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# High Aspect Ratio Ion Exchange Resin Bed – Hydraulic Results for Spherical Resin Beads

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**Abstract:** A principal role of the DOE Savannah River Site is to safely dispose of a large volume of liquid nuclear waste held in many storage tanks. An in-tank ion exchange unit is being considered for cesium removal to accelerate waste processing. This unit is planned to have a relatively high bed height to diameter ratio (10:1). Complicating the design is the need to cool the ion exchange media; therefore, the ion exchange column will have a central cooling core making the flow path annular. To separate cesium from waste the media being considered is made of resorcinol formaldehyde resin deposited on spherical plastic beads and is a substitute for a previously tested resin made of crystalline silicotitanate. This spherical media not only has an advantage of being mechanically robust, but, unlike its predecessor, it is also reusable, that is, loaded cesium can be removed through elution and regeneration. Resin regeneration leads to more efficient operation and less spent resin waste, but its hydraulic performance in the planned ion exchange column was unknown. Moreover, the recycling process of this spherical resorcinol formaldehyde causes its volume to significantly shrink and swell. To determine the spherical media's hydraulic demand a linearly scaled column was designed and tested. The waste simulant used was prototypic of the wastes' viscosity and density. This paper discusses the hydraulic performance of the media that will be used to assist in the design of a full-scale unit.

**Keywords:** Cesium removal, formaldehyde, resorcinol, small column ion exchange, spherical resin

## INTRODUCTION

The Savannah River National Laboratory (SRNL) assisted the Savannah River Site (SRS) Tank Farm to determine the pressure drop that can be

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expected in a high aspect ratio ion exchange (IX) column (1). This process unit will be used to remove cesium from alkaline high-sodium wastes, referred to as waste feed or simply feed, in several SRS tanks. The IX column is called the Small Column IX (SCIX), and was originally designed to use a resin made of crystalline silicotitanate (CST) (2). The SCIX is now being evaluated for another type of resin made of resorcinol formaldehyde that is deposited on spherical plastic beads (spherical RF or SRF).

Much of the past IX work done at SRNL has been limited to aspect ratios (bed height to bed diameter) of about one (3). However, the current plan is to remove cesium from several SRS waste tanks with an IX column in-situ that needs a tall column and to use of SRF resin instead of CST. The column will have an inside diameter of 0.69 m and the needed height is being investigated with computer modeling and this hydraulic test. In theory, the column could be 7.6 m tall. The column will be installed within a riser of the tank under treatment and process the feed to remove cesium to an acceptable level. Such geometry leads to a bed aspect ratio of approximately 10:1. Since the exact dimensions were not fixed at the time of this test other heights were also considered, specifically 3 and 4.6 m. A further complication was the need to actively cool the IX column with a central cooling core (4). The cooling core is currently designed to traverse the height of the resin and have an outside core diameter of 0.17 m, effectively giving the IX column a hydraulic diameter of 0.52 m and an annular flow channel that will have a larger pressure drop than a column without a core.

The pressure field in an IX column with such a high aspect ratio, containing SRF resin, and an annular flow path, was unknown. Past IX experiments (2) have shown pressure fields for very high aspect ratios, but with different flow environments and a different resin, in both shape and size, e.g., CST. There was a need to determine the hydraulic demand for the new annular column design containing SRF resin. This paper discusses a test with a scale version, referred to as the SCIX Hydraulic Test, of the planned SCIX full-size column shown in Fig. 1, utilizing SRF media. The parameters utilized were:

- The plant considered nominal resin bed height (BH) versus hydraulic diameter, aspect ratios of 6, 9, and 15 based on a full-size hydraulic diameter of 0.52 m. This test maintained these same ratios using a column with hydraulic diameter of 0.055 m.
- The dP/L was matched at both scales by using the same superficial velocities, based on the full-scale feed flow rates of approximately

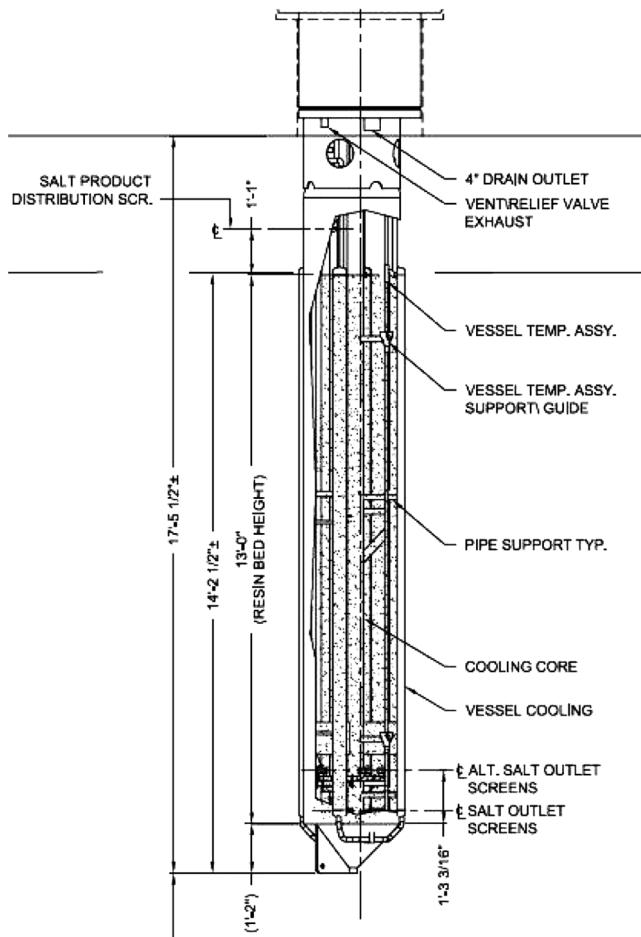


Figure 1. Full Scale SCIX (ORNL Drawing 1760-M-101, Rev. F, 9/8/2005).

20, 40, and 75 lpm. [ $dP/L$  is the pressure drop per length of the resin bed (or BH) during the processing of waste feed.]

- The pressure drop due to an annular flow was matched by using the same ratio of inner surface flow area. This was accomplished by linear scaling of the central cooling tube.
- The column temperature was ambient. It was not controlled, but it was measured.
- The operational procedures for a complete IX cycle followed a protocol developed during IX testing with SRF for the DOE River Protection Project – Waste Treatment Plant (3).

- Simulated feed and regenerated solutions were made to have similar mechanical properties to those that will be used in actual operation, specifically, having the same fluid densities and viscosities (5).
- Actual SRF resin was used to ensure an accurate flow regime.

## PREVIOUS EXPERIMENTAL WORK

### Oak Ridge National Laboratory

In developing a design for a 6 meter tall IX column for SRS that was to contain 4.9 m of resin, ORNL (2) carried out an experiment with a full height column, but with an inside diameter of 76 mm, compared to the full-size diameter of 0.69 m. The superficial velocity of the feed in down-flow ranged from 4.1 to 5.0 cm/min and the resin was made from the engineered form of crystalline silicotitanate (CST) known as Ionsiv<sup>TM</sup> IE-911 (ranging in size from 350 to 550 microns). At a 5 cm/min superficial velocity, the results showed a pressure drop of 51 kPa across the 4.9 m tall bed, or 10.4 kPa per meter of resin. The experiment was carried out at room temperature and the waste feed simulant had a sodium concentration of 5.6 M, which was developed at SRS (6) from what was considered an “SRS average waste composition.” The results of that experiment assisted in the design of the SCIX shown in Fig. 1, which was intended to hang from the top of a waste tank to be processed.

The IX unit included 3.96 m (13 ft) of media space, 0.33 m (13 in) of freeboard, 0.69 m (27.2 in) inside diameter column, and a 0.17 m (6.6 in) outside diameter central cooling core. Subsequent considerations changed the design to make the column taller and place it on the tank bottom. Another major change was to use spherical resorcinol formaldehyde (SRF) resin instead of crystalline silicotitanate (CST) media. SRF resin beads are more uniform in size and shape, leading to less resistance to flow, more resistant to fracturing, and SRF can be used over and over while CST must be discarded once it is fully loaded with cesium. Because SRF is different in shape than CST, and swells and contracts during the processes of regeneration and elution, it was necessary to obtain new hydraulic characteristics for proper design, thus leading to the current test.

### Savannah River National Laboratory

Fortunately, there existed extensive experience with SRF testing for 0.3 m (12 in) and 0.6 m (24 in) diameter columns at SRNL (3) in support of DOE’s River Protect Plan-Waste Treatment & Immobilization Project

(RPP-WTP). While these experiments dealt with resin beds that had an aspect ratio of  $\sim 1$  (bed height versus bed diameter) the information was very useful. These larger-column tests refined the operational IX protocol for RPP-WTP with the use of SRF media. This operational protocol included resin elution with acid, elution displacement with deionized water, resin regeneration with caustic, feed processing, feed displacement with a mild caustic, and followed by the displacement of the mild caustic with deionized water to repeat the complete IX cycle. Besides studying feed processing and resin regeneration, all other aspects of resin chemical and mechanical performances were analyzed. Due to these experiences SRS decided to use the same protocol to properly handle the resin for the SCIX Hydraulic Test.

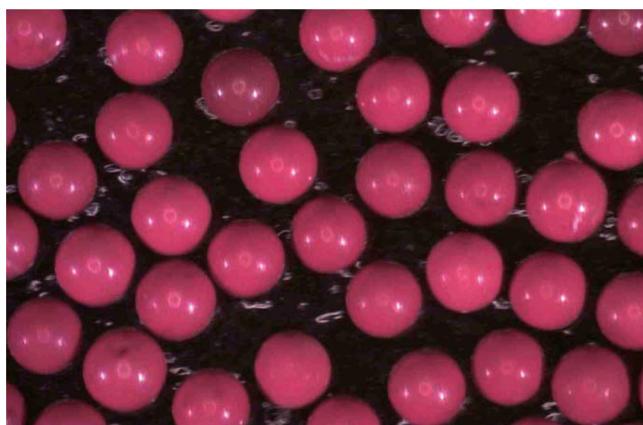
### Pacific Northwest National Laboratory

Tests with SRF were carried out at PNNL, but at smaller scales, i.e., 51 mm (2 in) and 76 mm (3 in) diameter columns (references 7 and 8, respectively), which were also done in support of the RPP-WTP ion exchange facility. These studies included hydraulic tests on various media, including SRF resin beads, to determine void fraction, permeability, and mechanical robustness under prototypic and non-prototypic conditions. Because the SCIX Hydraulic Test, of this paper, used a 76 mm diameter column the PNNL data (8) helped with predicting the pressure drop, so that instrumentation and measurement ranges could be planned. The lowest superficial velocity used for the 76 mm PNNL test was 7.2 cm/min, which was higher than the lowest superficial velocity planned for the 76 mm SCIX Hydraulic Test, i.e., 5.3 cm/min. Moreover, the PNNL feed viscosity was lower, i.e., 2.2 cP, than for the current test, i.e., 3.2 cP, and the PNNL bed aspect ratio was only 1.2:1, as opposed to the current test of 10:1; therefore, a direct comparison between the two experiments is not straight forward. However, by extrapolating the PNNL downflow results to a superficial velocity of 5.3 cm/min, and considering the more viscous feed, then the pressure drop would be approximately 10 kPa per meter of resin bed. This quantity gave a lower bound of the expected pressure drop because the annular flow channel for the SCIX column should produce a higher pressure drop than for the non-annular flow used by PNNL.

## CURRENT EXPERIMENTAL WORK

### The Ion Exchange Media

In its search for a replacement for its baseline resin the RPP-WTP IX facility at the DOE Hanford Site required a resin that was mechanically



**Figure 2.** New spherical resorcinol formaldehyde resin beads in H-form.

robust. An efficient ion adsorber is useless if it cannot be handled properly and exhibit good hydraulic characteristics. That is, past resins were shown to work effectively in a test tube by efficiently removing a target ion. However, as the scale increased to plant-size operational mechanical problems became evident. Past resins tended to break apart easily, creating many fines which led to plugging and unacceptable resistances to the flow of feed needing treatment. Even when they did not break down, irregularly-shaped and sharp-edged resin particles created prohibitive pressure drops, slurring problems, and non-uniform bed structures that caused resin plugs, and fissures that caused liquid channeling leading to less efficient operation. A candidate for a replacement resin was resorcinol formaldehyde (RF), which was developed in the 1980s (9) as an ion exchange media to separate radioactive cesium and strontium from nuclear waste. In 2002, the RF polymer matrix was deposited on spherical plastic beads by the Norwegian company Microbeads AS. Herein, this resin will be referred to as spherical resorcinol formaldehyde (SRF). The spherical shape not only provides a large exposure surface area, but it would be mechanically robust to minimize bead damage and be hydraulically favorable. Figure 2 shows a microphotograph of new SRF resin beads in acid form. Note how the beads are almost perfect spheres. Due to testing for RPP-WTP, the SRF media was also considered for use in the SCIX column at the DOE Savannah River Site and therefore is the subject of the current study.

New beads of SRF come in acid form (H-form), ready to be regenerated to sodium form (Na-form) in preparation to separate cesium from an alkaline high sodium waste. The new beads in H-form beads have an

average diameter of approximately 388 microns. While in sodium form the beads expand to approximately 450 microns. One of the principal drivers for this current test was to determine the frictional pressure drop caused by this spherical-shaped resin.

Because the SRS plant design for the working resin bed height had not been decided before this test began, three scaled heights were used: 0.32, 0.48, and 0.80 m. Knowing that the open-column flow area of the scaled unit was  $0.00386 \text{ m}^2$ , these heights lead to bed volumes of 1.2, 1.9, and 3.1 liters, respectively. New SRF resin was used for the SCIX Hydraulic Test and it was pretreated following a RPP-WTP protocol (10). Pretreatment conditions new resin that starts out in H-form with caustic so that it is ready to separate cesium from waste feed. Moreover, pretreatment allows the resin beads to expand to their largest size so that a working resin bed height can be set in the IX column.

### Feed and Regeneration Solutions

SRS, like RPP-WTP, was looking for an IX media to replace that which was originally planned and in the case of SRS this was crystalline silicotitanate (CST). An important reason that the SRF media was selected for study with the SCIX facility was because once it is loaded with cesium it can be regenerated and reused many times. CST resin must be removed and discarded after becoming loaded with its target ion, while SRF resin can be regenerated in-situ by stripping (eluting) it of the target ion so that it can be reused to process more waste. To regenerate SRF media it goes through a six-step process, collectively called a cycle, using five different solutions. Those steps are:

IX Cycle Step	Solution	Reason
1. Feed Treatment	Alkaline-based waste	Remove Cs
2. Feed Displacement	0.1 M Sodium Hydroxide	Prevent precipitation
3. Caustic Displacement	Deionized Water	Prepare for elution
4. Elution	0.5 M Nitric Acid	Strip resin of Cs
5. Acid Displacement	Deionized Water	Remove acid
6. Regeneration	0.5 M Sodium Hydroxide	Prepare resin to receive Cs

For the current test all of the solutions, except the simulated feed, were made at the test location and titrated to verify molarity. The hydraulic resistance to flow through the resin bed was obtained using a simulant that matched the feed's rheology and pH, i.e., greater than 13. At the time of the test the actual feed to be processed did not exist. That is, existing stored tank wastes are primarily in the form of a saltcake

**Table 1.** Average SRS waste feed simulant used in the SCIX CST test (Table 2.1 of 2)

Component	Average concentration ( <i>M</i> )
Na <sup>+</sup>	5.6
Cs <sup>+</sup>	0.00014
K <sup>+</sup>	0.015
OH <sup>-</sup>	1.91
NO <sub>3</sub> <sup>-</sup>	2.14
NO <sub>2</sub> <sup>-</sup>	0.52
AlO <sub>2</sub> <sup>-</sup>	0.31
CO <sub>3</sub> <sup>2-</sup>	0.16
SO <sub>4</sub> <sup>2-</sup>	0.15
Cl <sup>-</sup>	0.025
F <sup>-</sup>	0.032
PO <sub>4</sub> <sup>2-</sup>	0.010
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	0.008
SiO <sub>3</sub> <sup>2-</sup>	0.004
MoO <sub>4</sub> <sup>2-</sup>	0.0002

and to be processed through an IX column they require a significant amount of water to dissolve the solids. Those diluted wastes are expected to be a liquor with an adjusted sodium concentration of 6 M. To that end SRNL evaluated the expected feed streams from the SRS tanks to be treated (5), and compare these to the simulant used for the previous SCIX test done at ORNL (2), which is listed in Table 1.

That simulant investigation led to the following results shown in Table 2. The “Average SRS” waste feed shown in the table was used in the ORNL test, but was developed at SRS (6).

Of primary importance in measuring hydraulic resistance through a packed bed of resin beads is the liquid’s viscosity (11); therefore, based on Table 2, the “Average SRS” waste feed was chosen for the current

**Table 2.** Waste feed stream evaluation done by SRNL (Properties at 25°C)

Measured quantity	Waste feed mixture		
	Average SRS	Tank 1	Tank 3
Na, moles/Liter	6	6	6
OH <sup>-</sup> , moles/Liter	2.08	1.42	0.56
NO <sub>3</sub> <sup>-</sup> , moles/Liter	2.33	3.10	4.72
Viscosity, cP	3.24	3.04	2.36
Density, g/mL	1.273	1.285	1.294

test because it was the most viscous and would give a conservative result.

### Scaling Parameters

A scaled IX test facility was designed to bound the flow conditions for a preliminary column design. The planned full-scale SCIX column has a 0.69 m (27.2 in) inside diameter, a 0.17 m (6.6 in) central cooling tube, would be filled with approximately 4 m (13 ft) of SRF resin, and have approximately 0.3 m (1 ft) of freeboard above the resin bed, see Fig. 1. The full-scale dimensions, the scaled dimensions, and the results of planning analyses are summarized in Table 3. To better understand Table 3 some of its features are listed below:

The top half of Table 3 contains full-scale and small-scale column dimensions

- The superficial velocity was maintained at both scales to produce the same dynamic pressure drop.
- The ratio of the surface areas of the inside diameter of the column to the outside diameter of the cooling core was maintained, since this is a principal hydraulic parameter. This could be done with linear scaling; therefore, linear scaling was used throughout.
- Bed height was scaled linearly.

The remaining rows contain information on feed flow and flow regime to estimate pressure drop

- Feed properties were maintained prototypic.
- Flow regimes were maintained prototypic.
- Because of all of the above, the pressure drop per height was expected to be prototypic.

### Test Equipment

#### The Small-Scale Test Column

The 76 mm (3 in) inside diameter plastic tube, which actually measured to be 72.1 mm, used for the scaled test is shown in Fig. 3 and pressure taps were installed at locations thought to best capture the hydraulic performance of the resin. As seen in Table 3 the three scaled bed heights of resin in sodium form would be 319, 478, and 797 mm. The pressure taps were

Table 3 . Analyses done to design 72 mm scale SCIX Hydraulic Test

Scaling criteria parameter	Full-scale	Unit	Small-scale	Unit	Scale
HI = Resin Bed Height 1	3.05	m	31.88	cm	10
H2 = Resin Bed Height 2	4.57	m	47.82	cm	10
H3 = Resin Bed Height 3	7.62	m	79.70	cm	10
Percentage of height above resin to screen in CST column design <sup>a</sup>	7.69	%	7.69	%	1
Freeboard above highest resin height for the CST column design	0.59	m	6.13	cm	10
Maximum CST column screen-to-screen Height = H3 + Freeboard	8.21	m	85.83	cm	10
Screen-to-screen height for scaled column for RF resin expansion and fluidization	na	-	152.40	cm	na
Freeboard above highest resin height in the scaled column design	na	-	72.70	cm	na
D = Inside Diameter of Column <sup>b</sup>	0.69	m	7.21	cm	10
d = Outside Diameter of the Inner Core <sup>c</sup> (scaled by d/D)	0.17	m	1.75	cm	10
d = Outside Diameter of the Inner Core <sup>c</sup> (scaled with an available 5/8-inch tube stock)	0.17	m	1.71	cm	10
Dh = Hydraulic Diameter = 4 × Flow Area / Wetted Perimeter	0.52	m	5.50	cm	10
HI /Dh = Aspect Ratio 1	5.82	-	5.79	-	1
H2/Dh = Aspect Ratio 2	8.73	-	8.69	-	1
H3/Dh = Aspect Ratio 3	14.55	-	14.49	-	1
Flow Area	0.38	m <sub>2</sub>	40.87	cm <sup>2</sup>	92
Flow Area (annular)	0.35	m <sub>2</sub>	38.57	cm <sup>2</sup>	92
Flow Outside Surface Area, for HI	6.63	m <sub>2</sub>	722	cm <sup>2</sup>	92
Flow Outside Surface Area, for H2	9.94	m <sub>2</sub>	1084	cm <sup>2</sup>	92
Flow Outside Surface Area, for H3	16.57	m <sub>2</sub>	1806	cm <sup>2</sup>	92
Flow Inside Surface Area, for HI	1.61	m <sub>2</sub>	171	cm <sup>2</sup>	94
Flow Inside Surface Area, for H2	2.42	m <sub>2</sub>	257	cm <sup>2</sup>	94

Flow Inside Surface Area, for $H_3$	4.03	$m_2$	429	$cm^2$	94
Flow Out/In Surface Area = $D/d$ (at the same resin bed height)	4.11	—	4.21	—	1
Feed Flowrate 1	3.15E-04	$m^3/s$	3.44	$cm^3/s$	92
Feed Flowrate 2	9.46E-04	$m^3/s$	10.31	$cm^3/s$	92
Feed Flowrate 3	1.58E-03	$m^3/s$	17.18	$cm^3/s$	92
$V_1$ = Superficial Velocity 1	5.03	$cm/min$	5.03	$cm/min$	1
$V_2$ = Superficial Velocity 2	15.09	$cm/min$	15.09	$cm/min$	1
$V_2$ = Superficial Velocity 3	25.15	$cm/min$	25.15	$cm/min$	1
$V_{1a}$ = Superficial Velocity 1 (annular)	5.35	$cm/min$	5.35	$cm/min$	1
$V_{2a}$ = Superficial Velocity 2 (annular)	16.04	$cm/mm$	16.04	$cm/min$	1
$V_{3a}$ = Superficial Velocity 3 (annular)	26.73	$cm/min$	26.73	$cm/min$	1
<i>Feed and Packed-Bed Parameters for following analyses</i>					
$\rho$ = Solution Density @ $25^{\circ}C^d$	1.27	$g/mL$	1.27	$g/mL$	1
$\mu$ = Dynamic Viscosity @ $25^{\circ}C^d$	3.24	$cP$	3.24	$cP$	1
$V$ = Kinematic Viscosity @ $25^{\circ}C$	2.545E-06	$m^2/s$	2.545E-06	$m^2/s$	1
$\varepsilon$ = Void Fraction [5]	0.42	—	0.42	—	1
$D_p$ = SRF Particle Diameter with feed <sup>e</sup>	456	mm	456	mm	1
$\varphi$ = Particle Sphericity (RF bead assumed to be a perfect sphere) <sup>e</sup>	0.99	—	0.99	—	1
<i>Reynolds Number calculation to determine flow regime</i>					
Largest Circular Tube Reynolds No. = $D \times V_3/V$	1140	laminar	119	laminar	10
Largest Annular Tube Reynolds No. = $D \times V_{3a}/V$	917	laminar	96	laminar	10
<i>Particle Reynolds Number calculation to determine packed-bed flow regime</i>					
Largest Circular-Tube Particle Reynolds No. = $D_p \times V_3/[V \times (1-\varepsilon)]$	1.29	laminar	1.29	laminar	1
Largest Annular-Tube Particle Reynolds No. = $D_p \times V_{3a}/[V \times (1-\varepsilon)]$	1.38	laminar	1.38	laminar	1

*(Continued)*

Table 3. Continued

Scaling criteria parameter	Full-scale	Unit	Small-scale	Unit	Scale
<i>Expected Pressure Drops based on existing packed bed research</i>					
Largest Circular Tube $dP/L = 150 \times V3 \times m \times (1-\epsilon)^2 / [Dp^2 \times (\phi \times \epsilon^3)^f]$	45393	Pa/m	45393	Pa/m	1
Largest Annular Tube $dP/L = 150 \times V3a \times m \times (1-\epsilon)^2 / [Dp^2 \times \phi^2 \times \epsilon^3]^f$	48244	Pa/m	48244	Pa/m	1
Largest Annular Tube $dP/L$ (from factor below of $dP(\text{annul})/dP$ (circ.))	122513	Pa/m	122513	Pa/m	1
<i>Ratio of analytical <math>dp</math> for circular-to-annular flow with same length and orientation</i>					
$dP(\text{annular})/dP(\text{circular})$ [7]	2.54	—	2.54	—	1

<sup>a</sup>The full-scale CST DC column called for a nominal 4 m (13 ft) of fully swollen resin and the top screen to sit 0.33 m (13 inches) above the resin (see Figure 1)

<sup>b</sup>The full-scale CST IX column is to be made from a nominal O.D. of 28 inches, sch. Std pipe [winch has a 0.375-in wall or an ID = 27.25 in.(or 0.69 m)] and the scaled column was made from a 7.6-cm (3") plastic tube with an actual inside diameter =  $7.21 \pm 0.05$  cm.

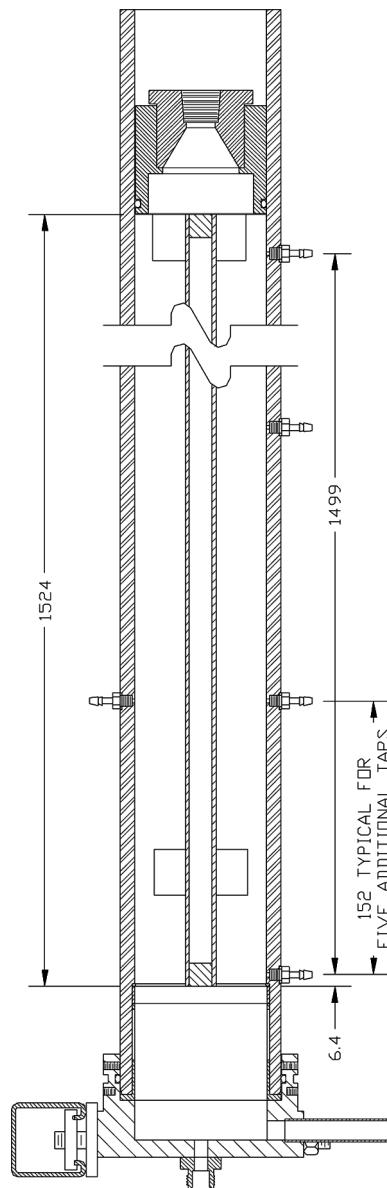
<sup>c</sup>The full-scale nominal outside diameter of the cooling core is to be made from 6 inches sch 40 pipe [O.D. = 6.625 in.(0.168 m)] and the scaled cooling core that was made from 5/8-inch tubing (1.59 cm) actually had an outside diameter of  $1.71 \pm 0.005$  cm.

<sup>d</sup>Properties are based on an Average SRS Salt Solution (6) that was adjusted to 6M sodium as shown in Table 2.

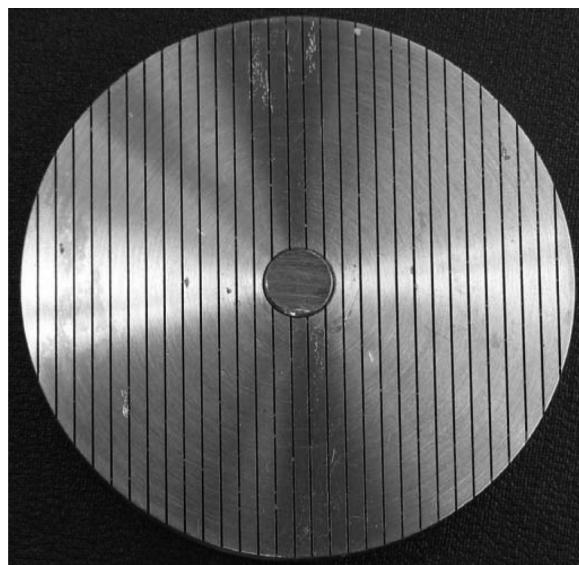
<sup>e</sup>Based on values determined in previous work (3).

<sup>f</sup>Blake-Kozeny equation (11) which is valid for laminar flow when Particle Reynolds No. < 10.

<sup>g</sup>See Reference (12).



**Figure 3.** Scale IX column used for SCIX Hydraulic Test [72 mm I.D.] [All dimensions are in millimeters].



**Figure 4.** Johnson screen used for the SCIX Hydraulic Test.

set at 152-mm (6 in) intervals starting at 6.4 mm (0.25 in) above the bottom resin screen. Other features included the central cooling core. The core was held in the center of the tube by metal fins. The bottom fins were linearly scaled to be at the same location as those in the prototypic column shown in Fig. 1. The centering fins are hard to see in Fig. 1; therefore, other drawings were used to secure the correct dimensions. Another feature was the top plug, which was made removable so that when the resin bed height needed increasing the plug could be removed.

#### Johnson Screen

SRF resin in the column was supported by a Johnson screen, Fig. 4. The overall diameter of the screen was 76 mm (3 in) and because the inside diameter of the scale IX column was actually 72 mm some of the tube wall was removed to properly seat the screen. The screen had a wire size of 2.24 mm, and the slots were a uniform 225 microns in width. This was sufficient to prevent the SRF resin from passing through. In acid form the smallest SRF resin diameter, when it is new, is approximately 380 microns. With these dimensions the flow area through the screen was estimated to be  $3.95 \text{ cm}^2$ , or approximately 10% of the flow area in the empty annular column, i.e.,  $\pi/4 \times (7.21 \text{ cm})^2 = 40.8 \text{ cm}^2$ . The center hole, which existed from previous work, was plugged because this is where the central cooling core sat.

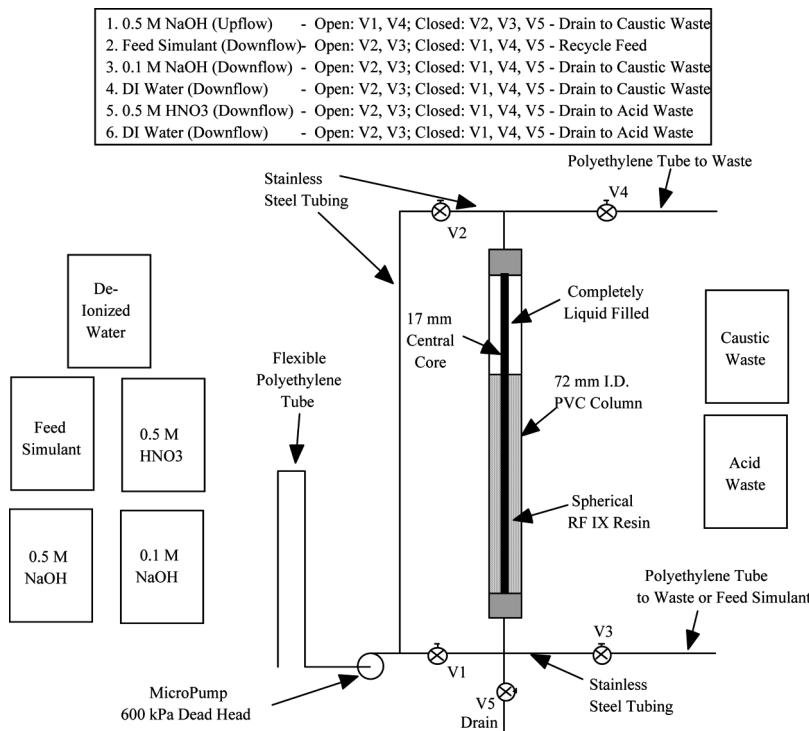
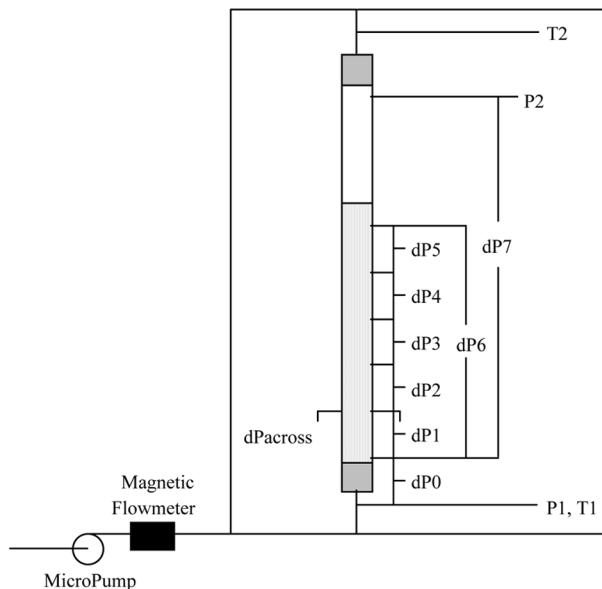


Figure 5. Schematic of the experimental setup.

## Test Setup

Figure 5 shows a schematic of the test setup that was made for simple operation, and as can be seen, only a minimum number of valves existed so that the column flow could be reversed between upflow and downflow easily and quickly. The column was made to be leak-free and the Micro-Pump was able to supply up to 600 kPa to the column for safe operation.

In most cases the operation of the scaled SCIX Hydraulic Test was an open system with once through flow of solutions. That is, water, caustic, and acid passed through the IX column and resin bed only once and then were discarded. However, most of the feed waste simulant was recycled in a closed system. When the feed was introduced it was allowed to flow through the column and discarded until three column volumes (CV) were processed. After three CV, the feed was then directed back to the feed tank to be recycled. The first three CV of simulant potentially contained residual regenerate fluid (dilute NaOH), and would therefore dilute the recycled simulant if not discarded. The recirculation of feed



**Figure 6.** Instrument locations.

reduced the simulant quantity needed and minimized the creation of unnecessary waste.

The instruments shown in Fig. 6 consisted primarily of pressure transducers that needed a complex series of sensor tubes and valves so that those tubes could be purged throughout the experiment. That is, to obtain accurate differential pressure measurements it was very important that the sensor tubes, as well as the transducers themselves contained the correct density fluid. Those tubes and valves are not shown in Fig. 6 because they are not necessary to understand the basic test operation and would obscure the salient features of instrument locations.

### Test Matrix

Requirements for this test were to operate the small scale SCIX column with SRF resin and to follow the same IX cycle protocol as was determined for use with the DOE River Protection Project – Waste Treatment & Immobilization Plant (RPP-WTP) ion exchange facility (3). Table 4 incorporates that IX operational protocol with modifications for the proper scaling. While the superficial velocities (SV) and Bed Volumes

**Table 4.** A single IX cycle for the scaled SCIX Hydraulic Test

Cycle steps	Cycle activity	Flow direction	Superficial vel. cm/min	Flowrate lpm	Duration min	Liquid processed liters	BV	Comment
A	0.5M NaOH Regeneration	Upflow	12.4	0.48	30	14.5	4.7	Process based on time
	a. Fluidization	No Flow	0.0		2			Process based on time
	b. Settling	Upflow	2.0	0.08	20	1.6	0.5	Process based on time
B	c. Stabilization							
	Feed Simulant Introduction	This is a RPP-WTP step to prevent its low aspect ratio resin bed from be disturbed while the dense feed simulant is introduced. The high aspect ratio resin bed of this test did not require this step, therefore feed simulant was introduced in Step Ca.						
C	Feed Simulant Loading <sup>a</sup>							
	a. Full-size flow of 95 lpm	Downflow	26.7	1.04	50	51.9	16.8	Process until stable dP
	b. Full-size flow of 57 lpm	Downflow	16.0	0.62	30	18.6	6.0	Process until stable dP
	c. Full-size flow of 19 lpm	Downflow	5.3	0.21	30	6.2	2.0	Process until stable dP
D	0.1M NaOH Displacement	Downflow	8.8	0.34	28	9.3	3.0	Processed volume = 1.4 CV

*(Continued)*

Table 4. Continued

Cycle steps	Cycle activity	Flow direction	Superficial vel. cm/min	Flowrate lpm	Duration min	Liquid processed liters	BV	Comment
E	DI Water Pre-Elution <sup>b</sup>	Downflow	13.3	0.52	16	8.0	2.6	Processed volume = 1.2 CV
F	0.5M HNO <sub>3</sub> Elution	Downflow	6.1	0.24	196	46.4	15.0	
G	DI Water Post-Elution	Downflow	13.3	0.52	16.0	8.0	2.6	Processed volume is 1.2 CV

1 CV = Volume of annulus + Volume above and below the screens =  $5.9 + 0.7 = 6.6$  liters < < Column Volume

1 BV = Bed Volume for 7.6-m<sup>c</sup> of resin scaled to 80-cm (ID, column 72 mm, OD, core of 17 mm) = 3.1 liters << Maximum [3] Bed Volume

<sup>a</sup>All three feed simulant loading superficial velocities were used during each cycle. However, Step Ca is longer than Cb and Cc to introduce the feed simulant and fill the column. Once the column was filled and the pressure readings were stable each flowrate was held for 30 minutes at the specified velocity to obtain stable dP readings.

<sup>b</sup>Because this test used three different resin bed heights the process volume was changed to 1.2 CV instead of the indicated 2.5 BV (3). This means the tallest bed experienced 2.6 BV and shorter beds slightly more. This change did not affect the test results but permitted uniform operation because a CV was fixed quantity.

<sup>c</sup>Three scaled resin bed heights were tested: 32, 48, and 80 cm representing full-size heights of 3, 4, 6, and 7.6 m, respectively.

(BV) are prototypic to RPP-WTP IX facility for all the cycle steps, the flow rates are scaled. However, the SV for feed had to be set to meet the current test requirement, as shown in the Simulant Loading, Step C, of Table 4.

Table 4 was the basic Test Matrix and shows a complete IX cycle, comprised of Steps A through G. The following are general highlights of the Test Procedure that was performed during each day of testing:

- During each day of testing the complete IX cycle of Table 4 was performed.
- Step B was not performed, but included for completeness. This is a RPP-WTP step to introduce feed very slowly in upflow. The slow upflow prevented the much heavier feed, density  $\sim 1.27$  g/mL, from disturbing the surface of the resin bed that occurred when it was introduced in downflow through the much lighter 0.5 M caustic, density  $\sim 1.0$  g/mL. This occurred in RPP-WTP tests at SRNL (3) because the resin beds had an aspect ratio of approximately 1:1 and the distance from the top of the IX column to the top of the resin bed was on the order of a column diameter. However, for the column used in the SCIX Hydraulic Test the resin bed aspect ratio was approximately 10:1 and the freeboard above the bed was multiple column diameters; therefore, feed was introduced in downflow because it was not expected to significantly disturb the tall bed of settled resin. The test showed that downflow introduction of feed did not affect the resin surface.
- The first 20 minutes of Step Ca allowed the column to experience three column volumes of simulated feed and to stabilize the resin bed pressure drop. At this point feed was recycled through the column to minimize waste.
- After the first 20 minutes of Steps Ca and Step D it was necessary to change the fluid in the pressure sensor tubes so that they contained the correct density to obtain accurate measurements. All the pressure zeros were checked after purging the lines.
- After the testing program began it was determined that the pressure sensor tubes also had to be purged after elution began, Step F. It seemed that some of the readings were remaining too high after acid filled the column. Purging the tubes eliminated this problem.
- On test days 3 and 5 the resin bed height was increased from 32 cm to 48 cm and then from 48 cm to 80 cm, respectively. To change the height, it was important to have the resin in sodium form, which means that the resin is expanded to its largest size. This is the size of the resin when it is processing feed. Every test day started by fluidizing the resin bed in order to regenerate the resin so that normal feed processing could be performed. However, on days 3 and 5, after the resin was

regenerated and swollen to its full size, i.e., after Step Aa, the column was then opened and filled to the next bed height. After closing the column the bed was re-fluidized for a short time before performing Steps Ab and Ac. Subsequently, those two steps were then carried out as normal to make sure that the resin beads were uniformly distributed and to relieve any built-up stresses throughout the bed.

- RPP-WTP recommends storage of resin in acid form soaking in water to minimize resin damage. Each day's testing ended with the resin in this form.
- Before the test program began a batch of new resin, which was in H-form, underwent pretreatment by following a required RPP-WTP protocol (10). The pretreatment conditioned the new resin so that it was ready to process caustic feed and to swell it to its full size so that the initial bed height could be set in the test column.

### Small-Scale Limitations

When utilizing a small-scale test facility it is important to be cognizant of its limitations. While most aspects of the test were maintained and operated prototypically, as the scale allowed, the full-scale resin bed height will be 10 times higher, as was discussed and as seen from the first three rows of Table 3. What is lost in the small scale is the full force the resin will experience at the bottom of the full-scale column, which is a combination of the weight of resin in simulant, accounting for buoyancy, and the accumulate drag force on the resin beads from the feed that flows down through the bed. The consequence of a taller, and therefore heavier, column is that the beads will be subjected to a higher compressive force than in the scaled unit. This stronger force may decrease the resin bed's void fraction, which in turn will decrease permeability leading to a larger pressure drop. Moreover, the stronger force will exert more pressure on the beads that may lead to damage. However, a previous study with SRF media indicated that the full-scale force will not have a detrimental effect on the bed's hydraulics. In a Bed Voidage and Permeability Test, using a small-scale facility (see Table 7-1 in Ref. 8) an attempt was made to create full-scale bottom pressures by using feed flows much greater than prototypic flows to increase the drag force on the resin. The SRF resin bed was subjected to superficial velocities of downflowing feed up to 347 cm/min, which was approximately 50 times greater than prototypic IX flows. The very high non-prototypic superficial velocity did not significantly affect the resin bed void fraction and the permeability unexpectedly increased, as long as the bed was allowed to be regenerated in a fluidizing upflow so that bed stresses could be relieved (as was done for

the SCIX Hydraulic Test). The increased permeability may be explained by the changing flow regime which decreased the drag coefficient around the spherical resin beads as the Reynolds number increased from the increasing flow of feed (13). Moreover, that Bed Voidage and Permeability Test (8) did not find any resin bead breakage at the highest flow rates, i.e., the highest pressure at the bottom of the column. The resilience of resin beads was also shown in a compression test of a bed of SRF with the dimensions of a 5 cm diameter and a 3.7 cm height (7). That test applied 267 N (60 lbf) of force from 138 kPa of pressure to the top of the resin resulting in a force of 356 N (80 lbf) at the bottom of that column with no bead breakage and only a 2% reduction in bed height. To reiterate, while the small-scale SCIX Hydraulic Test would not replicate the absolute pressures in the full-scale resin bed the results from previous studies (7 and 8) indicate that the pressure drop through the linearly scaled bed will be prototypic of the full-scale unit when using a prototypic feed superficial velocity because the bed void fraction and permeability are not significantly affected.

## RESULTS AND DISCUSSION

### Simulated Waste Feed

Because of the importance of simulant viscosity to hydraulic testing, it was measured before, during, and after the test. As already shown in Table 2 the target was 3.24 cP, which was measured at 25°C. The resulting simulant results are shown in Table 5.

Note, the temperatures shown in Table 5 for the measurements made at the test location were simply what existed at ambient, that is, they were not controlled. These temperatures are similar to those of the test since each day's property measurements were done around the mid-point of each testing cycle. Samples were also taken to make measurements under controlled conditions. However, all of the numbers are close and the simulant viscosity of 3.2 cP matched the target value of 3.24 cP (6). Flow resistance through the resin bed with this simulant was therefore expected to be prototypic, if not conservative, of real waste.

For the SCIX Hydraulic Test the waste feed simulant needed to match, primarily, the actual feed viscosity and of lesser importance, its density. To obtain a simulant with these properties it was not necessary to match all the analytes shown in the Table 1, after it was adjusted to 6 M Na, which was shown in Table 2 as the "Average SRS" waste feed. However, the principal analytes were used, i.e., greater than 0.5 M. To verify that the simulant matched experimental needs a sample was taken

Table 5. 6 M Na feed simulant rheology

Sample date	Temp.°C	Measurement made at PEDL			Measurement made at ACTL		
		Density gm/ml	Viscosity cP	Temp. °C	Density gm/ml	Viscosity cP	Comment
5/21/2007	21.3	1.317	4.09	25.0	1.299	3.80	Too Viscous - 6.6 M Na - water added
5/22/2007	21.7	1.291	3.69				Still too viscous - water added
5/22/2007	21.9	1.286	3.23	25.0	1.272	3.21	Final Simulant - 6.1 M Na
5/23/2007	21.8	1.286	3.43				Test Day 1 morning
5/23/2007	22.4	1.284	3.29				Test Day 1 afternoon
5/24/2007	22.0	1.287	3.33				Test Day 2
5/29/2007	24.3	1.283	3.08				Test Day 3
5/30/2007	25.7	1.276	2.97				Test Day 4
5/31/2007	24.7	1.284	3.02				Test Day 5
6/1/2007	24.1	1.283	3.11	25.0	1.270	3.12	Final Test Day
Averages* =		1.28	3.18		1.27	3.17	
Std. Dev. =		0.003	0.16		0.001	0.06	

\*The average values do not include those in the first two rows because the simulant was too viscous.

PEDL = Process Engineering Development Laboratory = SCIX Hydraulic Test Location.

ACTL = Aiken County Technology Laboratory = A laboratory at SRNL that was the provider of the simulant recipe.

**Table 6.** Some analytes measured in the pre-test simulant

Analyte	Sample 1 mg/L	Sample 2 mg/L	Average values		Target M
			mg/L	M	
Al	9690	9500	9595	0.36	0.34
Na	141000	140000	140500	6.1	6.0
NO <sub>2</sub>	26300	26200	26250	0.57	0.56
NO <sub>3</sub>	152000	151000	151500	2.4	2.3
SO <sub>4</sub>	15300	15300	15300	0.16	0.16

just before the experiment began. The results of that sample are shown in Table 6.

Within measurement uncertainty ( $\pm 15\%$ ) the simulant matched the target values for the 6 M Na solution. In fact, both the rheology (Table 5) and the analytes (Table 6) show that the simulant was within 5% of the target values.

### Precipitation

An IX cycle for SRF resin contains several steps to buffer the pH changes that must occur when switching from resin elution with acid to resin regeneration with caustic. Those steps include the use of a mild caustic and deionized water when switching between the principal treatment solutions. One reason for these extra steps is to minimize precipitation of solids that could lead to reduced IX performance. However, with long-term operation and planned or unplanned stoppages, some precipitation can be expected. Despite the presence of a small amount of solids during the current experiment they did not appear to have any impact on the hydraulic operations.

The current test was run once a day and shut down at the end of each day; that is, the column was not run in a continuous mode, which an actual plant will most likely operate. The test always ended by eluting the resin with 0.5 M nitric acid followed by replacing the acid with deionized water. This was done by flushing the IX column with enough water to replace the entire volume of acid. Then the test rig was shut down for the evening. From one day to the next there were approximately 15 hours for the water to absorb some of the acid still contained in the resin bed, resulting in a small amounts of precipitants. Figure 7 shows those solids.

Before a day's test began Fig. 7a shows that a fine white layer of precipitation was present (the yellow object was an unknown, but it did not seem to affect operations). After fluidization the light precipitants tended to break apart, Fig. 7b. During feed operation the solids would disappear altogether. Note that the lighting in the figure makes the resin in Fig. 7a

**Table 7.** Resin bed changes that occurred during the IX cycle steps

Test date	Bed liquid	Resin form	Bed dynamics	Flow direction	BH cm
Day 1 Test*: Target BH = 32 cm: BH/Bed Diameter = 5.8					
5/23/07	0.5 M NaOH	Sodium	Stable	No Flow	33.0
5/23/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	45.5
5/23/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	32.8
5/23/07	6 M Na Simulant	Sodium	Stable	Downflow	31.2
5/23/07	0.1 M NaOH	Sodium	Stable	Downflow	31.4
5/23/07	DI Water	Sodium	Stable	Downflow	31.7
5/23/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	25.8
5/23/07	DI Water	Acid	Stable	Downflow	25.8
Stable Sodium/Acid Bed Height Ratio = 1.21					
Stable Fluidized/Acid Bed Height Ratio = 1.76					
Stable Fluidized/Sodium Bed Height Ratio = 1.46					
Day 2 Test*: Target BH = 32 cm: BH/Bed Diameter = 5.8					
5/24/07	0.5 M HNO <sub>3</sub>	Acid	Stable	No Flow	25.7
5/24/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	45.2
5/24/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	32.8
5/24/07	6 M Na Simulant	Sodium	Stable	Downflow	32.0
5/24/07	0.1 M NaOH	Sodium	Stable	Downflow	32.7
5/24/07	DI Water	Sodium	Stable	Downflow	32.5
5/24/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	25.8
5/24/07	DI Water	Acid	Stable	Downflow	25.8
Stable Sodium/Acid Bed Height Ratio = 1.24					
Stable Fluidized/Acid Bed Height Ratio = 1.75					
Stable Fluidized/Sodium Bed Height Ratio = 1.41					
Day 3 Test: Target BH = 48 cm: BH/Bed Diameter = 8.7					
5/29/07	0.5 M NaOH	Sodium	Stable	No Flow	48.2
5/29/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	72.0
5/29/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	50.5
5/29/07	6 M Na Simulant	Sodium	Stable	Downflow	49.1
5/29/07	0.1 M NaOH	Sodium	Stable	Downflow	49.8
5/29/07	DI Water	Sodium	Stable	Downflow	49.3
5/29/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	40.0
5/29/07	DI Water	Acid	Stable	Downflow	40.0
Stable Sodium/Acid Bed Height Ratio = 1.23					
Stable Fluidized/Acid Bed Height Ratio = 1.80					
Stable Fluidized/Sodium Bed Height Ratio = 1.47					
Day 4 Test: Target BH = 48 cm: BH/Bed Diameter = 8.7					
5/30/07	0.5 M HNO <sub>3</sub>	Acid	Stable	No flow	39.9
5/30/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	72.0
5/30/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	49.2
5/30/07	6 M Na Simulant	Sodium	Stable	Downflow	48.9
5/30/07	0.1 M NaOH	Sodium	Stable	Downflow	49.4
5/30/07	DI Water	Sodium	Stable	Downflow	49.0
5/30/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	39.9
5/30/07	DI Water	Acid	Stable	Downflow	39.9
Stable Sodium/Acid Bed Height Ratio = 1.23					
Stable Fluidized/Acid Bed Height Ratio = 1.80					
Stable Fluidized/Sodium Bed Height Ratio = 1.47					
Day 5 Test: Target BH = 80 cm: BH/Bed Diameter = 14.4					
5/31/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	127.5
5/31/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	83.6

*(Continued)*

**Table 7.** Continued

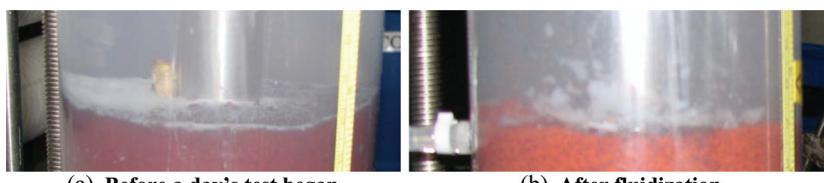
Test date	Bed liquid	Resin form	Bed dynamics	Flow direction	BH cm
5/31/07	6 M Na Simulant	Sodium	Stable	Downflow	81.3
5/31/07	0.1 M NaOH	Sodium	Stable	Downflow	82.0
5/31/07	DI Water	Sodium	Stable	Downflow	81.5
5/31/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	66.3
5/31/07	DI Water	Acid	Stable	Downflow	66.3
Stable Sodium/Acid Bed Height Ratio = 1.23					
Stable Fluidized/Acid Bed Height Ratio = 1.92					
Stable Fluidized/Sodium Bed Height Ratio = 1.57					
Day 6 Test: Target BH = 80 cm: BH/Bed Diameter = 14.4					
6/1/07	0.5 M HNO <sub>3</sub>	Acid	Stable	No Flow	66.4
6/1/07	0.5 M NaOH	Sodium	Fluidizing	Upflow	123.0
6/1/07	0.5 M NaOH	Sodium	Stabilizing	Upflow	84.6
6/1/07	6 M Na Simulant	Sodium	Stable	Downflow	82.7
6/1/07	0.1 M NaOH	Sodium	Stable	Downflow	83.1
6/1/07	DI Water	Sodium	Stable	Downflow	82.6
6/1/07	0.5 M HNO <sub>3</sub>	Acid	Stable	Downflow	66.6
6/1/07	DI Water	Acid	Stable	Downflow	66.4
Stable Fluidized/Acid Bed Height Ratio = 1.24					
Stable Fluidized/Acid Bed Height Ratio = 1.85					
Stable Fluidized/Sodium Bed Height Ratio = 1.49					

BH = Bed Height.

\*Odd-numbered test days always started with resin in sodium form because the resin height was changed.

\*Even-numbered test days always started with resin in acid form as it was left from the previous test day.

appear to be darker than in Fig. 7b, but it was just the reflection. Resin in acid form, Fig. 7a, is a brighter orange than in sodium form, Fig. 7b. Even though a small amount of precipitants were visible at the start of each day they generally were reabsorbed during the operation of caustic feeds, i.e., regeneration and simulated feed. Figure 8 shows a relatively clean resin surface while it was processing waste feed in downflow. The thin darkened surface layer of resin is thought to be resin damaged by oxygen.



**Figure 7.** Precipitation after the resin was sitting in water for 15 hours which followed acid elution.



**Figure 8.** Surface of SRF resin bed while it was processing waste feed in downflow.

### Performance of Spherical Resorcinol Formaldehyde Resin

#### Resin Bed during Regeneration and Elution

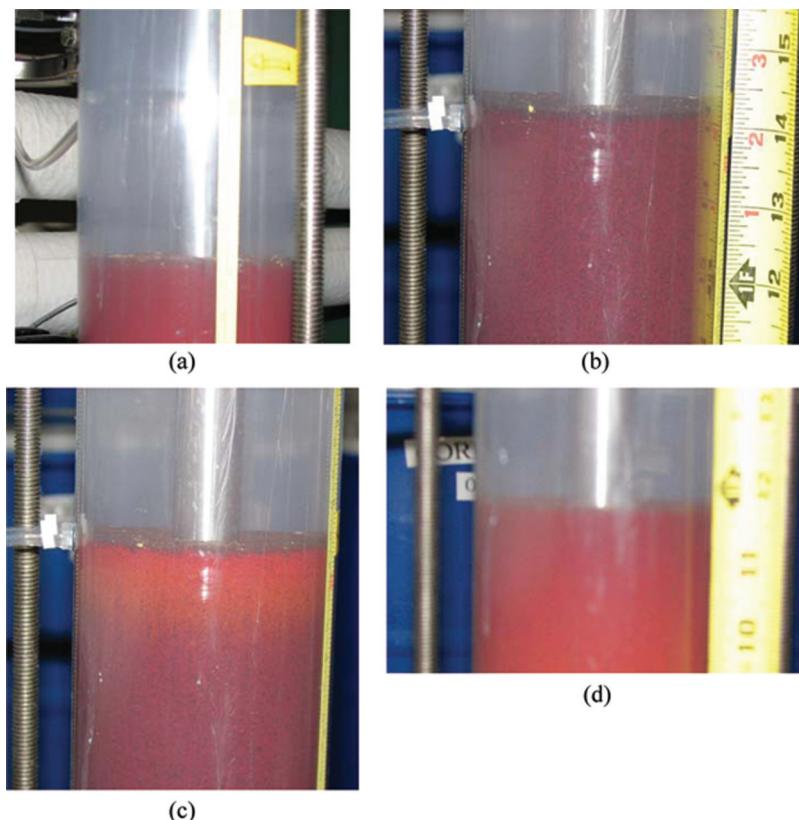
Besides determining the dynamic pressure as a simulated waste feed moved downwards through a bed of the resin the shrink/swell behavior of the high aspect ratio resin bed was of interest. In the process of performing an entire IX cycle the resin demonstrated its characteristics of shrinking in size when exposed to acid during elution, and rising in the column from both expanding in size and being lifted when exposed to caustic during regeneration in a fluidizing flow. For example, Fig. 9 shows several of the steps from starting with the resin in acid form soaking in water, Fig. 9a, to regeneration that prepares the resin to treat waste feed, Fig. 9b, to eluting the resin after the resin is loaded with the target ion, Fig. 9c, to finally returning the resin to acid form, Fig. 9d.

The overall movements of the resin bed in each of the tests are summarized in Table 7. Some of the key results shown in the table are explained below:

- After regenerating the SRF resin in caustic – the bed working volume (the sodium bed height), is 23% larger than its smallest volume in acid form (the acid bed height).
- After eluting the SRF resin in acid – the bed volume is at its smallest size (the acid bed height). As regeneration begins the bed is fluidized and the volume increases immediately by approximately 80%, or basically doubling in size. Further, this fluidized bed height is approximately 50% taller than the sodium bed height.

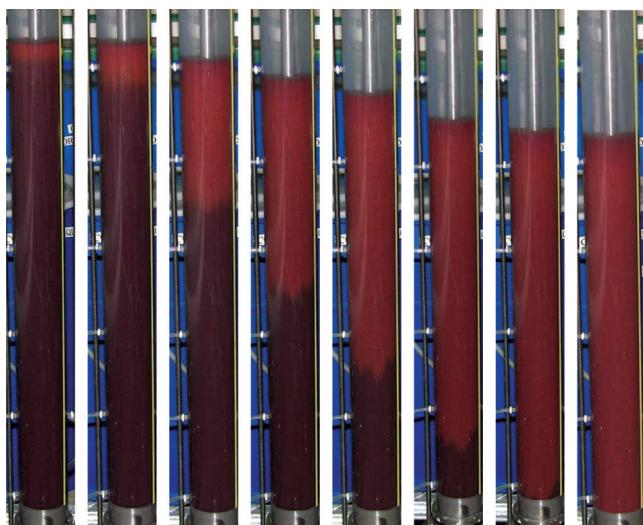
#### Resin Bed During Elution

As has been observed and previously reported (3), SRF resin changes color during regeneration and during elution. The process of regeneration



**Figure 9.** Several phases of an SRF IX cycle that occurred with the 32 cm bed height; (a) The resin initially starts in acid form where the bed was at its shortest height of 25.7 cm.; (b) After undergoing regeneration with the upflow of 0.5 M NaOH, the bed was allowed to settle in its fully swollen state so that feed processing can begin. In this picture simulant was flowing downwards and the bed height was 32.0 cm (12.6 in). [The ruler shows 14.2 in because there was a 1.6 in offset.]; (c) Once the resin was loaded the process of regeneration began by first stripping it of the unwanted ions with acid elution. The acid again shrank the resin and in the picture the bed has already begun to shrink. It was at a height of 31 cm.; (d) Elution takes several hours while the bed reduces to its smallest height. The figure shows that the bed returned to its smallest height of 25.8 cm (10.2 in). [That is, 11.8 in – 1.6 in offset]

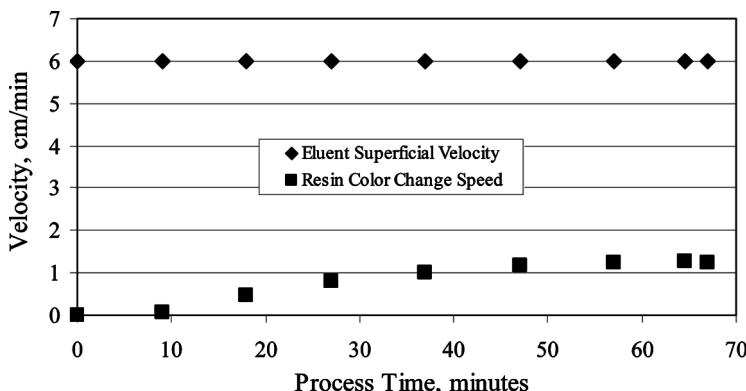
converts the resin bed to sodium form, which then changes from bright to dark orange. The bed is immediately raised during fluidization and each resin particle experiences a high flow of caustic, which causes the color changeover to be uniform throughout the entire resin bed. However, during elution the superficial velocity of nitric acid is one half that of regeneration



**Figure 10.** Elution of the spherical RF resin bed. Color changeover during the first 60 of the 196 minute elution process as the resin changed from Na form to H form.

and occurs in downflow; therefore, the color changeover of the settled bed is more pronounced and visually noticeable. Figure 10 shows the 80 cm resin bed undergoing elution during the first 60 of 196 minutes of acid flow.

Some aspects of the hydraulic performance of the resin during elution are quantified in the following figures. Figure 11 compares the eluent superficial velocity to the color change interface velocity. The color



**Figure 11.** Superficial velocity of eluent and velocity of color interface.

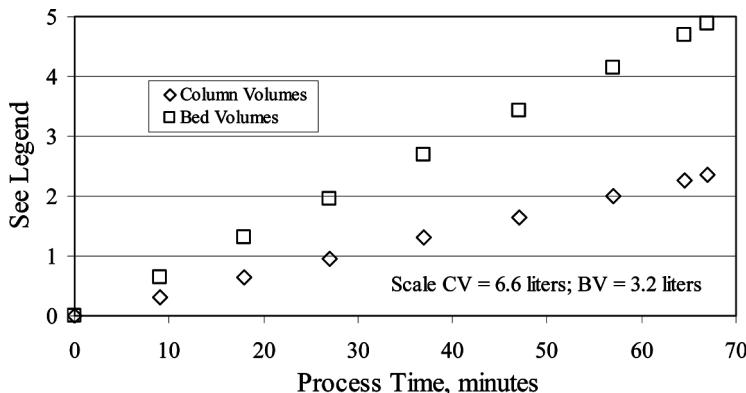


Figure 12. Volume of eluent processed during resin color change.

change interface moved much slower than the liquid velocity by a factor of 6. By the time the color of the resin was completely changed, Fig. 12, approximately two column volumes of acid were processed, which meant approximately 5 bed volumes for the 80 cm tall resin bed. Finally, during the first 60 minutes of elution, Fig. 13, the bed height dropped from 83 cm to 66 cm.

#### Pressure Drop through SRF with a 6 M Na Average SRS Waste Feed Simulant

Over the six test days the pressure drop was measured using three different resin bed heights, i.e., 32, 48, 80 cm and three different feed superficial

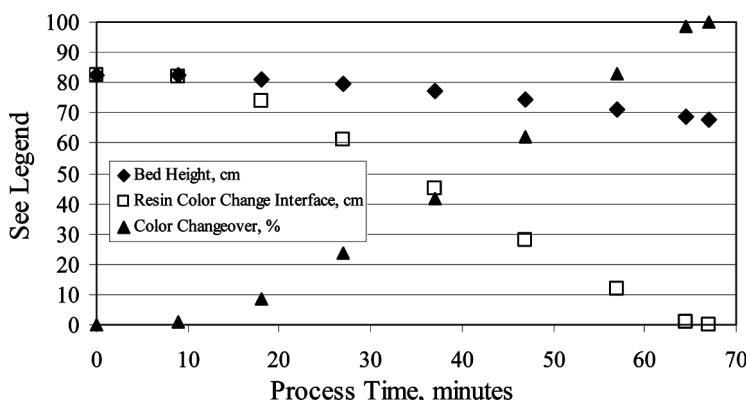
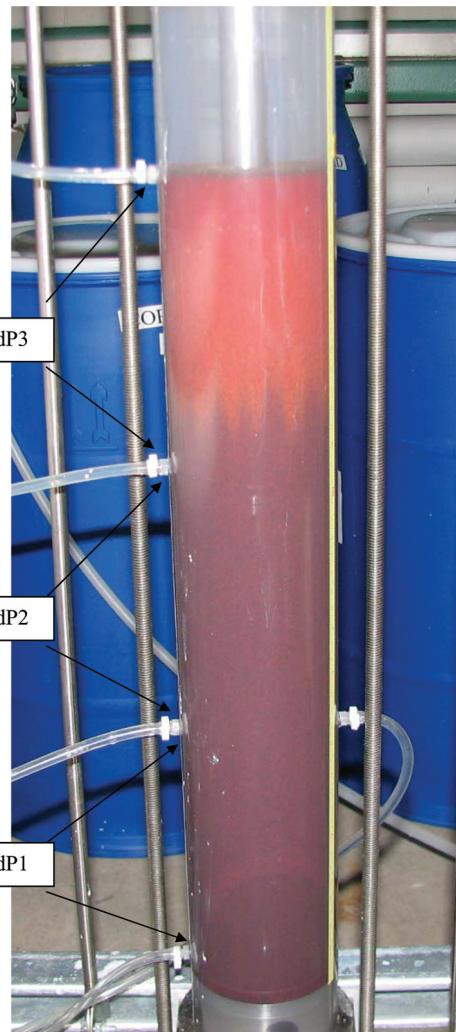
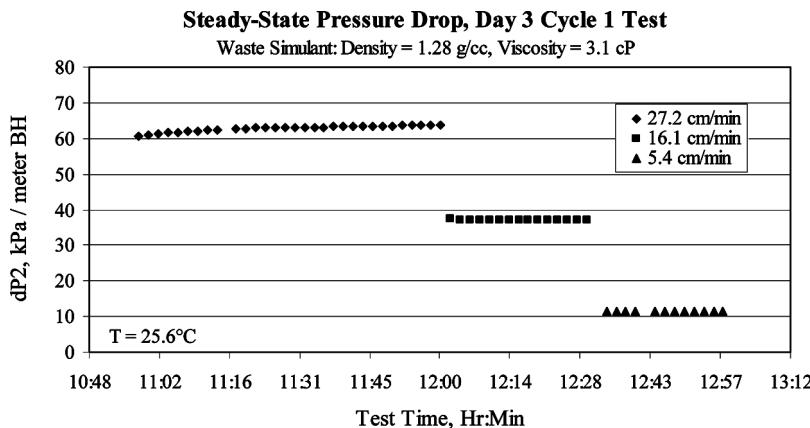


Figure 13. Quantities of resin color change.

velocities, i.e., approximately 5, 16, and 27 cm/min. Figure 14 shows one test that used a 48 cm bed height as evident from the pressure taps that were located every 152 mm starting at 6.4 mm above the bottom resin screen. (The bed height in Figure 14 is slightly shorter than 48 cm because it was undergoing elution which shrinks the bed.)

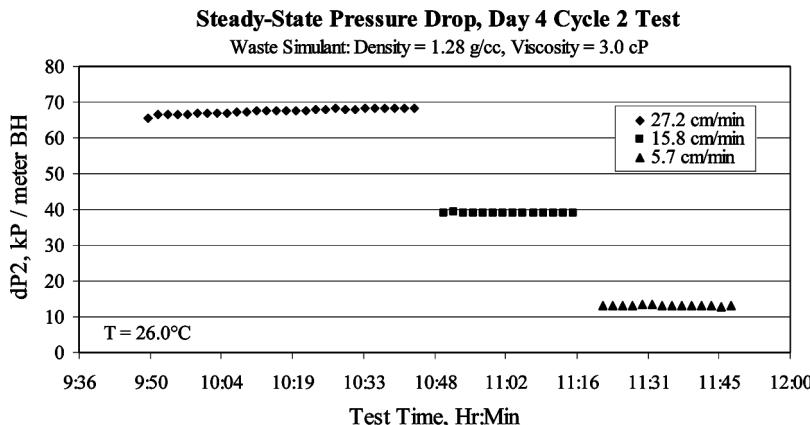


**Figure 14.** Scale SCIX during the 48 cm test. Here the bed height was approximately 46 cm after it began to shrink during elution with acid.



**Figure 15.** Pressure drop between dp2 pressure taps at 31.1 to 15.9 cm (see Figure 14) during the first day of the 48 cm Bed Height (BH) test.

Each test day started by regenerating the resin, followed by introducing the simulated waste feed into the column. As had already been seen in Table 4, the first of three periods of simulated feed was 50 minutes long, Cycle Step Ca. However, the initial 20 minutes of that 50-minute period were to introduce feed into the column and to approach a steady-state pressure drop. At a superficial velocity of  $\sim 27$  cm/min the 20 minutes allowed approximately three column volumes of solution to



**Figure 16.** Pressure drop between dp2 pressure taps at 31.1 to 15.9 cm (see Figure 14) during the second day of the 48 cm Bed Height (BH) test.

**Table 8.** Pressure drop across a 1 meter bed height (BH) of SRF resin with 6 M Na feed

Temp.°C	Bed height		AR	SV cm/min	Per METER of BH	
	Target cm	Actual cm			dP kPa	1xStd. Dev %
Feed Flowrate – Target SV = 5.3 cm/min						
Cycle 1	24.6	32	31.2	5.7:1	5.7	2.65%
Cycle 2	24.6	32	32.2	5.9:1	5.4	4.74%
Cycle 1	25.9	48	49.1	8.9:1	5.4	6.51%
Cycle 2	26.3	48	49.0	8.9:1	5.7	1.74%
Cycle 1	27.2	80	81.5	14.8:1	6.0	0.36%
Cycle 2	26.7	80	82.9	15.0:1	5.4	0.66%
		Avg = Std. Dev. =		5.6	12.1	2.8%
		Calibration Uncertainty =		0.1	1.1	1.1
Feed Flowrate – Target SV = 16.0 cm/min						
Cycle 1	24.7	32	31.2	5.7:1	16.3	40.4
Cycle 2	24.4	32	32.2	5.9:1	16.3	37.4
Cycle 1	25.6	48	49.1	8.9:1	16.1	38.5
Cycle 2	26.0	48	48.9	8.9:1	15.8	39.6
Cycle 1	27.0	80	83.1	15.1:1	16.1	38.0

Cycle 2	26.6	80	82.7	15.0:1	Avg =		
					Std. Dev. =		
					Calibration Uncertainty =		
Feed Flowrate - Target SV = 26.7 cm/min							
Cycle 1	24.7	32	31.2	5.7:1	26.9	68.5	0.84%
Cycle 2	24.3	32	32.0	5.9:1	27.7	64.3	1.34%
Cycle 1	25.3	48	49.1	8.9:1	27.2	65.3	1.37%
Cycle 2	26.1	48	48.9	8.9:1	27.2	68.7	1.32%
Cycle 1	26.8	80	81.4	14.8:1	28.0	66.5	1.70%
Cycle 2	26.4	80	82.7	15.0:1	27.5	60.4	0.90%
				Avg =	27.4	65.6	1.2%
				Std. Dev. =	0.4	3.1	
				Calibration Uncertainty =	0.1	1.1	

BH = Bed Height

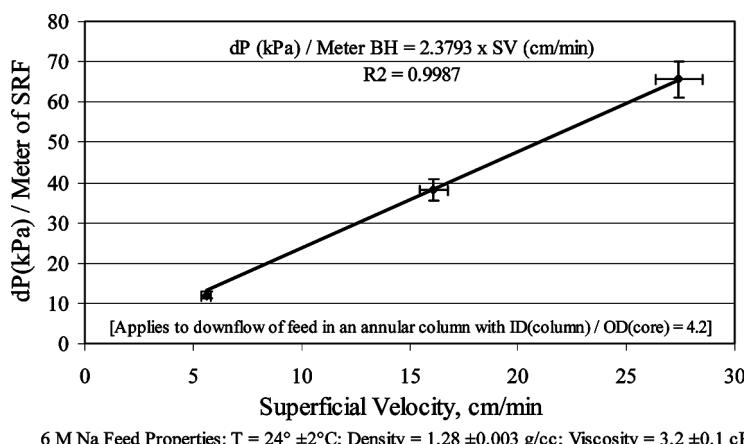
AR = Aspect Ratio =  $BH/D_h$  (based on actual BH for this table) $D_h$  = Hydraulic Diameter = 5.50 cm (see Table 3)

SV = Superficial Velocity

pass through the column. The next 30 minutes, and the subsequent two 30 minute periods, Cycle Steps Cb and Cc, were performed to obtain steady-state pressure drop readings. The 30 minute period was sufficient to attain steady-state flow. To illustrate the pressure drop measurements, results from transducer dP2 are shown in Fig.15 and Fig.16.

From Fig.15 and Fig.16 it appears that the pressure drop through the bed becomes stable relatively fast. This was typical for all of the tests. The average values among all participating pressure taps are listed in Table 8.

The standard deviations of the averaged data in Table 15 indicate that the pressure drop through the resin was relatively stable. Over all test days, the data that made up the pressure drop measurements had a standard deviation of 7% or less, with the overall average standard deviation of approximately 1.5%. This means the measurements were stable. The superficial velocity (which is basically the feed flow rate) was steady throughout and its standard deviation fluctuation was less than 5% over all days. The measurement uncertainty obtained from calibration was less than these standard deviations, which indicate that the stability was significant. An important aspect of the data in Table 8 is that pressure drop per unit bed height is not dependent on the depth of the bed. Only the overall pressure drop is dependent on the bed height, that is, the further feed needs to travel through the bed the larger the pressure drop. Finally, the SCIX Hydraulic Test pressure drop was found to be linearly dependent on feed flow rate, or superficial velocity, since the flow area was constant. This is shown in Fig. 17.



**Figure 17.** Pressure drop per meter of SRF Bed Height (BH) versus 6 M Na feed superficial velocity.

### Pressure Drop through SRF During all Other Steps

The resistance to the flow of all other solutions through the IX column was less than that which was experienced from the flow of the feed simulant. That is, while the superficial velocities of the other IX process solutions, i.e., NaOH, HNO<sub>3</sub>, and H<sub>2</sub>O, were comparable to that of the feed simulant, see Table 4, the pressure drops were significantly less because the viscosity of the feed simulant was a factor of 3 larger.

## CONCLUSIONS

A batch of spherical resorcinol formaldehyde resin was tested in a scaled 72 mm (2.8 in) inside-diameter column with a central core to represent a cooling tube that will exist in the full-scale column of a 0.69 m (27.2 in) inside diameter and a 0.17 m (6.6 in) central core. The scaled annular column was filled with three different bed heights of resin, i.e., 32, 48, and 80 cm to represent full scale heights of 3.0, 4.6, 7.6 m, respectively. The resin was prototypic as well as the simulated 6 M Na feed with a density of 1.28 g/cc and dynamic viscosity of 3.2 cP. The feed simulant was chosen to have a viscosity equal to, or larger than, the expected viscosity of the SRS tanks to be treated so that the hydraulic results would be conservative. Flow was prototypic in that the feed operated in downflow and the superficial feed velocities (i.e., volumetric flow rate divided by the empty column flow area) were 5.6, 16.1, 27.4 cm/min. From the results the following conclusions can be made:

1. With the downflow of 6 M Na feed of density 1.28 g/cc and viscosity of 3.2 cP the:
  - Average pressure loss was 12 kPa per meter of bed height at a 5.6 cm/min superficial velocity.
  - Average pressure loss was 38 kPa per meter of bed height at a 16.1 cm/min superficial velocity.
  - Average pressure loss was 66 kPa per meter of bed height at a 27.4 cm/min superficial velocity.
  - The pressure loss in the resin bed per unit height was independent of the resin height.
2. The introduction of 1.28 g/mL feed simulant into a 1.0 g/mL caustic regeneration solution in downflow did not disturb the resin bed at all. The freeboard, which for the tallest bed height of 80 cm was 72 cm, significantly damped turbulence in the downflow of feed before it reached the resin.

3. The spherical resin mechanically performed as seen in past hydraulic tests (3):
  - The working sodium-form resin bed volume was 23% larger than the acid-form bed.
  - The suspended resin bed during fluidization was approximately 80% larger in volume than the settled bed in acid form.
  - The suspended resin bed during fluidization was approximately 50% larger in volume than the settled bed in sodium form.
  - No fines or broken particles were observed.

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