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LAWPS Ion Exchange Column Gravity Drain of Spherical Resorcinol Formaldehyde Resin

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EXECUTIVE SUMMARY

Experiments at several different scales were performed to understand the removal of spherical resorcinol formaldehyde (sRF) ion exchange resin using a gravity drain system with a valve located above the resin screen in the ion exchange column (IXC). This is being considered as part of the design for the Low Activity Waste Pretreatment System (LAWPS) to be constructed at the DOE Hanford Site.

The testing was to determine the resin's behavior with the side-discharge valve concept and what additional efforts may be required to successfully remove any residual resin. The tests demonstrated the:

- Resin response to gravity drain through a side port, of different sizes, from a simulated IXC.
- Residual resin left in IXC after draining.
- Methods for removing the residual resin heel using liquid.
- Difference among multiple test solutions, that is, inhibited water (IW), simulated supernatant, a super saturated simulant (precipitants), and nitric acid elution solution on gravity draining of sRF resin.
- Impact of temperature on the resin's ability to drain by gravity.

In the order of IXC scale, the tests and platforms utilized were:

<u>4-inch inside diameter IXC</u>, made of plastic (PVC), at moderate temperatures, i.e., 25°C to 65°C, and stainless steel at the highest test temperature, i.e., 85°C. Both materials were tested at 65°C. The sRF was both new and oxidized in sodium form and the drain valve size was 3/8 inch.

<u>18-inch inside diameter IXC</u>, made of plastic (Acrylic), at 25°C, with a 1-inch, 2-inch, and 3-inch side drain. All four test solutions were used as well as the sRF being in hydrogen form and sodium form.

<u>40-inch inside diameter IXC,</u> made of plastic (Acrylic), at 25°C, with a 3-inch and a 4-inch side drain; however, the test solution was limited to the simulated supernatant due to the results of the previous 18-inch IXC tests, which showed that the difference in drain characteristics for the four test solutions to be insignificant.

Specific conclusions for each test platform are listed in the Conclusion Section of this report, but overall conclusions are:

- A slurry of liquid waste and resin should always drain freely after a valve is opened as long as an IXC is vented.
- For a non-vented IXC, the resin slurry will drain for discharge ports of 3 inches and greater after the drain valve is opened and when the created vacuum above the column is strong enough to prevent the IXC contents from initially exiting the valve.
- Draining from the IXC for a vented column is generally less than a few minutes.
- Draining from the IXC for a non-vented column increases the drain time by an order of magnitude over the time for a vented column with the same drain size.
- The use of plastic models of the IXC elicits drain data similar to that from stainless steel.
- Resin drain times are not significantly affected by temperatures of 65°C or less. Above 65°C, resin appears to drain slower possibly due to an increased adhesion forces among the resin beads.
- Resin heel volume after draining does not appear to be dependent on temperature.
- The volume of heel after each drain test did not show a dependence on temperature. However, the volume of remaining heel did show dependence on the size of the drain port: in general, the

larger the drain port size the smaller the heel. The 4-inch drain port performed the best such that approximately 10% of the ion exchange bed volume (BV) resin remained as a heel.

- IXC sRF resin slurry drain results appear to be approximately independent of suspension liquid, i.e., IW, supernatant simulant, and Nitric Acid. Furthermore, when the suspension was subjected to precipitation, the time to drain was similar to all other tests, but it initially did not drain until suspension liquid was upflowed for a few seconds to free up the resin so it could move.
- The remaining resin heels could be flushed out with distributor liquid, but as scale increased so did the amount of liquid needed.
  - For the 18-inch IXC, approximately 10 gallons were needed to reduce the heel to only a trace amount of resin.
  - For the full-scale IXC, approximately 50 gallons were needed. Considering the increase in IXC resin screen area, i.e., $(40.5 \text{ in.}/17.5 \text{ in.})^2$ = a factor of 5.4, then the 50 gallons necessary makes sense.

    However, at the larger scale, the amount of remaining heel could only be reduced to approximately 1.5% (~ 4 gallons) of the initial BV, which represented the removal of approximately 85% of the heel left after the drain.
- Loosely packed sRF resin H-form with water as the supernatant has a yield stress on the order of 100 Pa.

Based on the results some recommendations include:

- Employ a resin drain valve of 3-inch or larger to prevent pluggage that would impede the emptying of the IXC in the event of a non-vented drain.
- In the event of a non-vented drain, it will be helpful to include a small gas plenum above the IXC that would allow the resin slurry to initially drain until the vacuum in the gas plenum equals the weight of the remaining IXC contents, which would temporarily slow the draining process. However, the initial flow of resin slurry, while the pressure is equalizing, will help to allow air to enter the drain valve, thus permitting the draining to continue.
- Include a method of introducing a stream to rain down on the draining resin to help flush out the resin heel because a significant heel remains after a drain.
- A method of upflowing feed into the IXC during a power outage for a few seconds will help to drain the resin in the event of the resin being subjected to precipitants.

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NOMENCLATURE

BV	Ion exchange resin Bed Volume
BV/h	Flow rate through IXC based on the volume of the resin bed, bed volume per hour
H-form	Elution of ion exchange resin to hydrogen form to remove adsorbed cesium
IW	Inhibited water = 0.01 M NaOH solution
IX	Ion Exchange
IXC	Ion Exchange Column
IXC04	Refers to small-scale tests with 4-inch insider diameter IXC
IXC18	Refers to mid-scale tests with 18-inch insider diameter IXC
IXC40	Refers to full-scale tests with 40-inch insider diameter IXC
LAWPS	Low Activity Waste Pretreatment System
Na-form	Ion exchange resin condition with sodium so it is ready to adsorb cesium
PEP	Pretreatment Engineering Platform
PVC	Polyvinyl chloride plastic
RDT	Resin Disposal Tank
sRF	Spherical resorcinol formaldehyde (ion exchange resin)
SRNL	Savannah River National Laboratory
SS	Stainless Steel

1.0 Introduction

During the Advanced Conceptual Design of the Low Activity Waste Pretreatment System (LAWPS), an option was identified for spherical resorcinol formaldehyde (sRF) resin removal using a gravity drain system utilizing a valve located above the resin screen in the ion exchange column (IXC). The resin was drained from the IXC under gravity flow to the Resin Disposal Tank (RDT). This concept allows the resin to be discharged from the IXC to a holding tank to establish a geometrically favorable state for the resin.

The resin's response to gravity draining through an IXC is not completely understood. The goal of this testing was to determine the ability of the resin to gravity flow from a IXC, impacts of the exit configuration, quantify the residual resin heel after draining and demonstrate/determine the ability to remove residual resin.

Based on the proposed work [Duignan, 2015a] and the approved Technical Task and Quality Assurance Plan [Duignan and Herman, 2015], the testing was to determine the resin's behavior with the side-discharge valve/RDT concept and what additional efforts may be required to successfully remove any residual resin. The specific objectives for these tests included:

1. Determine the resin's response to gravity drain through a side port from a simulated IXC.
2. Determine the effects of the side port size on draining of resin from a simulated IXC.
3. Determine residual resin, i.e., heel, left in IXC after draining.
4. Demonstrate methods for removing the resin heel (after draining) using liquid.
5. Determine the difference among water, simulated supernatant, a super saturated simulant (precipitants), and a nitric acid elution solution on gravity draining of sRF resin from an IXC.
6. Demonstrate the impact of temperature on the resin's ability to flow.

For items 1 through 5, testing was performed in an ~18-inch inside diameter IXC. Items 1 through 4 testing was performed in a ~40-inch inside diameter IXC, since the mid-scale testing demonstrated insignificant differences of drain performance with the test solutions. Item 6 was performed in ~4-inch inside diameter IXCs. This report describes the tests in order of IXC size, i.e., the sections will describe 4-inch, 18-inch, and then the full size (40-inch) tests.

2.0 Experimental Setup

There were three different scales of the IXC used to demonstrate different aspects of the resin gravity drain mechanism in order to minimize cost and time. There is always an increased risk in using experimental scales smaller than full scale; however, it is not cost effective to address some issues at full scale especially when it appears that a smaller scale can adequately capture the data needed. To understand the effect of higher temperature on the flow of resin from a column, a small scale was chosen because it was much easier to design columns that could be uniformly maintained at temperatures up to 85°C for up to 14 days. Scale was not expected to have a significant impact on differences due to temperature. Furthermore, it would be cost prohibitive to develop a full scale test to evaluate temperature of up to 85°C due to the equipment needed and operations to perform. Where scale was considered important, e.g., resin flowing along wide expanses and under prototypic pressures, then full scale was used to demonstrate that smaller scale results accurately represent what happens at a larger scale. Each of the scale tests are explained below.

2.1 Small-Scale Drain Tests: Effect of Temperature

Tests were conducted on a small scale to determine the impact of temperature and the flow properties of sRF resin. A series of tests were conducted in which resin samples immersed in simulant were held at a range of different temperatures between 25 and 85ºC for periods of up to 14 days. Furthermore, a test was done with resin that was exposed to oxygen in order to demonstrate the flow characteristic of degraded resin at 25°C. To perform these tests, the test vessels were made of a 4-inch inside diameter tube that had18-inch of height above the "resin screen[1]," Figure 1.

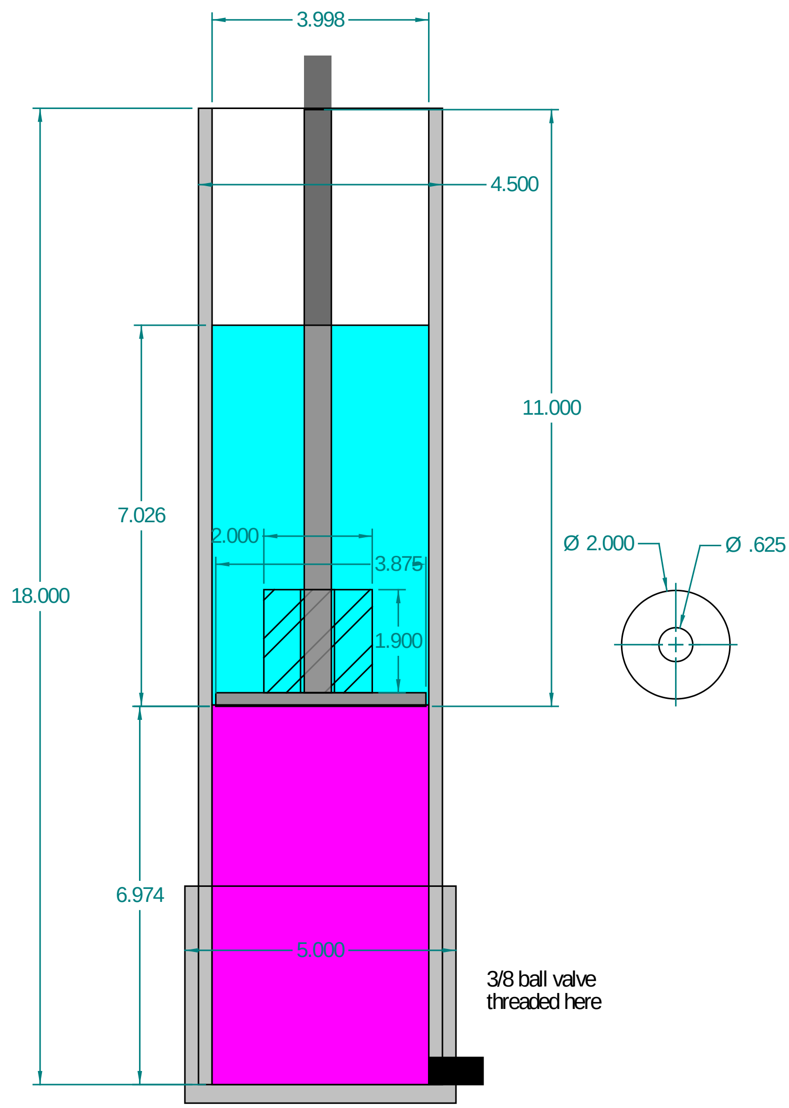

Figure 1. Design drawing of 4-inch IXC. The dimensions are in inches.

Also note the weight located on top of the resin bed. This was a Teflon plunger with a 0.250-inch base that spanned the full surface area of the resin on which a 2-inch diameter cylinder sat. The plunger surface was perforated to prevent the restriction of flow. This weight was made to compensate for the

[1] The small-scale column did not have a resin screen, but the actual bottom screen is expected to be close to flush with the resin drain port at the bottom of the IXC.

short height of the resin bed so the resin used experienced an equivalent force if the column were filled to the prototypic height of 50.4 inches of resin, which will be used in the actual IXC. That is, the weight compensated for the lack of resin height so that the resin was subjected to the same pressure that resin would experience at the bottom of a full-scale IXC. In total, six columns were fabricated with three made of polyvinyl chloride (PVC) plastic and three made of 304L stainless steel in order to understand if the column material affected the resin drain process. Figure 2 depicts the three plastic column used. Note the weight on top of the resin bed.

Figure 2. Three plastic columns in preparation for a test.

The three stainless steel columns are shown in Figure 3. Besides being made of steel, the other difference between the two column types is that the drain tube and port on the steel columns was flush with the steel bottom plate of the flat-bottom column; however, the valve in each plastic column was located in the middle of an oval bottomed end fitting that was glued to the upper transparent straight section.

Figure 3. Three stainless steel columns in preparation for a test.

That is, for the plastic tubes, the bottom pipe end cap would be filled approximately half way, i.e., ~2 inches, with resin before matching the vertical location of the drain valve.

However, flow differences between bottom column designs for the plastic and steel were not expected to be a factor. Until the resin is almost drained from a column the resin completely covers the bottom of any column and as the resin drains, its movement out the drain is basically layers of resin on resin, with the suspension liquid between layers. That is, the bottom of the column may only play a role in the draining process at the last few seconds, if at all.

A summary of the general procedure followed for each drain test with the 4-inch columns is listed below. However, as the tests progressed some variation occurred, which will be explained in the results section.

Summary IXC04 Test Procedure

1. Stage liquid simulant and resin (in Na-form and either new or oxygen damaged resin).
2. Preheat oven temperature to target value.
3. Measure mass of each column that is clean and dry, with cap on column.
4. Measure mass of compensation weight.
5. Fill each column with 7 inches of resin above the drain port.
6. Set compensation weight on resin in each column.
7. Fill columns with simulated liquid waste to double the resin height, i.e., 14 inches.
8. Cap column with loose fitting top.
9. Measure mass of each filled column.
10. Fill a sample bottle with excess resin and simulant.
11. Place columns and sample in oven for a fixed period, e.g. 3 to 14 days.
12. Check columns periodically to replace the water that has evaporated.
13. Remove columns and with sample from the oven.
14. Top the liquid off in each column.
15. Reweigh columns.
16. Prepare to drain resin by securing compensation weight in place to prevent its traveling with resin.
17. Drain each column and record the time for resin slurry to drain.
18. After draining, measure the mass of the columns and height of the resin drained.

2.2 Mid-Scale Drain Tests: Effects of Waste Streams, Pressure, and Drain Valve Size

For the midscale test, existing equipment, i.e., 18-inch IXC and a 5 M Na Hanford simulant, were repurposed to minimize time and cost. Figure 4 shows a schematic of the overall resin drain test equipment for the 18-inch (17.6-inch actual inside diameter) IXC. The drawing is not to scale but gives an idea of the entire test apparatus. As seen in Figure 5, the IXC itself is located on a second level mezzanine in the Engineering Develop Laboratory. The figure shows the IXC as it was being tested with water. It is tall enough to allow a single bed volume (BV) of resin, 53 gallons (or 50.4[2] inches of height) and a single BV of free board liquid above the resin bed for a total height of 100.8 inches, which is approximately 2-inches from the bottom of the top resin screen. Note the 50.4 inches represents the expected full height of resin in the LAWPS IXC.

[2] For the tests with 0.5 M HNO_3, the resin height was lowered to 50.4 inches x 0.76 = 38.3 inches assuming this will be the resin height after elution and the sRF resin has shrunk 20% to 25% as seen in previous tests [Duignan et al. 2008].

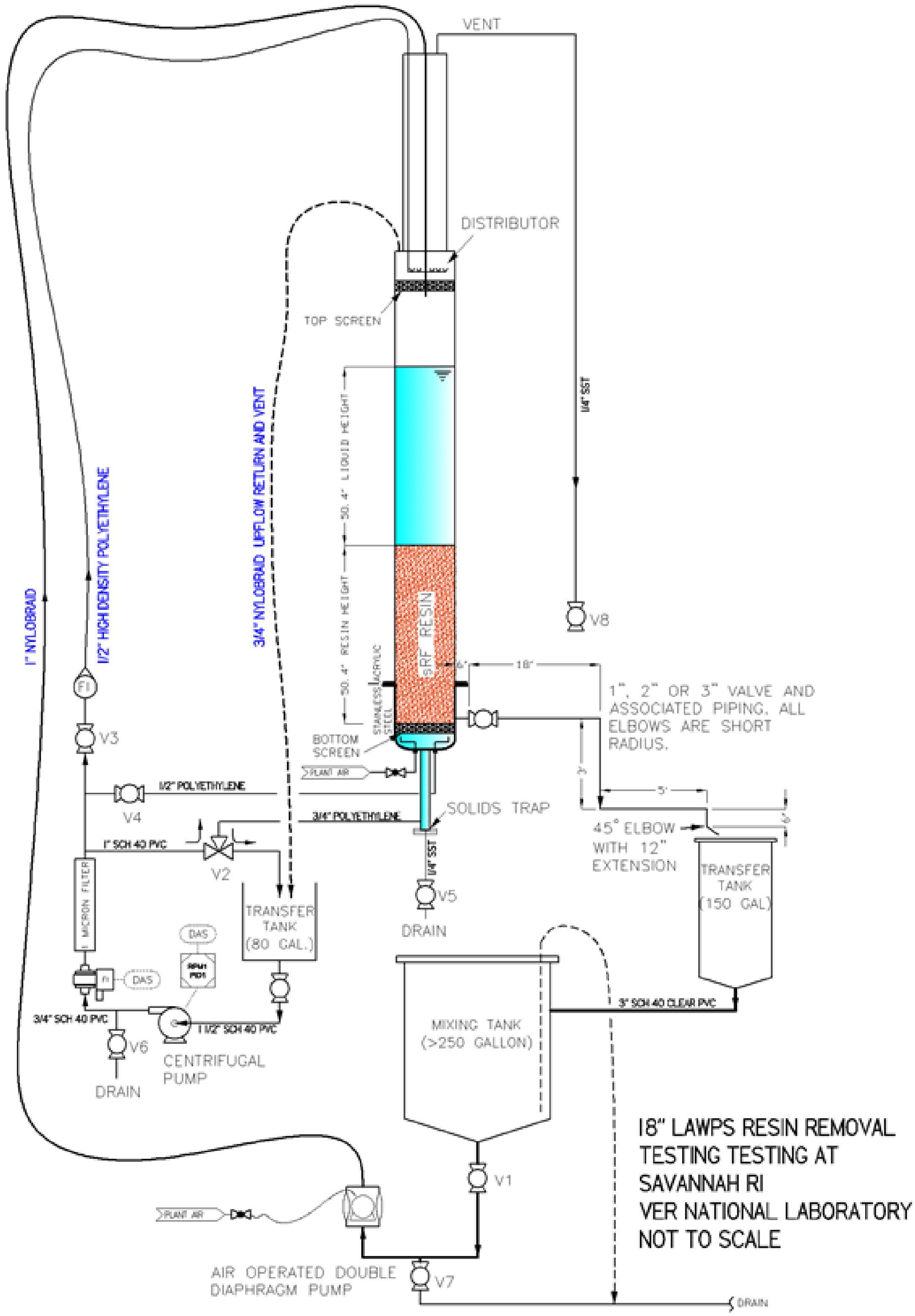

Figure 4. Schematic of 17.6-inch resin drain test equipment.

Figure 5. IXC assembled and filling with water and in this picture the water was at 6 inches from top resin screen.

Figure 6 shows the top resin screen and the feed distributor. Both the top and bottom screens were made of stainless steel with 60 micron openings and were supported by a perforated 0.5-inch thick PVC disk. The distributor was made of 0.5-inch PVC tube shaped into an approximately 12-inch diameter ring and had 3/16-inch holes placed every 1.5 inches along its top. After the IXC was filled with resin and the test liquid to the correct heights, one of the test valves was opened to initiate a drain. The IXC contents then traveled through the valve and down a short section of the drain pipe[3]. Figure 7 shows the 3-inch drain pipe, which runs below the mezzanine to the transfer tank on the first level mezzanine. That drain pipe, shown in the Figure 4 schematic, is only a short section of the drain pipe planned for the actual plant. However, it contains all of the expected bends as it makes its way to the RDT, which for this test is being called the Resin Slurry Receipt tank. All three drain pipe sizes, i.e., 1-, 2-, and 3-inch pipe had the same orientation and pipe section lengths. The transfer tank acted as a funnel to direct the resin slurry to its final destination, i.e., the receipt tank. The two photographs of Figure 8 show the drain into the receipt tank and the three test valve, i.e., 1-, 2-, and 3-inch valves. Also seen in this figure is the bottom of the IXC that was 12.5-inch deep from the top of its metal flange to the top of the bottom resin screen. Figure 9 shows the bottom screen and it location with respect to the drain ports, and Figure 10 shows the PVC support plate before the screen was attached. Figure 11 is a close-up of the drain ports with drain plugs to minimize the effect the holes would have on the resin movement during a drain. That is, only one drain was used at a time; therefore, the other two drains ports would be plugged at those times. The small white blocks seen in the figure were there to lock the resin screen assembly in place.

[3] The choice of the drain system utilized, i.e., valve and fitting, was made from a combination of the actual IXC requirements and experimental needs. During the planning for this 18-inch IXC resin drain test, the existing plant design for the drain pipe from the IXC is roughly sketched as Figure 110 in Appendix A.

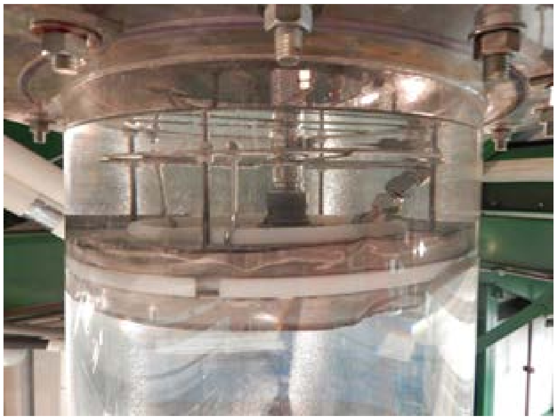

Figure 6. Water level is above top resin screen. Liquid distributor is the horizontal poly tube above screen. The large vertical tube in the center is the resin discharge.

Figure 7. 3-inch drain line into the top of the transfer tank.

Figure 8. Water discharging into the 350-gal tank from the 3-inch test valve fully opened. The 1- and 2-inch drain ports are internally plugged.

Figure 9. Bottom resin screen[4] in IXC with a view of drain holes. Screen was flush with holes.

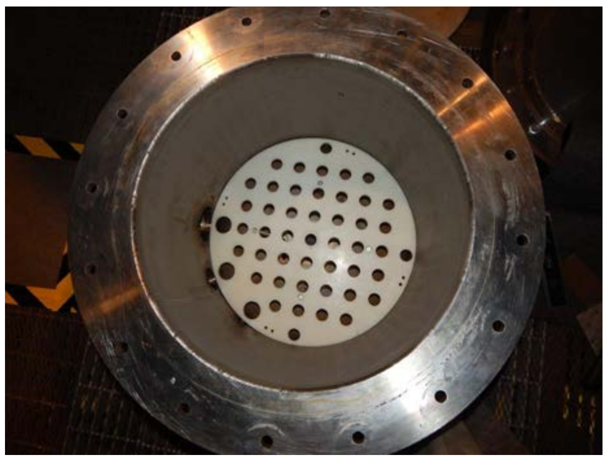

Figure 10. Resin screen support plate before screen installation.

Figure 11. Three drain port with plugs to minimize effect on resin due to presence of holes.

[4] The resin screen shows that it was not completely flat on the screen support plate; however, the lighting and angle gives the impression the surface was significantly undulated. This was not the case. The height of the waves on the surface was less than a few millimeters. Furthermore, the very flexible screen would be held flat under the weight of the resin.

This IXC is being reused from a previous test, but it is very useful in the current test in that it contains just enough metal at the bottom to mimic the metal bottom of a real IXC, meaning that surface roughness is potentially prototypic. Furthermore, the upper clear acrylic IXC allowed visualization of the resin movements. The resin screens used are not Johnson screens, which have 177 micron slits that are oriented parallel on the screen face and are commonly used in IXCs. However the screen used, which had 60-micron openings, are made of stainless steel. The different screens are not expected to result in a significant difference in performance because most of the resin drains out of the IXC before exposing the screen. That is, most of the movement of resin as it leaves the IXC through the drain port is resin on resin, until the screen is exposed.

During the non-vented test (referred to as the "glug-glug" test), the contents inside the IXC are not vented to the atmosphere to determine if draining is still possible by air rising from the receipt tank through opened drain valve. To perform these tests, it was necessary to seal all possible vents, see Figure 4. There are a 1-inch Flex Hose that delivers slurry, but remains empty during each test, the 0.75-inch Flex Hose that allow test liquid to return to the feed tanks during upflow, and a small 0.25-inch tube dedicated to venting the IXC. The vent pathways are closed to perform the no-vent test and when determined necessary one of those three tubes are used to apply a vacuum to better represent what may happen in the actual IXC.

Finally, all the 18-inch tests were done at ambient temperature. No special control was made to maintain the temperature constant, but in the Engineering Development Laboratory it is generally kept at 25°C ±5°C. Note, that because the test IXC was on the second mezzanine in the laboratory the temperature is roughly 6°C higher than on the main floor of the building. However, while the column itself would have been slightly warmer than 25°C, the resin and simulant was fed from the main floor and thus closer to 25°C. The very short time of feeding the resin slurry to the IXC, i.e. < 1 hour, is not enough time for the large mass of resin and liquid to significantly increase in temperature before they were drained back down to the main floor. Therefore, the 25°C ±5°C for the test solutions is expected to be a fair estimate of the operational temperature.

Summary IXC18 Test Procedures

Basic Operation for the 18-inch IXC tests was:

<u>Prepare IXC for test.</u>

1. Pump resin and supernatant from Resin Receipt Tank into IXC until sufficient resin is transferred to meet the target resin height of 50.4 inches (53 gallons) for Na-form resin, see Figure 12, or 38.3 inches for the H-form resin.
   a. The pump was started and stopped several times to allow resin to settle and find its level, until the correct level was reached.
   b. The drain valve was used to then trim[5] the resin level to the target[6] test height.
2. Allow the liquid to continue to fill IXC from Receipt Tank until it is about 2 feet from top resin screen, see Figure 13.

[5] Actually setting the height was very difficult. It entailed a lot of stopping and starting of the resin. After each stop the resin had to settle to measure the height and was repeated several times. Unfortunately, with each transfer of the resin the concentration of resin in the test liquid was always different and very often the target heights were missed, sometimes by large margins. Initially, the testing was simply performed at the higher resin level because adjusting the height by opening the drain valve was thought to affect the test results or because dumping the resin and restarting was very time consuming. This is the reason some of the photographs and videos shows a resin height above the target level. However, after several tests it was found that adjusting the resin level by opening the drain valve had no noticeable effect on the test results. The resin height was therefore adjusted by gradually opening the drain valve for the subsequent tests.

[6] It was rare to have the resin height exactly at the target because after the bed was fluidized and settled the true height varied. However, the height was generally within ± 0.5 inch, after using the drain valve to trim the height, as explained.

3. Continue to fill IXC with liquid from liquid feed tank in upflow at approximately 9 BV/h (~8.5 gpm) to fluidize bed.
4. Stop flow and allow resin to settle.
5. Finish filling IXC with downflow from the liquid feed tank at approximately 4 BV/h (~3.5) gpm until the upper target level is reached of 100.8 inches ±0.5 inches (106 gallons = 53 gallons of resin and 53 gallons of liquid freeboard, which does not count the interstitial liquid. Of course, for the H-form tests with the resin height set at 38.3 inches, the freeboard would be larger because the overall height of the column contents was kept at 100.8 inches.)

   <u>IXC is ready for test.</u>
6. Open valve over a 10 second interval.

   Options for test

a. No distributor flow (4 BV/h), but turn on flow after drain to assist resin removal.
b. Distributor flow (4 BV/h) initiated just before opening drain valve.
c. No vent, but starting at atmospheric pressure in top of column, which allowed the column height to drop until the weight of the IXC contents just equaled the vacuum created in the expanded overhead gas space.
d. No vent, but apply a vacuum to prevent column contents to drop on opening the drain valve.

Figure 12. Filling the IXC with resin slurry from receipt tank.

Figure 13. Upflowing supernatant at ~9 BV/h to adjust the bed.

2.3 Full-Scale Drain Tests: Effect of Scale

For the full-scale test, existing equipment, i.e., 40-inch IXC, and a 5 M Na Hanford simulant, were repurposed to minimize time and cost. Figure 14 shows a schematic of the overall resin drain test equipment for the 40-inch (40.5-inch was the actual inside diameter) IXC. The drawing is not to scale but gives an idea of the entire test apparatus. As seen in Figure 15, the IXC itself was located on the main floor of the Engineering Develop Laboratory. The figure shows the IXC filled with simulant to the target height of 100.8 inches and the resin was in the process of settling down to its target value of 50.4 inches, which represents the expected full height of sodium form resin used in the LAWPS IXC. The test in the picture was of the 4-inch drain valve and a vented IXC.

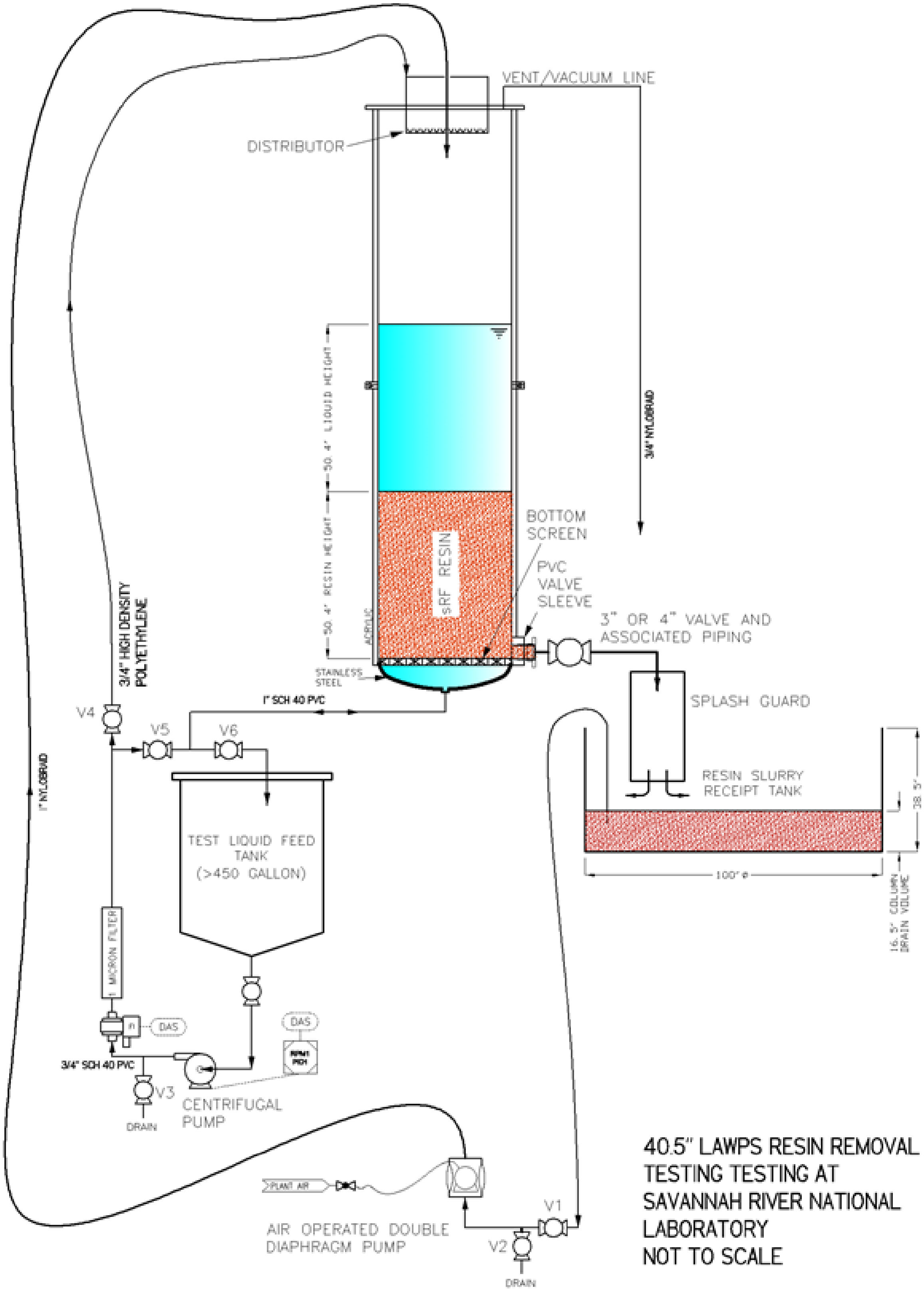

Figure 14. Schematic of 40.5-inch resin drain test equipment.

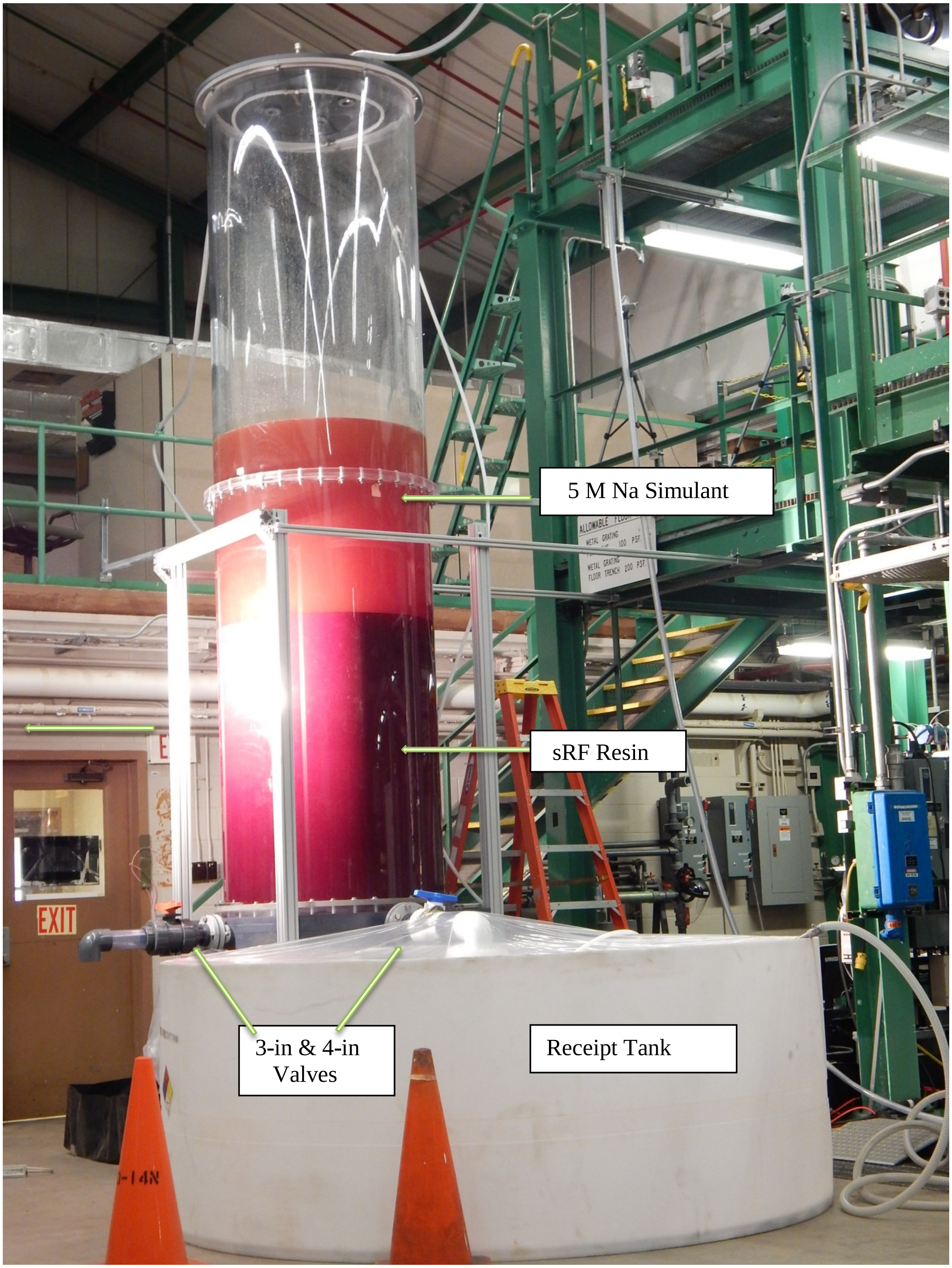

Figure 15. Assembled IXC with sRF resin and simulant at 100.8-inch mark.

Figure 16 shows the feed distributor. The distributor was made of 0.75 inch HPVC tube shaped into an approximately 33-inch diameter ring and had 3/8-inch holes placed every 3 inches along its top. For this test, no top resin screen was used because the existing IXC was much taller than 100.8 inches, so the resin would never reach the top of the IXC. After the IXC was filled with resin and the test liquid to the correct heights, one of the test valves was opened to initiate a drain. The IXC contents then travel through the valve and down through a 90° elbow[7] fitting directly into the 8-foot diameter 38-inch tall receipt tank. Figure 17(a) shows the two valves used for 40-inch IXC tests, i.e., the 3-inch and 4-inch drain valves. Note that the receipt tank is not shown in the figure, but shows a smaller tank used during water shakedown tests to understand the dynamics of the discharging fluid. Because the bottom of the receipt tank was approximately 3 feet from the bottom of the elbow, Figure 17(b) shows the splash guard that was utilized to minimize the spread of test slurry outside the boundary of the receipt tank.

Figure 18(a) shows the 6-inch thick valve sleeve, which contained the four drain ports, i.e., 2-, 3-, and two 4-inch valves[8]. Also seen in Figure 18(a) is the 1-inch thick PVC support plate for the resin screen, which was glued into the valve sleeve before the screen was attached. Just above the support plate, the drain ports can be seen and their relative location to the resin screen. The photograph shows the two 4-inch drain ports and that they were flush with the bottom screen. Figure 18(b) shows the 2-inch and 3-inch ports, which were 1 inch and 0.5 inch above[9] the plate surface, respectively. Note that when a drain port was used for one of the drain tests, the other drain ports were closed off with either a blank flange or closed valve. No effort was made to block those idle ports, and make them flush to the inside diameter of the IXC, as was done for the 18-inch IXC tests (e.g., see Figure 8 in Duignan, 2015c) because there was interest to see how resin fills those dead-end spaces during a test. Also seen in Figure 18(b) is the resin screen while it was being installed. The resin screen used was not a Johnson screen, which has 177-micron sized slits that are oriented parallel on the screen face and are commonly used in IXCs. However the screen used, which had 60-micron openings, was made of stainless steel. The different screens are not expected to result in a significant difference in performance because most of the resin drains out of the IXC before exposing the screen. That is, most of the movement of resin, as it leaves the IXC through the drain port, is resin on resin, until the screen is exposed.

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[7] The choice of the drain system utilized, i.e., valve and fitting, was made from a combination of the actual IXC requirements and experimental needs. Per the customer, the system in the plant will be a valve that allows the drain to be directed immediately out and down from the IXC in a 90° fashion, which had changed from the plant design that existed while planning the 18-inch IXC drain test and shown in Appendix A. The exact valve and fittings are still subject to change, but the current proposed design is as a sketch, Figure 111, in Appendix B. For this test, a simple ball valve was used with two short sections of horizontal pipe to connect the valve to the appropriate drain port and to a 90° elbow with the elbow directed down. The horizontal length of the 4-inch valve system was approximately 30 inches from the opening of the valve port on the inside wall of the IXC to the centerline of the elbow. The valve was approximately in the middle of this arrangement. That 30-inch length allowed the horizontal pipe section between the IXC and the valve, which was transparent plastic, to be long enough to have an approximately 2-inch section through which the resin and simulant could be observed. There was a concern the resin in this section may cause plugging. Furthermore, the 30 inches of the drain system was long enough to fit a receipt tank under the elbow, but far enough away from the tank to provide splash protection. The exit of the elbow sat approximately 3 feet off the bottom of the tank, which meant significant splashing of test solution was expected, especially during the first minute of opening the valve for the vented IXC tests. The other drain valve systems, e.g., 3-inch, were set at the same lengths.

[8] There were three valve sizes and four locations due to early planning; however, the 18-inch IXC tests [Duignan, 2015c] demonstrated that the 2-inch valve did not successfully drain with a non-vented IXC; therefore, it was not used for the 40-inch IXC tests. The second 4-inch drain port was to demonstrate draining through two valves at the same time, but the necessary short duration of the overall project did not allow this test to be included.

[9] The method of fabrication of the valve sleeve required the drain port holes to be in the center of the sleeve height, so the largest ports, i.e., 4 inches, were made flush to the resin screen resulting in the smaller ports to be above the screen.

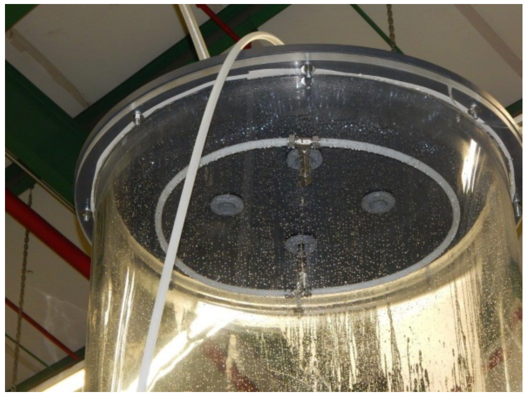
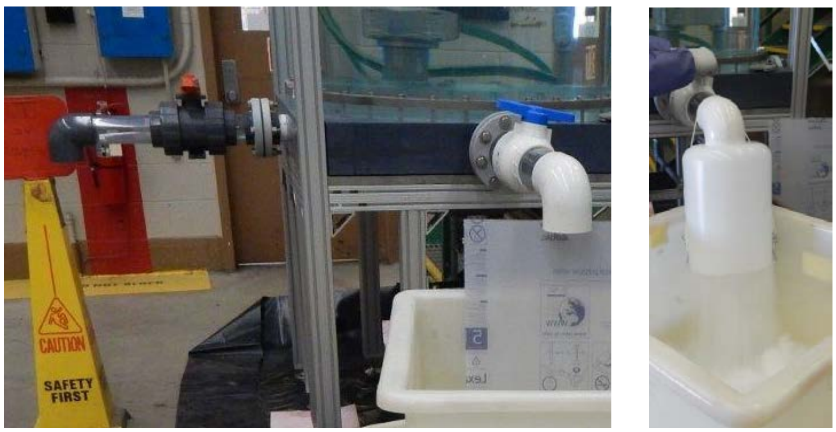


Figure 16. Liquid distributor was the horizontal poly tube. Flow was directed upwards and fell by gravity. No resin screen was used because resin would not, or could not, reach the top of the IXC.



(a) (b)

Figure 17. (a) The flow from the 3-inch (left) and 4-inch (right) drain valves were simply directed down through a 90° elbow, (b) but protected with a splash guard during drains.

(a) (b)

Figure 18. (a) Bottom resin screen support and (b) screen in the IXC with a view of drain ports. Screen was flush with the 4-inch drain holes. No effort was made to fill in drain port not being used in order to see what happed to the resin lodged in these dead-end locations during the test.

During the non-vented test (also referred to as the “glug-glug” test), the contents inside the IXC are not vented to the atmosphere, in order to determine if draining is still possible by air rising from the receipt tank through the opened drain valve. To perform these tests, it was necessary to seal all possible vents, see Figure 14. There was a 1-inch Flex Hose that delivers slurry, but remains empty during each test, and a 0.75-inch Flex Hose that was dedicated to vent the IXC. The vent pathways were closed to perform the no-vent test and the vent hose was used to apply a vacuum to better represent what may happen in the actual IXC. That is, a vacuum is applied that would just counter the weight of the IXC contents, which would prevent the contents from immediately draining the IXC upon opening the drain valve. It is important that the action of air entering the drain valve should be the only mechanism to affect the vacuum and allow the resin to drain. This was desired to more closely mimic what is expected in the actual IXC.

Finally, all the 40-inch tests were done at ambient temperature. No special control was made to maintain the temperature constant, but in the Engineering Development Laboratory it is generally kept at 25°C ±5°C.

Summary IXC40 Test Procedures

Basic Operation for the 40-inch IXC tests was:

<u>Prepare IXC for test</u>

1. Pump resin and supernatant from Resin Receipt Tank into IXC until sufficient resin was transferred to meet the target resin height of 50.4 inches (281 gallons) for Na-form resin, see Figure 19.
    a. The pump was started and stopped several times to allow resin to settle and find its level, until the correct level was reached.
    b. The drain valve was used to then trim[10] the resin level to the target[11] test height.
2. The liquid was allowed to continue to fill IXC from Receipt Tank until it reached the target level of *100.8 inches.*
3. Because of the IXC was much taller than needed, the liquid could not reach the top to return for a circulation of upflow. Therefore, the liquid was then pumped down to approximately 2 inches above the top of the resin bed into the liquid feed tank.
4. The liquid was then returned to the column from the feed tank in upflow at approximately ~5 BV/h (~25 gpm) to fluidize bed, see Figure 20. (The resin generally reached about 80 to 85 inches in height before the upflow was stopped.)
5. The flow was then stopped to allow resin to settle. (This took about 15 minutes because the resin settled at an approximate rate of 2 inches a minute.)
6. If test liquid was needed, then downflow was initiated from the liquid feed tank at approximately ~4 BV/h (~18.5) gpm until the upper target level was reached of 100.8 inches ±0.5 inches. (At this level the IXC contained 562 gallons = 281 gallons of resin and 281 gallons of liquid freeboard, which does not count the interstitial liquid.)

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[10] Actually, setting the height was very difficult, as it was for the 18-inch IXC. It entailed a lot of stopping and starting of the resin transfer pump. After each stop the resin had to settle to measure the height and this action was repeated several times. Unfortunately, with each transfer of the resin the concentration of resin in the test liquid was always different and very often the target heights were missed. For the first simulant test, the resin height was trimmed to exactly 50.4 inches, from 55 inches, and then the liquid height was set at 100.8 inches. However, setting the resin height caused the upper surface of the resin to be slightly uneven due to the draining. For the remaining three tests, the resin height was not adjusted down, which was generally a few inches above the target. This allowed the resin top surface to be level at the start of each test. Furthermore, from the 18-inch test it was found that variation of resin height by several inches did not significantly impact results.

[11] It was rare to have the resin height exactly at the target because after the bed was fluidized and settled the actual height varied.

<u>IXC is ready for test</u>

7. The drain valve was then opened over a 10-second interval.

Options for test

a. Vented IXC to the atmosphere.
b. No vent, but apply a vacuum to prevent column contents from dropping on opening the drain valve.

Figure 19. Filling the IXC with resin slurry from receipt tank.

Figure 20. Upflowing supernatant at ~5 BV/h to fluidize the resin bed.

2.4 <u>Quality Assurance</u>

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

As with the equipment description, the results are presented in order of the scale of each test, that is, 4-inch, 18-inch, and then the 40-inch IXC. To distinguish among the different tests at each scale, the nomenclature for the tests will be prefixed with the scale name, i.e., IXC04-x, IXC18-x, and IXC40-x, respectively.

3.1 Small-Scale Drain Tests: Effect of Temperature

3.1.1 Conditioning Spherical RF Resin

Before the tests began the resin was conditioned by changing it to Na-form. However, the resin had already been converted to perform the mid-scale IXC test, so a portion of that resin was used. See Section 3.2.1 to read the discussion of the conversion. The resin used in the small scale temperature tests was new resin provided by Washington River Protection Solutions. All of the small scale temperature tests were run using the Pretreatment Engineering Platform (PEP) recipe simulated salt solution.

3.1.2 Overall IXC04 Tests

The original test matrix for the small-scale test contained activities other than resin drain tests, for example, oxidizing the resin, shakedown tests, etc. To facilitate understanding of the test performed, Table 1 is limited to the drain tests and the test numbers are in chronological order.

Table 1. The test matrix performed.

Test (1) IXC04-	Drain Date	Temp °C (2)	Resin state (3)	Columns Used (4)	Duration days (5)	Comment (6)
1	9-Oct-15	25	oxidized	3PVC	3	Test 9
2	12-Oct-15	50	new	3PVC	3	Test 6
3	15-Oct-15	25	new	3PVC	3	Test 4
4	29-Oct-15	65	new	1PVC	3	Test 7
5	9-Nov-15	65	new	1PVC, 1SS	14	Test 10
6	4-Dec-15	65	new	2PVC, 1SS	14	Test 10b
7	10-Dec-15	85	new	3SS	3	Test 8
8	14-Dec-15	40	new	3PVC	3	Test 5

Notes:
(1) The test numbers are in chronological order.
(2) ±5°C
(3) The resin in Na-form for Test IXC04-1 was oxidized with air until it turned dark red in color, before the test.
(4) Used either 1 or 3 columns made of either plastic (PVC), or stainless steel (SS).
(5) Duration = time columns were held at the listed temperature before test.
(6) These are the original test matrix test numbers, which contained activities that were not drain tests, for example, oxidizing the resin. Furthermore, the order was not chronological. The new test numbers are drain tests only.

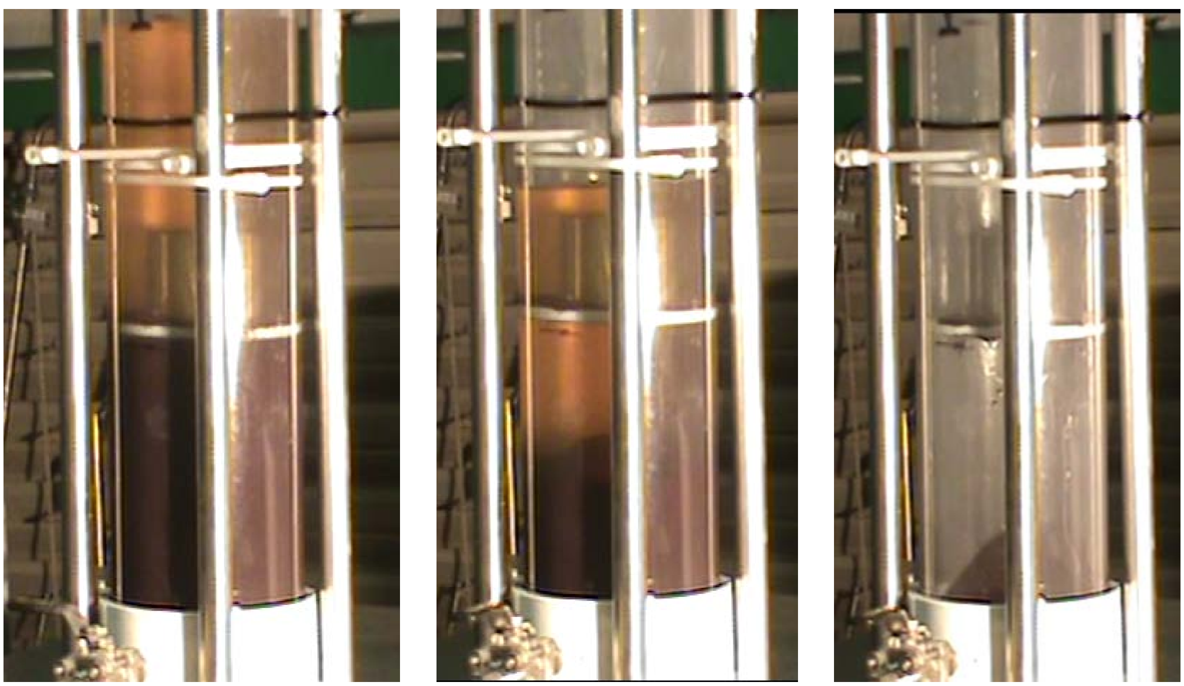
The following sections describe each of the tests by test solution. The photographs included are only examples of the test operations. Pictures and videos were taken of each test, but because they look basically the same, only a few are shown.

3.1.3 Test IXC-04 Results

The small scale temperature tests utilized 6 columns to measure the drain rate, three made of plastic, Figure 2, and three of stainless steel, Figure 3. The plastic was to facilitate observation and the metal allowed testing up to 85°C, at which the PVC plastic was not safe. Both materials were used at 65°C to determine if the material had an effect. Each of the tests is briefly described below in chronological order, which will be followed by a summary of the entire test matrix.

Test IXC04-1: 25°C Test: Oxidized Resin, 3-Day Duration

Three of the PVC columns, i.e., Columns 1-3, were used and specifically tested sRF resin in the oxidized state. Before the test began a batch of resin[12] was subjected to air bubbling through the bed for approximately 8 hours until its color turned a dark red, thus indicating oxidized resin. This first drain test went smoothly and all three columns gave the same results. That is, after sitting in an oven for 3 days at an average temperature of 26.4°C ±2.4°C (1 standard deviation), the columns drained readily. On opening the ball valve, the resin immediately began to flow and all three columns took approximately 1 minute to drain, shown in Figure 21(a) and Figure 21(b) with the data in Table 2. From the starting BV of 1.44 liters of resin, the remaining heel in each of the columns ranged from 13 to 16% of the BV, Table 3. The highest heel level was on the column wall opposite of the drain port and then the heel smoothly tapered down to the exit, Figure 21(c).



(a) (b) (c)

Figure 21. Test IXC04-1 at 25°C with oxidized sRF resin at the (a) start, (b) midpoint, and (c) end of the test showing the resin heel. Pictures are typical of all the test runs.

[12] The resin used was from the 18-inch test that was old resin stored at SRNL to better ensure the resin was oxidized, because that resin had been used for many previous tests and stored for more than 10 years, see Figure 26.

Test IXC04-2: 50°C Test: New Resin, 3-Day Duration

Three of the PVC columns, i.e., Columns 1-3, were used with new sRF resin. This second test went smoothly and all three columns gave slightly different results, but that was probably from handling. Firstly, this was the first test above room temperature and during the 3 days at 50°C, the three IXCs lost water from evaporation, so the draining began with the liquid level a few inches under the target, which was 1BV freeboard of liquid above the resin bed. Secondly, for Columns 1 and 3, the compensation weight did not stay locked in position as the resin drained; therefore, the weight began to lower with the resin, which probably affected the lag time to begin draining and affected the drain rate. The weight was secured in place after about the first 5 seconds; therefore, the drain rate of all three columns is in question and the lag times of Columns 1 and 3 is questionable. However, the filled IXCs did sit in an oven for 3 days at an average temperature of 52.2°C ±1.0°C (1 standard deviation), and the columns drained readily after a lag time. On opening the ball valve, the columns took approximately from 44 to 50 seconds to drain, which would be slightly longer if the liquid levels began at the target height. The data are in Table 2. From the starting BV of 1.44 liters of resin, the remaining heels ranged from 14 to 20%, Table 3, with the highest heel level on the column wall opposite of the drain port, which then smoothly tapered down to the exit. This was slightly more than the average 14% from the 25°C test. However, for this test, the resin took some (lag) time before the resin slurry began to drain. The lag times ranged from 12 to 55 seconds, but the 55-second lag from Column 2 is a good data point because while Columns 1 and 3 started with the compensation weight still in place on top of the resin for the first ~5 seconds, the weight was properly secured for Column 2, Table 2.

Lag Time – This is the period when the IXC has its drain valve open, but the resin is not freely flowing. This issue will be discussed further in the summary of data.

Test IXC04-3: 25°C Test: New Resin, 3-Day Duration

Three of the PVC columns, i.e., Columns 1-3, were used with new sRF resin. This third test at 25°C is considered the baseline test for the small scale temperature testing since the large scale tests were performed (approximately) at this temperature. The test went smoothly and all three columns drained very similarly to the first 25°C test, which used oxidized resin. Once again, the filled IXCs sat in an oven for 3 days at an average temperature of 24.1°C ±1.7°C (1 standard deviation), Table 3, and the columns drained readily with no lag time. On opening the ball valve, the resin immediately began to flow and all three columns took just under 1 minute to drain, Table 2. In fact, the columns drained approximately 6 seconds faster than for Test IWX04-1, but that small difference is probably not significant. From the starting BV of 1.44 liters of resin, the remaining heel in each column ranged from 7 to 21%, with an average of 14%, Table 3. This average is the same as for the first 25°C test, so it appears that oxygen-damaged and new resin drain similarly. The photographs of this test look exactly as those seen in Figure 21.

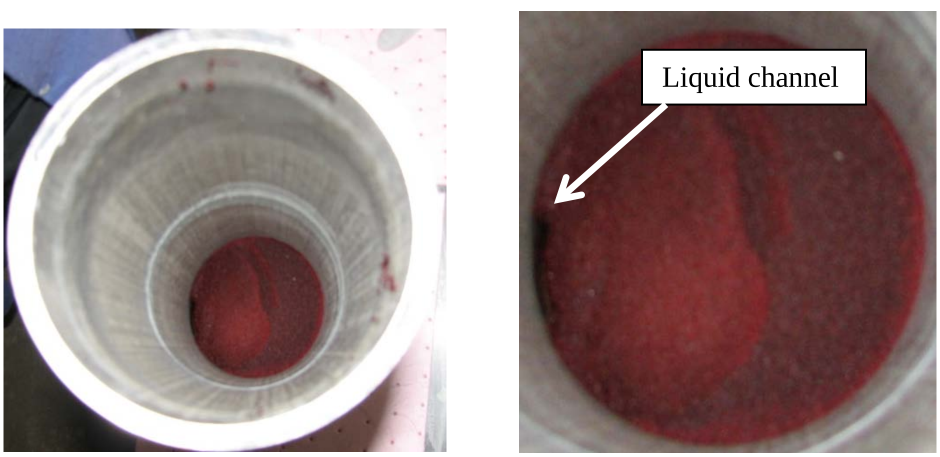
Test IXC04-4: 65°C Test: New Resin, 3-Day Duration

Only one of the PVC columns, i.e., Columns 2, was used with new sRF resin. This was the initial 65°C test and it did not have a lag time and the drain time was short, but it suffered from the same problems encountered for the 50°C test, IXC04-2. That is, water had evaporated over the 3 days it was held at the target temperature and when the test started, the compensation weight was not secured in its position, so for the first ~5 seconds the weight dropped with the resin. The results are questionable, but the 3-day test was a good trial to perform the next 65°C, which would be held for 14 days at the target temperature. The filled IXC did sit in an oven for 3 days at an average temperature of 64.5°C ±2.1°C (1 standard deviation), and the column drained readily with no lag time. On opening the ball valve, the columns only took 37 seconds to drain, which would be slightly longer if the liquid levels began at the target height. The data are in Table 2. From the starting BV of 1.44 liters of resin, the remaining heel was 27% of the BV, Table 3, with the highest heel level on the column wall opposite of the drain port and then the heel smoothly tapered down to the exit. The heel volume was slightly more than the largest heel of 21% from the 50°C test.

Test IXC04-5: 65°C Test: New Resin, 14-Day Duration

A comparison between PVC and stainless steel was the goal for this test and Columns 1 and 4 were used with new sRF resin. This fifth test, second at 65°C, went smoothly and both columns drained at approximately the same rate, i.e., 50 seconds for the PVC and 60 seconds for the SS, Table 2. Before draining, the filled IXCs sat in an oven for 14 days at an average temperature of 62.6°C ±2.8°C (1 standard deviation), Table 3, and the columns drained readily with a very short lag time, from 5 to 7 seconds. With respect to lag and drain times, the two materials appear to be similar. However, there was a complication discussed below that affected the draining of the stainless steel (SS) column.

From the starting BV of 1.44 liters of resin, the remaining heel in the PVC was 11% and the heel in the SS was 43%, Table 3. However, this large heel in the SS column is not accurate. As learned from the previous higher temperature tests it was necessary to periodically add makeup water to compensate for evaporation. However, the draining of the columns for this test occurred on 9 November 2015, which was a Monday after 3-day weekend. The evaporation rate was such that liquid level was below the top of the resin bed. That is, all the liquid freeboard was gone and some height below the top of the resin bed may have been dry too. Unfortunately, because the column was stainless steel this fact could not be verified visually. The evaporation problem was not as strong for the PVC because the plastic has a thermal conductivity of two orders of magnitude smaller than the steel and therefore acts like an insulator; that is, the plastic experienced a much lower rate of evaporation. When it was time to test, the liquid levels in both columns were brought up to the target levels, but if the top few inches of resin in the SS column were indeed dry then that added water may have affected the bead-to-bead structure that was created during the 14 days at 65°C. That is, the wetted resin at the start of the temperature period probably had different structure than from the resin that was wetted just at test time. The result, shown in Figure 22, was that when the resin drained to the bottom of the column the structure of the heel was more like a flat plate, instead of a sloping surface commonly seen in all other tests.



(a) (b)

Figure 22. Test IXC04-5 at 65°C. (a) Looking down into IXC04 SS Column 4 after drain was complete. (b) Shows a larger view of the top surface that does not slope down to the drain port but the liquid drained through the small channel shown to the left. A height measurement confirmed this and the heel volume was 43% of the BV.

Instead of a heel with a sloped surface that is high on the wall opposite of the drain port, the resulting resin heel surface was basically flat across the entire surface, except right above the drain port. This was verified by measuring the height of the heel across the surface. The shadow on the resin heel surface seen in Figure 22 gives the impression there was a slope; however, the impression was actually made from the mark left when the resin surface separated from the compensation weight during the draining process. There was a small crevice/hole just above the drain port, which allowed just the liquid to drain. Note that when this test was repeated (IXC-04-6) and the resin remained whetted, the flat plate structure did not form and the resin drained similarly to the other tests.

Test IXC04-6: 65°C Test: New Resin, 14-Day Duration

One last 14-day test was performed using both PVC and SS columns, specifically Columns 1 and 3 (PVC) and Column 4 (SS). More columns were not included because of the lack of physical space in the oven. This sixth test, third at 65°C, went smoothly, but the SS column drained slightly slower than the PVC columns, i.e., ~58 seconds for the PVC and 73 seconds for the SS, Table 2. Before draining, the filled IXCs sat in an oven for 14 days at an average temperature of 67.0°C ±2.9°C (1 standard deviation), Table 3, and the columns drained readily. However, Table 2 shows the lag times varied significantly. The PVC Column 1 had a lag time of 195 seconds and Column 3 had a lag time of 30 seconds. The SS Column 4 had a lag time of 29 seconds, matching the second PVC column so the data appear to be random. The general conclusion is that a lag time exists at higher temperatures before smooth draining commences. From a starting BV of 1.44 liters of resin, the remaining heels were 7% for both the PVC and SS columns, Table 3.

Test IXC04-7: 85°C Test: New Resin, 3-Day Duration

The test at 85°C was for a 3-day period and was performed only using the three SS columns, i.e., Columns 4, 5, and 6. The plastic PVC columns could not be safely used at this temperature. This seventh test, went smoothly, and all columns drained close to 2 minutes, slightly longer than the 1 minute experienced from the other tests, i.e., 149 seconds, 134 seconds, and 107 seconds for Columns No. 4, 5, and 6, respectively, Table 2. Before draining, the filled IXCs sat in an oven for 3 days at an average temperature of 85.2°C ±1.7°C (1 standard deviation), Table 3, and the lag times ranged from 22 to 38 seconds, Table 2. From a starting BV of 1.44 liters of resin, the remaining heel in Column 4 was 11%, Table 3. The heels were not measured in Columns 5 and 6.

Test IXC04-8: 40°C Test: New Resin, 3-Day Duration

A final test was performed at 40°C, which was maintained for a 3-day period using the three PVC columns, i.e., Columns 1, 2, and 3. This eighth, and final test went smoothly, and all drained close to 1.5 minutes, which is slightly longer than the 1 minute from the lower temperature test, and shorter than the 2 minutes from the 85°C test, i.e., 81 seconds, 94 seconds, and 94 seconds for columns No. 1, 2, and 3, respectively, Table 2. Before draining, the filled IXCs sat in an oven for 3 days at an average temperature of 41.9°C ±0.7°C (1 standard deviation), Table 3, and the lag times ranged from 0 to 18 seconds, Table 2. From a starting BV of 1.44 liters of resin, the remaining heels in all three columns were similar, i.e., 23%, 21%, 21% respectively, Table 3.

3.1.4 Summary 4-inch IXC Test Results

All of the drain and lag times are listed in Table 2 to better compare the results.

Table 2. Small scale IXC temperature tests

Test	Temp	Resin	Columns	Period	Col. No. 1		Col. No. 2		Col. No. 3		Col. No. 4		Col. No. 5		Col. No. 6		
IXC04-	°C	state	Used (2)	days	Lag	Drain	Lag	Drain	Lag	Drain	Lag	Drain	Lag	Drain	Lag	Drain	Comments
					s.	s.	s.	s.	s.	s.	s.	s.	s.	s.	s.	s.	
1	25	oxidized	3PVC	3	0	61	0	65	0	62							
2	50	new	3PVC	3	15	44	55	50	12	50							
3	25	new	3PVC	3	0	55	0	59	0	56							
4	65	new	1PVC	3			0	37									Trial with 1 column only
5	65	new	1PVC,1SS	14	5	50					?	60					No lids on columns (3)
6	65	new	2PVC,1SS	14	195	59			30	57	29	73					Repeat with lids (3)
7	85	new	3SS	3							36	149	38	134	22	107	
8	40	new	3PVC	3	0	81	18	94	0	94							
		Drain Time Averages				61		64		67		94		134		107	Only full column
		Drain Time Standard Deviation				14		24		19		39		0		0	averages.

Notes:
(1) Column 1 to 3 were of plastic (PVC), and 4 to 6 were of stainless steel (SS).
(2) PVC = Plastic column, SS = Stainless steel column
(3) After the 1st 14-day test at 65°C where the evaporation was high a loose-fitting lid was constructed for each column to minimize the loss of water.
[green] = Tests in which the compensation weight was accidently left in place, on the resin, after the drain valve was opened, either during the lag time or during the drain time, if there was no lag time. However, the time left in place was less than about 5 seconds.
[yellow] = Tests when the column liquid level was below the target level because of evaporation. Liquid was added to the other tests to make up what evaporated.

To illustrate some of the trends from the results Figure 23 compares the resin drain rates with temperature for both PVC and SS. Due to data variability and uncertainty from 25° to 65°C, it appears that temperature does not affect the time it takes to drain resin under gravity in a vented column. Furthermore, at 65°C it appears that the material of the column does not significantly affect drain rate. The resin drains between 1 to 1.5 minutes in all cases. However, for the highest temperature used, i.e., 85°C, there was a significant jump in the drain time to just over 2 minutes. From the two SS IXC tests, i.e., 65°C and 85°C, the drain time doubled. There were three (3) SS columns used for the 85°C test and the drain times varied from 107 to 149 seconds, showing a significant variability. Furthermore, the ~ 2 minutes of drain time was only approximately double that at temperatures greater > 65°C so it would appear that while temperature does have an effect, it is not a large affect. Due to the plastic not being able to safely operate at 85°C, the temperature effect could not be confirmed.

Another insight to the draining process is the time necessary to begin draining, referred to as lag time. This is the period when the IXC has its drain valve open, but the resin is not freely flowing. Observation shows that on opening the valve a little bit, liquid drops of the supernatant exit the valve. Through the resin, which is packed in place, the liquid must squeeze by, as if the resin is acting like a filter. As time goes on, the drops become low flowing intermittent stream with some resin beads, as if the resin is being entrained in single and multiple packets. Eventually, the resin becomes fully fluidized and then the stream flows as a liquid freely. For all vented larger scale test, this lag phenomenon was not experienced; therefore, it may be related to the small scale. The opening of the drain valve was 0.375 inch (9.5 mm), which means it was only approximately 21 times larger than the 0.45-mm resin beads. When the drain is first opened, the 21-bead wide front may be bridged, caused by the higher temperature, and only after some of the beads in the front are entrained and fluidized does the resin bed become a slurry and act like a fluid. In fact, it was noted that on cleaning the resin after the test, that the resin beads seemed to be slightly sticky, causing some of the beads to form clumps, which may also help to answer the question about the cause of lag time. This "clumping" may imply that the rheological yield stress of wet resin increases with temperature. However, no stress measurements were made with the clumpy resin.

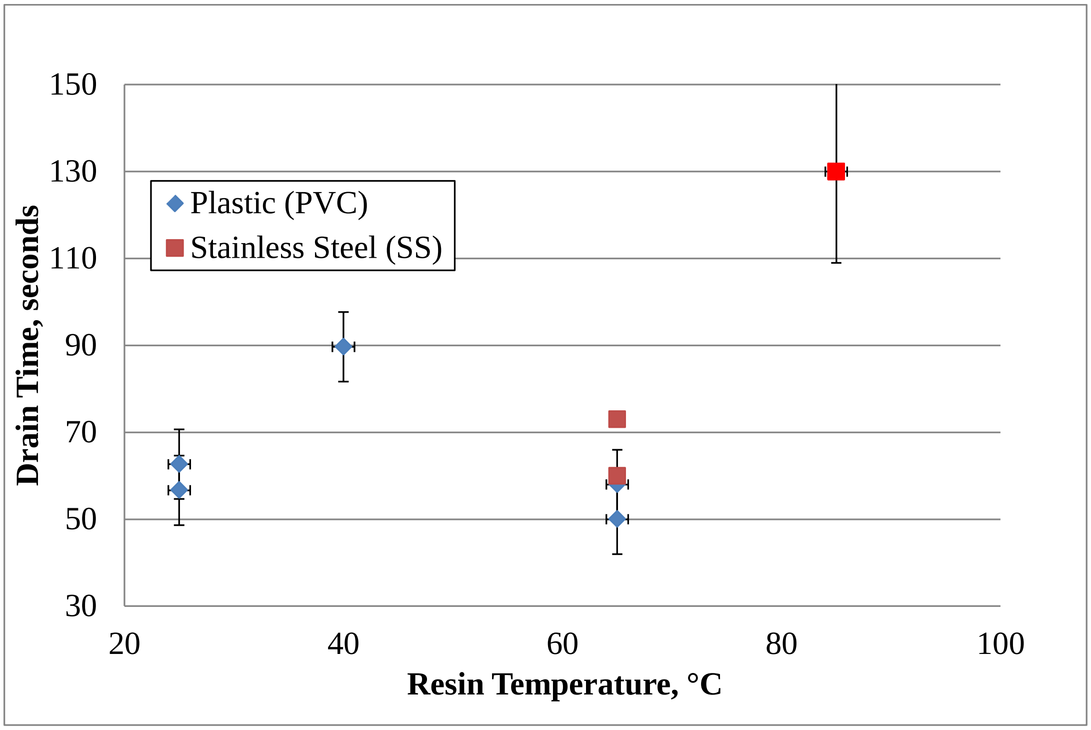


Figure 23. The effect of temperature on resin drain rate.

Figure 24 shows all the lag times versus temperature as a function of IXC material. Besides the one low lag-time result at 65°C, it appears that as temperature increases the lag time increases. The effect is larger for PVC, but there is a slight increase in lag time for the SS columns too, but it appears to be insignificant due to measurement uncertainty. For all tests done at 25°C, there was no lag time and for all tests above this temperature, there was a measurable lag time, but with a large variation from 5 seconds to 195 seconds, Table 2. After each test, the higher temperature test, i.e., 40°C to 85°C, the drain resin appeared to be "stickier" than the resin at 25°C. This change in adhesion probably helped to form a bridge in the small drain opening of 9.5 mm, but because of the large range of lag times it appears the process is stochastic and cannot be easily predicted. Fortunately, there did not appear to have any significant lag times for the larger scale test; therefore, it is probably related to the size of the valve opening and resin bead size, but this is only speculation. It is important to note that the relation of size to lag time cannot be answered with the larger scale tests because they were all done at room temperature, i.e., 25°C ±5°C, so it is not known if a lag would exist at the larger scales at higher temperatures.

Another issue is the amount of resin left in the small column after draining is complete. That information can be found in Table 3 along with the actual temperatures used and their variation over the length of the constant temperature period, as indicated by the standard deviation of each data stream. The way the heels were measured was simply by measuring the high level of the heel, which invariably was on the column wall directly opposite of the drain port and the lowest level of the heel, which would be, in most cases, right at the drain port. There was one exception, which will be explained later. With those measurements, the thickness of the heel was roughly estimated by assuming a uniform heel that had a diameter of the inside diameter of the IXC and with one half the height of the measured thickness. The actual heel was more of a parabolic shape, so the estimate made in the table is conservative in that the true heel volume should be slightly smaller.

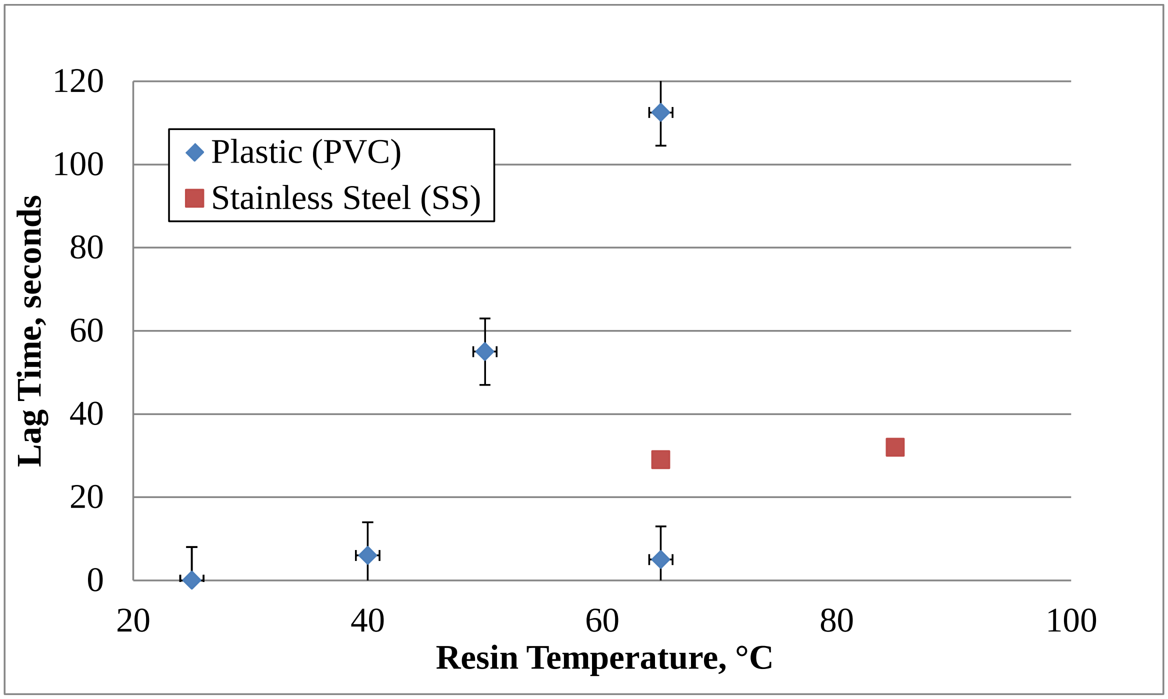


Figure 24. The effect of temperature on resin drain lag time.

Table 3. Actual test temperatures and remaining resin heel after drain.

Test (1) Number IXC04-	Test Drain Date	Target Temp. °C	Actual Temp. °C (1)	Standard Deviation °C (1)	Initial Resin BV liters (2)	Columns 1 or 4 Heel Volume liters	%	Columns 2 or 4 Heel Volume liters	%	Columns 3 or 6 Heel Volume liters	%
1	9-Oct-15	25	26.4	2.4	1.44	0.23	16	0.21	14	0.18	13
2	12-Oct-15	50	52.2	1.0	1.44	0.28	20	0.21	14	0.21	14
3	15-Oct-15	25	24.1	1.7	1.44	0.21	14	0.31	21	0.10	7
4	29-Oct-15	65	64.5	2.1	1.44	(3)		0.39	27	(3)	
5	9-Nov-15	65	62.6	2.8	1.44	0.15	11	(3)		0.62	43
6	4-Dec-15	65	67.0	2.9	1.44	0.10	7	0.10	7	0.10	7
7	10-Dec-15	85	85.2	1.7	1.44	0.15	11	(4)		(4)	
8	14-Dec-15	40	41.9	0.7	1.44	0.33	23	0.31	21	0.31	21

Notes:

(yellow) = Heel form a block. Assumed because of the lack of water

(heavy box) = Stainless Steel column used. Col. No. 4, 5, or 6. All the rest were of PVC Col. No. 1, 2, or 3.

(1) These are the average temperatures over the duration of each test, i.e., 3 or 14 days and the standard deviation is from the fluctuations over those periods.

(2) All volumes, initial Bed Volume (BV) or heel, are based on (resin height) X (column flow area) of IXC.

(3) No column tested.

(4) Heel not measured.

Taking all the data in Table 3, the estimated heel volumes are shown as a percentage of the BV used for this small scale test, i.e., 1.44 liters, and are shown in Figure 25. The shot-gun pattern of the data implies that the heel is not a function of temperature or the material of the IXC. Furthermore, the baseline temperature data, i.e., 25°C showed no difference whether the resin was new or oxidized. If fact, from the table, the new resin heel data at 25°C range from 7% to 21%, while the oxidized resin heel data at the same temperature range from 13% to 16%. Coincidentally, they both have heel averages of 14%.

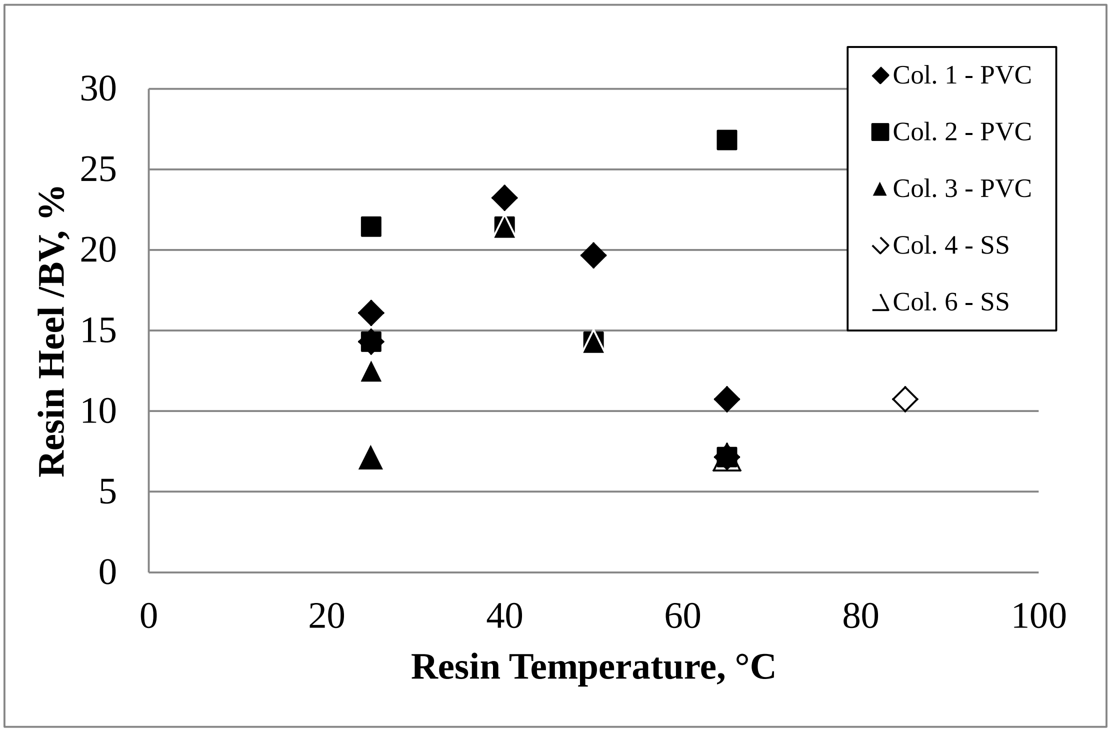


Figure 25. The effect of temperature on resin drain rate.

Finally, the one unusual data point, mentioned above, occurred for the Test IXC04-5 at 65°C in the stainless steel IXC. In the section for that test, this anomaly was explained in detail and will not be repeated here. However, the unusually large heel of 43% occurred because some percentage of the resin was dried during the long period that evaporated water was not replaced, having occurred over a long weekend. When water was replaced in the SS column before the draining occurred, the rewetted resin more than likely had a different adherence structure and that structure remained intact when the draining stopped because a small channel was made just above the drain port. The resulting heel was thick and in the form of a hockey puck with one little hole at the drain. The datum point is not included in Figure 25 because it is not representative of an expected prototypic heel. More than likely all of the heels for the small scale are not prototypic because the method of mimicking the full column weight of a 50.4-inch resin bed by using a weight. The method has its limitations, like wall effects, differences in drain when the weight is removed, etc.

3.2 <u>Mid-Scale Drain Tests: Effects of Waste Streams. Pressure, and Drain Valve Size</u>

These tests began by using two caustic solutions, that is, 0.01 M NaOH and the PEP simulant, which is a simulated salt solution; however, before testing the sRF resin was conditioned to be in Na-form. The final set of tests was performed with an acid test solution, that is, 0.5 M HNO_3; therefore, the sRF had to be first returned to hydrogen form. Normally these processes are performed in the actual column. However, because a specific bed volume was required, i.e., 53 gallons in Na-form, and many tests were required, which means the IXC had to be refilled multiple times, then the resin was conditioned separately in the receipt tank. Subsequently, the resin was introduced into the IXC to set up each test.

3.2.1 Conditioning Spherical RF Resin

Before the test began, the resin was conditioned by changing it to Na-form for the tests with 0.01 M NaOH and PEP simulant and then returning it to H-form for the tests with 0.5 M HNO_3. (Note that this was legacy resin received stored at SRNL and had been used in previous tests.) Samples were taken before converting the resin, (Figure 26), after converting to Na-form (Figure 27), and after it was converted to H-form (Figure 28). The actual form of the stored resin, seen in Figure 26, was in doubt because the stored liquid measured a pH~9. (Note that this resin used in the mid-scale testing was legacy resin used in previous testing in 2005.) Therefore, while the resin may have been H-form, the liquid was slightly caustic. However, the range of resin diameter shown in the figure indicated the actual form may have been mixed along with suffering from some O_2 damage from being used many times in the past.

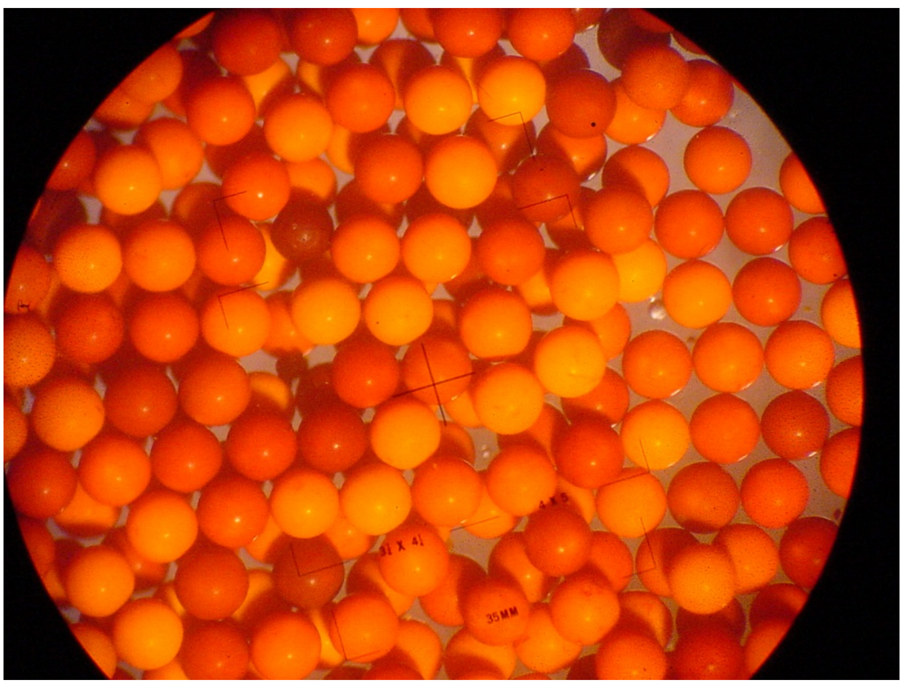


Figure 26. H-form resin with sizes of ranging from 410 to 440 microns.

In converting the resin to Na-form, the concentration of caustic used was 1.8 M NaOH instead of the traditional concentration of 0.5 M NaOH. This higher concentration allowed the use of much less solution and, after soaking in the caustic for over a weekend, the concentration dropped to approximately 0.2 M NaOH based on a measurement of the solution density. Clearly the resin adsorbed much of the sodium and the resin size increased to that shown in Figure 27. At this point, the solution was replaced with 0.01 M NaOH to begin the inhibited water tests. Furthermore, if the average values of the resin sizes are used, shown in Figure 27 and Figure 28, then the resin bed volume grew by approximately $[1+(460-430)/430]^3 = 1.224$, or 22%, which was expected [Duignan et al. 2008].

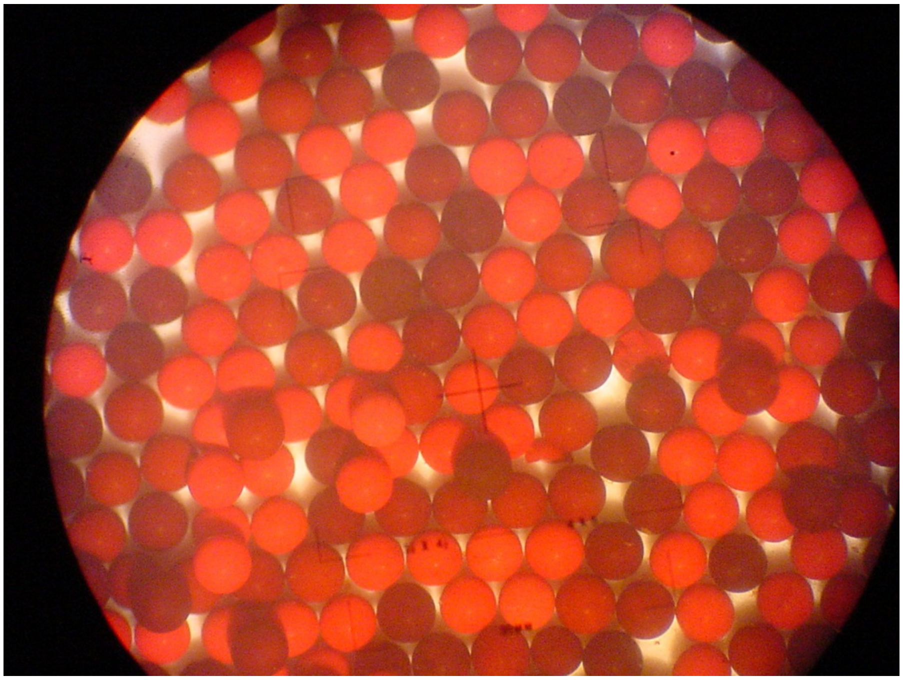


Figure 27. Sodium-form resin with sizes of approximately 460 microns and an overall color change to a burnt orange.

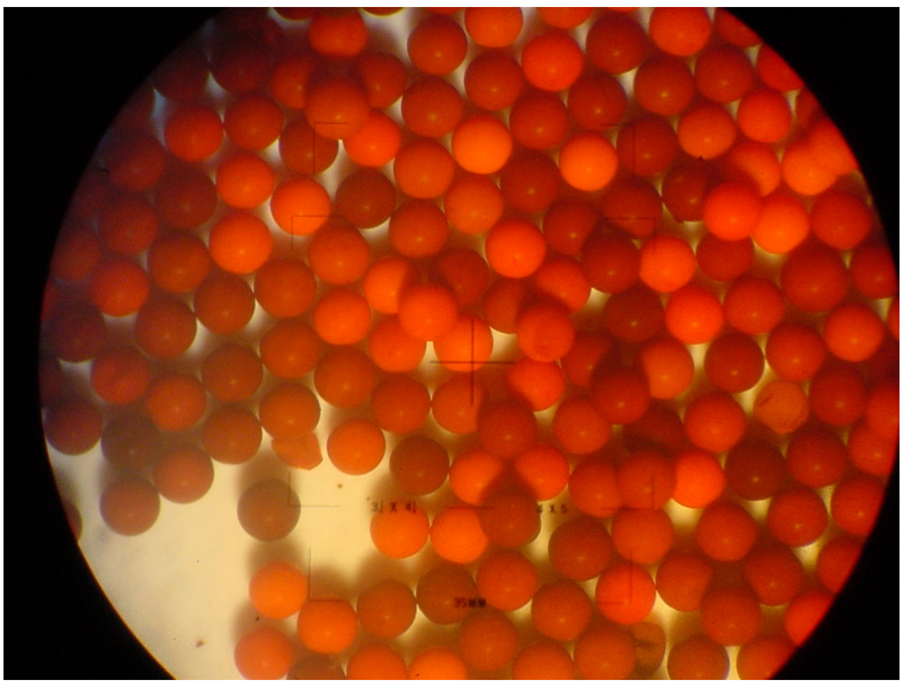


Figure 28. H-form resin after converting resin with sizes of approximately 430 microns. The resin was clearly lighter in color than the Na-form, which is not easily seen from the photograph, but it didn't attain the bright orange of new H-form resin.

3.2.2 Overall IXC18 Tests

As shown in Table 4, in chronological order, a total of 22 tests were performed using 3 different size drain valves, i.e., 1-, 2-, and 3-inch. Three different test solutions were used, i.e., inhibited water (IW = 0.01 M NaOH), PEP (Hanford salt simulant, 5 M Na), as well as PEP with a precipitation agent[13], and resin elution solution (0.5 M HNO_3).

Table 4. Resin drain tests performed with 17.6-inch inside diameter IXC

Test Name	Daily Tests	Test Date	Valve (in)	Test Liquid
IXC18-1	1	31-Aug-15	3	0.01 M NaOH
IXC18-2	2	31-Aug-15	3	0.01 M NaOH
IXC18-3	3	31-Aug-15	1	0.01 M NaOH
IXC18-4	4	31-Aug-15	1	0.01 M NaOH
IXC18-5	1	2-Sep-15	1	PEP-5 M Na
IXC18-6	2	2-Sep-15	1	PEP-5 M Na
IXC18-7	3	2-Sep-15	3	PEP-5 M Na
IXC18-8	1	3-Sep-15	3	PEP-5 M Na
IXC18-9	2	3-Sep-15	3	PEP-5 M Na
IXC18-10	3	3-Sep-15	3	PEP-5 M Na
IXC18-11	4	3-Sep-15	2	PEP-5 M Na
IXC18-12	1	8-Sep-15	2	PEP-5 M Na
IXC18-13	1	9-Sep-15	3	PEP-5 M Na
IXC18-14	2	9-Sep-15	3	PEP+$Al(NO_3)_3$ (1)
IXC18-15	3	9-Sep-15	3	PEP+$Al(NO_3)_3$ (1)
IXC18-16	1	22-Sep-15	3	0.5 M HNO_3
IXC18-17	2	22-Sep-15	3	0.5 M HNO_3
IXC18-18	3	22-Sep-15	3	0.5 M HNO_3
IXC18-19	1	23-Sep-15	3	0.5 M HNO_3
IXC18-20	2	23-Sep-15	3	0.5 M HNO_3
IXC18-21	3	23-Sep-15	2	0.5 M HNO_3
IXC18-22	4	23-Sep-15	2	0.5 M HNO_3
Note: (1) $Al(NO_3)_3$ was the precipating agent				

The following sections describe each of the tests by test solution. The included photographs are principally shown for the first phase of testing, i.e., inhibited water, because for all succeeding tests the visual appearance were similar; therefore, do not add information. However, photographs of key moments of those other tests are shown when they better illustrate the accompanying discussion.

[13] The precipitation agent was a 32.8 wt% solution of $Al(NO_3)_3 \cdot 9H_2O$, which was added at a concentration of 8.7 wt% of the agent to the PEP simulant. These concentrations are based on a bench-top test that determined an amount of precipitated solids sufficient to cover the bottom of the test beaker.

3.2.3 Test Solution - Inhibited Water – 31 August 2015

Test IXC18-1: 3-inch Drain Valve with No Distributor Flow During Resin Drain

To initiate the testing, the valve was opened over a 10 second period, Figure 29 and Figure 30. From the start of the countdown to the completion of the resin and supernatant draining, the duration was 1 minute, Figure 31, Figure 32, and Figure 33. During the last few seconds, the supernatant reached the top of the 3-inch valve port. At this point the drain rate increased, but it also entrained some of the resin heel in the bottom of the IXC. After completion, there was a heel of approximately 8 inches on the IXC wall directly across from the drain port and the resin surface had a gentle slope downwards until about the center of the IXC, Figure 34. From the center to the drain port, the slope of the resin was precipitous with the appearance of an avalanche. The angle of repose for the remaining resin excluding the zone around the discharge valve was approximately 25 degrees. Therefore, there is the potential for a greater heel of resin as the column diameter increases. At no time did the drain pipe from the drain valve to the transfer tank show any plugging, Figure 35 and Figure 36 About 90% to 95% of resin drained from the IXC. Roughly, there was about 4 gallons of resin remaining, Figure 37. With less than 5 minutes of follow-up distributor flow (less than 15 gallons), only trace resin was left on the resin screen, Figure 38. The drain pipe was completely clean from this action, too, Figure 39.

Figure 29. Test IXC18-1 – Open up valve over 10-second period.

Figure 30. Test IXC18-1 – After a few second with valve fully open.

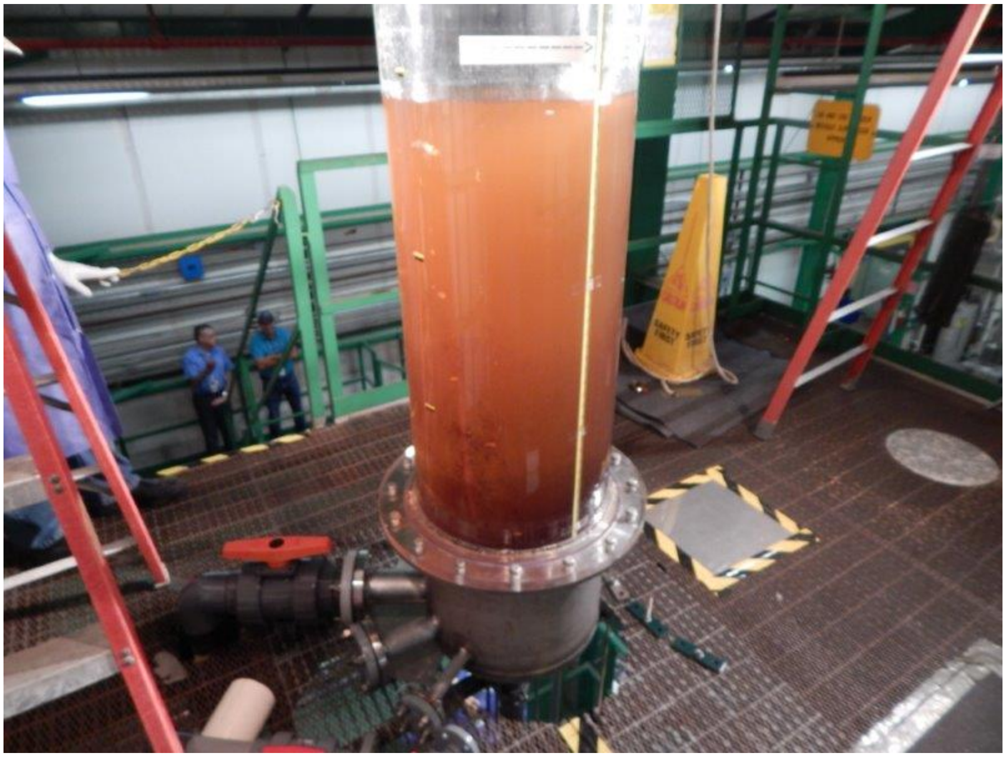


Figure 31. Test IXC18-1 – Drain continues with liquid level present.

Figure 32. Test IXC18-1 – Liquid and resin continues to drain as liquid approaches top of drain port.

Figure 33. Test IXC18-1 – After 61 seconds drain is complete with resin heel remaining.

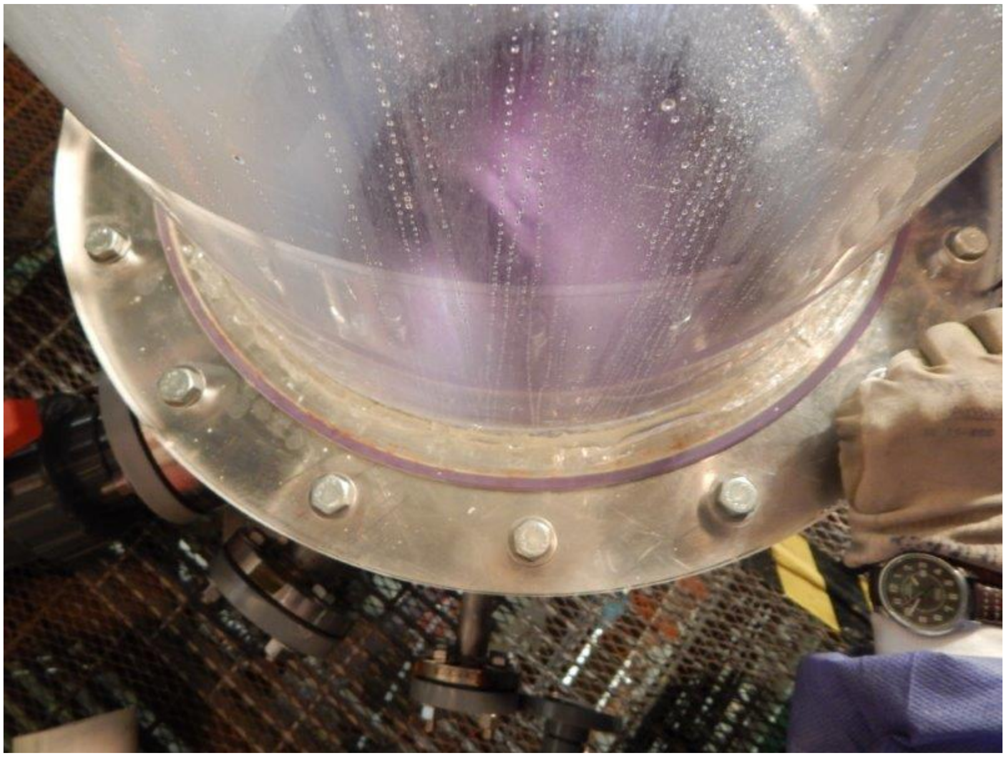


Figure 34. Test IXC18-1 – Resin heel gently slopes to IXC center, then drops precipitously.

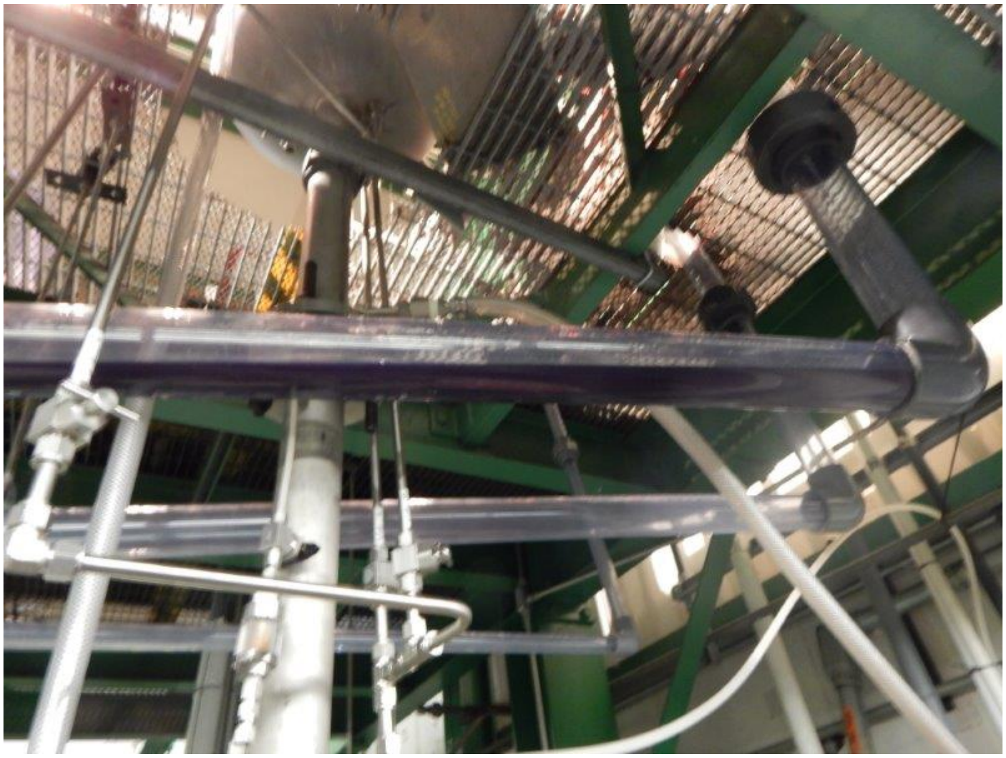


Figure 35. Test IXC18-1 – Initial part of drain pipe with slight resin bed remaining.

Figure 36. Test IXC18-1 – Final part of drain pipe with slight resin bed remaining.

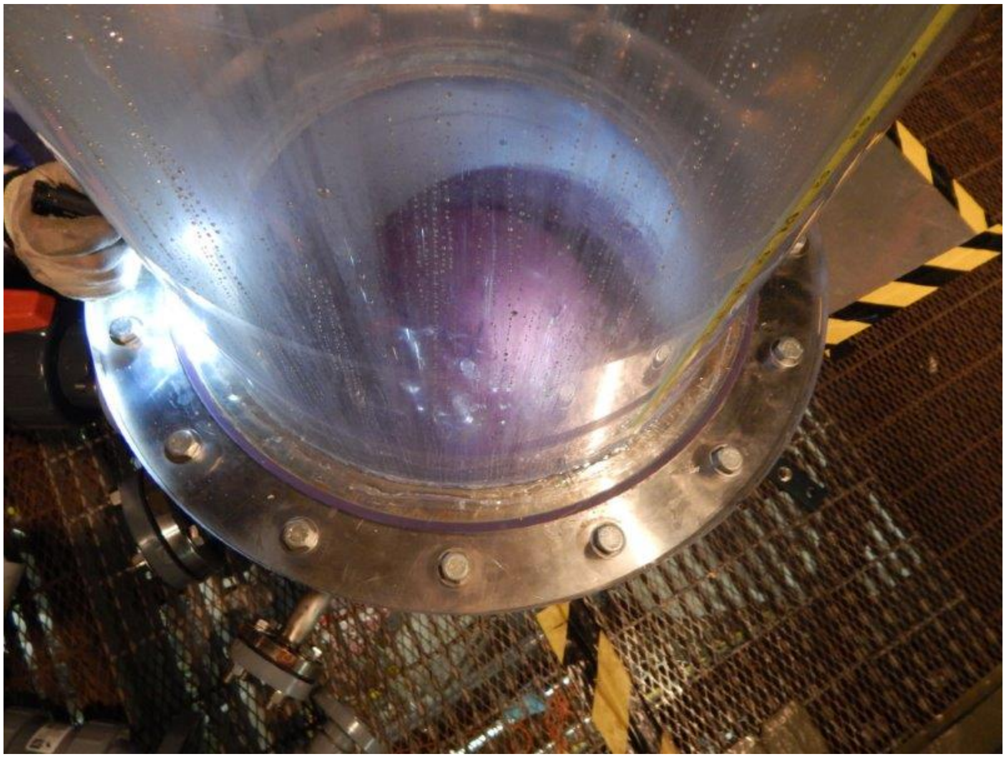


Figure 37. Test IXC18-1 – Distributor flow of 4 BV/h initiated and resin heel is collapsing.

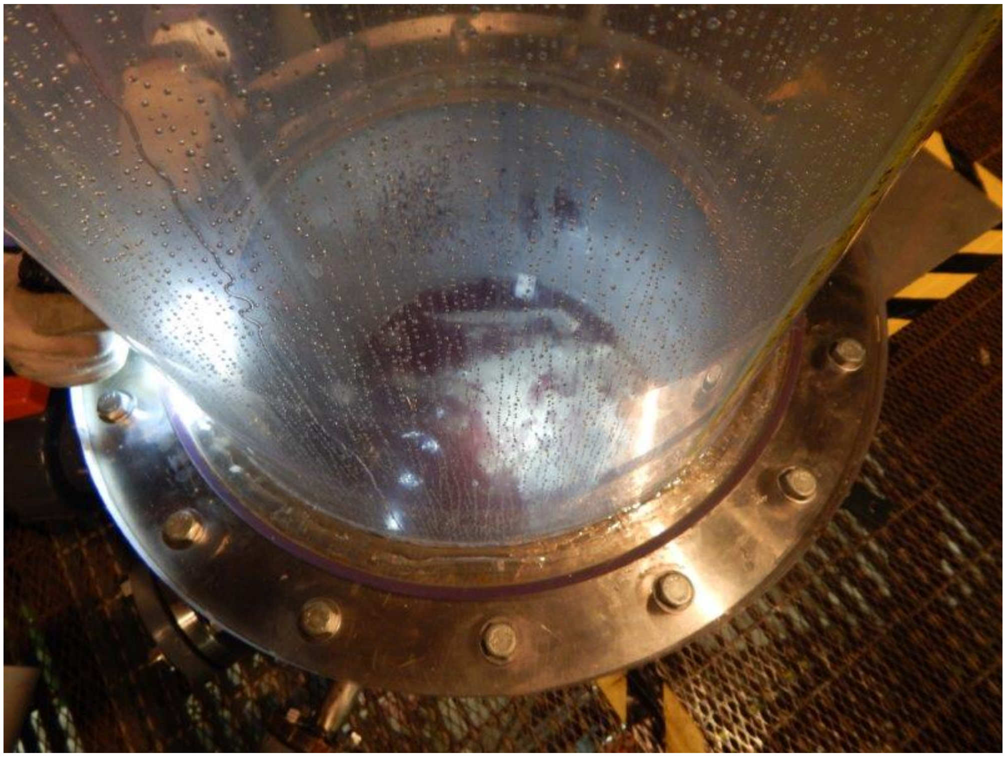


Figure 38. Test IXC18-1 – After a few minutes of distributor flow of 4 BV/h, only trace resin remains.

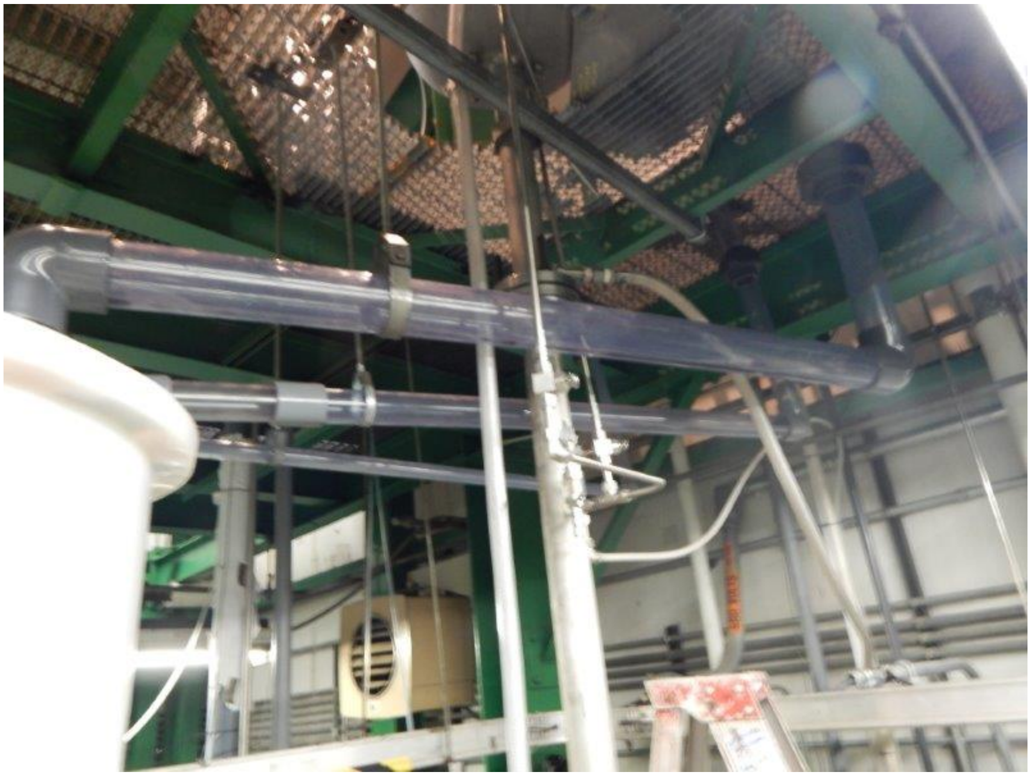


Figure 39. Test IXC18-1 – Drain pipe is completely clear of resin after distributor flow.

Test IXC18-2: 3-inch Drain Valve Distributor Flow During Resin Drain at ~ 4 BV/h

The distributor flow was first initiated at approximately 4 BV/h and then the drain valve was opened over a 10 second period. From the start of the countdown to the completion of the resin and supernatant draining took 1.1 minutes. Because of the continued distributor flow, the liquid level was higher as the drain progressed and no resin heel accumulated at the completion of the drain. The distributor liquid was allowed to flow for a few minutes (less than 15 gallons) after all the IXC contents exited to help carry the remaining resin in the drain system to flush to the receipt tank.

After terminating the test, only trace resin was left on the resin screen.

Note that the resin height was much higher than desired; see Figure 40, because it is very difficult to see the resin height while loading. However, because of the difficulty in removing resin without using the drain valve, the higher level was used. The height of total IXC contents was the same, that is, 100.8 inches, which means the overlaying liquid freeboard was shorter by the amount of resin overage. It was also thought that the extra resin height would produce a more extreme test in that if all the resin was removed than a smaller volume would be less of a problem.

Figure 40. Test IXC18-2 – Ready to test – Difficulty to set resin height caused a 12-gallon overage.

The major difference between this test and Test IXC18-1 is that distributor flow was flowing during the entire drain process to demonstrate the operation. Figure 41 show the draining action was immediate and fast. During the ~10 seconds of opening the valve the resin height already dropped by 15 inches. In less than 30 seconds almost one half the resin had drained, Figure 42. During the drain, the distributor flow flowed through the upper resin screen at random locations, some on the column wall and some through the center, Figure 43 and Figure 44. After most of the resin was drained and the liquid level reached the drain port, the liquid agitation increased as air rose from the valve, Figure 45. Even after most of the contents of the IXC drained, the distributor flow continued to rinse the resin screen of the remaining resin, Figure 46. The IXC and drain pipe had trace resin after the distributor flow was stopped, Figure 47.

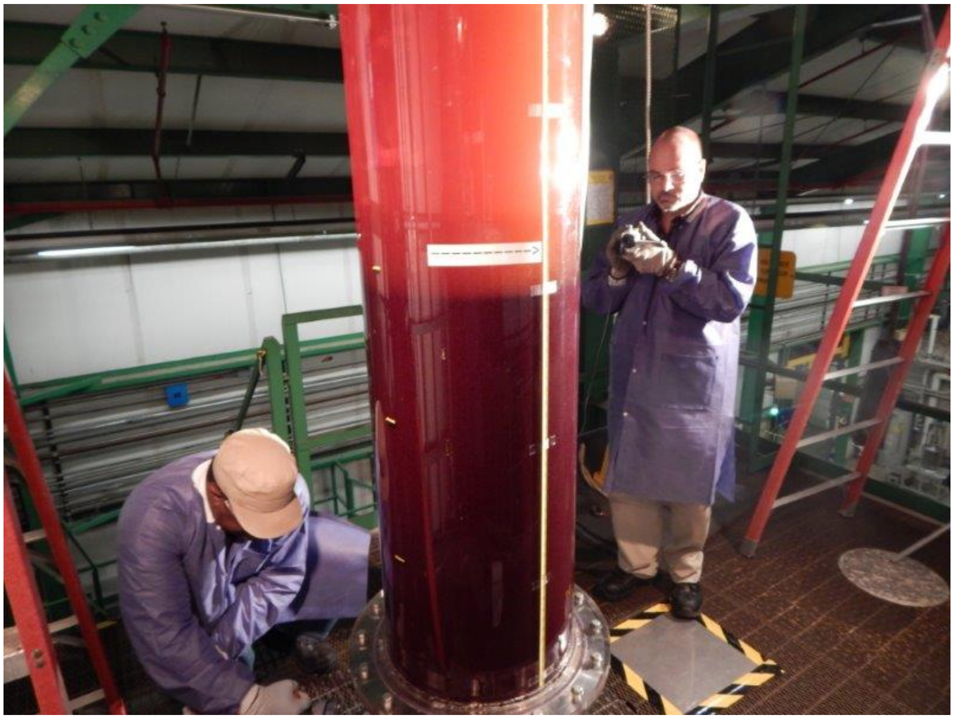


Figure 41. Test IXC18-2 – While in the process of opening the 3-inch valve, near the end of the 10-second opening duration, the resin already dropped about 15 inches (or about 16 gal).

Figure 42. Test IXC18-2 – IXC contents continue to drain.

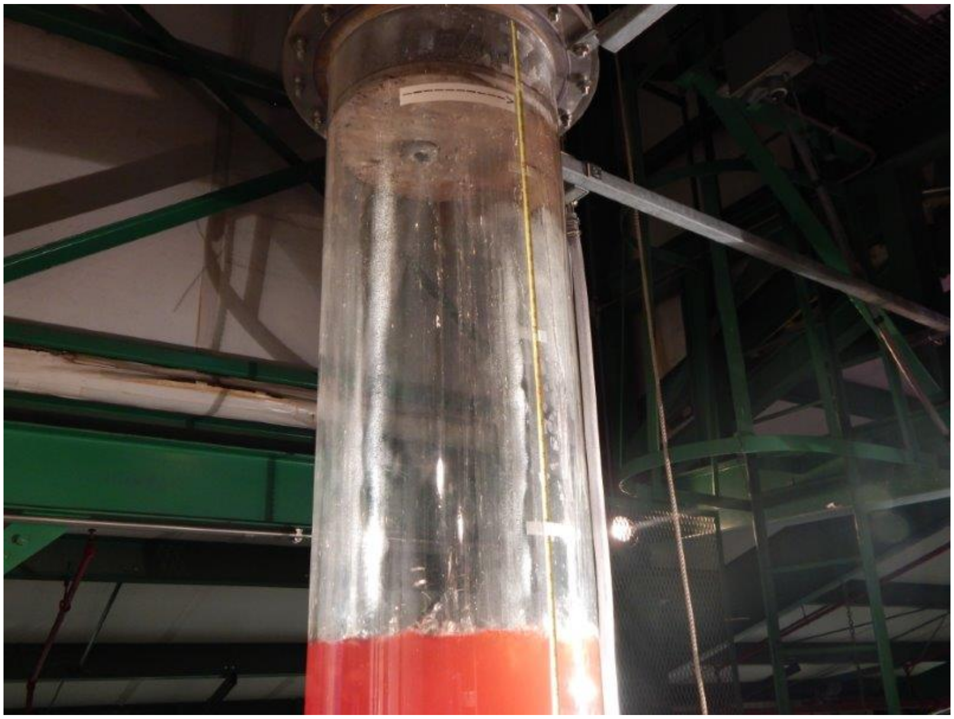


Figure 43. Test IXC18-2 – During draining 4 BV/h of solution entered the IXC through the distributor.

Figure 44. Test IXC18-2 – Closer look of the uneven distributor flow. Most ran down the IXC wall.

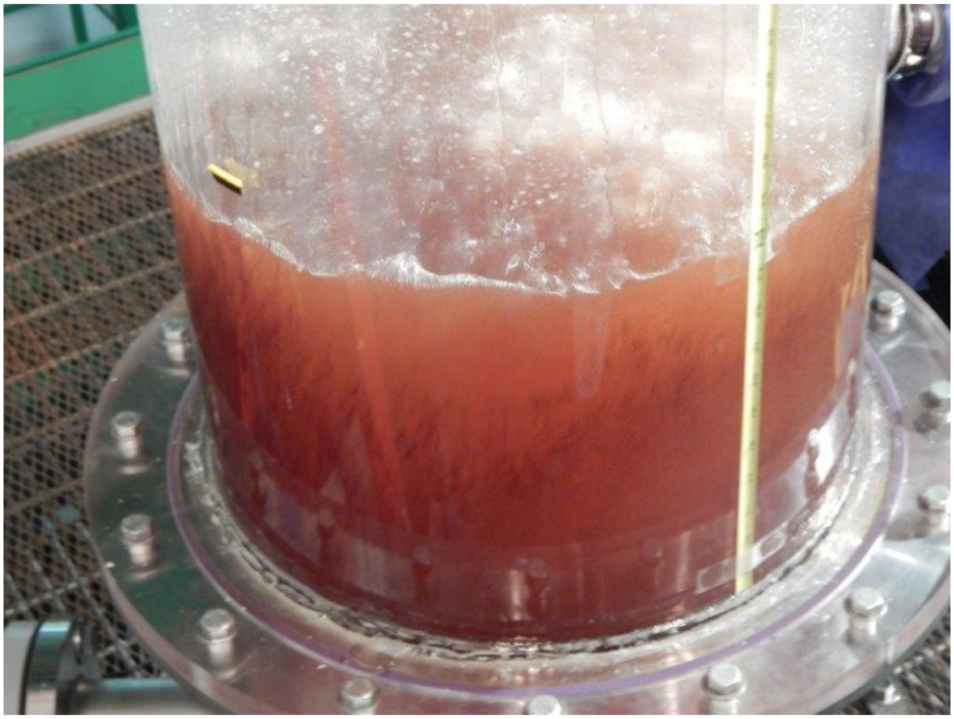


Figure 45. Test IXC18-2 – Note the streaks of resin as the drain violently stirs up the contents.

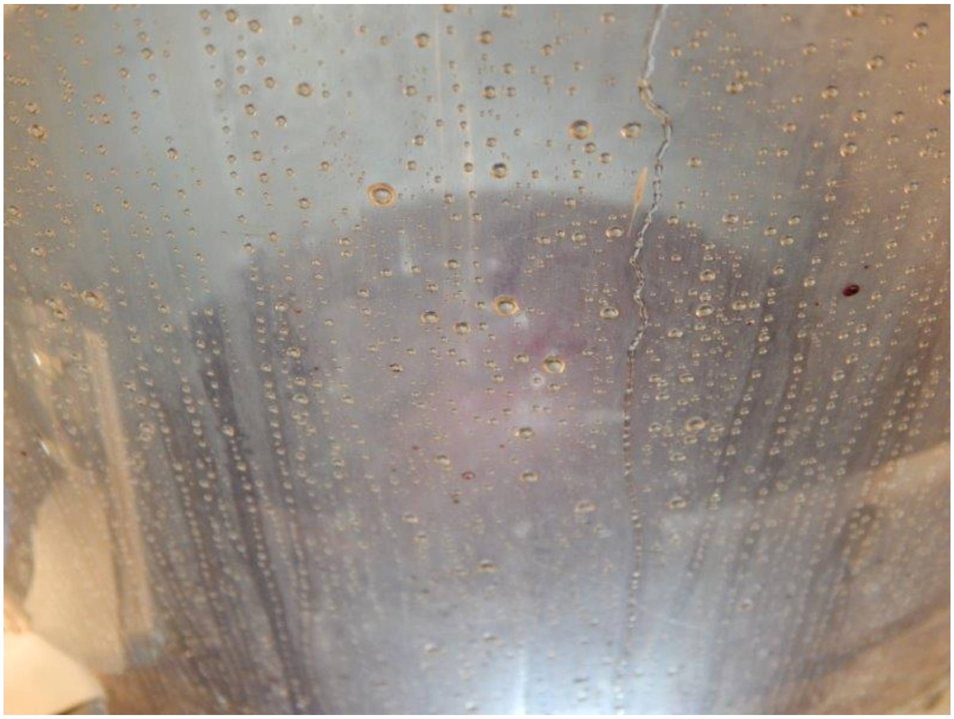


Figure 46. Test IXC18-2 – With the drain complete, the distributor flow continues for a few minutes, but the resin screen is already visible.

Figure 47. Test IXC18-2 – Only trace resin sits in the bottom of the drain pipe.

Test IXC18-3: 1-inch Drain Valve with No Distributor Flow During Resin Drain

As in Test IXC18-1, after the IXC was filled to the appropriate height of resin and liquid, Figure 48, the 1-inch valve was opened over a 10 second period, Figure 49, and from the start of the countdown to the completion of the resin and supernatant draining; however, this time the duration was 5.6 minutes, or 5.5 times slower than with the 3-inch valve, Figure 50 and Figure 51. After completion, there was a heel of approximately 15 inches on the IXC wall directly across from the drain port, which was approximately 3 inches above the flange of the IXC metal bottom, Figure 52. Once again, the resin surface had a gentle slope downwards until about the center of the IXC. From the center to the drain port, the slope of the resin was precipitous with the appearance of an avalanche. The 15-inch height opposite the drain valve would correspond to an angle of repose of approximately 40 degrees.

Roughly there was about 8 gallons of resin remaining, Figure 53, which would be about 15% of 53 gallons of starting resin volume. With less than 5 minutes of follow-up distributor flow (less than 15 gallons), only trace resin was left on the resin screen.

Figure 48. Test IXC18-3 – IXC set with 53 gal of resin and 53 gal of freeboard supernatant.

Figure 49. Test IXC18-3 – Open drain valve over a 10-second duration.

Figure 50. Test IXC18-3 – Much slower flow through 1-inch drain valve.

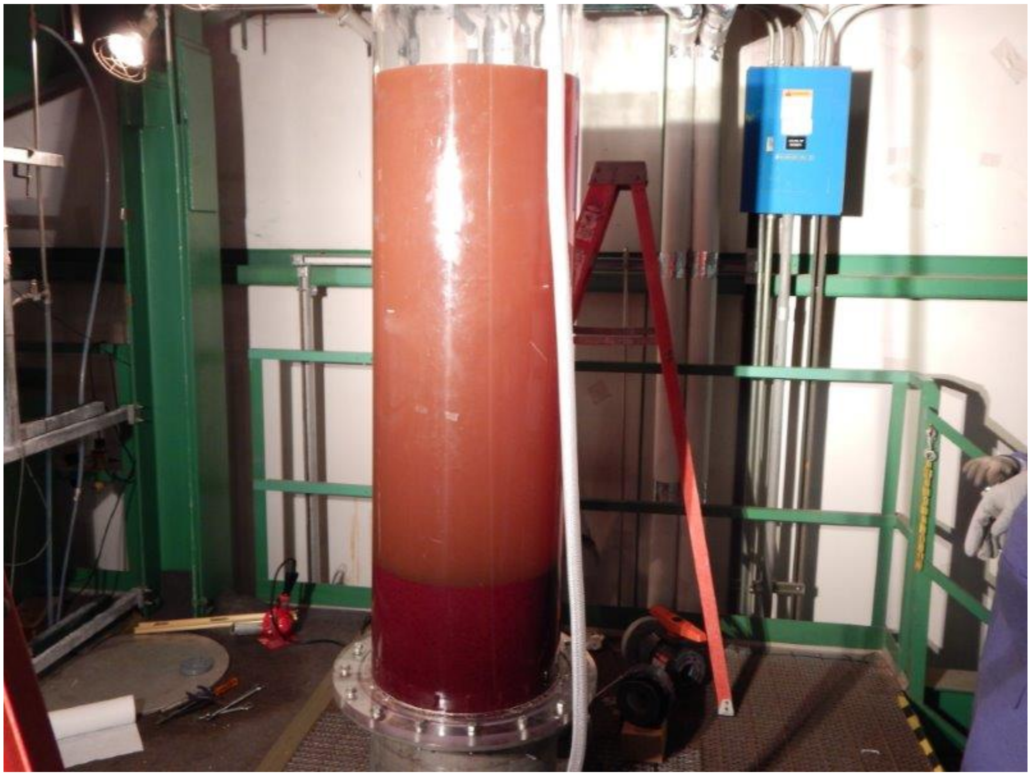


Figure 51. Test IXC18-3 – Resin is about half drained.

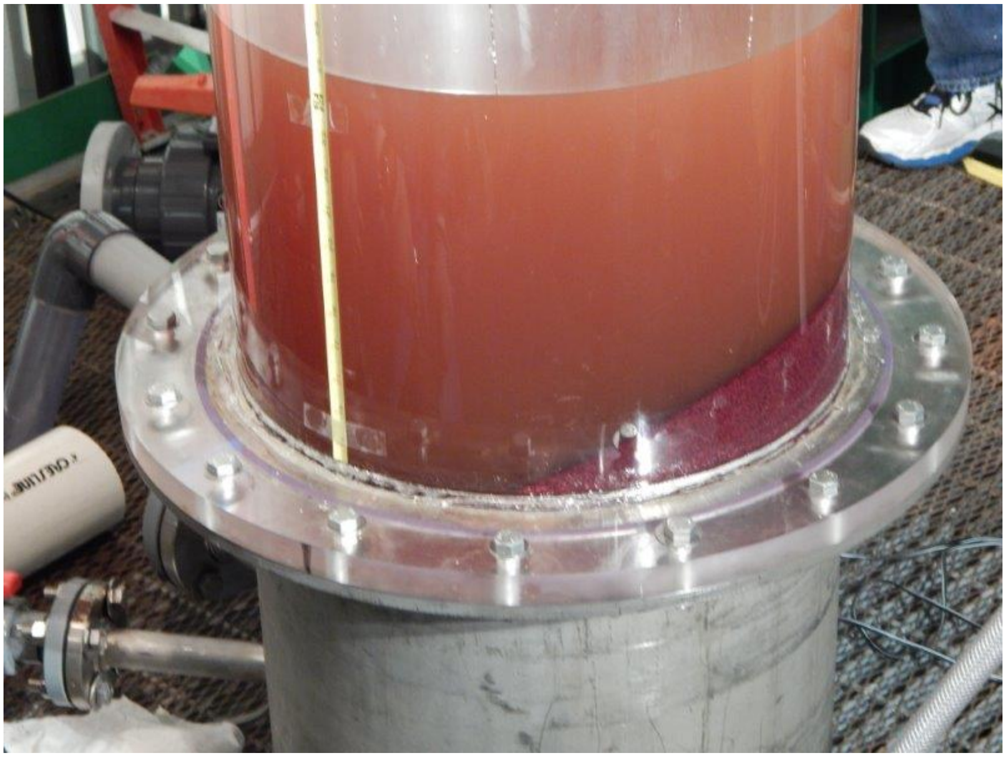


Figure 52. Test IXC18-3 – Resin heel forming.

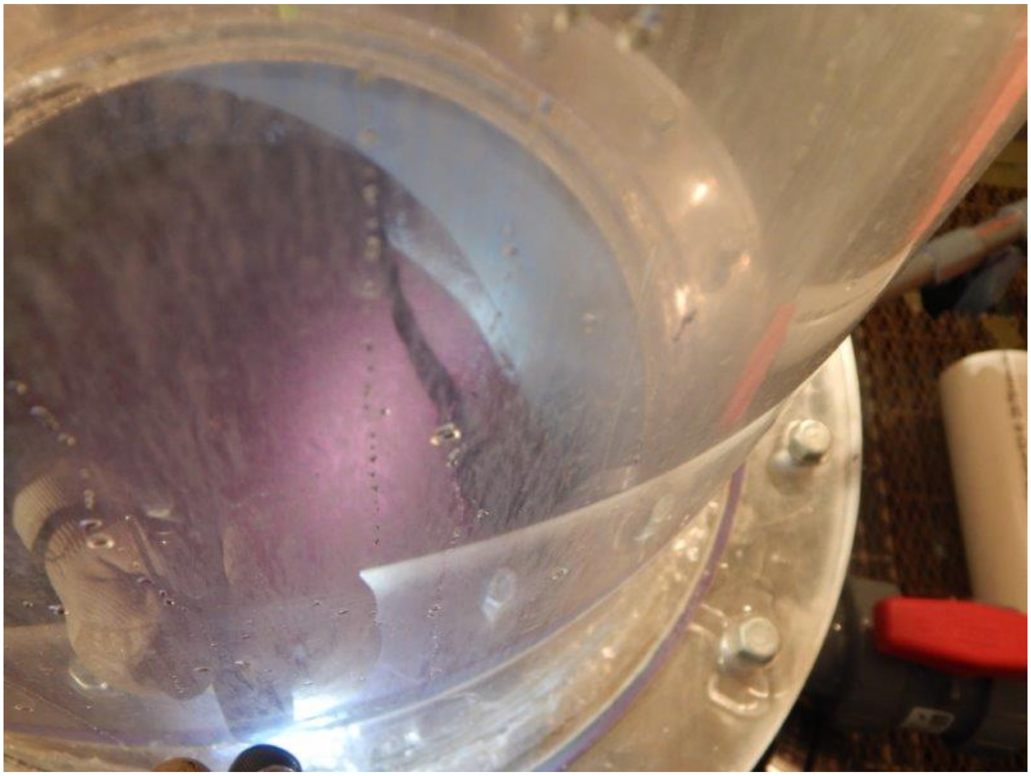


Figure 53. Test IXC18-3 – Drain complete. Viewing resin heel from above.

Test IXC18-4: 1-inch Drain Valve Distributor Flow During Resin Drain at ~ 4 BV/h

As in Test IXC18-2, the distributor flow was first initiated at approximately 4 BV/h and then the drain valve was opened over a 10 second period. From the start of the countdown to the completion of the resin and supernatant draining took 6.4 minutes seconds. Because of the continued distributor flow, the liquid level was higher as the drain progressed and no resin heel accumulated at the completion of the drain. The distributor liquid was allowed to flow for a few minutes (less than 15 gallons) after all the IXC contents exited to help carry the remaining resin in the drain system to flush to the receipt tank.

After terminating the test, only trace resin was left on the resin screen.

3.2.4 Test Solution – PEP – Hanford Waste Simulant – 2, 3, 8, and 9 September 2015

Test IXC18- 5: 1-inch Drain Valve with No Distributor Flow During Resin Drain

Starting with a resin height of 55.1 inches and a total height of 100.8 inches, the valve was opened over a 10 second period, Figure 54, and from the start of the countdown to the completion of the resin and supernatant draining, the duration was 5.8 minutes, which was similar to the 5.6 minutes for the 1-inch valve with IW without distributor flow. Both show the drain rate to be about 5 minutes longer than using the 3-inch valve. Once again, the resin surface had a gentle slope downwards until about the center of the IXC. From the center to the drain port, the slope of the resin was precipitous with the appearance of an avalanche, Figure 55. The 15-inch height opposite the drain valve would correspond to an angle of repose of approximately 40 degrees.

Roughly, there was about 8 gallons of resin remaining, which would be about 15% of 53 gallons of starting resin volume. With about 2 minutes of follow-up distributor flow (less than 6 gallons), only trace resin was left on the resin screen, Figure 56.

Figure 54. Test IXC18-5 – Starting test with a 10-second opening duration for the 1-inch valve.

Figure 55. Test IXC18-5 – Heel remaining after draining though the 1-inch valve.

Figure 56. Test IXC18-5 – Only trace resin on resin screen after ~2 minutes of 4 BV/h distributor flow.

Test IXC18-6: 1-inch Drain Valve Distributor Flow During Resin Drain at ~ 4 BV/h

The test was started at a resin height of 54.9 inches and a column height of 100.8 inches. As for the IW tests, with distributor flow, the flow was first initiated at approximately 4 BV/h and then the drain valve was opened over a 10 second period. From the start of the countdown to the completion of the resin and supernatant draining took 6.8 minutes. Figure 57 shows the resin starting height to a few inches above the target level. However, the drain rate was similar to the IW test with the 1-inch valve of 6.4 minutes. Because of the continued distributor flow the liquid level was higher as the drain progressed and no resin heel accumulated at the completion of the drain, see Figure 58. The distributor liquid was allowed to flow for about 2 minutes (less than 6 gallons) after all the major IXC contents exited only trace resin was left on the resin screen, see Figure 59.

Figure 57. Test IXC18-65 – Starting test with a 10-second opening duration for the 1-inch valve with distributor flow turned on.

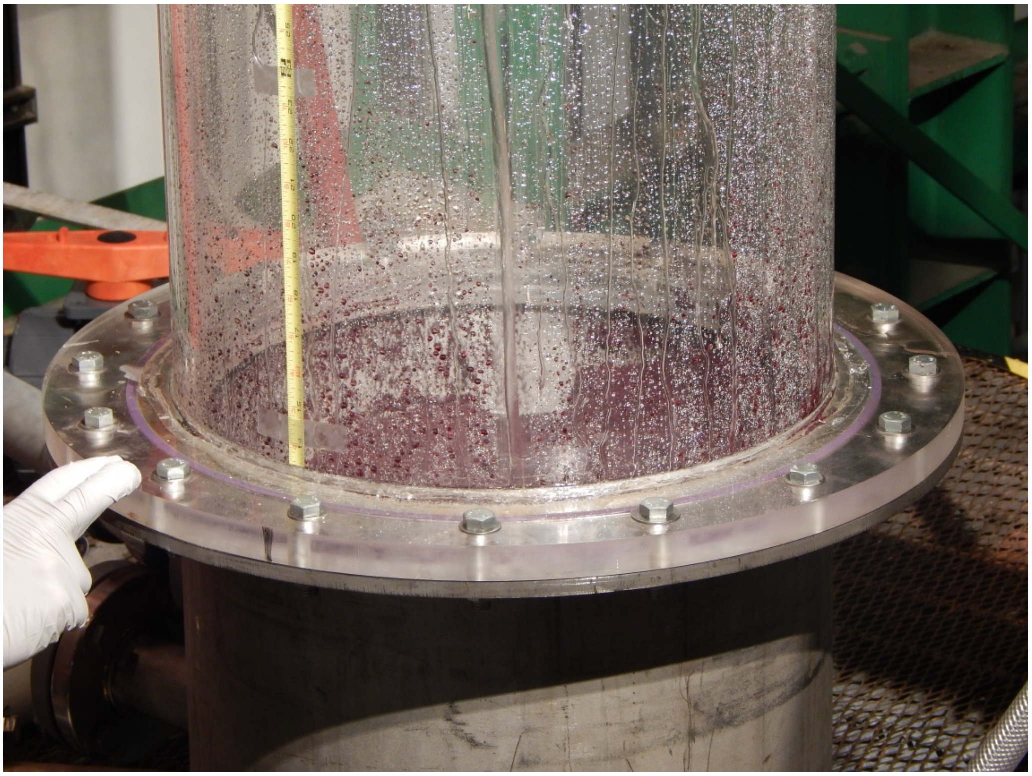


Figure 58. Test IXC18-6 – The continuous distributor effectively removes the resin and a heel does not develop.

Figure 59. Test IXC18-6 – Only trace resin remains.

Test IXC18-7: 3-inch Drain Valve with No Distributor Flow During Resin Drain – No Vent

This test started with a resin height of 50.9 inches and a column height of 100.8 inches. Unfortunately, the vent line for this test was accidently left closed, which was only noticed after starting the test due to the slow drain. It was allowed to continue and the total time was 7.9 minutes to drain. Gas exchange occurred through the drain valve allowing the resin slurry to drain. The inlet of the gas bubbles resulted in significant turbulence in the IXC. The column drained well and the resin heel was surprisingly small due to the high agitation from the gas exchange fluidizing the resin and helped to minimize the heel. However, because of not knowing the exact starting parameters, the test was repeated on the following day.

Test IXC18-8: 3-inch Drain Valve with No Distributor Flow During Resin Drain

The initial filling of the column with resin resulted in too much resin, so the IXC was drained and refilled. The starting resin height was 51.1 inches and the column height was 100.8 inches. The valve was opened over a 10 second period and from the start of the countdown to the completion of the resin and supernatant draining the duration was 1.2 minute. This was a repeat from the previous day, but the column was properly vented. The results are very similar to the IW results under the same conditions.

Test IXC18-9: 3-inch Drain Valve with No Distributor Flow During Resin Drain – No Vent

This test was the first purposely-performed non-vent test. The resin height was set at 50.1 inches and the column height was set at 100.8 inches. The drain valve was opened over a 10 second period and from the start of the countdown to the completion of the resin and supernatant draining the duration was 6.8 minutes. While this test took longer to drain, the heel was approximately half the heel of the vented test. That is, approximately 2 gallons of resin remained, or less than 4% of the starting resin volume. The high agitation of the rising bubbles from the drain port effectively mixed the column contents, especially when the level reach the bottom third of the column height. The small heel was easily flushed out with about 3 gallons of liquid.

Test IXC18-10: 3-inch Drain Valve with No Distributor Flow During Resin Drain

This test repeated the vented drain in Test IXC18-8 for this day because of the importance of a vented drain with a 3-inch valve. The drain time was to be verified. The resin height was set at 51.1 inches and the column height was set at 100.8 inches. The valve was opened over a 10 second period and from the start of the countdown to the completion of the resin and supernatant draining the duration was 0.9 minute, which is very similar to the 1.2 minutes, obtained from Test IXC18-8. Approximately 8% of the resin remained as a heel and it was effectively flushed out with approximately 6 gallons of liquid, leaving only trace resin on the resin screen.

Test IXC18-11: 2-inch Drain Valve with No Distributor Flow During Resin Drain

The resin height was set at 50.0 inches, Figure 60, and the column height was set at 100.8 inches. The valve was opened over a 10 second period and from the start of the countdown to the completion of the resin and supernatant draining the duration was 1.8 minutes. This was approximately double the time for the 3-inch valve (Tests IXC18-8, and -10), but one third the time for the 1-inch valve (Test IXC18-5). The heel, Figure 61, was slightly larger than for the 3-inch valve drain. It was approximately 10% of the starting resin, but it was still effectively flushed out with approximately 6 gallons of liquid, leaving only trace resin, Figure 62, on the resin screen.

Figure 60. Test IXC18-11 – Starting test with 2-inch drain valve with PEP simulant and a vented column.

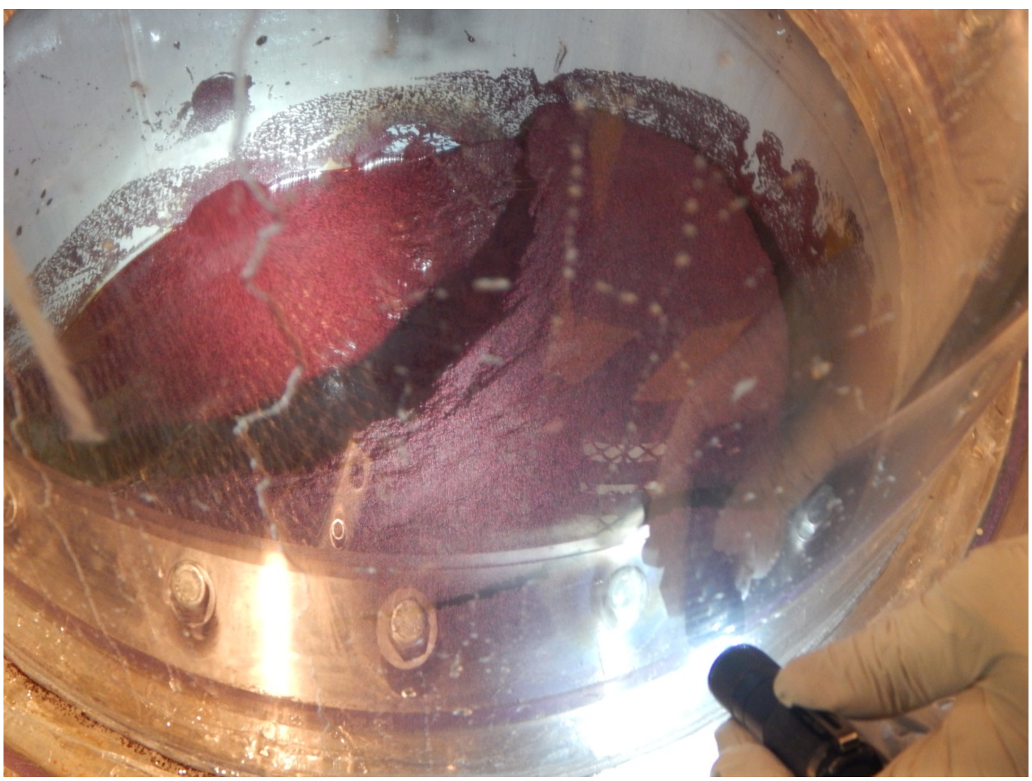


Figure 61. Test IXC18-11 – Remaining resin heel.

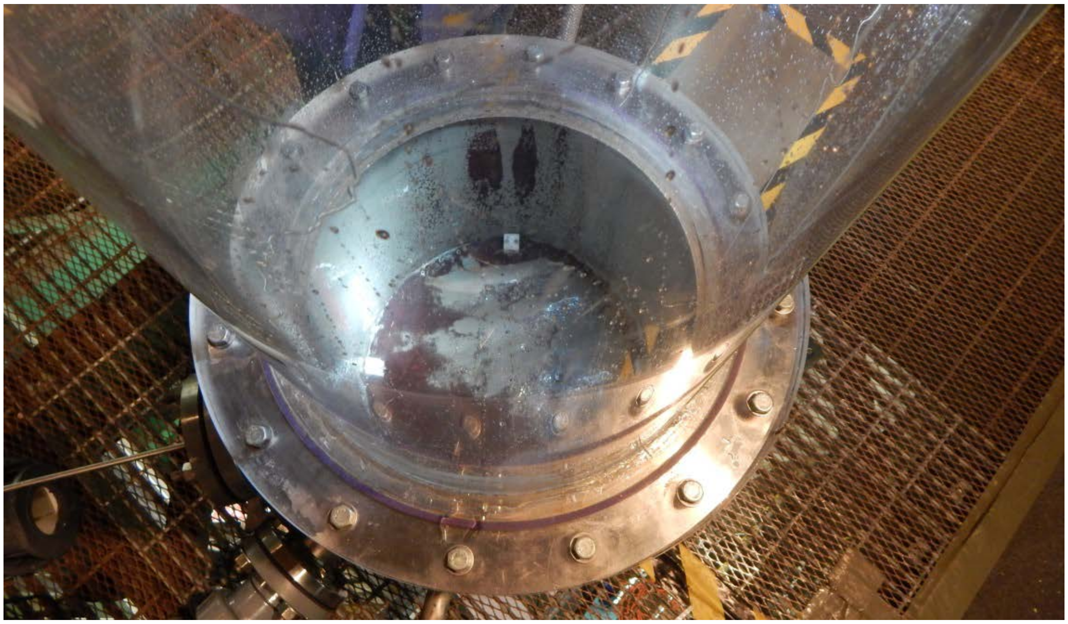


Figure 62. Test IXC18-11 – After ~6 gallons of flushing only trace resin remains.

Test IXC18-12: 2-inch Drain Valve with No Distributor Flow During Resin Drain – No Vent

This test was to determine if the 2-inch drain valve could effectively drain a column that was not vented. In filling the column, the resin initial height was well beyond the target of 50.4 inches. However, this time the drain valve was opened to adjust the height instead of simply draining the contents and starting over. The resin height was then adjusted to 50.4 inches and the column height was set at 100.8 inches. The drain valve was opened over a 10 second period. As occurred with the 3-inch no vent test, the resin level immediately dropped to approximately 78.5 inches, Figure 63, as the vacuum above the column just matched the weight of the liquid, which stopped the flow. However, for the 3-inch column the flow slowed, but it never stopped. For this test, the flow of resin basically stopped. There were drops coming out of the drain, but this stayed stable for more than 20 minutes. It was clear if it did drain, it may be many hours, if not a day to drain. Therefore, the vent was then opened which allowed the drain to continue.

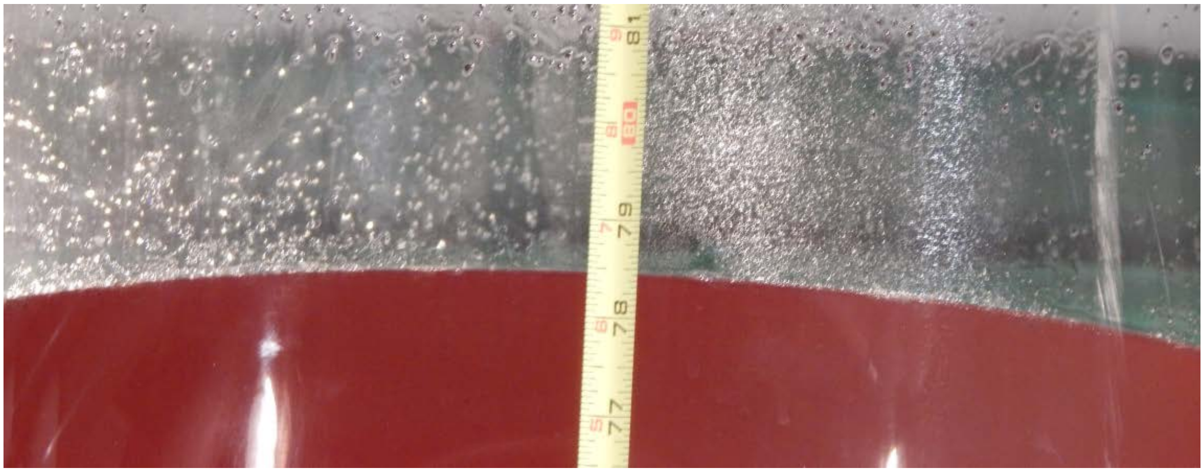


Figure 63. Test IXC18-12 – Draining stopped vacuum matched the weight of the column contents.

Test IXC18-13: 25/100 - 3-inch Drain Valve with No Distributor Flow During Resin Drain

This test was not originally planned, but on talking with the customer there was a suggestion based on the first letter report [Duignan, 2015b] that more of the resin heel could be removed during a vented drain if there were simply more freeboard liquid above the resin bed. That is, if there were more liquid it could better flush out the heel. Therefore, this test was included and is referred to as the 25/100 test. The “25” refers to the bed height, which was reduced from 1 BV = 53 gallons (for the 17.6-inch column = 50.4 inches to ½ BV, or ~25 inches). The “100” refers to the liquid height, which was kept at 100.8 inches, but this mean the liquid freeboard height would be now 1½ BV. The column was not designed to simply increase the liquid height above the target value of 100.8 inches, therefore, the resin bed was lower, which effectively added approximately an extra 2 feet of liquid above the resin bed. The resin height was set at 24.9 inches and the column height was set at 100.8 inches, Figure 64. As for all other tests, the valve was opened over a 10 second period and from the start of the countdown to the completion of the resin and supernatant draining the duration was 0.9 minutes, which is approximately equal to all vented 3-inch valve, e.g., Tests IXC-8 and -10. The heel appeared to be the same as all other 3-inch drain valve tests, so there was no improvement. However, this clearly showed that once the heel is formed, the extra liquid had no significant flushing capacity. It is interesting to note that even with only ½ BV of resin the heel was approximately the same as for a full BV of resin, about 8% of the full BV.

Figure 64. Test IXC18-13 – Starting the 25/100 test.

3.2.5 Test Solution – PEP – Hanford Waste Simulant + Precipitation

Test IXC18-14: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Vented Column: Non-Homogeneous Mixture of Resin and Precipitants

For this test it was necessary to add material to the simulant to induce precipitation that would create solids that could possibly hinder the draining of the column of resin. There was considerable discussion of what to use and how to add it to the column. It was clear the precipitated solids should be both in the liquid and within the resin bed. The precipitating agent decided upon was a 32.8 wt% solution of $Al(NO_3)_3 \cdot 9H_2O$ and it was added at to the 5 M Na PEP simulant at a concentration of 8.7 wt%. To minimize the risk of precipitated solids plugging all the feed systems, i.e., liquid and resin, the resin was first introduced and set to near the target height, which turned out to be 50.6 inches. Approximately 2 inches of PEP simulant sat above the resin, then ~6 gal of the precipitation agent were introduced, Figure 65. The intent then was to fill the column with PEP in upflow, which would fluidize the bed and allow the introduced liquid to circulate throughout the column, Figure 66. However, the precipitation of solids was so fast and produced such a large amount of floating solids that it seemed the column would not drain at all, Figure 67 and Figure 68. Due to this distribution of precipitants the test is referred to as the non-homogeneous precipitation test. After filling the column to the full height of 100.8 inches, the 3-inch drain valve was opened over a 10 second period. Surprisingly, from the start of the countdown to the completion of the resin and supernatant draining, the duration was only 0.9 minute. Figure 69 gives an idea on how fast the drain occurred. However, there was a very large heel remaining, Figure 70. Most of the IXC contents drained to the receipt tank, Figure 71, but a very "hard" heel remained in the column. It took approximately 40 minutes with the flow of the PEP liquid impinging directly onto the heel to erode the heel and flush it out. The precipitating agent appeared to adhere to clumps of resin forming brick-like solids that were difficult to break down, even by hand.

Figure 65. Test IXC18-14 – Introducing precipitation agent into IXC

Figure 66. Test IXC18-14 – Upflowing the IXC contents to fluidize. Precipitants are floating and in the bed.

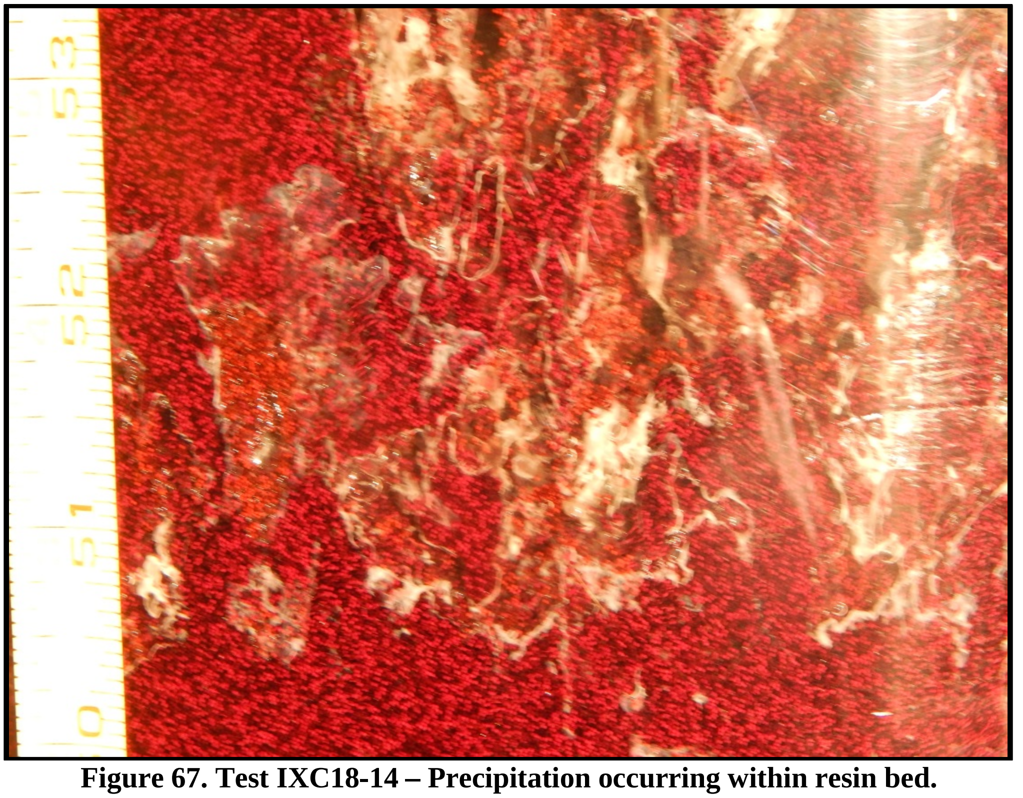


Figure 67. Test IXC18-14 – Precipitation occurring within resin bed.

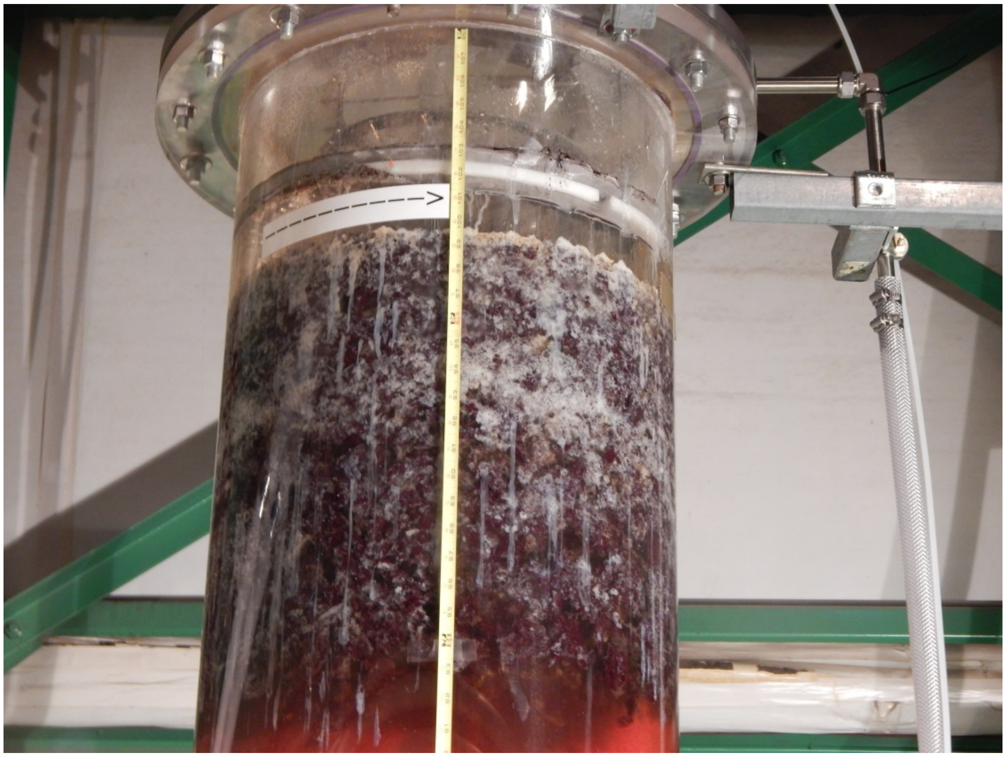
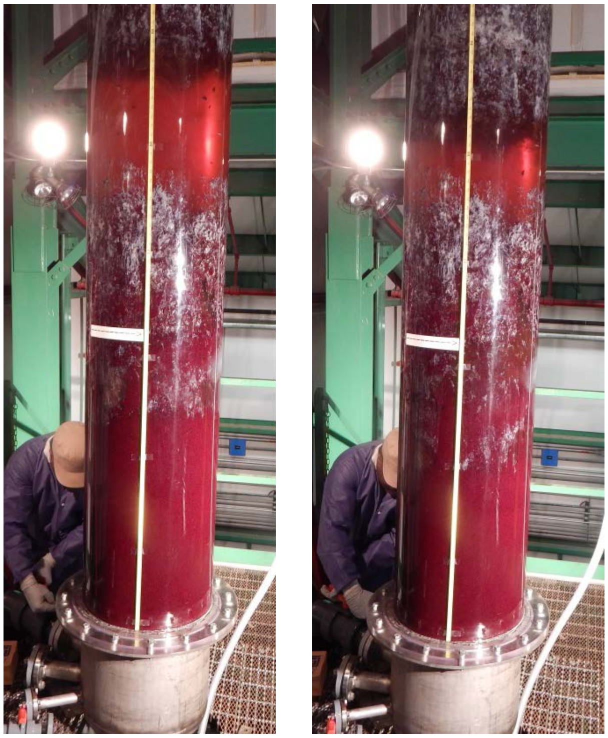


Figure 68. Test IXC18-14 – IXC almost full and ready to start test.



(a) (b)

Figure 69. Test IXC18-14 – (a) Starting to open 3-inch drain valve and (b) valve fully opened within 10 seconds. Notice how fast solids drop.

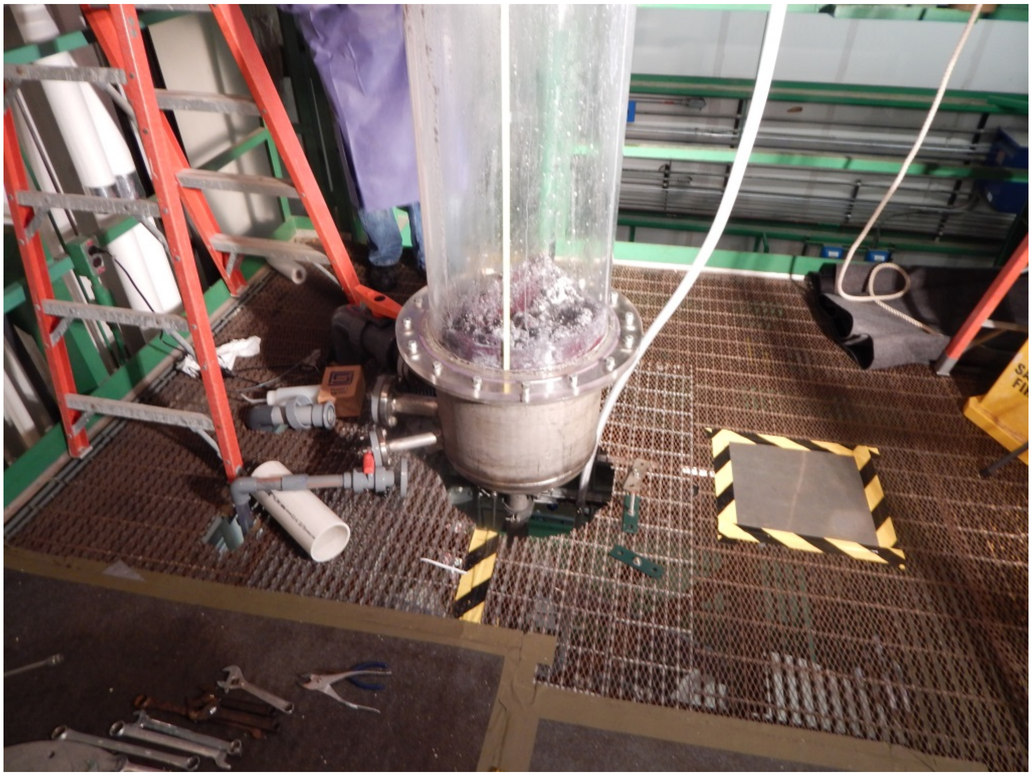


Figure 70. Test IXC18-14 – Drain complete within 1 minute, but a large heel remains.

Figure 71. Test IXC18-14 – Resin, PEP, and precipitated solids enter the receipt tank.

Test IXC18-15: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Vented Column: Homogeneous Mixture of Resin and Precipitants

After all the resin was removed from the IXC and incorporated into the receipt tank, the contents were well mixed. After a while the appearance was as if nothing was added to the PEP. That is, it appeared that all the large clumps of resin were broken down and the precipitants were well distributed through resin-PEP mixture. At this point, the precipitation drain test was repeated and called the Homogeneous test. The column was filled with resin, but initial resin height was higher than target resin height. Therefore, the 3-inch valve was opened slightly to reduce the resin, but this time the resin did not drain. After opening the valve a little, nothing came out and after opening the valve 100%, still nothing came out. Even after opening and closing the valve three times, no draining occurred. This was never seen before. The valve was then closed and upflow was initiated for a few seconds. Fortunately, when the drain valve was opened again, the resin began to drain. While the column of resin appeared, Figure 72, as it always did before the precipitation test, Test IXC18-14, there did seem to be slight differences in the resin behavior.

Figure 72. Test IXC18-15 – Start of 3-inch drain valve test with a homogenous mixture of PEP+ Precipitated solids.

The resin height was set at 50.7 inches and the column height was set at 100.8 inches. After opening the valve during a 10-second period, the column was fully drained in 0.9 minute, which is the same as all other 3-inch valve drains with a vented column. The remaining resin heel was very similar to all the heels left after a 3-inch drain without precipitation. The heel may have been slightly smaller but the exact difference is not known. This may have occurred because the resin appeared to flow slightly smoother after adding the precipitating agent. The appearance was more of a shiny plastic flow stream. It could have been more visco-elastic, shear thinning, which would explain why upflowing the resin mixture allowed it to flow more smoothly. However, rheological measurements were not made on the slurry.

3.2.6 Test Solution – 0.5 M HNO_3 – Elution Solution – 22 and 23 September 2015

The remaining 7 tests were done with 0.5 M HNO_3, the resin elution solution, and using the 2- and 3-inch drain valve, with and without venting the IXC. There were no surprises here and it appeared that the acid+resin drains the same as both the IW+resin and PEP+resin, and leaves approximately the same amount of resin heel. All the photographs look exactly the same too, so they will not be repeated in this section. However, the big difference between the acid tests and the caustic tests is the non-vented drain tests. The series of test begin with a simple vented drain through the 3-inch valve, with and without distributor flow, which are then followed by a several non-vented tests. The first one was done by venting the column to compare to the Test IXC18-9, but then tests were done with an applied vacuum in the gas space above the column. This was done in order to better represent the actual IXC where it is expected that there will not be a large gas gap above the column liquid. This means that when the drain valve is opened the vacuum that is created between the liquid-metal surface at the top of the column should be equal to the weight of the liquid contents of the column above the drain valve, divided by the applied surface area. This is basically the “thumb over the straw” scenario. As calculated in Note (5) of Table 5, the vacuum necessary to just equal the weight of either a column of acid or a column of caustic is estimated. Non-vent tests were done at three pressures after the column vents were sealed: atmospheric (gas will expand until vacuum equals column weight), a vacuum close to the weight of the column, and a vacuum slightly above the weight of the column. Those tests results are elaborated below.

Also, not discussed here is the fact that before the acid tests the resin had to be converted to H-form. Because of the addition of the precipitation agent, the procedure to convert the acid is not elaborated here. However, it entailed first washing the resin with deionized water to help remove most of the PEP simulant and dropping the pH to approximately pH=12. This was followed with ~2 BV of 1 M HNO_3 that was allowed to soak for overnight for at least 12 hours. The following day the acid was drained and replaced with ~3 BV of 0.5 M HNO_3 to begin the test. The pH of that final mixture was a stable pH~0; therefore, the resin was then in H-form.

Before testing with acid began, the IXC was marked with new target resin height, see Figure 73. The volume of the resin decreased, which is expected after the elution, but was not known a priori. However, it normally shrinks on the order of 20% to 25% and a number used from past tests [Duignan et al., 2008] was 24%; therefore, the 53 gallons of Na-form resin was expected to shrink to ~40 gallons, for a drop in resin height of approximately 12 inches from the Na-form height. It was exactly 38.3 inches.

Figure 73. Test IXC18-16 – First test with 0.5 M HNO_3 with the reduced volume of H-form resin.

Test IXC18-16: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Vented Column

Test began with the resin height set at 38.8 inches and the column height at 100.8 inches. After opening the drain valve over a 10 second period and from the start of the countdown to the completion the drain took 0.9 minute, which is the same as the time for IW and PEP test under the same conditions. The remaining heel and the amount of flush liquid were the same as previous tests.

Test IXC18-17: 3-inch Drain Valve with Continuous Distributor Flow of ~4 BV/h & Vented Column

The test began with the resin height set at 38.7 inches and the column height at 100.8 inches. After opening the drain valve over a 10 second period and from the start of the countdown to the completion the drain took 0.9 minute. The continuous flow was able to flush out all of the resin, except for a trace amount.

Test IXC18-18: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Non-Vented Column

Test began with the resin height set at 38.9 inches and the column height at 100.8 inches. All three vent paths were closed, but no vacuum was applied. After opening the drain valve over a 10 second period, the start of the countdown to the completion of the drain took 6.0 minutes. Initially the column contents dropped from 100.8 inches to ~83 inches while the above-column gas expanded to create a vacuum to hold up the column. After the draining paused, at the 83-inch mark, it slowly continued to drain as air bubbles rose from the drain port. The high agitation at the end of the column drain helped to flush out some of the heel, leaving less than half the heel of a vented drain. Approximately 3 gallons of flush liquid removed the remaining heel.

Test IXC18-19: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Non-Vented Column with an Applied Vacuum of 10 inches of Hg.

Test began with the resin height set at 38.4 inches and the column height at 100.8 inches. All three vent paths were closed, but a vacuum of 10 inches of Hg was applied. After opening the drain valve over a 10 second period, the start of the countdown to the completion of the drain took ~52 minutes. Initially the column contents did not drop and it took approximately 20 minutes for resin to drop 2 inches. After approximately 40 minutes the resin was still at ~34.5 inches. After 50 minutes, the resin dropped to ~32.5 inches, but that seemed to be an important height because the contents became much more agitated, as more air was entering from the valve. During the remaining 2 minutes of the drain, the remaining resin bed was suspended and the column appeared to be uniformly mixed. The remaining heel was very small, less than 2% of the starting volume. It was easily flushed out with a few gallons of test liquid.

Test IXC18-20: 3-inch Drain Valve with No Distributor Flow During Resin Drain & Non-Vented Column with an Applied Vacuum of 7.5 inches of Hg.

Test began with the resin height set at 37.5 inches and the column height at 100.8 inches. All three vent paths were closed, but a vacuum of 7.5 inches of Hg was applied. After opening the drain valve over a 10 second period, the start of the countdown to the completion of the drain took ~33 minutes. This drain acted the same as the previous drain with vacuum, but faster because of the lower vacuum strength. The key observation from this test was the resin level of ~32.5 inches is an important hydraulic limit for the scale IXC. As soon as the resin dropped to that height the drain rate increased, more air entered, and the entire resin bed was fluidized and well mixed as it drained out. The remaining heel was still very small, but slightly larger than the previous test, approximately 4% of the starting volume.

The last two tests were performed with the 2-inch drain valve to measure the difference to the 3-inch drain valve

Test IXC18-21: 2-inch Drain Valve with No Distributor Flow During Resin Drain & with a Vented Column.

Test began with the resin height set at 38.3 inches and the column height at 100.8 inches. After opening the drain valve over a 10 second period and from the start of the countdown to the completion, the drain took ~1.6 minutes. The result is similar to the test with PEP, Test IXC18-11, where the drain time was 1.8 minutes. The slightly faster time may be due to the shorter resin bed of the less dense acid solution. Everything else was the same as Test IXC18-11, indicating the different solution does not have a significant effect.

Test IXC18-22: 2-inch Drain Valve with No Distributor Flow During Resin Drain & Non-Vented Column with an Applied Vacuum of 7.5 inches of Hg.

Test began with the resin height set at 38.6 inches and the column height at 100.8 inches. All three vent paths were closed, but a vacuum of 7.5 inches of Hg was applied. After opening the drain valve over a 10-second period the column did not drain similar to what occurred for Test IXC18-12. There was an initial small drop in resin height to 33.6 inches, but then it remained at that height for 30 minutes without change. In fact, there was a very small amount of liquid draining, but no visible resin. It appeared as if the resin was acting like a filter. The vent was then opened and the contents were allowed to drain.

3.2.7 Summary 18-inch IXC Test Results

Table 5 shows the results for all 22 tests performed with the 18-inch IXC drain test.

Table 5. Summary 18-inch Inside Diameter Ion Exchange Column Test Results

Test IXC18-	Daily Test	Test Date	Valve inch	Test Liquid	Name	Duration min (1)	Comment
1	1	31-Aug-15	3	0.01 M NaOH	Drain with no distributor flow	1.0	Distributor flow of ~4 BV/h used after drain to remove heel.
2	2	31-Aug-15	3	0.01 M NaOH	Drain with continuous distributor flow	1.1	Distributor flow of ~4 BV/h
3	3	31-Aug-15	1	0.01 M NaOH	Drain with no distributor flow	5.6	Distributor flow of ~4 BV/h used after drain to remove heel.
4	4	31-Aug-15	1	0.01 M NaOH	Drain with continuous distributor flow	6.4	Distributor flow of ~4 BV/h
5	1	2-Sep-15	1	PEP-5 M Na	Drain with no distributor flow	5.8	Distributor flow of ~4 BV/h used after drain to remove heel.
6	2	2-Sep-15	1	PEP-5 M Na	Drain with continuous distributor flow	6.8	Distributor flow of ~4 BV/h
7	3	2-Sep-15	3	PEP-5 M Na	No Vent -Drain with no distributor flow	7.9	No vent was not intended, but allowed to continue once started test.
8	1	3-Sep-15	3	PEP-5 M Na	Drain with no distributor flow	1.2	Distributor flow of ~4 BV/h used after drain to remove heel.
9	2	3-Sep-15	3	PEP-5 M Na	No Vent -Drain with no distributor flow	6.8	Column contents dropped from 100.8" to ~81" when gas vacuum = weight of contents. (2, 3)
10	3	3-Sep-15	3	PEP-5 M Na	Drain with no distributor flow	0.9	Distributor flow of ~4 BV/h used after drain to remove heel.
11	4	3-Sep-15	2	PEP-5 M Na	Drain with no distributor flow	1.8	Distributor flow of ~4 BV/h used after drain to remove heel.
12	1	8-Sep-15	2	PEP-5 M Na	No Vent -Drain with no distributor flow	NA	After initial drop in IXC height, when the gas vacuum equaled the IXC weight, the draining stopped. After ~18 min of no draining the vent was opened. Total
13	1	9-Sep-15	3	PEP-5 M Na	25-inch resin - Drain with no distributor flow	0.9	1/2 BV used but full 2 BV Column Contents
14	2	9-Sep-15	3	PEP+$AlNO_3$	Precipitation - Drain with no distributor flow	0.9	Non-Homogeneous - Large heel remained
15	3	9-Sep-15	3	PEP+$AlNO_3$	Precipitation - Drain with no distributor flow	0.9	Homogeneous
16	1	22-Sep-15	3	0.5 M HNO_3	Drain with no distributor flow	0.9	Distributor flow of ~4 BV/h used after drain to remove heel.
17	2	22-Sep-15	3	0.5 M HNO_3	Drain with continuous distributor flow	0.9	Distributor flow of ~4 BV/h
18	3	22-Sep-15	3	0.5 M HNO_3	No Vent -Drain with no distributor flow	6.0	Column contents dropped from 100.8" to ~83" when gas vacuum = weight of contents. (2, 4)
19	1	23-Sep-15	3	0.5 M HNO_3	No Vent -Drain with no distributor flow	51.8	10 in. Hg vacuum - IXC contents did not drop after opening 3-inch drain valve. (5)
20	2	23-Sep-15	3	0.5 M HNO_3	No Vent -Drain with no distributor flow	33.0	7.5 in. Hg vacuum - IXC contents did not drop after opening 3-inch drain valve. (5)
21	3	23-Sep-15	2	0.5 M HNO_3	Drain with no distributor flow	1.6	Distributor flow of ~4 BV/h used after drain to remove heel.
22	4	23-Sep-15	2	0.5 M HNO_3	No Vent -Drain with no distributor flow	NA	7.5 in. Hg vacuum - IXC contents did not drop after opening 3-inch drain valve. (5) After 33 minutes & no change in resin height, the small vent was opened. The total drain time was 50.1 minutes.

Notes to Table 5

Nomenclature: IW - Inhibited Water = 0.01 M NaOH, PEP = Hanford Simulant: 5 M Na, Acid = Elution Liquid: 0.5 M HNO_3, and NA = Not Applicable.

(1) Time uncertainty of ±0.2 min is principally based on the end of the drain cycle because it was difficult at times to perceive when the drain completed.
(2) If the column contents did not initially drop after the drain valve was opened then the vacuum above the contents was strong enough to prevent that drop to occur. In the cases when the contents did initially drop, while the vents were closed, this meant that the gas space above the column, which was initially at atmospheric pressure, had to expand for the pressure of the gas to reduce just enough to balance the remaining weight of the column contents.
(3) When the column contents first stopped draining, the height was ~ 81 inches. With the PEP density = 1.23 g/cm^3 then the weight of the liquid in the unvented column was [81" - 3" (valve port)] (2.54 cm/in) (1.23 g/cm^3) x (29.53 inches of Hg / 1019.72 g/cm^2) = 7.1 inches of Hg vacuum.
(4) When the column contents first stopped draining, the height was ~ 83 inches. With the acid density = 1.024 g/cm^3 then the weight of the liquid in the unvented column was [83" - 3" (valve port)] (2.54 cm/in) (1.024 g/cm^3) (29.53 inches of Hg / 1019.72 g/cm^2) = 6.0 inches of Hg vacuum.
(5) To prevent the column contents from immediately exiting the IXC on opening, the drain valve, the vacuum above the column needs to, at least, match the weight of the full column. For a full column of 0.5 M HNO_3, when using a 3-inch valve, is [100.8" - 3" (valve port)] (2.54 cm/in) (1.024 g/cm^3) (29.53 inches of Hg / 1019.72 g/cm^2) = 7.4 inches of Hg vacuum. For a full column of PEP, when using a 3-inch valve, the equalization is [100.8" - 3" (valve port)] (2.54 cm/in) (1.23 g/cm^3) (29.53 inches of Hg / 1019.72 g/cm^2) = 8.8 inches of Hg vacuum.

3.2.8 Summary of 3-Inch Drain Valve Times – All Solutions

From the data shown in Figure 74, it appears that the rate to drain sRF resin through a 3-inch valve is independent of the condition of the resin, be it in Na-form, H-form, being mixed with precipitants, or submerged in solutions of water, 5 M Na Hanford waste, or 0.5 M nitric acid. Within the uncertainty of the time measurement, which is on the order of the size of the symbols in the graph, it appears the only important factor is the size of the drain port through which the resin drains. These results are limited to an IXC that is fully vented. In the event the column vent is closed, the results are different, as discussed in the following section.

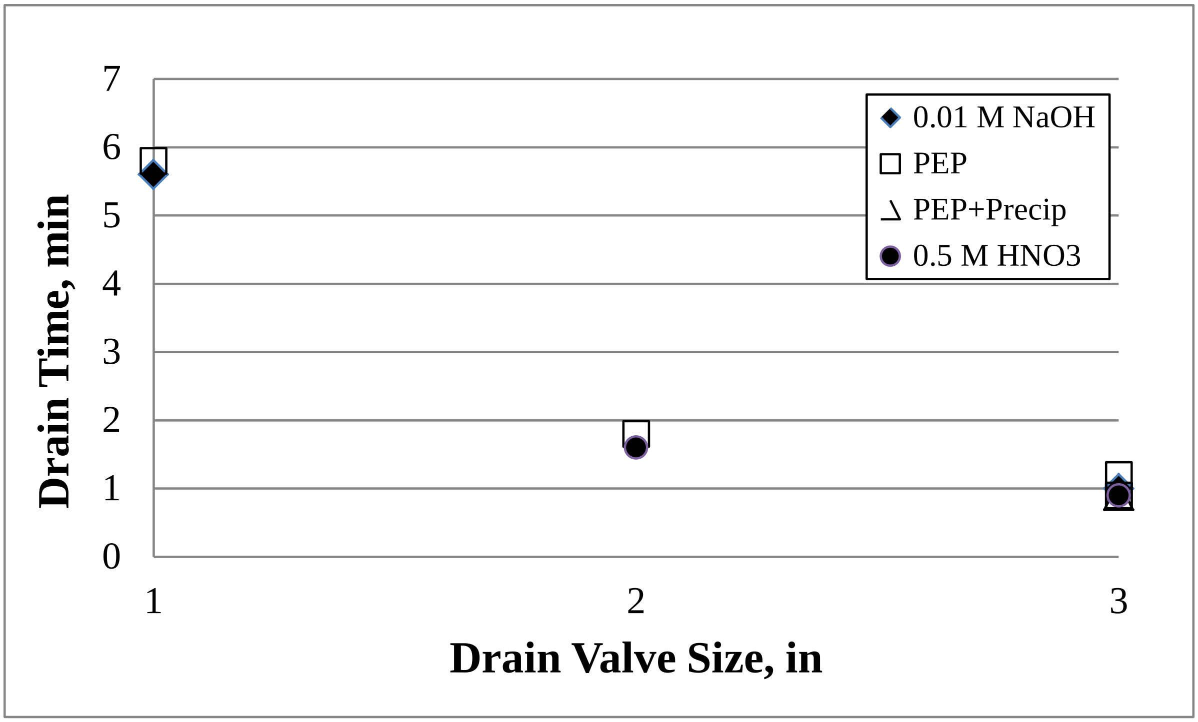


Figure 74. 18-Inch IXC drain times of sRF through various valve sizes and a vented column.

3.2.9 Summary of No Vent Drain Valve Times – All Drain Valves and Solutions

In the event of the IXC that is not vented, the times to drain will depend on the valve size and the strength of the vacuum above the column. The important feature seen in Figure 75 is that for a 3-inch drain valve the contents of the column always drained and this appears to be independent on whether the surrounding liquid is the processing waste stream or the elution acid solution. Furthermore, because of the high agitation during the draining process as rising gas violently stirs the contents of the column, which occurs at approximately the lower third of the column, most of the resin heel is washed out. The remaining heel is at least one-half that of a vented drain and is roughly less than 5% of the initial resin contents. Resin did not drain through the 2-inch valve; therefore, the 1-inch drain valve was not included in the no-vent drain tests.

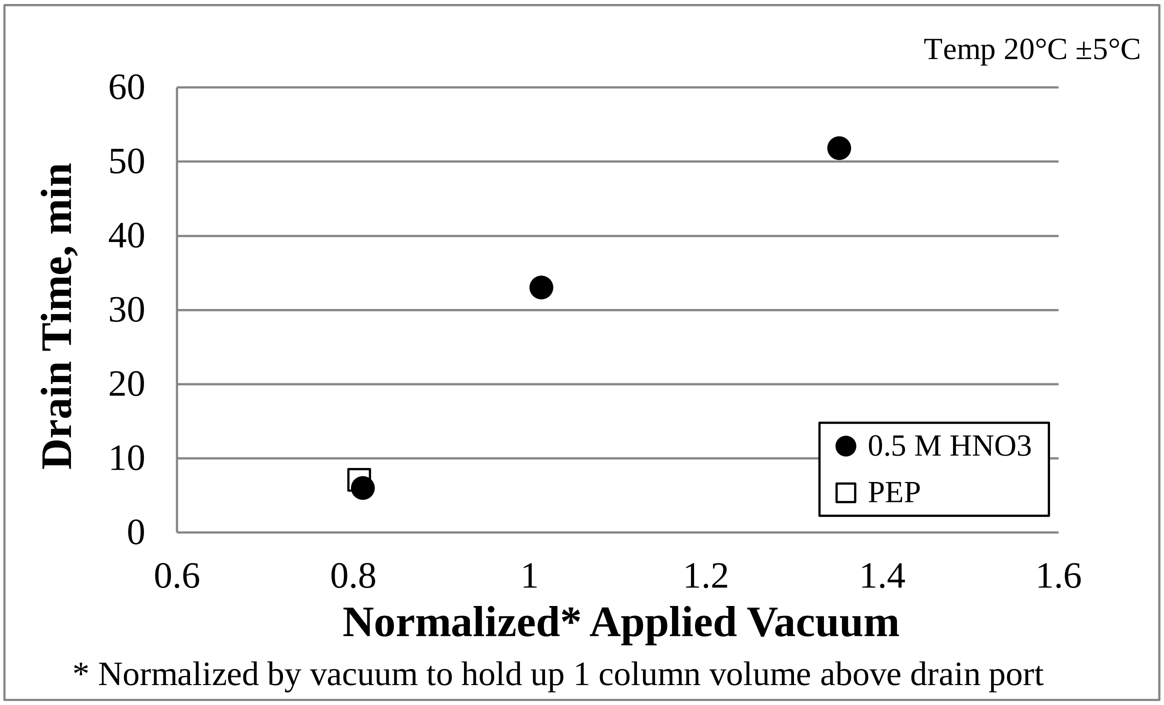


Figure 75. 18-Inch IXC drain times of sRF through a 3-inch valve and unvented column

3.3 Full-Scale Drain Tests: Effect of Scale

Before the tests with the PEP test solution, the approximate 200 gallons of new sRF resin in H-form was conditioned to be in Na-form. Normally, conditioning is performed in the actual column but a specific bed volume was required, i.e., 50.4-inch IXC height (or 281 gallons = 1 BV) in Na-form. Because several tests were required, needing to refill the IXC multiple times, the resin was conditioned separately in the 8-foot diameter receipt tank and then was introduced into the IXC to prepare for each test.

3.3.1 Conditioning Spherical RF Resin

Before the tests began, the resin was conditioned by changing it to Na-form with the 5 M Na PEP simulant. (Note that this was new resin received from WRPS specifically for this test.) Samples were taken after converting to Na-form (Figure 76) and the measured diameter was approximately 460 microns, indicating it was fully converted. In converting the resin to Na-form, the concentration of caustic used was 1.0 M NaOH instead of the traditional concentration of 0.5 M NaOH. This higher concentration was allowed in order to use less solution and, therefore, less waste. The mixture was soaked over a weekend. After the conversion, the solution was replaced with the caustic simulant to begin the tests.

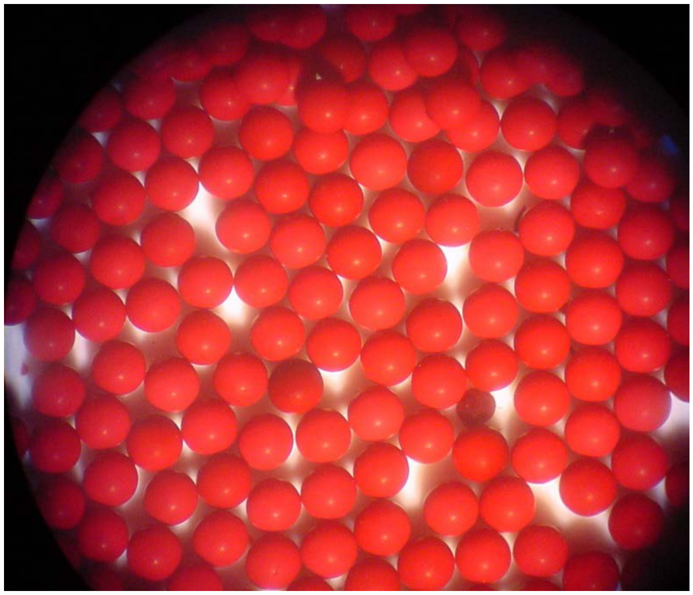


Figure 76. Sodium-form of new resin after conversion with sizes of approximately 460 microns. The color is much more uniform than the used resin of the 18-inch column test[14].

3.3.2 Overall IXC40 Tests

As shown in Table 6, in chronological order, a total of 6 tests were performed using 2 different size drain valves, i.e., 3-inch and 4-inch. Two different test solutions were used, i.e., process water[15] and PEP (Hanford salt simulant, 5 M Na). The results from the 18-inch IXC tests showed that the other solutions used, i.e., inhibited water and 0.5 M HNO_3 did not demonstrate different resin drain results from the PEP solution [Duignan, 2015c]; therefore, this larger scale test was limited to the PEP test solution. However, water was initially used to shakedown the test equipment.

Table 6. Resin drain tests performed with 40.5-inch inside diameter IXC

Test IXC40-	Test Date	Valve (in)	Test Liquid	IXC Pressure (1)
1	20-Oct-15	4	Water	0 inches Hg
2	20-Oct-15	4	Water	- 5.5 inches Hg
3	21-Oct-15	4	PEP-5 M Na	0 inches Hg
4	21-Oct-15	4	PEP-5 M Na	- 8 inches Hg
5	23-Oct-15	3	PEP-5 M Na	0 inches Hg
6	23-Oct-15	3	PEP-5 M Na	- 8 inches Hg
(1) Air pressure difference from atmospheric above IXC contents				

The following sections describe each of the 6 tests. The photographs included are only a few of the key moments.

[14] The total column volume of resin in Na-form was approximately 307 gallons. After conversion the volume shrank to 239 gallons, which means that it reduced by (307-239) / [(307+239)/2] x 100% = 24.9% as expected [Duignan et al. 2008].
[15] Process water is the well water supplied the laboratory building for general purpose use.

3.3.3 Test Solution - Water – 20 October 2015

Test IXC40-1: 4-inch Drain Valve with a Vented IXC

Because this test was only water with no resin, it was performed to check for leaks and the operation of the 40-inch IXC. There were several leaks that were fixed after the shakedown tests. Furthermore, it was important to know how much protection was needed to prevent simulant and resin from splashing, or being ejected, out of the receipt tank. However, it was also important to be able to see the draining material to understand the dynamics of the process. Several tests were performed, with the only difference being the amount of splash protection used. Figure 77 shows the liquid jet as it exits the elbow and expands outward as it entrained air. The elbow was then covered with a modified carboy as the principal splash guard, but it, and the tank, were also covered with a plastic tarp, Figure 78, which contained the splashing while allowing observation during a drain.

Figure 77. Test IXC40-1 – 4-inch valve test with water to determine time and splash potential during vented drain test.

The receipt tank used for the water tests was much smaller than the 8-foot diameter receipt tank because the larger tank contained the resin, which was being conditioned. Therefore, for the water test the full IXC contents could not be drained. However, for the vented test, approximately a third of the IXC was drained in ~20 seconds, which means the total drain time would be close to 1 minute for the ~560 gallons of test solution.

Figure 78. 4-inch discharge elbow was covered with a modified carboy and a plastic tarp to contain splashing and is shown here during one of the resin tests.

Test IXC40-2: 4-inch Drain Valve with a Non-Vented IXC

A non-vented test was done with water to determine how well the IXC would sustain the needed vacuum. The test was also performed to observe the gas-liquid interaction during the drain. The IXC was filled with water to 105 inches, which was just above the target liquid height of 101.8 inches. Then the vent pathways were closed. The vacuum pump was connected to the principal vent line and the vacuum was slowly increased. The target was at least 8 inches of Hg, because at least that amount was necessary to prevent the test simulant from dropping, once the drain valve was opened. That is, a lower vacuum would allow the contents to drop as the air space above the liquid in the column expanded just enough to balance the weight of the IXC contents. Fortunately, the applied vacuum did increase, thus indicating a sealed column. Unfortunately, when the vacuum reached ~5.5 inches of Hg one of the 8 bolts, which secured the top of the IXC to the top column flange, caused the flange to crack due to bending stress, Figure 79.

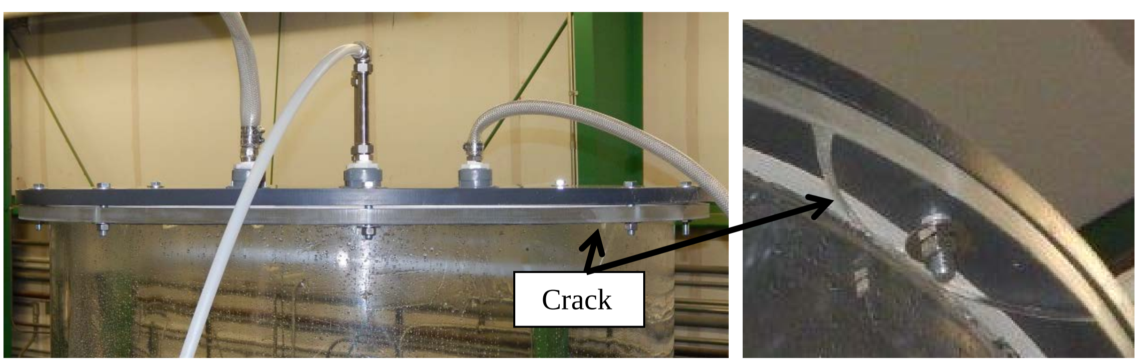


Figure 79, IXC top with crack in flange.

Despite the crack, the seal still held the vacuum and it was then increased to 6 inches of Hg. However, it was thought best to not risk further damage; therefore, the vacuum was reduced to 5.5 inches of Hg to perform the non-vented test[16]. The valve was opened and as expected the water level immediately

[16] After the test with water, all 8 flange bolts were loosened to minimize the bending stress they transmitted during the bowing of the top lid, as a result of the vacuum. The PVC lid was calculated to bow ~0.5 inches at 10 inches Hg.

dropped from 105 inches to 96 inches to sustain the weight of the liquid. Roughly, a vacuum of 7 inches of Hg would be needed to prevent water from dropping. Once the 96-inch level was reached, the water level stopped dropping and allowed small and large bubbles to enter the 4-inch drain valve port, Figure 80. The water also allowed one to see the air bubbles entering the valve port from the drain valve, Figure 81.

Figure 80. 4-inch valve test with water to observe how IXC operated under vacuum of 5.5 inches of Hg.

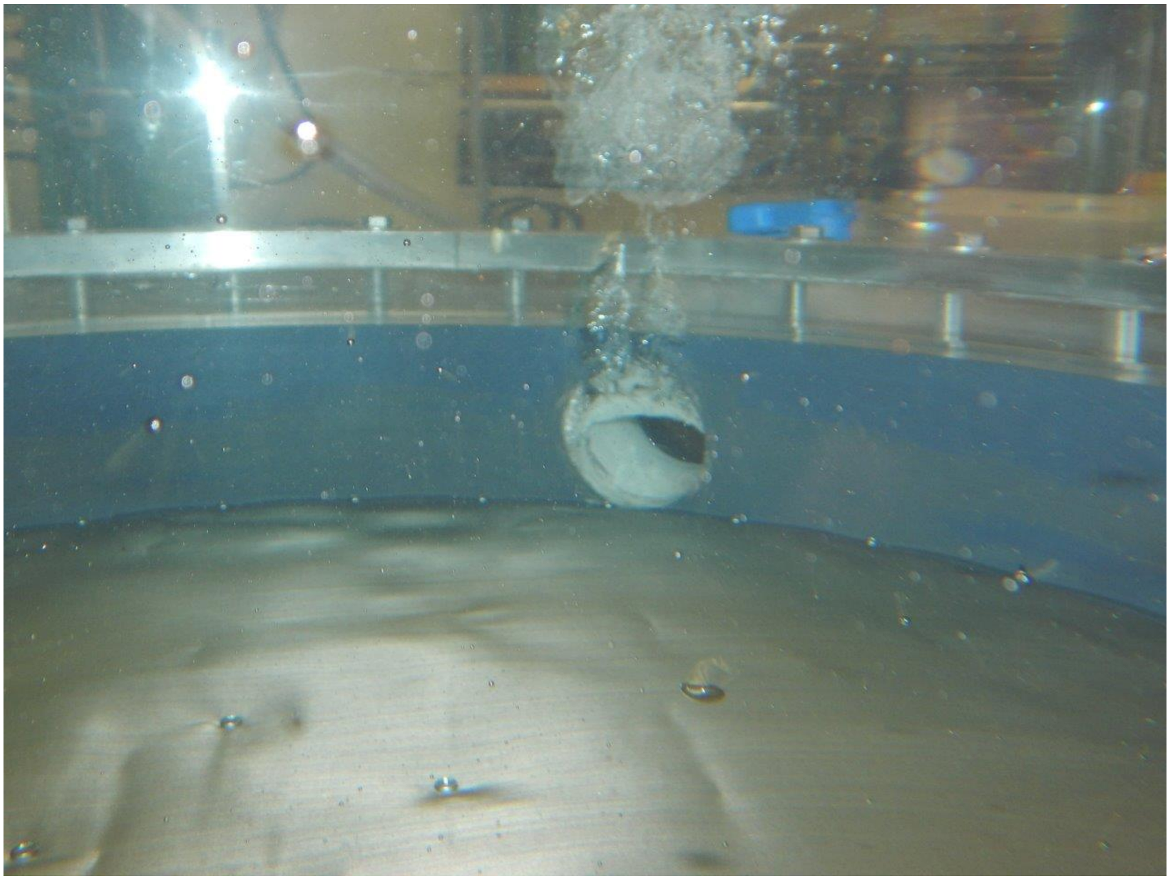


Figure 81. Observation on inside of 4-inch valve during draining of water with 5.5 inches of Hg.

3.3.4 Test Solution – PEP – Hanford Waste Simulant – 4-inch Valve – 21 October 2015

Test IXC40-3: Vented IXC Drain with the 4-inch Valve

Starting with a resin height of 55 inches and a total height of 100.8 inches, the 4-inch drain valve was opened just enough to trim the resin height to 50.4 inches, Figure 82. However, the top of the resin surface became slightly wavy because of draining action. Subsequently, the test began by opening the drain valve over a 10-second period. The contents of the IXC drained smoothly and took approximately 2.4 minutes, Figure 83.

The surface of the resin heel that remained had many different slopes, Figure 84. On the IXC wall, opposite of the drain valve, the heel was its highest at about 23 inches above the resin screen, but it dropped very fast. At the midpoint, the resin on the wall was approximately 9 inches above the screen. The very rough terraced surface had the appearance of a stadium or a precipitous mud slide.

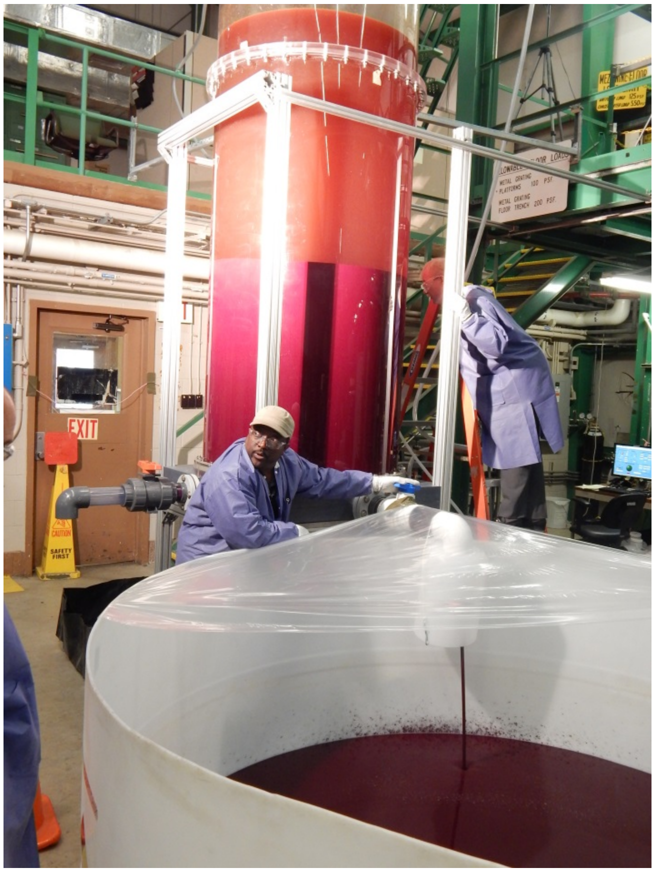


Figure 82. Adjusting the resin height.

Figure 83. Test IXC40-3 was started with a 10-second opening duration for the 4-inch valve. Note, the liquid level in this photograph already reached the top of the drain valve port.

(a) (b)

Figure 84. The remaining heel contained approximately 10%, ~28 gallons of the starting resin volume. Shown is the heel from a (a) top view and (b) a view from the drain port.

A rough estimate of the heel volume is that there was about 28 gallons of resin remaining, which would be about 10% of 280 gallons of starting resin volume. Subsequently, the IXC overhead distributor flow of feed solution was initiated at 12.5 gpm for 2 minutes. What remained was a heel of approximately 6 gallons of resin, or about 2% of the resin BV. This rinse was followed with another 1 minute of 12.5 gpm of distributor flow. The heel then reduced to approximately 4 gallons of resin, or 1.5% of the resin BV. After a final 1-minute of 12.5 gpm of distributor flow, the heel volume did not significantly reduce. The resulting flat surface just directed the liquid flow directly out of the drain port, Figure 85.

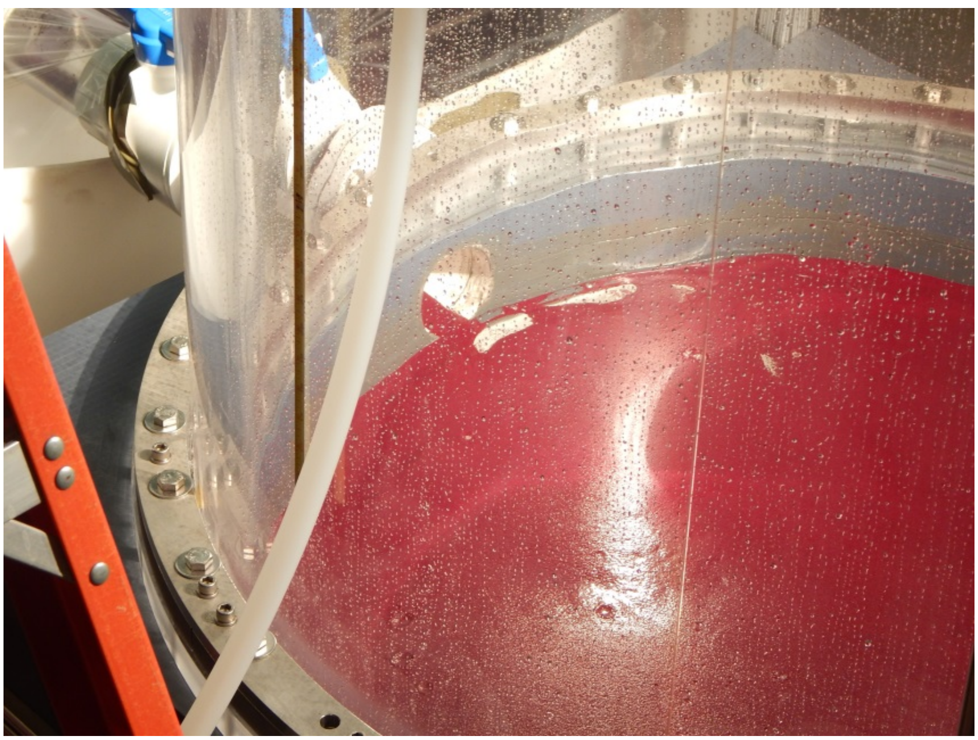


Figure 85. Remaining resin heel of ~4 gallons spread over entire large surface of resin screen preventing further resin to be easily removed.

Test IXC40-4: Non-Vented IXC Drain with the 4-inch Valve

After filling the IXC with 49.5 inches of resin, the bed was fluidized and after settling the height was 53.3 inches, Figure 86. At this point both the customer and principal investigator decided not to adjust the resin height to 50.4 inches because:

1. The 18-inch IXC drain tests demonstrated that resin bed heights varying several inches did not have a significant impact on drain times or the resin heels left.
2. The action of lowering the bed height in Test IXC40-3 to the target height caused the resin surface to change from a level surface to a slightly wavy surface; therefore, the amount of resin was kept at the slightly higher level.

Figure 86. Test IXC40-4 was the non-vented 4-inch valve drain with resin at 53.3 inches.

The test began by applying a vacuum to the IXC. The target was at least 8 inches of Hg, but the vacuum was increased very slowly because of the flange crack experienced on the previous day. As already mentioned, the top lid bolts were slightly loosened to hopefully minimize the stress on the flange. The vacuum was applied over a 25-minute period. The vacuum held and no further damage to the IXC was experienced. However, the vacuum was stopped at 8 inches of Hg, Figure 87, to not risk further damage, and assuming it was sufficient to keep the contents from dropping once the drain valve was opened. The IXC was ready to drain.

Figure 87. Vacuum above IXC contents was set at 8 inches of Hg.

Over a 10-second period, the drain valve was opened. The contents in the column did not noticeably drop and the vacuum held steady at 8 inches of Hg. However, the contents did start to drain very slowly, Figure 88. After about 20 minutes, the liquid level in the tank reached the top of the 4-inch drain port, which increased the drain rate and the amount of air entering the drain, Figure 89. After about 28 minutes, the principal draining was complete, which left a resin heel that was completely different from the vented test of Test IXC40-3.

Figure 88. Immediately after opening the 4-inch drain valve the IXC contents drained slowly.

Figure 89. Drain rate increased as air entered the drain port, increasing agitation.

The heel was basically flat across its upper surface until it was close to the drain port, Figure 90. The remaining heel was about 8.9 inches above the screen on the IXC wall opposite of the drain port. Instead of the heel dropping fast to the drain port, as occurred in Test IXC40-3, it smoothly angled down towards the drain. At the midpoint (~20 inches), the resin height was still approximate 7.8 inches, which then dropped down to the zero near the drain port.

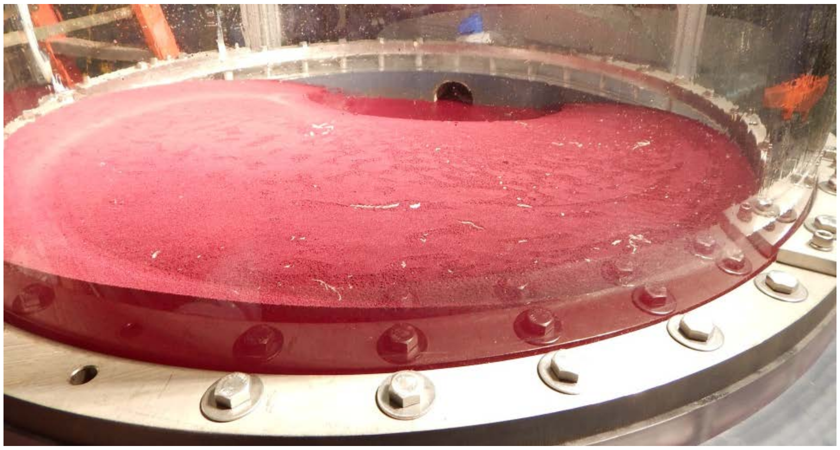


Figure 90. For Test IXC40-4 the remaining heel contain approximately 12%, ~34 gallons, of the starting resin volume.

Roughly, if it is assumed that the resin were redistributed to be level across the bottom of the column it would be approximately the height of the plastic bottom flange, which was approximately 6 inches above the resin screen. This would mean a heel volume = $(3.14/4)(40.5/12)^2(6/12)$ = 4.5 ft^3 (~ 33.5 gallons), which is about 12% of the starting resin bed volume of 281 gallons.

Surprisingly, this was more than the vented Test IXC40-3 and the opposite of the result elicited in the 18-inch IXC test. It appears the much wider surface area, that is, the longer distance from the drain port, prevented more resin from exiting. Furthermore, the level of agitation in the 40-inch IXC seemed to be less than in the 18-inch test. The larger scale probably affects movements of liquid and solids. That is, there is a larger flatter resin surface and a longer distance to move resin.

An initial distributor flow of feed was initiated at about 12.5 gpm for 1 minute. This rinse resulted in a heel that was about 4 inches above the resin screen on the IXC wall, opposite of the drain port, and sloped evenly down to the drain port. Assuming the average thickness across the resin plate is half the 4 inches, then the remaining heel volume was approximately = $(3.14/4)(40.5/12)^2((4 \text{ x } ½)/12)$ = 1.5 ft^3 (~ 11 gallons, or about 4% of the starting resin bed with using 12.5 gallons of liquid to rinse out the heel.

A second distributor flow of feed was initiated at about 12.5 gpm for 1 minute. This second rinse resulted in a remaining heel of about 2 inches above the resin screen on the opposite of the drain port and sloped evenly down to the drain port. Assuming the average thickness across the resin plate is half the 2 inches, then the remaining heel volume was approximately = $(3.14/4)(40.5/12)^2((2 \text{ x } ½)/12)$ = 0.75 ft^2 ~ 6 gallons, or about 2% of the starting resin bed with using 25 gallons of liquid to rinse out the heel.

A third distributor flow of feed was initiated at about 12.5 gpm for 1 minute. This third rinse resulted in a remaining heel of about 1.5 inches above the resin screen on the opposite of the drain port and sloped evenly down to the drain port. Assuming the average thickness across the resin plate is half the 1.5 inches, then the remaining heel volume was approximately = $(3.14/4)(40.5/12)^2((1.5 \text{ x } ½)/12)$ = 0.56 ft^3 (~ 4 gallons), or about 1.5% of the starting resin bed with using 38 gallons of liquid to rinse out the heel.

A fourth, and final, distributor flow of feed was initiated at about 12.5 gpm for 1 minute. However, the last rinse did not result in significantly more resin removal; therefore, no more flow was used. For this rinse the remaining heel was about 1.3 inches above the resin screen opposite of the drain port and sloped evenly down to the drain port. Assuming the average thickness across the resin plate is half the 1.3 inches, then the remaining heel volume is approximately = $(3.14/4)(40.5/12)^2((1.3 \text{ x } ½)/12)$ = 0.48 ft^3 (~ 3.6 gallons), or about 1.3% of the starting resin bed with using 50 gallons of liquid to rinse out the heel, Figure 91. Note that this volume is similar to the volume of 4 gallons obtained in Test IXC40-3 after its last rinse.

Figure 91. In Test IXC40-4 the final heel that remained was approximately ~1.3% (~3.6 gallons) of the starting BV after 4 rinses with feed flow raining down from the feed distributor.

3.3.5 Test Solution – PEP – Hanford Waste Simulant – 3-inch Valve – 23 October 2015

Test IXC40-5: Vented IXC Drain with the 3-inch Valve

After filling the IXC with resin and test solution to 100.8 inches, the settled resin height was 48.9 inches. The IXC resin was then fluidized and after the resin settled the resin bed height was 51.8 inches, Figure 92. No effort was made to trim the height to the target of 50.4 inches, as previously discussed. The test began by opening the 3-inch drain valve over a 10-second period. The contents of the IXC drained smoothly and took approximately 3.7 minutes, which was about 80 seconds longer than what resulted in the 4-inch drain valve test.

As in the non-vented 4-inch test, the surface of the resin heel had many different slopes, Figure 93. The remaining heel was approximately 11 inches high on the IXC wall opposite of the drain valve and had very irregular surfaces down to the drain port. If the resin were redistributed to be level across the bottom of the column, then an estimate of the level height is 6 inches, or 5.5 inches above the bottom of the bottom portion of the 3-inch drain valve, giving a remaining volume ~31 gallons of resin, or 11% of the resin BV.

Figure 92. Test IXC40-5 – 3-inch drain valve for a vented IXC.

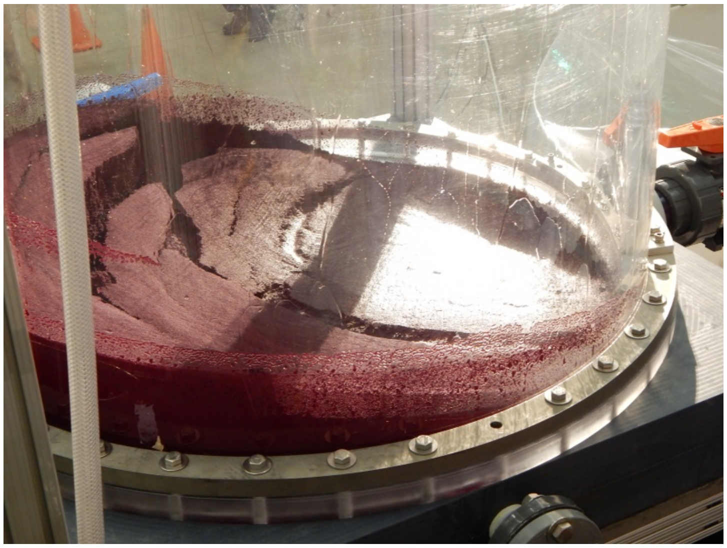


Figure 93. Remaining heel containing approximately 11%, ~31 gallons, of the starting resin volume.

To begin rinsing out the resin heel, an initial 1-minute distributor flow was directed to the heel at ~12.7 gpm, or ~2.7 BV/h. This action left a heel that was 3 inches high on the opposite IXC wall from the drain port. The heel sloped gently down to the drain being about 3 inches tall at the midpoint on one side and 2 inches on the other side and about 1.8 inches at the drain port. Using an average of 2.4 inches above the entire screen surface and taking into account that the 3-inch valve port was 0.5 inches above the screen surface, then the average was 1.9 inches, giving a remaining volume of 8.95 ft^2 x (1.9/12) x 7.48 gallons / ft^3 = 10.6 gallons, or approximately 10.6/281 ~ 3.8% of the resin BV.

A second 1-minute distributor flow of ~12.7 gpm, or ~2.7 BV/h resulted in a remaining heel that was 2 inches high on the opposite IXC wall to the drain port. The heel sloped gently down to the drain, but it was still about 2 inches in height at the midpoint on both sides and about 1.5 inches at the drain port. Using an average of 1.8 inches above the entire screen surface and taking into account the 3-inch valve port was 0.5 inches above the screen surface, then the average was 1.3 inches, giving a remaining volume of 8.95 ft^2 x (1.3/12) x 7.48 gallons / ft^3 = 7.2 gallons, or approximately 7.2/281 ~ 2.6% of the resin BV.

A third 1-minute distributor flow of ~12.7 gpm, or ~2.7 BV/h resulted in almost no change to the remaining heel of 2 inches high on the opposite IXC wall to the drain port. The heel sloped gently down to the drain, but it was still about 1.5 inches in height at the midpoint on both sides and about 1.5 inches at the drain port. Using an average of 1.5 inches above the entire screen surface and taking into account the 3-inch valve port was 0.5 inches above the screen surface, then the average was 1 inch, giving a remaining volume of 8.95 ft^2 x (1.0/12) x 7.48 gallons / ft^3 = 5.6 gallons, or approximately 5.6/281 ~ 2.0% of the resin BV.

A final 1-minute distributor flow of ~12.7 gpm, or ~2.7 BV/h resulted in little effect. It appeared that maybe ¼ inch of heel was further removed. If true, this would amount to another 8.95 ft^2 x (0.75/12) x 7.48 gallons / ft^3 = 4.2 gallons, or approximately 4.2/281 ~ 1.5% of the resin BV. As for the 4-inch drain test, it appeared the approximately 4 gallons of resin cannot be removed without an increased effort of removal, see Figure 94.

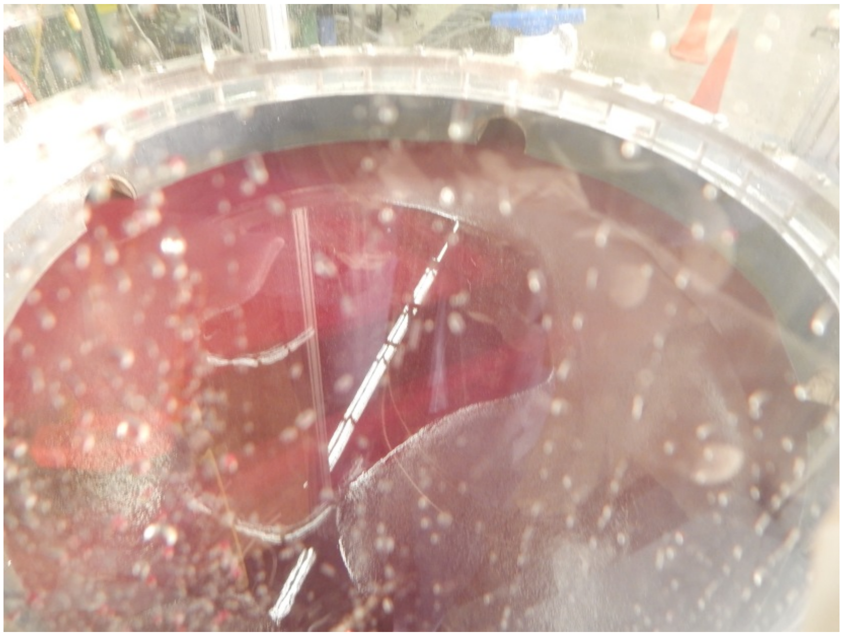


Figure 94. In Test IXC40-5, the remaining resin heel was ~4 gallons spread over entire large surface of resin screen preventing further resin to be easily removed.

Test IXC40-6: Non-Vented IXC Drain with the 3-inch Valve

The test preparation began by filling the IXC with 49.6 inches of resin. The resin bed was then fluidized and after settling the final height was 53.4 inches. This was followed by applying a vacuum to the IXC and the target was at least 8 inches of Hg, which was increased very slowly. This time an attempt was made to increase the vacuum to 8.5 inches Hg, but it appeared that 8 inches Hg was the equipment limit, so the test was done at that pressure again. Over a 10-second period, the drain valve was opened. The contents in the column did not noticeably drop and the vacuum held steady at 8 inches of Hg. However, the contents did start to drain immediately, but very slowly, as previously shown in Figure 88. This time the slow drain was different from the 4-inch non-vented drain in that the exiting resin-liquid stream appeared to contain less liquid. That is, after about 5-10 minutes of slow poring of wet resin, the resin stream appeared drier[17], Figure 95. This drier stream lasted at least 10 to 15 minutes. The draining was slow with intermittent air bubbles entering the drain port, Figure 96, but many bubbles remained trapped in the resin just above the valve opening. Furthermore, a cycle was set up in the resin bed and the slurry, which was apparent in the changing slope of the resin upper surface in the IXC. As draining progressed, the resin plane just above the drain port dropped as resin poured out of the valve, leaving the resin plane higher on the opposite side of the IXC. Then air would enter the drain port raising the resin level at that side of the IXC, which reduced the resin slope across the IXC. This process is sketched in Figure 97. The cycle of the changing resin slope started with the entire upper surface of the resin basically flat and level. As the draining progressed, the slope in the resin surface slowly increased with the height difference across the IXC increasing from 4, 8, 12, 15, to 18 inches. These height differences existed on the resin upper plane just before air entered the drain port, which caused the slope of the resin upper surface to reduce a bit and then the cycle repeated until the liquid level reached the drain port.

Figure 95. Test IXC40-6 resulted in a drier stream of resin exiting from the 3-inch drain valve non-vented test.

[17] This may be the reason why the 2-inch drain valve in the non-vented 18-inch IXC tests did not permit resin to drain. The consistency of the resin slurry at the drain port may have been too viscous to exit the drain.

Figure 96. The draining was slow with intermittent air bubble entering the drain port, but many bubbles remained trapped in the resin just above the valve opening.

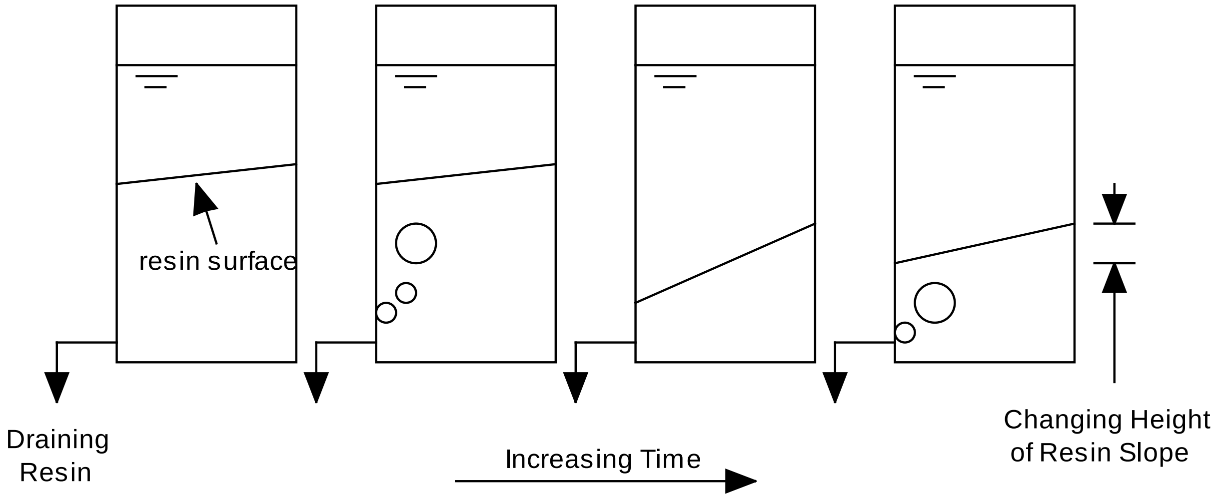


Figure 97. Sketch of draining progress in Test IXC40-6 as resin level drops through the 3-inch drain port during a non-vented test.

After approximately 57 minutes of pouring, the liquid level reached the top of the 3-inch drain port and then the slurry flow rate increased through the port, Figure 98. For the rest of the draining process, the IXC contents were more agitated with air readily entering the drain port, which fluidized some of the resin, but a heel began to form opposite of the drain port, Figure 99.

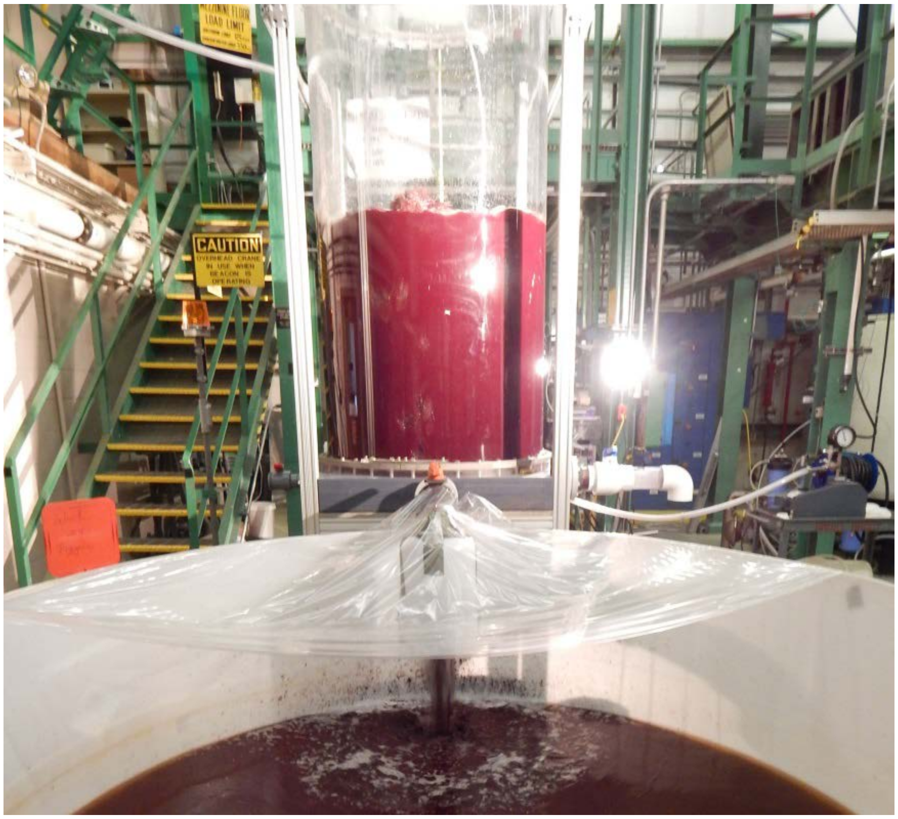


Figure 98. Liquid level reached the top of the 3-inch drain port and draining rate increased.

Figure 99. Liquid level reached the top of the 3-inch drain port while much of the remaining resin formed a heel opposite of the drain port.

As the contents of the IXC dropped further the mixture was being agitated by the incoming air, Figure 100; however, the agitation was not sufficient to remove the heel, Figure 101, as it was removed in the 18-inch IXC drain tests. The larger surface area and volume had an effect. The remaining heel was fairly uniform and flat. It was approximately 7 inches high on the IXC wall opposite of the drain valve, about 6 to 7 inches on one side of the column, and about 5 to 6 inches on the other side, and close to 0 inches near the drain port. If the resin were redistributed to be level across the bottom of the column, then an estimate of the level height is 5.5 inches above the resin screen, or 5.0 inches above the bottom of the bottom portion of the 3-inch drain valve, because that valve port sat 0.5 inch above the resin screen, giving a remaining volume of ~28 gallons of resin, which is similar to the amount left from the non-vented test.

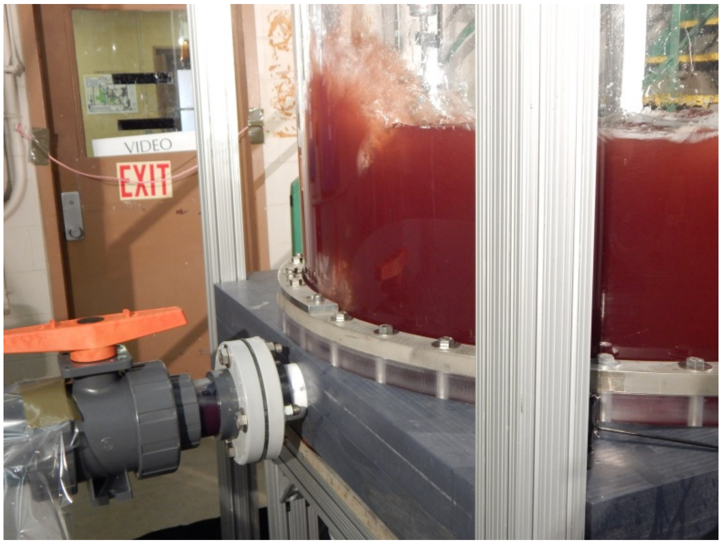


Figure 100. In Test IXC40-6 near the end of the draining process, more liquid was exiting the IXC than resin, which left a heel across the resin screen.

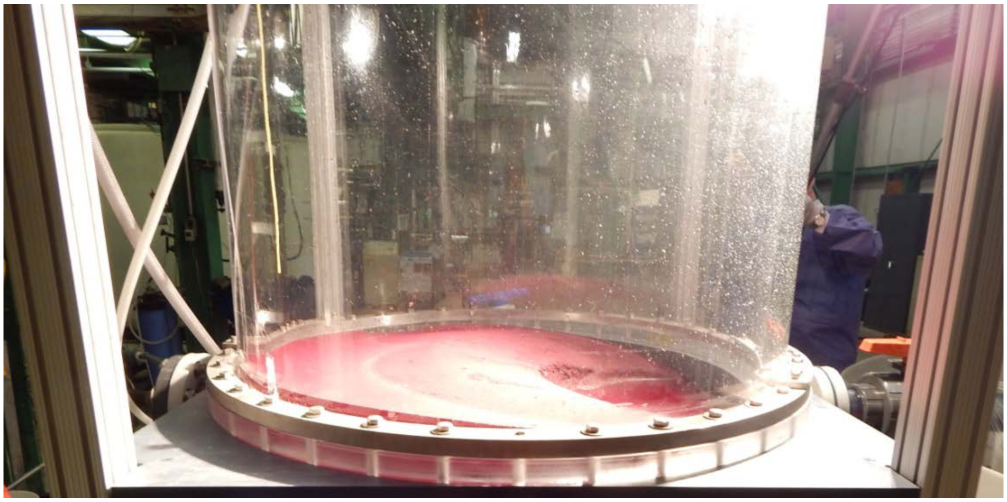


Figure 101. Test IXC40-6 had a relatively flat resin heel with an approximate volume of 28 gallons.

First Rinse – Figure 102 shows the resin heel during a 1-minute distributor flow as the liquid rained down at ~12.8 gpm, or ~2.7 BV/h. The remaining heel was approximately 4 inches high on the opposite IXC wall to the drain port. The heel sloped gently down to the drain being about 3.5 inches tall at the midpoint on one side and 2.0 inches on the other side. Using an average of 3 inches above the entire screen surface and taking into account that the 3-inch valve port was 0.5 inches above the screen surface, then the average was 2.5 inches, giving a remaining volume of 8.95 ft^2 x (2.5/12) x 7.48 gallons / ft^3 = 14 gallons, or approximately 14/298 ~ 4.7% of a BV.

Second Rinse – A 1-minute distributor flow was directed to the remaining heel at ~12.8 gpm, or ~2.7 BV/h. This left a heel that was approximately 2 inches high on the opposite IXC wall to the drain port. The heel sloped gently down to the drain, but it was still about 2 inches height at the midpoint on both sides and about 2 inches near the drain port. Using an average of 2 inches above the entire screen surface and taking into account the 3-inch valve port was 0.5 inches above the screen surface, then the average was 1.5 inches, giving a remaining volume of 8.95 ft^2 x (1.5/12) x 7.48 gallons / ft^3 = 8.4 gallons, or approximately 8.4/298 ~ 2.8% of a BV.

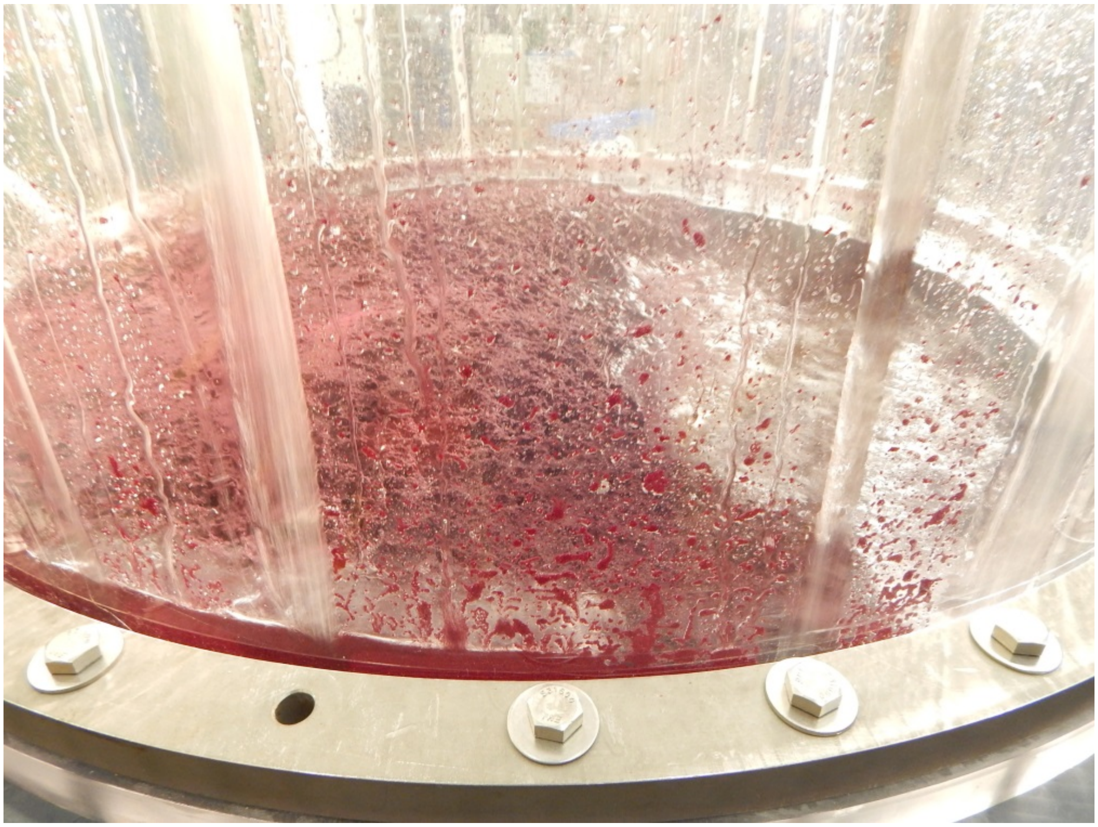


Figure 102. A photograph of distributor flow raining down on the resin heel pelting the surface. The flow ran down along the IXC as well as through the center.

Third Rinse – This time the rinse was performed differently from previous rinses. Both upflow and downflow were used. A ~1.5-minute upflow was directed to the remaining heel at ~25.2 gpm, which was then followed with 0.5 minute of downflow at 12.8 gpm. (The downflow would have been longer, but the feed liquid ran out.) The entire bed of resin was lifted off the resin screen approximately 4 inches before dropping after the flow was reversed. The change to the resin heel was not dramatic, but it appeared to reduce the heel by about 0.5 inches, or 1.0 inch from the bottom of the screen, giving a remaining volume of 8.95 ft^2 x (1.0/12) x 7.48 gallons / ft^3 = 5.6 gallons, or approximately 5.6/298 ~ 1.9% of a BV, Figure 103. A fourth rinse was not made because the distributor liquid was depleted; however, based on the vented Test IXC40-3, after 3 rinses the heel was approximately 5.6 gallons, too, which implies that the action of the upflow was not very useful. Furthermore, a fourth rinse was not expected to be different from Test IXC40-3, or from the 4-inch tests, which all indicated the final remaining resin heel is approximately 4 gallons.

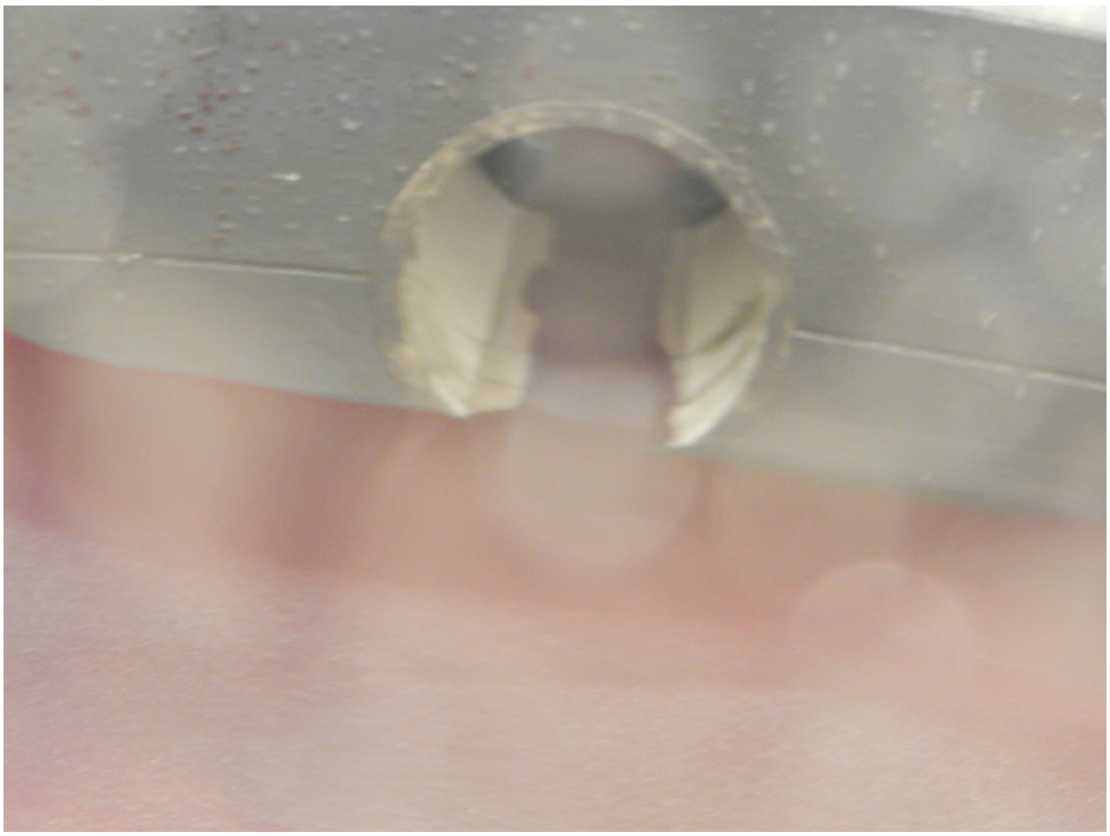


Figure 103. A heel of approximately ~1.9% (~5.6 gallons) of the starting BV remained after 3 rinses with feed flow raining down from the feed distributor and using upflow on the final rinse.

3.3.6 Summary 40-inch IXC Test Results

Table 7 shows the results for all 6 tests performed with the 40-inch IXC drain test, including the shakedown test with water.

Table 7. Summary 40-inch Inside Diameter Ion Exchange Column Test Results

Test IXC40-	Daily Test	Test Date	Valve inch	Test Liquid	Name	Duration min (1)	Comment
1	1	20-Oct-15	4	Water	Vented Drain	1.0	Distributor flow of ~4 BV/h used after drain to remove heel.
2	2	20-Oct-15	4	Water	Non-Vented Drain	7.0	5.5 in. Hg vacuum - IXC contents did drop 9 inches after opening 4-inch drain valve. (2)
3	1	21-Oct-15	4	PEP-5 M Na	Vented Drain	2.4	Distributor flow of ~4 BV/h used after drain to remove heel.
4	2	21-Oct-15	4	PEP-5 M Na	Non-Vented Drain	28.0	8.0 in. Hg vacuum - IXC contents did not drop after opening 4-inch drain valve. (3)
5	1	23-Oct-15	3	PEP-5 M Na	Vented Drain	3.7	Distributor flow of ~4 BV/h used after drain to remove heel.
6	2	23-Oct-15	3	PEP-5 M Na	Non-Vented Drain	62.0	8.0 in. Hg vacuum - IXC contents did not drop after opening 3-inch drain valve. (3)

Notes to Table 7

Nomenclature: PEP = Hanford Simulant: 5 M Na

(1) Time uncertainty of ±0.2 min is principally based on the end of the drain cycle because it was difficult to perceive when the drain stopped. The water drain times are only rough estimates because they are based on approximately a third of the IXC draining due to the smaller receipt tank used.

(2) When the column contents first stopped draining the height was ~ 96 inches. With the water density = 0.997 g/cm^3 (at 25°C) then the weight of the liquid in the unvented but equalized contents would be [96" - 4" (valve port)] (2.54 cm / in) (1.0 g/cm^3) (29.53 inches Hg / 1019.72 g/cm^2) = 6.7 inches Hg vacuum. The applied 5.5 inches Hg was not sufficient to prevent the contents from dropping.

(3) To prevent the column contents from immediately exiting the IXC on opening the drain valve, the vacuum above the column needed to, at least, match the weight of the full column. For a full column of 5 M Na PEP simulant, when using a 4-inch valve, is [100.8" - 4" (valve port)] (2.54 cm / in) (1.23 g/cm^3) (29.53 in.Hg / 1019.72 g/cm^2) = 8.7 inches Hg vacuum. (8.8 inches Hg vacuum for the 3" valve.) However, the 8 inches Hg used seemed sufficient to prevent the contents from dropping.

3.3.7 Resin Lodged within Dead-end Areas of the IXC

Observations were made of the resin that managed to enter the dead-end locations in the drain valves that were idle during other drain valve tests. That is, during the 4-inch drain valve tests, the resin deposited in the transparent pipe section leading to the 3-inch drain valve was observed and during the 3-inch drain valve tests the resin deposited in the transparent pipe section leading to the 4-inch drain valve was observed. Figure 99 (and others) shows both valves with the 3-inch valve to the left with the orange handle and the white colored 4-inch valve to the right. In that photograph it can be seen that between the valves and the IXC valve sleeve there was a small section of horizontal straight pipe. Approximately 2 inches of that transparent section was used to perform observations. Figure 104 shows that the resin in the 3-inch valve filled the pipe section about half full during the 4-inch drain valve test. Figure 105 shows that the resin in the 4-inch valve filled the pipe section about half full during the 3-inch drain valve test. This was fairly consistent for all four tests. It appears that resin does not pack this section of pipe, but separates to about the same concentration of slurry resin in the IXC. That is, the IXC had 1 BV of resin and 1 BV of freeboard liquid. During the draining of resin the contents of resin in the dead-end pipe sections did not appear to be significantly affected. However, this may not be indicative of when a resin remains in an IXC for dozens of feed and elution cycles, that is, when the resin physically changes size by shrinking and expanding. The life cycle of resin within such enclosed locations may result in different concentration of resin. Furthermore, the surface difference of PVC pipe and stainless steel pipe may also lead to a different result.

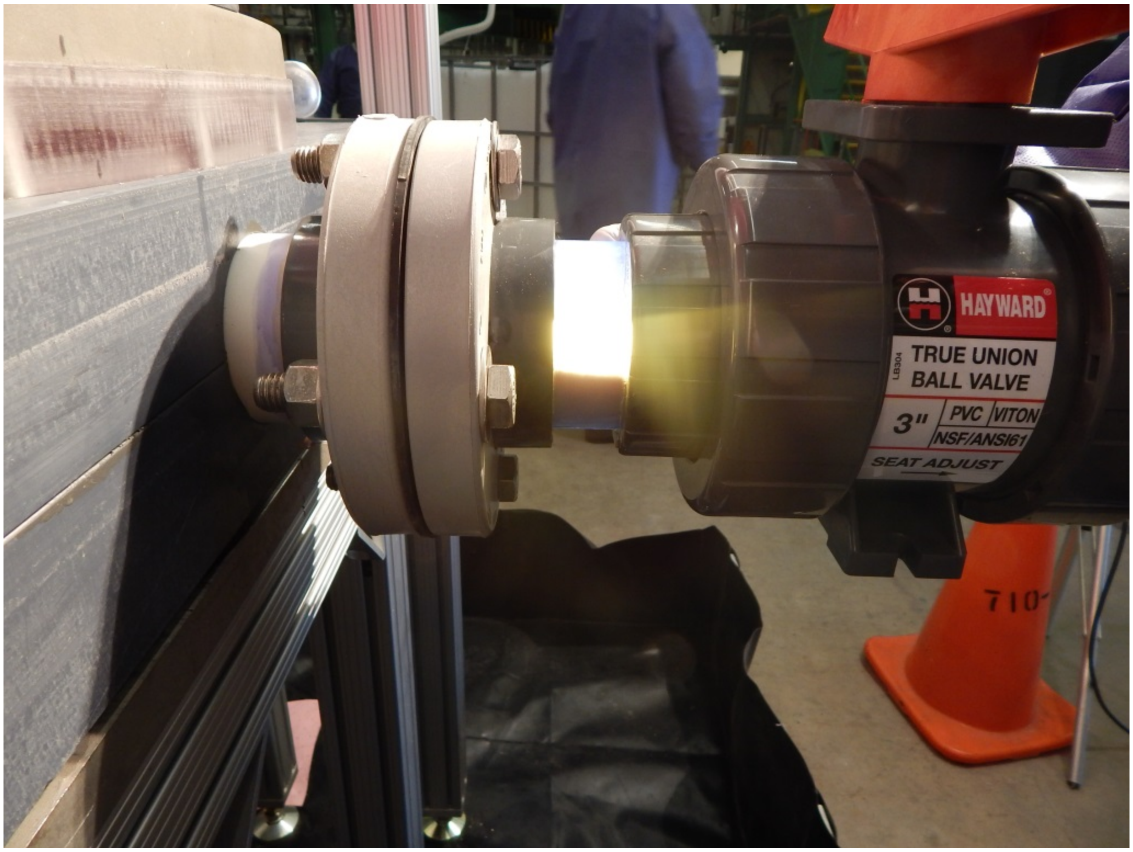


Figure 104. View through 2-inch window of 3-inch valve connection during 4-inch valve drain.

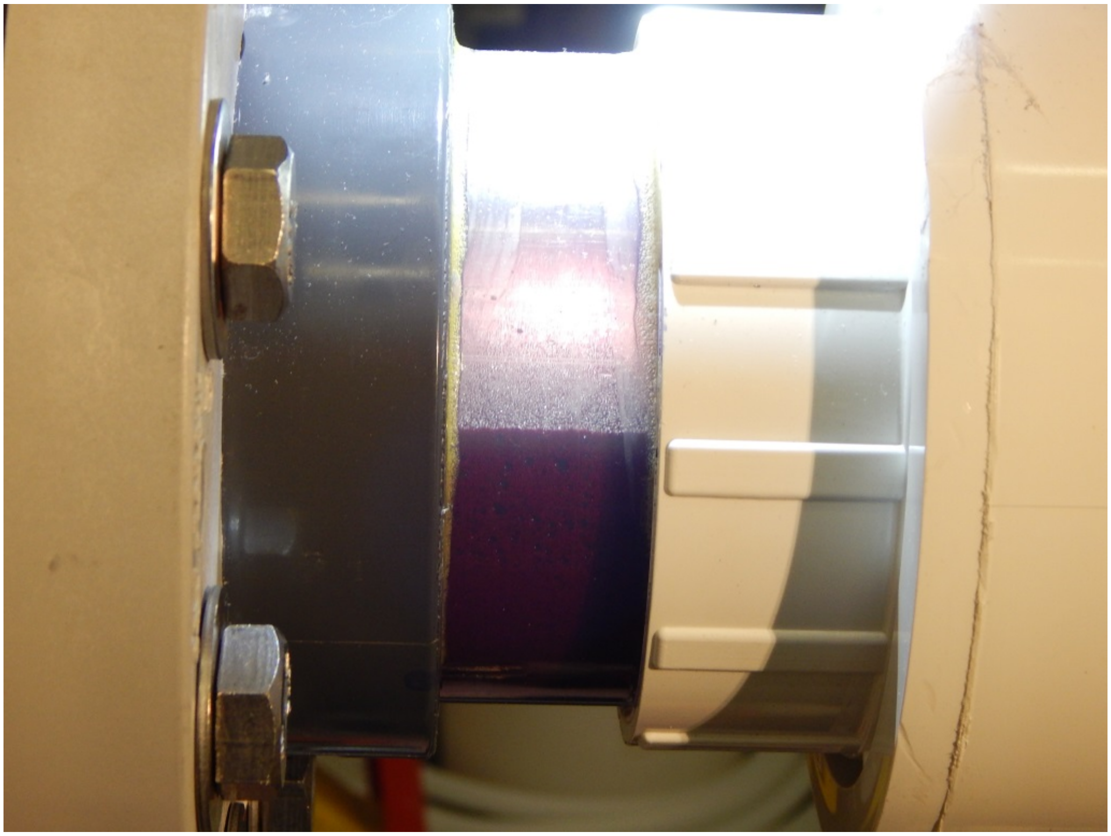


Figure 105. View through 2-inch window of 4-inch valve connection during 3-inch valve drain.

3.4 Temperature Effect on sRF Resin Yield Stress

At the end of all testing a sample of the sRF resin was taken from the storage containers of the new resin received from Hanford for the large scale tests. The resin was in H-form and kept in water. That sample was used to perform rheological measurements to hopefully better understand some of the IXC drain results, e.g., the lag times experienced in the small scale tests shown in Table 2.

Using a Haake rheometer and vane rotor FL-A[18] several shear stress measurement were made to estimate the Yield Stress of the sRF in water and if temperature had an effect. The resin sample obtained was split into two, approximately, equal batches. Each batch was then mixed and allowed to settle for three days. During that time one batch was maintained at room temperature (~22°C) and the other at 65°C before the measurement began. Figure 106 shows the two batches just before the yield stress measurement began. Note how the higher temperature H-Form resin turned from a light to a dark orange, very similar to the color change when sRF resin is converted to sodium form. Both batches were in plastic containers.

To perform the measurements, the vane rotor was lowered into the resin at a depth of approximately 16 mm, which is the height of the vane. That is, in Figure 106 the vane was lowered until the bottom of the white tape on the rotor shaft touched the top of the resin bed. Three measurements were made at each temperature with the results shown in Figure 108 and Figure 109.

[18] The dimensions of the vane for this rotor are a height of 16 mm and a sweep diameter of 22 mm. To operate accurately the top of the vane must at a depth of 16 mm, or greater, below the top surface of the solution being measured and have 8 mm, or greater, distance from the bottom of the vane to the bottom of the vessel. These conditions were met.

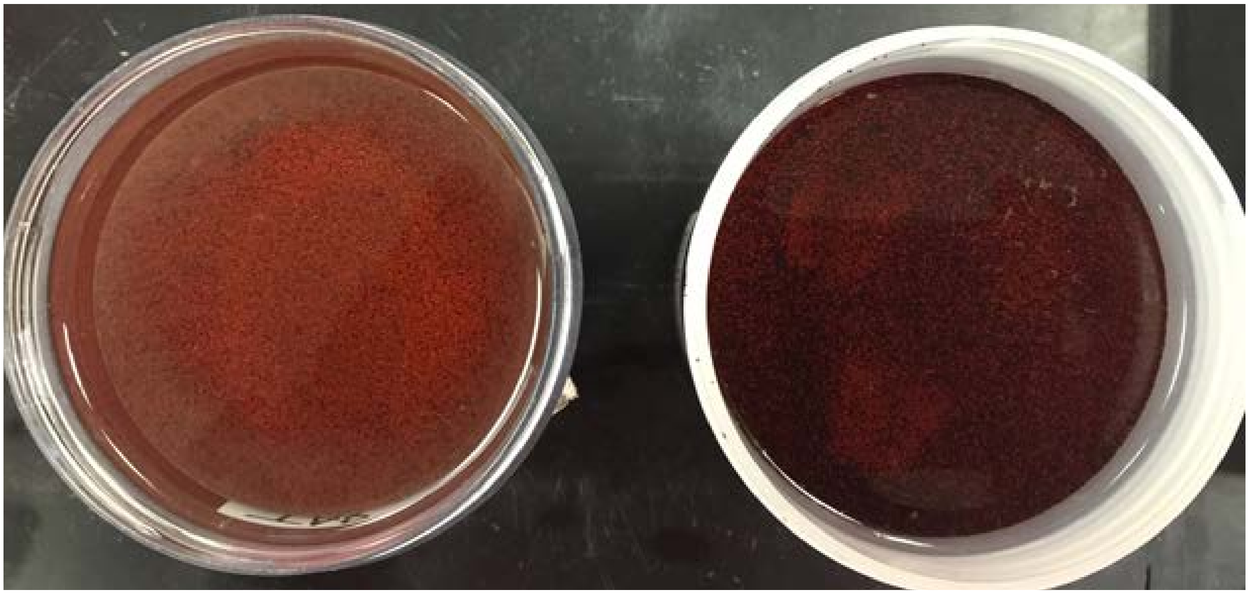


Figure 106. sRF in water after 3 days of settling at room temperature (left) and at 65°C.

Figure 107. The FL-A vane being inserted into the sRF for the room-temperature measurement.

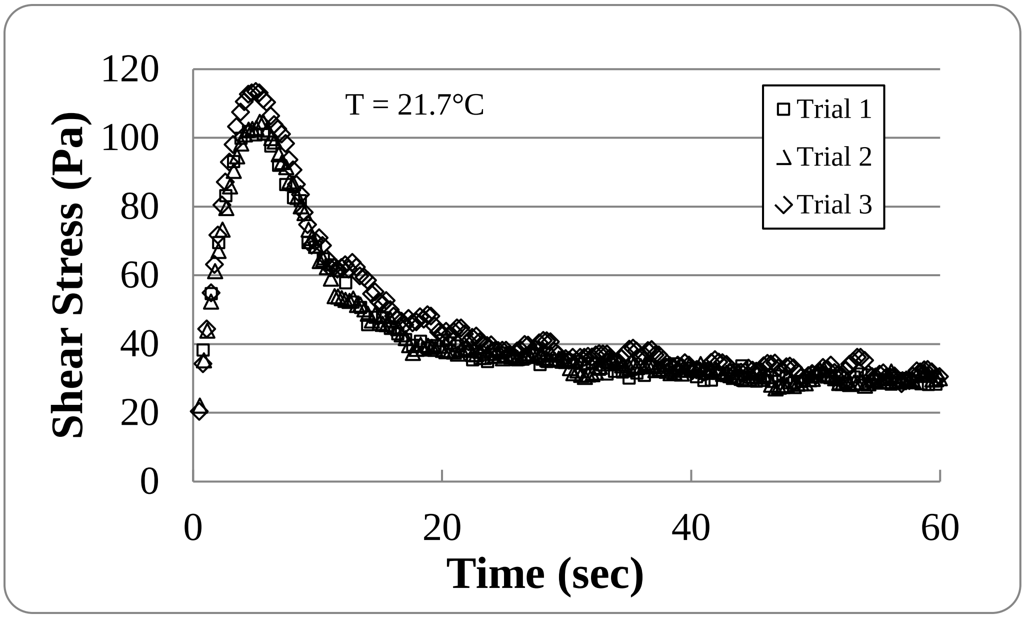


Figure 108. Yield stress measurement of H-Form sRF IX resin after 3 days of settling at room temperature.

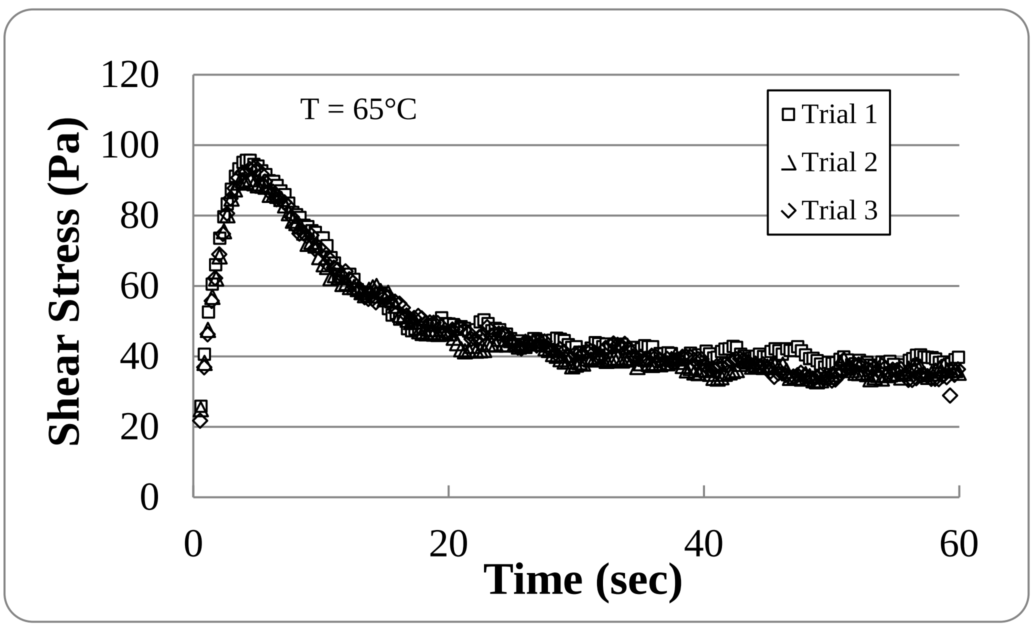


Figure 109. Yield stress measurement of H-Form sRF IX resin after 3 days of settling at 65°C.

Table 8 shows the overall data set and the averages of the resin yield stresses. There appears to a slight difference in the yields stress of the resin that drops as temperature increases. However, at the 95% confidence level, which is 3 standard deviations of the data, the difference in the yield stress at the two temperatures tested is statistically insignificant. Furthermore, the result does not explain why the lag time to begin draining from the IXC increased for increasing temperatures in the small scale tests. What can be stated is that for sRF resin in H-form, with water as the supernatant, the yield stress is on the order of 100 Pa. It is important to note that this yield stress value is for loosely packed resin. As stated, the rheometer rotor vane was placed only a short distance under the surface of the resin and no effort was made to pressurize the overlying surface to replicate packed resin under varying depths. As the resin experiences more surrounding pressure it will take more force to initially move, which means the yields stress will be higher.

Table 8. Yield stress measurements of loosely packed sRF IX resin in water.

Temp	Trial 1	Trial 2	Trial 3	Average	1xStd Dev	3xStd Dev
21.7°C	101.7	104.6	113.5	**106.6**	6.1	18.4
65°C	95.6	90.2	93.4	**93.1**	2.7	8.0
Note: Yield Stress values are in Pascal= Newton/m^2						

4.0 Conclusions

4.1 Small-Scale (IXC04) Drain Tests: Effect of Temperature

- Resin drained freely at all the temperatures used.
- Resin drained freely from IXC made from PVC or stainless steel.
- The rate of resin draining up to 65°C did not appear to be dependent on temperature.
- The rate of resin draining at 85°C did appear to have some dependence on temperature and is estimated to take approximately twice as long.
- The rate that resin drained did not appear to be dependent on different materials of the IXCs used, i.e., PVC and SS. Note that the valves for both types of columns were made of stainless steel.
- The time at which a drain valve is opened and when the resin slurry began to exit the drain as a fluid, that is, lag time, appeared to be dependent on temperature. However, because such lag times were not experienced in the larger scale IXCs, under vented conditions, this phenomenon may solely be due to scale. That is, the size of the drain port and the size of the resin bead, which was 21 to 1, respectively. However, the larger scale tests were not performed at elevated temperatures, this conclusion is only an assumption.
- The volume of the resin heel after every test ranged from approximately 5% to 25% of the initial BV, with an average of 15%. However, there did not appear to be any dependence on temperature, material, or the newness of the resin.

4.2 Mid-Scale (IXC18) Drain Tests: Effects of Waste Streams. Pressure, and Drain Valve Size

- All three drain valve sizes used, i.e., 1-, 2-, and 3-inch valves, allows the contents of a vented IXC to drain independent of suspension liquid, i.e., IW, PEP supernatant simulant, and Nitric Acid.
    - Only a small heel remains that is easily removed with 5 to 10 gallons of liquid.
    - The smaller the size of the drain valve, the longer the time to drain.
- The larger the drain size, the smaller the resin bed. For vented drain the remaining heel is approximately:
    - 5% to 10% of the resin BV for the 3 inch valve.
    - 10% to 15% of the resin BV for the 2 inch valve.
    - 15% to 20% of the resin BV for the 1 inch valve.
- The 3-inch drain valve allows a non-vented IXC to fully drain with both PEP and Nitric Acid as the suspension liquid.

- Only a small heel remains, which is approximately half the heel remaining after that left from a vented drain. The smaller heel may be the result of the intense mixing that occurs from gas rising from the drain port.
- A 2-inch valve does not effectively allow slurry resin to drain. This implies that a 1-inch valve will not effectively drain either, but it was not tested.
- The strength of the vacuum above a column controls the rate a column drains. For a vacuum that is just equal to the force necessary to hold up one column volume the drain time was approximately 30 minutes.

- As shown in Test IXC18-13, increasing the freeboard of liquid above the resin bed does not effectively improve the removal of a resin heel. Doubling the freeboard to 2 BV still left approximately the same amount of heel as a drain in which the IXC had 1 BV of liquid freeboard.
- All resin heels were easily flushed out with 5 to 10 gallons of liquid, which leaves only trace resin on the resin screen.
- With the continuous distributor flow of 4 BV/h during draining, only a trace amount of resin was left on the resin screen (Test IXC18-2, -4, -6, and -17).
- The column is able to drain with precipitates intermixed within the resin, though in severe cases a brief upflow may be required to initiate the drain.

4.3 Full-Scale (IXC40) Drain Tests: Effect of Scale

- Both drain valve sizes used, the 3- and 4-inch valves, allowed the contents of a vented IXC to drain with sRF resin in PEP 5 M Na test solution.
  - The remaining resin heel was on the order of 10 to 12% of the initial BV, i.e., 28 to 34 gallons. After approximately 50 gallons of rinsing using distributor flow the heel reduced to about 2% or 4 gallons.
  - The smaller the size of the drain valve, the longer the time to drain.
- Both the 3-inch and 4-inch drain valves allowed a non-vented IXC to fully drain the PEP 5 M Na simulant.
  - The resin heel that remained for the non-vented drain tests was approximately the same as for the vented drain tests. However, the form of the resin heel was different:
- The vented tests left a very uneven bed with a surface that changed rapidly across the IXC diameter, with the tallest resin location on the wall opposite of the drain valve.
- The non-vented tests left a flat and fairly level resin bed across most of the IXC resin screen surface.
  - The smaller the size of the drain valve, the longer the time to drain.
- The drain size did not result in significant differences of the remaining resin bed.
  - For vented drain the remaining heel was approximately:
    - ~11% of the resin BV for the 3 inch valve.
    - ~10% of the resin BV for the 4 inch valve.
  - For non-vented drain the remaining heel was approximately:
    - ~10% of the resin BV for the 3 inch valve.
    - ~12% of the resin BV for the 4 inch valve.
- Most of resin heels could be flushed out with about 50 gallons of liquid, but the remaining heel in all the tests had a fairly constant volume of about 1.5%, or ~4 gallons on the resin screen. Even a short upflow of feed to lift the resin off the resin screen did not result in a significant reduction of the final heel.
- Resin that enters the dead-end pipe location at the bottom of the IXC, e.g., drain valves not being used, appears to separate to approximately the concentration of the initial contents of the IXC, which for these test was 1 BV of resin and 1 BV of freeboard liquid.

4.4 Overall Conclusions

Based on the conclusions obtained at the different scales some overall observations can be made:

- A slurry of liquid waste and resin should always drain freely after a valve is opened as long as an IXC is vented.
- For a non-vented IXC, the resin slurry will drain freely for discharge ports of 3 inches and greater after the drain valve is opened and when the created vacuum above the column is strong enough to prevent the IXC contents from initially exiting the valve.
- Draining from the IXC for a vented column is generally less than a few minutes.
- Draining from the IXC for a non-vented column increases the drain time by an order of magnitude over the time for a vented column with the same drain size.
- The use of plastic models of the IXC elicits drain data similar to that from stainless steel.
- Resin drain times are not significantly affected by temperatures of 65°C or less. Above 65°C, resin appears to drain slower possibly due to increased adhesion forces among the resin beads.
- Resin heel volume after drain does not appear to be dependent on temperature.
- A rough comparison of the volume of resin heel to the ratio: (drain port diameter)/(IXC diameter), for all scales of a vented drain at 25°C is provided:

Small scale:	0.375”/4”	= 0.094	7-27%BV heel
Middle scale	1”/17.5”	= 0.057	15-20%BV heel
Middle scale	2”/17.5”	= 0.118	10-15%BV heel
Middle scale	3”/17.5”	= 0.171	5-10%BV heel
Large scale	3”/40.5”	= 0.074	11%BV heel
Large scale	4”/40.5”	= 0.099	10%BV heel

The only exception of these heel results is when the PEP simulant included a precipitant which caused the resin to adhere to itself. The heel was much larger than the percentages listed above and atypical of a standard drain.

- IXC sRF resin slurry drain results appear to be approximately independent of suspension liquid, i.e., IW, supernatant simulant, and Nitric Acid. Furthermore, when the suspension was subjected to precipitation the time to drain was similar to all other tests, but it initially did not drain until suspension liquid was upflowed for a few seconds to free up the resin so it could move.
- The remaining resin heels could be flushed out with distributor liquid, but as scale increased so did the amount of liquid needed.
    - For the 18-inch IXC approximately 10 gallons were needed to reduce the heel to only a trace amount of resin.
    - For the full-scale IXC, approximately 50 gallons were needed. Considering the increase in IXC resin screen area, i.e., $(40.5”/17.5”)^2$ = a factor of 5.4, then the 50 gallons necessary makes sense.

  However, at the larger scale the amount of remaining heel could only be reduced to approximately 1.5% (~ 4 gallons) of the initial BV, which represented the removal of approximately 85% of the heel left after the drain.
- The remaining resin heels could be flushed out with distributor liquid, but as scale increased so did the amount of liquid needed.
- Loosely packed sRF resin in the H-form with water as the supernatant had a yield stress on the order of 100 Pa.

5.0 Recommendations

From the results it would be prudent to:

- Employ a resin drain valve of 3-inch or larger to prevent pluggage that would impede the emptying of the IXC in the event of a non-vented drain.
- In the event of a non-vented drain, it will be helpful to include a small gas plenum above the IXC that would allow the resin slurry to initially drain until the vacuum in the gas plenum equals the weight of the remaining IXC contents, which would temporarily slow the draining process. However, the initial flow of resin slurry, while the pressure is equalizing, will help to allow air to enter the drain valve, thus permitting the draining to continue.
- Include a method of introducing a stream to rain down on the draining resin to help flush out the resin heel because a significant heel remains after a drain.
- A method of upflowing feed into the IXC during a power outage for a few seconds will help to drain the resin in the event of the resin being subjected to precipitants.

6.0 References

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Duignan, M. R. and Herman, D. T., 2015, "Task Technical and Quality Assurance Plan – LAWPS Resin Removal Test," SRNL-RP-2015-00631 (13 August)

Duignan, M.R., C.A. Nash, and T.M. Punch, 2008. *High Aspect Ratio Ion Exchange Resin Bed – Hydraulic Results for Spherical Resin Beads*, Separation Science and Technology, Vol. 43, pp. 2943-2979.

Appendix A. Early design of LAWPS IXC Drain system

This was the sketch obtained from WRPS of the possible resin drain system for the LAWPS IXC. It was utilized to design the 18-inch IXC pilot scale drain system.

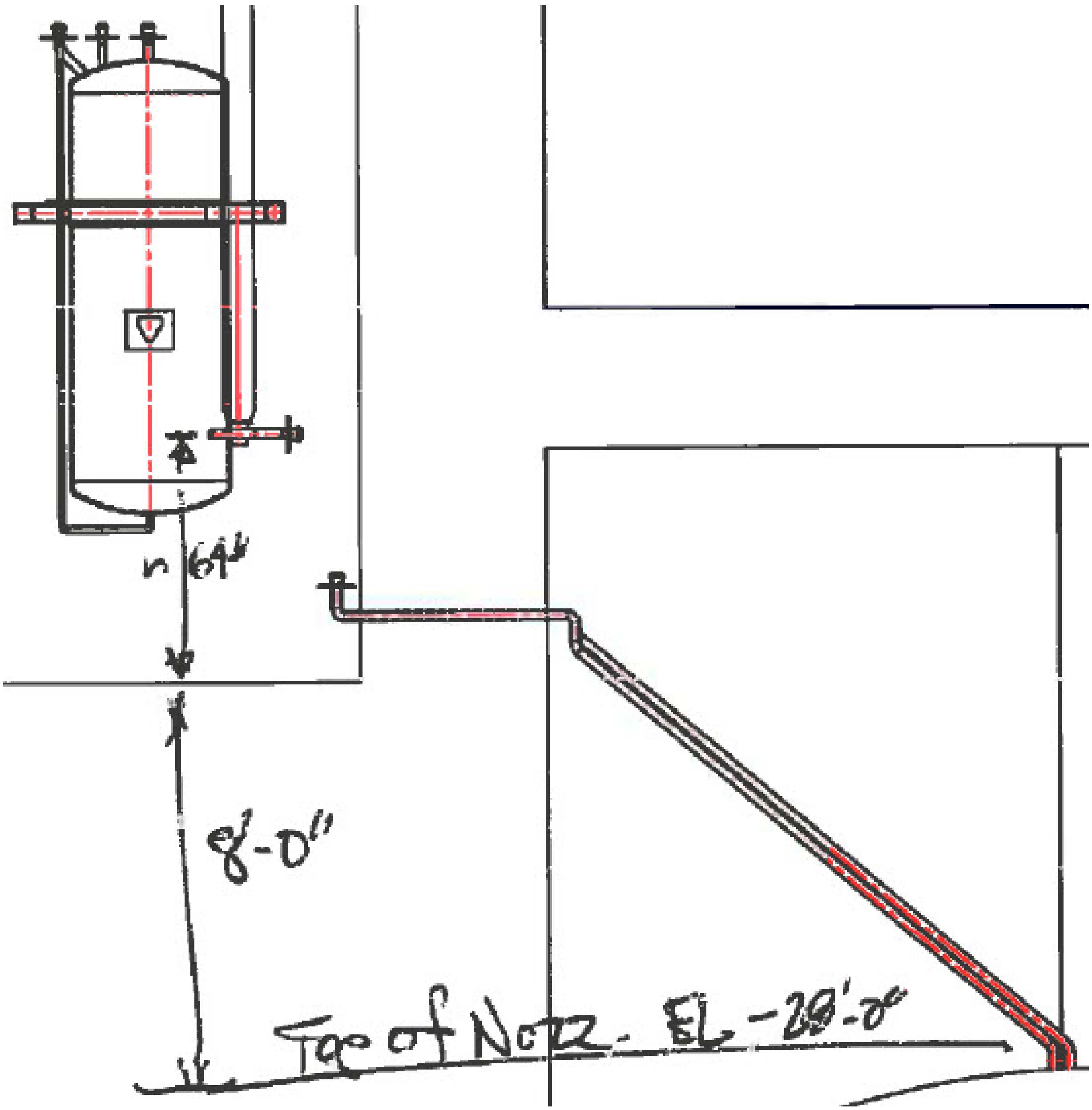

Figure 110. Sketch of the design of resin drain system for the LAWPS Ion Exchange Column as it existed during the planning for the 18-inch IXC resin drain test.

Appendix B. Revised design of the LAWPS IXC Drain system

This was the sketch obtained from WRPS of the possible resin drain system for the LAWPS IXC. It was utilized to design the 40-inch IXC pilot scale drain system. For this system, the drain valve will not be connected to any drain pipe, but the draining resin is to simply be caught be a large basin that will sit under the column.

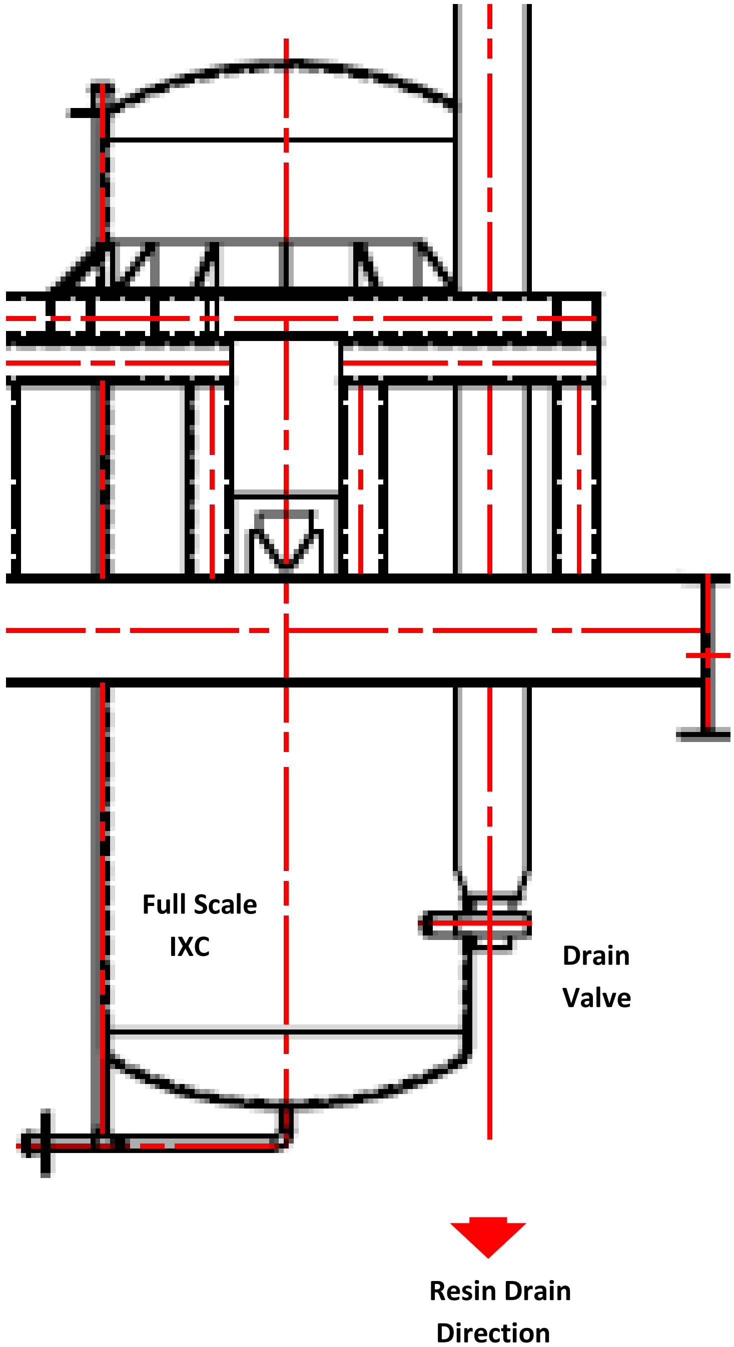

Figure 111. Sketch of the design of resin drain system for the LAWPS Ion Exchange Column as it existed during the planning for the 40-inch IXC resin drain test.

Distribution:

SRNL-Savannah River Site
D. J. Adamson, 999-W
T. B. Brown, 773-A
P.R. Burket, 773-42A
H. H. Burns, 773-42A
M. E. Cercy, 773-42A
A.D. Cozzi, 999-W
D. E. Dooley, 773-A
M. R. Duignan, 786-5A
A. P. Fellinger. 773-42A
S. D. Fink, 773-A
K.M. Fox, 999-W
E.K. Hansen, 999-W
C. C. Herman, 773-A
D. T. Herman, 735-11A
D. T. Hobbs, 773-A
E. N. Hoffman, 999-W
D.J. McCabe, 773-42A
F. M. Pennebaker, 773-42A
M. L. Restivo, 773-42A
G. N. Smoland, 773-42A
M. E. Stone, 999-W
A.L. Washington, 773-42A
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