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## Nitrate Conversion of HB-Line Reillex™ HPQ Resin

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## EXECUTIVE SUMMARY

Reillex™ HPQ ion exchange resin is used by HB Line to remove plutonium from aqueous streams. Reillex™ HPQ resin currently available from Vertellus Specialties LLC is a chloride ionic form, which can cause stress corrosion cracking in stainless steels. Therefore, HB Line Engineering requested that Savannah River National Laboratory (SRNL) convert resin from chloride form to nitrate form in the Engineering Development Laboratory (EDL).

To perform this task, SRNL treated two batches of resin in 2012. The first batch of resin from Reilly Industries Batch 80302MA was initially treated at SRNL in 2001 to remove chloride. This batch of resin, nominally 30 liters, has been stored wet in carboys since that time until being re-treated in 2012. The second batch of resin from Batch 23408 consisted of 50 kg of new resin purchased from Vertellus Specialties in 2012. Both batches were treated in a column designed to convert resin using downflow of 1.0 M sodium nitrate solution through the resin bed followed by rinsing with deionized water. Both batches were analyzed for chloride concentration, before and after treatment, using Neutron Activation Analysis (NAA).

The resin specification [Werling, 2003] states the total chlorine and chloride concentration shall be less than 250 ppm. The resin condition for measuring this concentration is not specified; however, in service the resin would always be fully wet. Measurements in SRNL showed that changing from oven dry resin to fully wet resin, with liquid in the particle interstices but no supernatant, increases the total weight by a factor of at least three. Therefore, concentration of chlorine or chloride expressed as parts per million (ppm) decreases by a factor of three. Therefore, SRNL recommends measuring chlorine concentration on an oven dry basis, then dividing by three to estimate chloride concentration in the fully wet condition.

Chloride concentration in the first batch (#80302MA) was nearly the same before the current treatment (759 ppm dry) and after treatment (745 ppm dry or ~248 ppm wet). Treatment of the second batch of resin (#23408) was very successful. Chloride concentration decreased from 120,000 ppm dry to an average of 44 ppm dry or ~15 ppm wet, which easily passes the 250 ppm wet criterion.

Per guidance from HB Line Engineering, SRNL blended Batch 80302 resin with Batch P9059 resin which had been treated previously by ResinTech to remove chloride. The chloride concentrations for the two drums of Batch P9059 were 248 ppm dry (~83 ppm wet)  $\pm 22.8\%$  and 583 ppm dry (~194 ppm wet)  $\pm 11.8\%$ . The blended resin was packaged in five gallon buckets.

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## LIST OF ABBREVIATIONS

ADS	Analytical Development Section
BV	bed volume
EDL	Engineering Development Laboratory
eHAP	electronic Hazards Assessment Plan
NAA	Neutron Activation Analysis
ppm	parts per million
psi	pounds per square inch
SVOA	Semi-volatile organic analysis
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TIC	total inorganic carbon
TOC	total organic carbon
VOA	volatile organic analysis

## 1.0 Introduction

### 1.1 Background

The current mission at H-Canyon involves the dissolution in nitric acid of an inventory that contains plutonium metal. HB-Line purifies the resulting plutonium solution using anion exchange, precipitates the plutonium as oxalate and calcines the oxalate to form plutonium oxide ( $\text{PuO}_2$ ). The  $\text{PuO}_2$  will provide feed material for the Mixed Oxide (MOX) Fuel Fabrication Facility, and the anion exchange raffinate will be transferred to H-Canyon [M. A. Jones, 2012]. The anion exchange resin, Reillex™ HPQ, has been used at SRS for ten years to remove plutonium from liquid streams. The polymer is intended to contain only carbon, hydrogen and nitrogen, but the resin is normally sold with chloride loaded in the active sites, because this form is not very combustible when dry. However, chloride is a problem at SRS because of stress corrosion cracking of stainless steel equipment, which is present in the HB line processing equipment.

In 1998 SRNL purchased Reillex™ HPQ from Lot 80302MA in chloride form from Reilly Industries. An attempt was made to convert the resin to nitrate form using batch mode in a carboy. Crooks, et al. [1999], Kyser [2000] and Crooks [2001] re-treated the same resin to nitrate form by washing with one molar sodium nitrate solution in a column followed by rinsing with water. This resin has been stored at SRNL since that time.

In 2004 a supplier, ResinTech of West Berlin NJ, sold Reillex™ HPQ from Batch P9059 which they had converted to the nitrate form. SRS purchased two steel drums each containing one cubic foot of resin. The as-received total damp weight was 35 kg. Resin in nitrate form must be kept damp or wet because it is combustible when dry. One-half of the ResinTech resin from one drum was subsequently used and the remainder was stored in the two original drums.

In 2012 SRS purchased an additional 50 kg of Reillex™ HPQ (Batch 23408) from Vertellus Specialties LLC in chloride form. Vertellus was not interested in converting the resin to nitrate form. Therefore, HB Line Engineering wrote a Technical Task Request [Christopher, 2011] requesting that SRNL perform the conversion. SRNL responded by preparing a Task Technical and Quality Assurance Plan [Leishear 2011a].

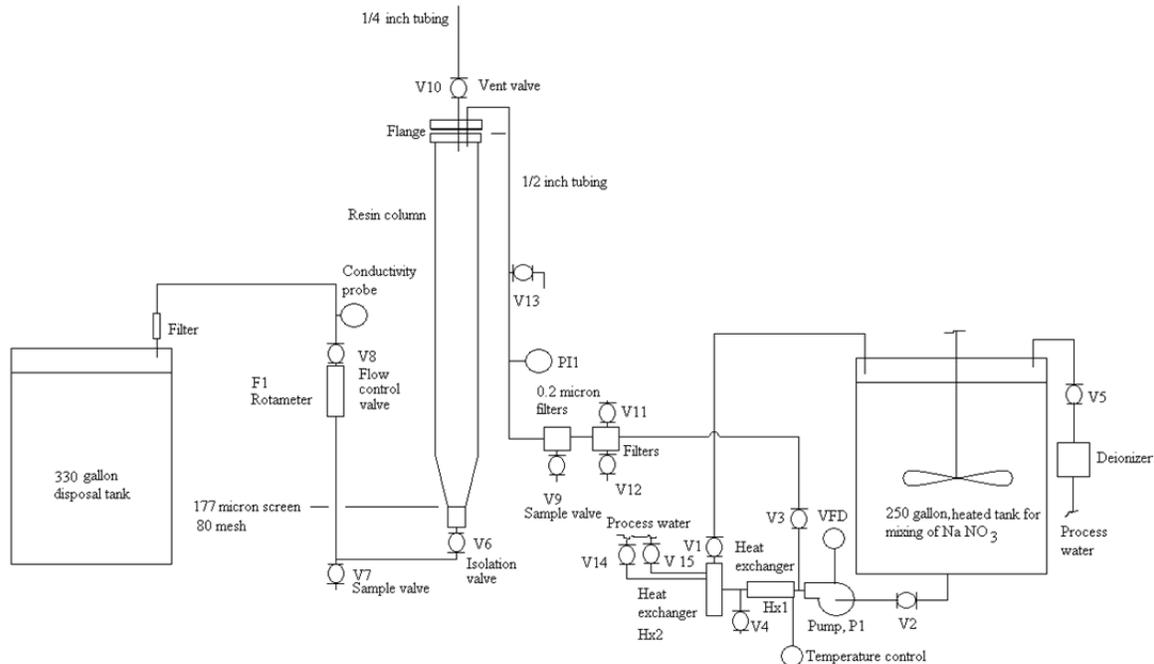
### 1.2 Characteristics of Reillex™ HPQ

Reillex™ HPQ is poly(4-vinylpyridene), cross-linked, methyl chloride quaternary salt and a strong base anionic ion exchange resin. The MSDS number is 45223-1. The resin is in the form of white to yellowish beads. SRS [Werling, 2003] specified that at least 90% of the mass is in the size range from 30 to 60 mesh (250  $\mu\text{m}$  – 595  $\mu\text{m}$ ). SRS also specified that the ion exchange capacity is 3.6 eq/kg (dry). When fully loaded with chloride that corresponds to 128,000 ppm chloride on a dry basis. Only the oxidation state 4+ of plutonium forms an anionic nitrate complex that loads onto Reillex™ HPQ [Kyser, 2000]. SRS also specified that the resin chloride concentration must be less than 250 ppm. However, the specification did not say what resin water content was used to determine the 250 ppm.

## 2.0 Experimental Procedure

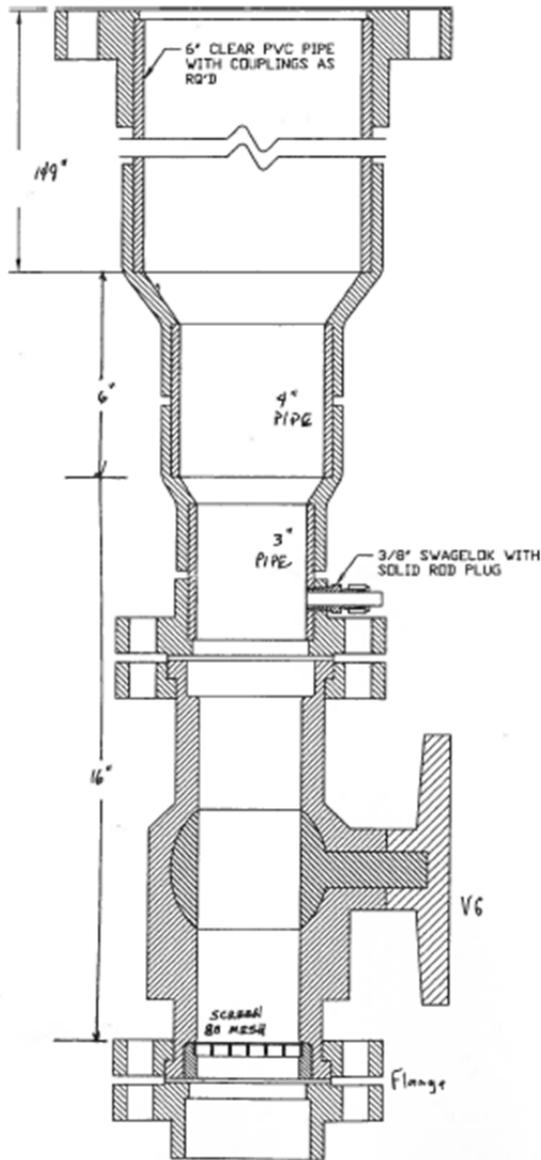
### 2.1 Description of Apparatus

The resin treatment apparatus is shown in Figure 1. The tank on the right side of the figure was used to mix one molar sodium nitrate solution. The tank has a heater and agitator to enhance dissolution of the solid sodium nitrate and a heat exchanger for subsequently cooling the tank contents. Downstream of the tank are a pump, filters, and a pressure gage. The inlet to the column is at the top, where there is also a vent point to allow removal of air from the column. The treatment column is primarily made from 6" sch. 40 clear CPVC pipe. The bottom of the column has an 80 mesh screen to hold up the resin. Downstream of the column is a pressure gage, a rotameter to measure flow, a throttle valve, a conductivity probe and a waste liquid collection tank.



**Figure 1 Piping and Instrumentation Diagram**

Figure 2 is a detail drawing of the original form of the treatment column which was used for the re-treatment of archived resin. The column size was based on the need to process as much as 50 kg of resin and on an estimated bulk density of  $25.2 \text{ kg/ft}^3$  ( $0.89 \text{ kg/L}$ ) for the resin. Therefore SRNL was expecting a maximum resin volume of 56 liters. The original form of the column included 149" of clear 6" pipe and shorter lengths of 4" and 3" pipe giving a calculated volume of 73.7 liters. For the treatment of new resin it was necessary to lengthen the column by adding 12" of 6" pipe or an additional 6 liters. The resin is supported on an 80 mesh screen. There is a solid  $3/8$ " plug in the side of the column which could be replaced with tubing to allow sparging of the resin to facilitate its removal from the column. However, sparging was never necessary.



**Figure 2 Design of Treatment Column**

## 2.2 Summary of Procedure

A Work Instruction [Leishear, 2012] was written to control this work. Data and observations were recorded in notebook SRNL-NB-2011-00156. A brief summary follows. After the apparatus was built, it was leak checked, cleaned and hydro-tested. That water was drained. Then deionized water and sodium nitrate in the proportion to give a 1.0 molar solution were added to the mixing tank while it was being heated and agitated. Sodium nitrate was procured with a chloride concentration less than 10 ppm, and the solution was filtered. After all solids in the mixing tank had dissolved, the agitator was de-energized and the tank contents, a one molar solution, were cooled to ambient temperature.

The treatment column was partially filled with deionized water. Then the top flange was removed from the column, and resin was scooped in. The top flange was replaced and flow of

nitrate solution was initiated. Flowrate was monitored using the rotameter and adjusted as necessary. Conductivity and chloride concentration of the column effluent were monitored using a conductivity probe and Quantab chloride test strips, respectively, and the results were recorded in the lab book. Sodium nitrate solution was pumped through the column in downflow at a superficial velocity of 2.7 cm/min to the disposal tank. Then, deionized water was pumped through the column to the disposal tank. Most of the water in the column was drained out using V7. The top flange was removed, and a resin sample was collected from the top. Valve V6 was closed and the bottom flange, 80 mesh screen, and a bottom resin sample were removed. The resin was then allowed to flow out of the column into a 55 gallon drum and a sample was collected from the middle of the column as the column was emptied.

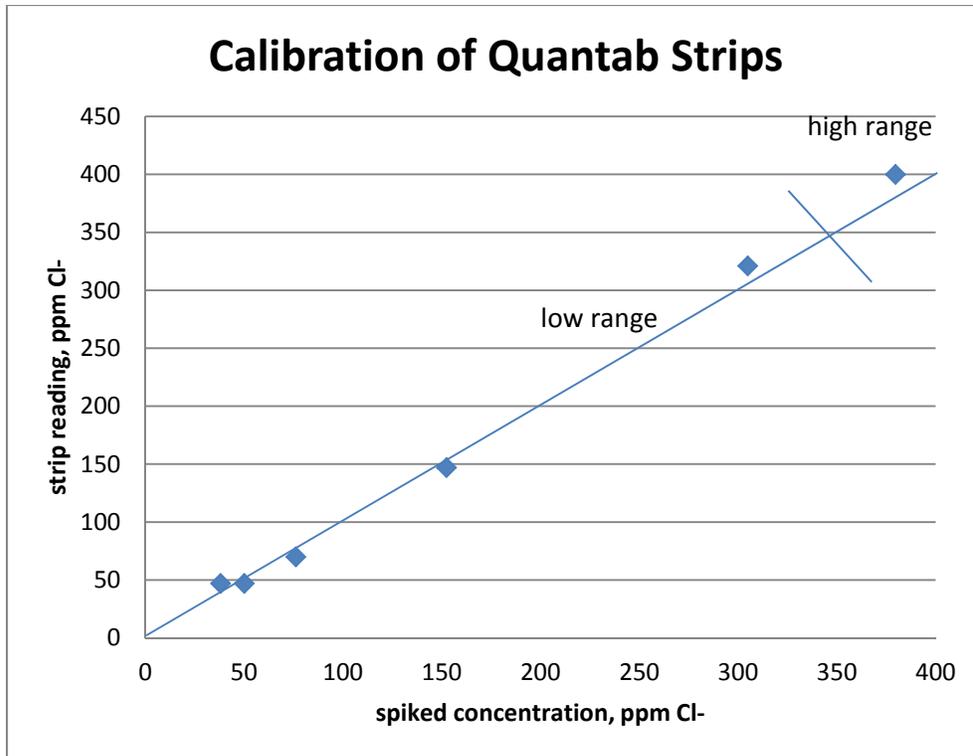
At the completion of treatment including washing with water, the resin was sampled to verify that chloride concentrations meet the 250 ppm specification in Z-SPP-H-00008. Samples were collected at the top, middle, and bottom of the resin, and were analyzed using Neutron Activation Analysis (NAA). The resin was observed to be very combustible in its dry form following nitrate conversion, and so was kept damp during processing, packaging and handling.

### 2.3 Chloride Test Strips

Chloride concentrations in the column effluent were measured using Quantab test strips. This aided in tracking the conversion process. The high and low range strips cover the concentration ranges 300 ppm to 6000 ppm chloride and 30 ppm to 600 ppm, respectively. The test strips were calibrated using solutions of sodium chloride in one molar sodium nitrate. The results are shown in Figure 3 and show good accuracy.

### 2.4 Conductivity Probe and Rotameter

Effluent stream conductivity was measured using an Omega conductivity meter, TR-03884. This also aided Liquid flowrate was measured using a Fischer-Porter rotameter, TR-00133, with a maximum flowrate of 20 gal/h.



**Figure 3 Calibration of Quantab Test Strips**

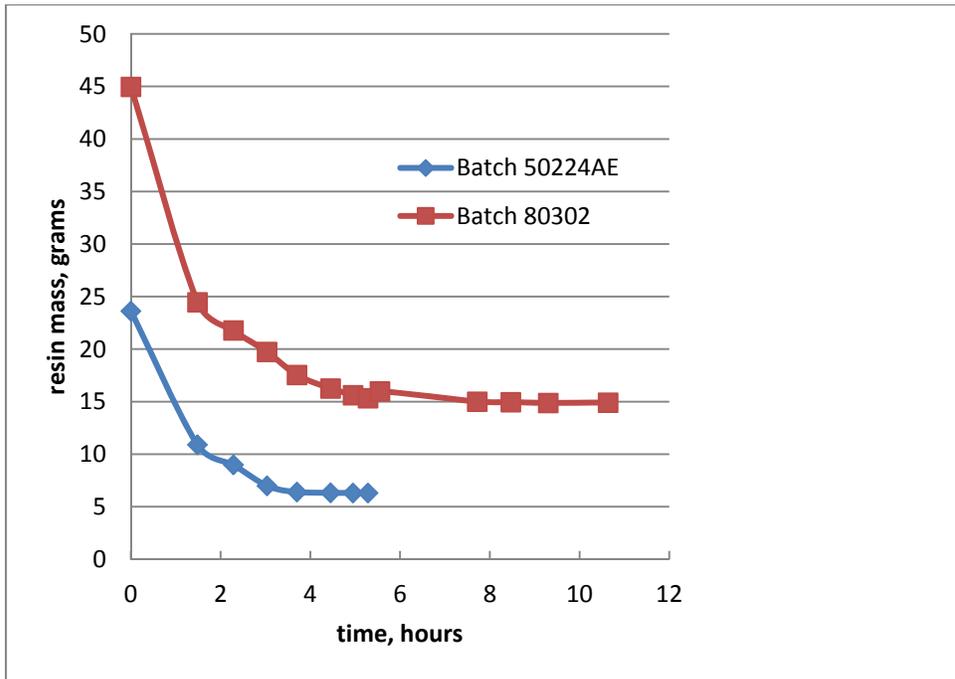
### 2.5 Neutron Activation Analysis

Analytical Development Section (ADS) of SRNL used NAA to determine the concentration of chloride in the resin. In this method, a sample is bombarded with neutrons, and the different elements present in the sample emit gamma rays at different energies. Concentrations of different elements may be determined by measuring the number of gamma photons with energies corresponding to the different elements. In this case NAA was calibrated by placing a quantity of water with a known amount of chloride in the sample holder. It is important to calibrate NAA in the approximate range of concentrations of interest. For relatively low concentration of chloride measurement, accuracy may be improved using longer counts and larger sample masses.

Nitrate treated Reillex™ HPQ is stored in the wet state. However, wet resin can contain a substantial amount of water, which increases the mass of the resin/water mixture and decreases the concentration of chloride expressed as ppm. To have reproducible results that allow for meaningful comparisons the resin was dried in an oven at 60 °C until the weight stopped changing. That temperature was chosen based on a report by Wu, et al [1996]. At higher oven temperatures static effects make it difficult to work with the resin.

To relate measured ppm of chloride on an oven dry basis to expected ppm chloride on a fully wet basis one must measure the change in mass between oven-dry resin and fully wet resin. Samples of fully wet Reillex from two batches, Batch 80302 and Batch 50224AE, were filtered to remove drainable water. Then both samples were dried at 60 °C and periodically weighed. The results are shown in Figure 4. The weight of the two samples decreased by at least a factor of three. Because there was some weight loss associated with draining, the weight loss going from fully wet to oven dry would have been more than a factor of three. For the purpose of this report the weight ratio between fully wet Reillex™ HPQ and oven-dry Reillex™ HPQ will be assumed to

be three. Therefore, chloride concentrations measured on an oven dry basis will be divided by three to estimate chloride concentrations on a fully wet basis.



**Figure 4 Resin Drying**

ADS reported chloride concentrations in ppm and one sigma uncertainties (68% confidence) as shown in Appendix A.

### 3.0 Results and Discussion

#### 3.1 Measured Chloride Concentrations for Reillex Resin Not Treated at EDL

In 2004 SRS purchased two drums each containing one cubic foot of Reillex™ HPQ resin from ResinTech (Batch P9059). The resin performance was tested [Pierce 2012] and found to be satisfactory. A representative resin sample from each drum was dried and analyzed by NAA. (See the summary of NAA results in Appendix A.) Chloride concentrations for the partial and full drum were 248 ppm dry (~83 ppm wet)  $\pm 23\%$  and 583 ppm dry (~194 ppm wet)  $\pm 12\%$ , respectively. It is not known why there was a significant concentration difference between the two drums.

Kyser previously treated two carboys of Reillex™ HPQ resin (Batch 80302MA) using 1.0 M sodium nitrate at ambient temperature. EDL mixed the contents of the carboys, collected and dried a sample. ADS analyzed the sample using NAA. The resulting chloride concentration was 759 ppm dry (253 ppm wet)  $\pm 8\%$ . This is a higher concentration than for the resin purchased from ResinTech.

Kyser archived a sample of never used and never treated Reillex HPQ resin (Batch 50224AE). This sample was dried and the chloride concentration measured by NAA (sample 300295921) was 77,000 ppm dry, although ADS characterized this measurement as semi-quantitative. The reason for the large uncertainty is that NAA works best if the operator has been provided an

estimate of the concentration to allow a calibration in the approximate range. Recall that the theoretical chloride concentration for new, dry Reillex™ HPQ is 121,000 ppm.

After new, untreated Reillex™ HPQ was received from Vertellus (Batch 23408), a sample was dried and analyzed by NAA. This time ADS was advised of the approximate concentration to expect. The chloride concentration was 120,000 ppm dry  $\pm 4.3\%$ , which is close to the expected value.

### 3.2 Observations and Measurements for EDL Resin Treatment of Batch 80302MA Resin

Two batches of Reillex™ HPQ resin were treated in the EDL: two carboys of resin that Kyser had previously treated in 1998 (Batch 80302MA) and newly purchased resin from Vertellus (Batch 23408). The Batch 80302MA resin was retreated for three reasons: to make it consistent with the Vertellus resin, because the pedigree of the previous treatment was somewhat uncertain, and because it was thought that the concentration of chloride could be decreased. In addition, there was a third batch (P9059) of stored resin which had been purchased from ResinTech in the nitrate form.

Two 25 L carboys containing Batch 80302MA resin were characterized. First the supernate over the resin was characterized. Test strips showed that the chloride concentration was  $\sim 10$  ppm. Some of the supernate was evaporated and the dissolved solids were determined to be 4 wt %. Assuming the solute is sodium nitrate, these dissolved solids correspond to a 0.5 M solution. Conversion with nitrate solution was planned with 1.0 M solution, so this nitrate measurement offered the potential that more chloride could be driven from the resin. Then, the contents of the two original carboys were mixed in a 50 liter carboy and a Coliwas sampler was inserted to collect a representative sample. The resin sample was dried in an oven at 60 °C and was submitted for NAA determination of chloride, sample 295920. As was noted earlier, the concentration for the 80302MA resin was  $759 \pm 8\%$  ppm dry, where the uncertainty is based on one sigma or 68% confidence. Therefore, approximately 99.4% of the original chloride had been removed.

On February 2, 2012, the Batch 80302MA resin was loaded to the treatment column. Deionized water was pumped through the resin at a flowrate of 10.5 gal/h to verify system operation and the column discharge was sent to a Buchner funnel with a filter paper to catch any fines. The funnel started to fill and initially there was concern that fines were plugging the filter paper. Later inspection of the filter paper showed no fines. The Buchner funnel was replaced with a 1  $\mu\text{m}$  bag filter. At no point in the resin treatment process for either batch of resin were fines detected in the bag filter. A total of five gallons of water was pumped through the resin as part of system verification.

The water was drained from the column and on Feb. 6 one molar nitrate solution was pumped through the resin at a flowrate of 8 gal/h. Initially, the effluent from the column had a conductivity of 194  $\mu\text{S}/\text{cm}$  but it increased when the nitrate solution broke through the column. After 42 minutes, the conductivity increased to 20,000  $\mu\text{S}/\text{cm}$  and pegged the meter. Test strips showed that chloride concentration in the effluent never exceeded 20 ppm (0.8 units). Nitrate solution was pumped through the column for a total of four hours and 32 gallons (121 liters) or 3.5 BV. Less than 10 BV was pumped through the resin because the resin had been previously treated and the test strips showed that little chloride was being removed. On Feb. 7 the resin was rinsed with 24 gallons of deionized water. The final effluent conductivity was 230  $\mu\text{S}/\text{cm}$ . At the end of testing the resin was calculated to occupy 34 liters of volume in the column.

When the resin was removed from the column, samples were collected from the top, middle and bottom. After drying, the samples were submitted for NAA analysis. ADS reported measured chloride concentrations, Appendix A, which were 510 ppm dry  $\pm$  41%, 893 ppm dry  $\pm$  20% and 822 ppm dry  $\pm$  20%, respectively. The variation in measured concentrations may simply be the result of large measurement uncertainty. The nominal average concentration was 742 ppm dry ( $\sim$ 247 ppm wet), which is about the same as before re-treatment in the EDL. Therefore, little or no additional chloride was removed during the current treatment. Because the uncertainties listed above are so large, another NAA analysis with a larger sample and a longer counting time was performed on a mixed sample of resin and the result was 745 ppm dry ( $\sim$ 248 ppm wet)  $\pm$  3%. Therefore, the more accurate measurement using a mixed sample is fully consistent with earlier measurements of resin samples collected from top, middle and bottom of the column. The remaining resin was stored in a 50 liter carboy where it occupied 30 liters.

### 3.3 Observations and Measurements for EDL Resin Treatment of New Resin

On Feb. 21, 2012, some of the new untreated Batch 23408 resin was sampled, dried and sent for NAA analysis. The result (300297017) was 120,000 ppm dry  $\pm$  2%.

The treatment column was partially filled with water and then 50 kg of new resin was loaded. As was previously stated, this resin was expected to have a volume of 56 liters. However, the resin occupied more volume than expected and it was necessary to lengthen the column. The initial resin volume in the column was 75 liters and after treatment and rinsing the volume in the column was 72 liters.

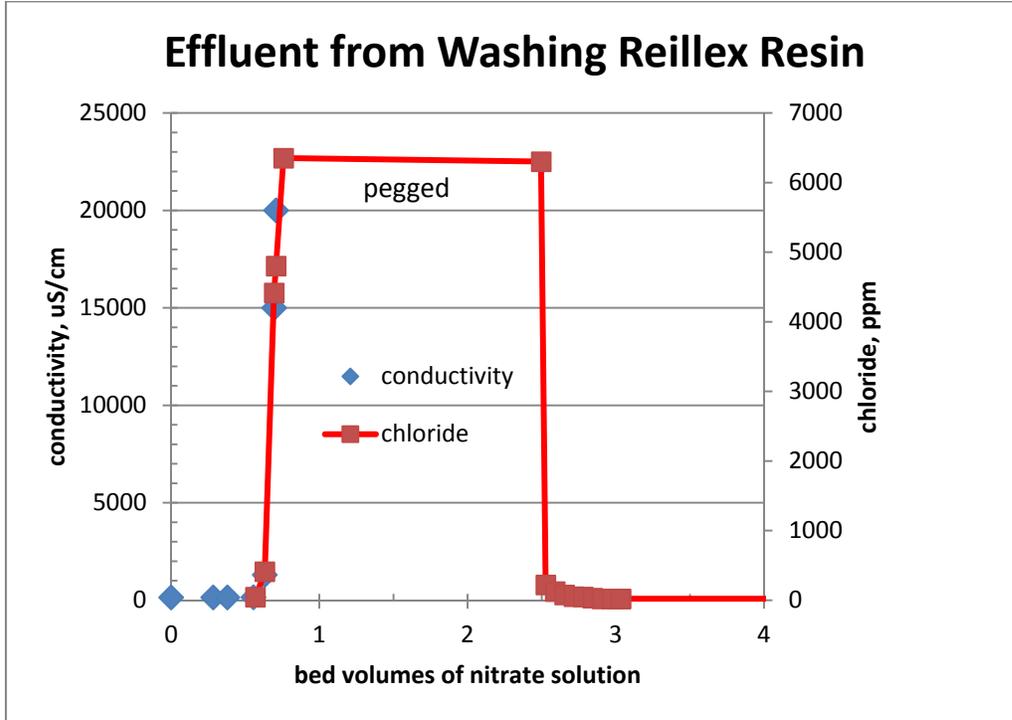
After loading the resin it was noted that the water at the bottom of the column had acquired a yellow tint. Some of the water was drained and was passed through a 0.2  $\mu$ m filter. The liquid still had the yellow tint and had a pH of 7 and a chloride concentration of 93 ppm. The liquid was then submitted for chemical analysis as shown in Table 2. The method abbreviations in the table are total inorganic carbon (TIC), total organic carbon (TOC), volatile organic analysis (VOA) and semi-volatile organic analysis (SVOA). To put the chemical analysis in perspective, after loading 50 kg of resin to the column, less than 1 gram of organic and inorganic material was observed to be released to column water.

**Table 1 Analysis of Yellow Tinted Column Effluent**

Component	method	Result	Result	1 sigma % uncer.	Units
total carbon	TIC/TOC		149	10	ug C/mL
inorganic carbon	TIC/TOC		6.4	10	ug C/mL
organic carbon	TIC/TOC		143	10	ug C/mL
Acetone	VOA		0.018	20	mg/L
2-butanone	VOA		0.083	20	mg/L
tetrahydrofuran	VOA		0.2	20	mg/L
all other analytes	VOA	<	0.005	20	mg/L
di-isooctyladipate (plasticizer)	SVOA		1.6	20	mg/L
all other analytes	SVOA	<	0.1	20	mg/L

On Feb. 23 the resin was rinsed with deionized water at 8 gal/h for 5.25 hours. On Feb. 27 treatment of the resin with one molar nitrate solution at 8 gal/h began. Conductivity and chloride concentration of the effluent were monitored and the results are shown in Figure 5. Both chloride and conductivity were low for the first 0.6 BV, then both increased sharply as nitrate solution broke through and chloride was driven from the resin. After 2.5 BV the chloride decreased

sharply when the chloride inventory of the resin became depleted. Conductivity remained high because sodium nitrate solution continued to be pumped through the column. At the same time that the chloride concentration decreased (at 2.5 BV) the column flowrate dropped by about one-third, suggesting that the resin bed flow resistance was 1.5 times as large, where flow resistance is proportional to pressure drop divided by flowrate. The throttle valve was readjusted to restore the target flowrate. This process of flow decrease and valve readjustment happened a total of four times, suggesting a total five-fold (1.5 to the fourth power) increase in flow resistance through the resin bed. Apparently a dimensional change in the resin during the treatment process increased flow resistance.



**Figure 5 Conductivity and Chloride Concentration of Effluent**

On March 6, an additional pressure gage was installed to allow measurement of frictional pressure drop across the column. On March 6 and 7 the resin was rinsed for a total of 10 hours at a flowrate of 8 gpm, and the frictional pressure drop was 3.25 psid. On March 8, one resin sample each was collected from the top and bottom of the column. When the resin was unloaded to a drum another sample was collected from the middle. Samples were dried and sent for NAA analysis. The analysis results, Appendix A, for the resin at top, middle and bottom of the column were 45.5 ppm dry (~15 ppm wet)  $\pm 22\%$ , 31.0 ppm dry (~10 ppm wet)  $\pm 30\%$  and 56.6 ppm dry (~19 ppm wet)  $\pm 17\%$ , respectively. The error bars overlap so there may not really be any difference in the three chloride concentrations. The average chloride concentration was 44 ppm dry (~15 ppm wet), therefore, the treatment of new Batch 23408 Reillex™ HPQ resin was very successful.

### 3.4 Resin Blending and Packaging

The original plan had been to blend all three batches of Reillex™ HPQ resin; new Batch 23408 resin, vendor treated Batch P9059 resin and archived Batch 80302MA resin. However, based on

the relatively large chlorine or chloride concentration for Batch 80302MA resin, only the other two batches were blended. The relatively dry Batch P9059 resin was added to the very wet Batch 23408 resin in a 55 gallon drum. In addition, some small samples of treated resin from those two batches were added. The Batch P9059 resin absorbed enough water so the resin would not blend readily. Therefore, five liters of deionized water were added to create a fluid mixture. The drum was turned on its side and rolled on the floor for 30 minutes. Then the drum was placed upright.

Resin was placed in five gallon buckets with sealing lids. The buckets had markings on the side for volume in quarts. These marks were calibrated by filling two buckets to the 11 quart mark and weighing the water and converting to liters. The two buckets had the same calibrations. The initial resin loading to buckets resulted in a variation in the proportion of resin and water based on appearance of the resin. Extra water was added to buckets having a dry appearance so that all buckets contained a fluid mixture of resin and water. Lids were attached to the buckets and they were gently rolled to allow air bubbles to disengage. Then resin was moved between buckets so that all have nearly the same volume of resin. Then excess water was removed so the liquid level was at the top of the resin with no extra supernate.

The net weight and resin volume were measured for each bucket and the results are listed in Table 2. The uncertainties for weight and volume are 0.5% and 5%, respectively. The uncertainty in bulk density is 5% and is dominated by uncertainty in volume.

**Table 2 Bucket Weights, Volumes and Densities**

bucket	mass, kg	nominal volume quarts	actual volume liters	density kg/L	
1	11.140	11	9.97	1.12	
2	11.405	11.5	10.42	1.09	before sample removal
2	9.240	9	8.16	1.13	after sample removal
3	11.870	11	9.97	1.19	
4	11.825	11	9.97	1.19	
5	11.875	11.25	10.20	1.16	
6	12.000	11.5	10.42	1.15	
7	11.670	11	9.97	1.17	
8	10.690	10.75	9.74	1.10	
9	11.175	11.25	10.20	1.10	
total volume L before 2 L sample			90.86	1.141	average density, kg/L
total volume L after 2 L sample			88.60	1.145	average density, kg/L

#### 4.0 Conclusions

Two batches of Reillex™ HPQ resin were treated at the EDL to replace chloride with nitrate: Batch 80302MA archived, previously treated resin, and new Batch 23408 resin. The re-treatment of Batch 80302MA archived resin was not successful in further reducing the concentration of chloride or chlorine, and the final concentration was 745 ppm dry or ~248 ppm wet. A likely reason for the lack of success was that chlorine was chemically bound to the resin and could not

be removed the treatment. The treatment of new Batch 23408 resin was much more successful. The chloride concentration in the new resin was decreased from 120,000 ppm dry to an average of 44 ppm dry (~15 ppm wet) or reduction by a factor of 2700. The method of manufacture of Reillex may have changed to decrease the amount of chlorine that cannot be removed. A third batch of resin, P9059, which had been vendor treated was shipped from H Area to EDL. The partial and full drums of this resin have chloride concentrations of 248 ppm dry (~83 ppm wet) and 583 ppm dry (~194 ppm wet), respectively. Batch 23408 and Batch P9059 have sufficiently low concentrations of chloride.

Per guidance of HB Line Engineering, SRNL blended Batch 23408 and Batch P9059. The blended resin was packaged in five gallon buckets with about 10 kg of wet resin each.

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## Appendix A Summary of NAA Results for Chloride

HB-Line, Reillex HPQ Resin, Neutron Activation Sample Results for Chloride									
Sample name	Chloride ppm, micro-gram / gram	percent uncertainty, 1 sigma	NAA Report Date	Sample ID	Batch number	oven dried?	Comments		
SRT-ATS-2001-00113	42.1	10	10/24/01	n/a	5109-36	no	Potassium nitrate conversion. Suction filtration performed before NAA.		
SRT-ATS-2001-00113	11	7	10/24/01	n/a	5109-36	no	Nitric acid, 8 M, 85C treatment. Suction filtration performed before NAA.		
spike	77,100	na	1/12/12	3002395921	50224AE	yes	purchased prior to 1998 and never converted		
P9059 Reillex partial	248	23	2/24/12	300237096	P9059	yes	Reillex stored in H-Area, partial container, washed by vendor		
P9059 Reillex full	583	12	2/24/12	300237097	P9059	yes	Reillex stored in H-Area, full container, washed by vendor		
P9059 Reillex partial	107	11	4/4/12	300238274	P9059	yes	treated with 1 molar NaNO <sub>3</sub> , then water rinse		
P9059 Reillex full	400	4	4/4/12	300238275	P9059	yes	treated with 1 molar NaNO <sub>3</sub> , then water rinse		
Reillex Nitrate NAA	84.2	36	12/18/11	3002395740	80302MA	no	Previously treated sample from E. Kyser. As received. Wet sample taken from one of two carboys. Resin purchased 1998.		
Carboy Reillex	759	8	1/12/12	3002395920	80302MA	yes	Previously treated sample from E. Kyser. As received after two carboys combined. Oven dried sample.		
top Reillex sample treated	510	41	2/14/12	3002367398	80302MA	yes	Previously treated sample from E. Kyser. After 2.9 BY of NaNO <sub>3</sub> , then water rinse. Sample from top of column before emptying.		
middle Reillex sample treated	893	20	2/14/12	3002367399	80302MA	yes	Previously treated sample from E. Kyser. After 2.9 BY of NaNO <sub>3</sub> , then water rinse. Sample from middle of column while emptying.		
bottom Reillex sample treated	822	20	2/14/12	3002368000	80302MA	yes	Partially washed sample from E. Kyser. After 2.9 BY of NaNO <sub>3</sub> , then water rinse. Sample from bottom of column before emptying.		
archived treated Reillex	745	3	4/4/12	3002382270	80302MA	yes	Composite sample of archived resin. Partially washed sample from E. Kyser. After 2.9 bed volumes of NaNO <sub>3</sub> added to 9 gallons of resin submerged in water.		
HBL Reillex, dry	164	33	2/24/12	300237007	5109/962F	yes	Reilly washed sample provided by E. Kyser for resin presently installed at HB-Line. Submerged in 0.48 molar NaNO <sub>3</sub> as received. Oven dried, although visually dry before heating.		
New Reillex untreated, dry	120,000	2	2/24/12	300237017	23408	yes			
new treated resin, top	45.5	22	4/4/12	300238271	23408	yes	treated with 1 molar NaNO <sub>3</sub> , then water rinse		
new treated resin, mid	31	30	4/4/12	300238272	23408	yes	treated with 1 molar NaNO <sub>3</sub> , then water rinse		
new treated resin, bottom	56.6	17	4/4/12	300238273	23408	yes	treated with 1 molar NaNO <sub>3</sub> , then water rinse		

**Distribution:**

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