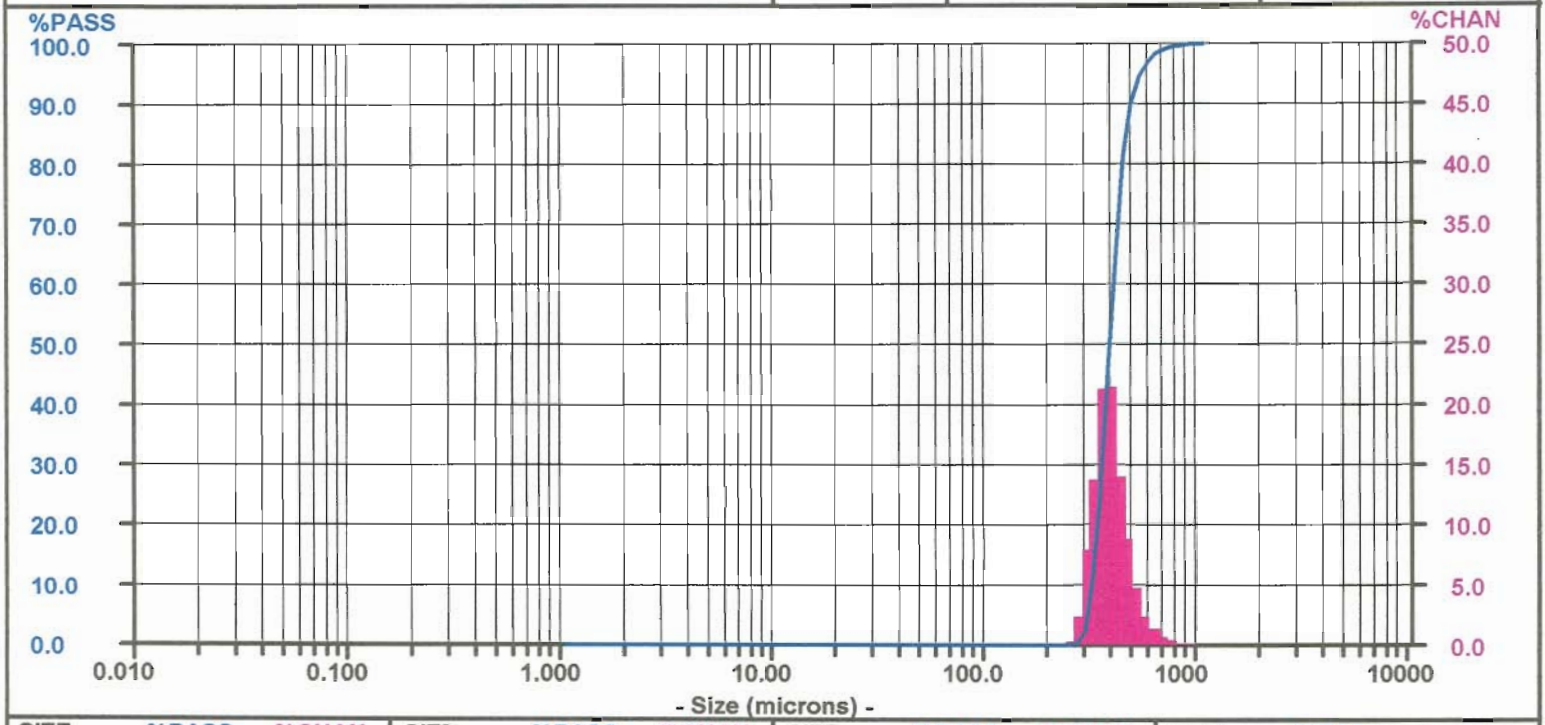
Date: 06/16/06 Meas #: 163
Time: 13:33 Pres #: 1

CSC #73 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable):

Summary		Percentiles			
mv	= 469.6	5% =	305.5	60% =	406.2
mn	= 406.4	20% =	343.7	70% =	425.4
ma	= 442.8	30% =	360.8	80% =	452.8
cs	= 0.014	40% =	376.7	90% =	498.0
sd	= 66.16	50% =	390.4	95% =	547.4

Dia	Num%	Width
390.5	100%	132.4



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1086	100.00	0.03	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.97	0.07	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.90	0.13	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	99.77	0.25	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	99.62	0.41	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	99.11	0.69	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	98.42	1.30	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	97.12	2.38	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	94.74	4.76	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	89.98	8.79	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	81.19	14.03	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	67.16	21.51	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	45.65	21.38	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	24.27	13.79	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	10.48	7.85	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	2.63	2.28	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.35	0.35	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			
161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00			
148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00			
135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00			

Distribution: Number
Progression: Geometric Root8
Upper Edge: 1086
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 81

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.6980
Transmission: 0.93
Above Residual: 0.04
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

WCB 6/19/06

Serial Number: S3177
Range: 0.021 -1408 um

MICROTRAC - S3000

V:9.1.15
DB : Recalc

061206 Resin Anal - SKF

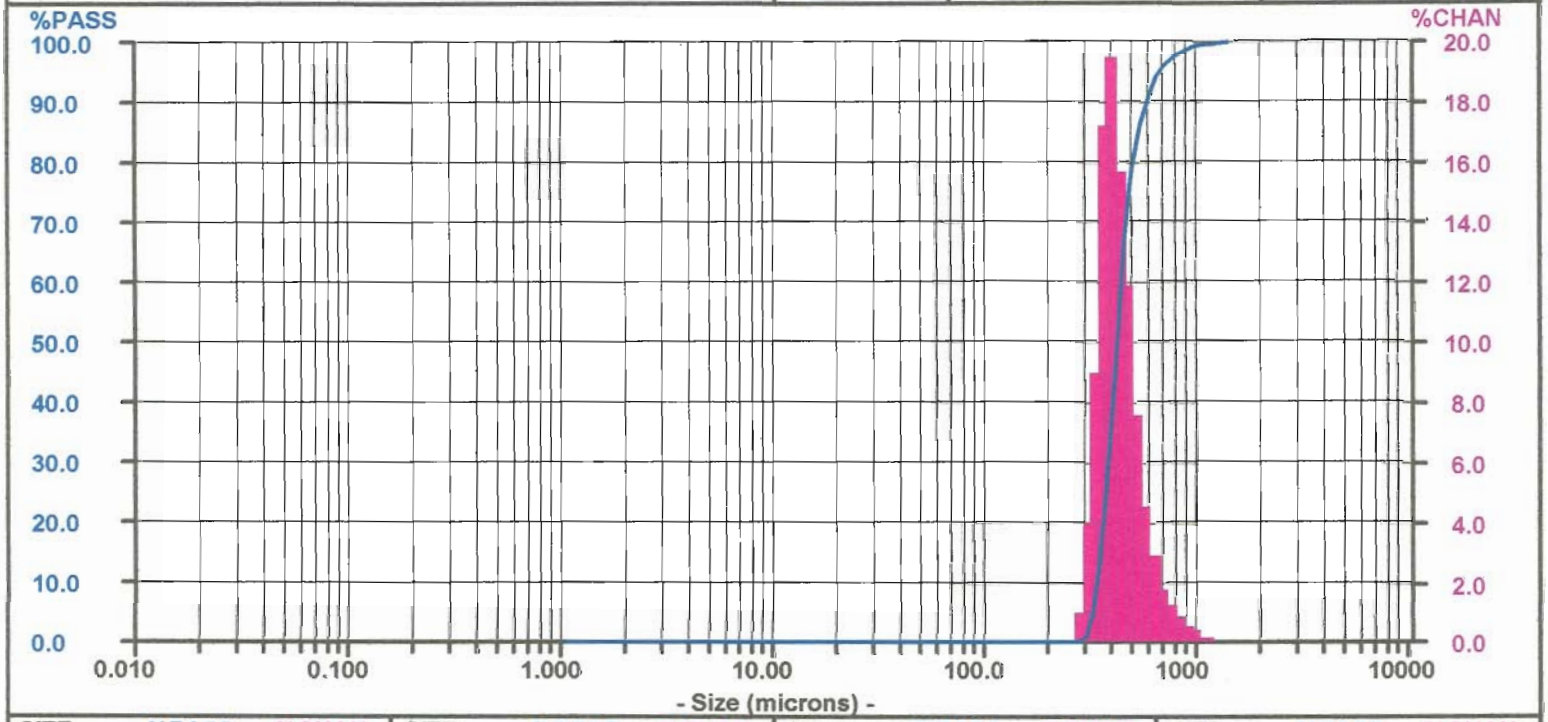
061506WCB01

Date: 07/10/06 Meas #: 163
Time: 16:00 Pres #: 1

BSC #73 Na-Form,
SK Fiskum, 05/18/06

Aliquot: 1
Re-run# (if applicable):

Summary		Percentiles			Dia	Area%	Width
mv = 476.6		5% = 321.2	60% = 438.1		416.3	100%	169.2
mn = 406.8		20% = 363.2	70% = 465.6				
ma = 445.5		30% = 381.1	80% = 502.7				
cs = 0.013		40% = 398.2	90% = 575.7				
sd = 84.60		50% = 416.3	95% = 664.2				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.08	161.4	0.00	0.00	18.50	0.00	0.00	2.121	0.00	0.00
1291	99.92	0.12	148.0	0.00	0.00	16.96	0.00	0.00	1.945	0.00	0.00
1184	99.80	0.14	135.7	0.00	0.00	15.56	0.00	0.00	1.783	0.00	0.00
1086	99.66	0.22	124.5	0.00	0.00	14.27	0.00	0.00	1.635	0.00	0.00
995.6	99.44	0.39	114.1	0.00	0.00	13.08	0.00	0.00	1.499	0.00	0.00
913.0	99.05	0.60	104.7	0.00	0.00	12.00	0.00	0.00	1.375	0.00	0.00
837.2	98.45	0.92	95.96	0.00	0.00	11.00	0.00	0.00	1.261	0.00	0.00
767.7	97.53	1.33	88.00	0.00	0.00	10.09	0.00	0.00	1.156	0.00	0.00
704.0	96.20	1.89	80.70	0.00	0.00	9.250	0.00	0.00	1.060	0.00	0.00
645.6	94.31	2.98	74.00	0.00	0.00	8.482	0.00	0.00			
592.0	91.33	4.68	67.86	0.00	0.00	7.778	0.00	0.00			
542.9	86.65	7.62	62.23	0.00	0.00	7.133	0.00	0.00			
497.8	79.03	11.98	57.06	0.00	0.00	6.541	0.00	0.00			
456.5	67.05	15.82	52.33	0.00	0.00	5.998	0.00	0.00			
418.6	51.23	19.59	47.98	0.00	0.00	5.500	0.00	0.00			
383.9	31.64	17.32	44.00	0.00	0.00	5.044	0.00	0.00			
352.0	14.32	9.02	40.35	0.00	0.00	4.625	0.00	0.00			
322.8	5.30	4.17	37.00	0.00	0.00	4.241	0.00	0.00			
296.0	1.13	1.02	33.93	0.00	0.00	3.889	0.00	0.00			
271.4	0.11	0.11	31.11	0.00	0.00	3.566	0.00	0.00			
248.9	0.00	0.00	28.53	0.00	0.00	3.270	0.00	0.00			
228.2	0.00	0.00	26.16	0.00	0.00	2.999	0.00	0.00			
209.3	0.00	0.00	23.99	0.00	0.00	2.750	0.00	0.00			
191.9	0.00	0.00	22.00	0.00	0.00	2.522	0.00	0.00			
176.0	0.00	0.00	20.17	0.00	0.00	2.312	0.00	0.00			

Distribution: Area
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.972
Residuals: Disabled
Number Of Channels: 84

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Resin
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN9~1\WCBRESIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.6980
Transmission: 0.93
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0084
Analysis Gain: 2

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