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CLEANUP OF TMI-2 DEMINERALIZER RESINS

W. D. Bond L. J. King J. B. Knauer

Oak Ridge National Laboratory* P. O. Box X Oak Ridge, Tennessee 37831

K. J. Hofstetter

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GPU Nuclear Corporation TMI Power Station Middleton, Pennsylvania 17057 DE85 011431

J. D. Thompson

EG&G Idaho, Inc./TMI P. O. Box 88 Middleton, Pennsylvania 17057

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CLEANUP OF TMI-2 DEMINERALIZER RESINS

W. D. Bond, L. J. King, J. B. Knauer Oak Ridge National Laboratory, P. O. Box X, Oak Ridge, TN 37831

K. J. Hofstetter

GPU Nuclear Corporation, TMI Power Station, Middleton, PA 17057

J. D. Thompson

EG&G Idaho, Inc./TMI, P. O. Box 88, Middleton, PA 17057

Radiocesium is being removed from Demineralizers A and b (DA and DB) by a process that was developed from laboratory tests on small samples of resin from the demineralizers. The process was designed to elute the radiocesium from the demineralizer resins and then to resorb it onto the zeolite ion exchangers contained in the Submerged Demineralizer System (SDS). The process was also required to limit the maximum cesium activities in the resin eluates (SDS feeds) so that the radiation field surrounding the pipelines would not be excessive. The process consists of 17 stages of batch elution. In the initial stage, the resin is contacted with 0.18 M boric acid. Subsequent stages subject the resin to increasing concentrations of sodium in $NaH_2BO_3-H_3BO_3$ solution (total B = 0.35 M) and then 1 M sodium hydroxide in the final stages. Results on the performance of the process in the cleanup of the demineralizers at TMI-2 are compared to those obtained from laboratory tests with small samples of the DA and DB resins. To date, 15 stages of batch elution have been completed on the demineralizers at TMI-2 which resulted in the removal of about 750 Ci of radiocesium from DA and about 3300 Ci from DB.

This paper discusses: (1) the development of a process for removing the cesium activity from the makeup and purification demineralizers at the Three Mile Island Nuclear Power Station, Unit 2 (TMI-2), and (2) the results obtained to date using the process at TMI-2. The removal of cesium from the TMI-2 demineralizers is still in progress and about 4000 Ci of 137 Cs have been removed. The process flowsheet was largely based on experimental results obtained in hot-cell experiments at Oak Ridge National Laboratory (ORNL) using rather small samples of the demineralizer

resins which were provided by General Public Utilities Nuclear Corporation (GPU Nuclear). The process consists of eluting the cesium from the resin and then sorbing it on the zeolites in the Submerged Demineralizer System (SDS). The demineralizer cleanup activities reported here were performed as part of a ntinuing program at TMI-2 for decontant.ation of the reactor system components.

Conceptual studies of the various alternative methods for cleanup of the demineralizers (1) indicated that the most desirable method was elution of the cesium and subsequent sorbing it on the zeolites in the Submerged Demineralizer System (SDS), provided experimental work with samples of resin indicated it to be feasible. This concept alleviated the high-level radiation problems associated with the eventual removal of the degraded resin beds and with management of the resin wastes. In elution, it was necessary that chemical reagents be employed that were compatible with the ionic solution chemistry of SDS feed solutions. This requirement dictated that Cs elution be accomplished by its displacement with sodium ions using sodium borate or sodium hydroxide solutions. The elution process was also required to limit the cesium activity in eluates to levels that were no greater than about 1 mCi/ml. At this maximum activity level, the planned dilution (maximum of 20/1) of eluates in an eductor device or my pump mixing methods produces feeds that pose no excessive radiation fields around the SDS pipeline.

As a consequence of the accident at TMI-2 on March 28, 1979, the two demineralizers (DA and DB) in the water makeup and purification system were severely contaminated with fission product radionuclides.

Since the time of the accident, most of the radionuclides have decayed and caused significant radiolytic and thermal degradation to the resin beds in the demineralizers. The principal radionuclide activity remaining on the resin beds when demineralizer cleanup activities began is due to the relatively long lived 137Cs (t_{1/2} = 30.1 Y) and 134Cs $(t_{1/2} = 2.06 \text{ Y})$. Prior to the inception of the present investigation, nondestructive assay (NDA) methods had been employed to estimate the quantity of cesium activity and material content of each demineralizer (Table I) (2). Comparison of the post accident resin bed volumes with that of preaccident showed severe shrinkage (~55%) of the resin beds had occurred and indicated that the beds had been significantly degraded as expected. It was known that the beds had not only been subjected to high radiation dosages ($\sim 10^9$ rads) but also to high temperatures because of the radioactive decay heat. The necessity to isolate the demineralizers from liquid flow at about 19 h after the accident prevented effective removal of the decay heat and estimates indicate that centerline bed temperatures may have been as high as about 540°C (1000°F). The demineralizers were sampled by GPU Nuclear personnel in early 1983 and it was observed that the DA vessel contained only dry, caked, resin. However, the caked bed was apparently deagglomerated after water addition and sparging so that resin samples were obtained and liquid-resin contact was improved for the subsequently planned elutions of the resin bed.

Since the effects of the resin bed degradation were unknown with regard to the quantitative elution behavior of cesium and the quality of

Loadings	Initial	DA vessel	DB Vessel
Resin		 	
Volume, ft ³	50	22	22
Weight, lb	2,139	1,025	1,025
$137C_{\rm s}$, C1	0	3,500	7,000
¹³⁴ Cs, Ci	0	270	540
Liquid			
Volume, ft ³	44	3	3
Weight, 1b	2,746	193	193
Debris			
U, 1b		5	1
Core debris, 1b		95	19
137Cs, Ci		177	35
¹³⁴ Cs, Ci		16	3
¹⁰⁶ Ru, C1		21	4
144Ce, C1		28	5
125 Sb. C1		116	23
TRU, Ći		0.5 ^b	0.1b

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Table I. Estimated Demineralizer Vessel Loadings Based NDS Characterizations^a

^aData from M. K. Mahaffey, et al (2). b_{α} activity only.

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the resultant eluates; it was necessary to obtain samples of the degraded resins and experimentally determine the feasibility of eluting the cesium and then fixing it on the zeolites in the SDS system. Even if cesium elution proved feasible, it was not certain that eluates could be satisfactorily processed in the SDS. Cesium loading of the SDS zeolite beds might be seriously impaired by the presence of degradation products in eluates, depending on their nature and quantity of degradation present in the eluates. Finely dispersed solids (or colloids) from resin particle breakage and degradation may not be readily separable and cause plugging of the zeolite bed. Soluble organic compounds and emulsified oils might be sorbable on zeolites and foul or block the exchange sites for cesium.

Process Flowsheet Description and Process Operations

The schematic process flowsheet is shown in Figure 1. After removal of the existing liquid in the demineralizers, cesium is eluted in stage wise batch contacts of the resin with the eluent solutions. As elution proceeded, the sodium concentrations of the eluent solution is increased from 0 to 1 M (0 to 23,000 ppm Na) to facilitate elution of the residual cesium on the resin. Liquid-to-solid phase volumetric ratios of ~1.5 are used in each stage which is governed by the available free volume in the demineralizer vessels. Batch contacts of the liquid eluent solutions with the resin was accomplished by upflowing the liquid into the demineralizer and then air sparging the liquid above the resin bed to promote mixing for 8 h. The air sparging was then stopp and the 24 to 72 h was allowed for the settling of suspended solids. Typically 3 to 4 F



Figure 1. Flowsheet for Elution of the TMI-2 Makeup and Purification Demineralizer.

drums (55-gal each) of eluent solution were added per batch content. Eluates were withdrawn through a suction hose from a depth about 1 ft below the liquid surface to ensure against the withdrawal of any solids which might be floating on the liquid. The removal was accomplished using either an eductor or a pump to lift the liquid. The eluates are then filtered through a sintered, stainless steel, filter frit (pore size 20 μ m) before being diluted with processed water (800 ppm B), transferred to inplant neutralizer tank storage, and finally processed through the existing SDS. The SDS has been previously described. (3,4)

Dilution was necessary to minimize personnel exposure during transfer through piping to in-plant tanks. Most of the eluates were transferred from the demineralizer using the eductor which diluted the eluates (up to 20-to-1) before filtration. Flow totalizers were used to measure volume of liquid transfer and flow of eluate from the demineralizer would continue until the pump or eductor lost suction leaving a considerable residual volume (V_T) of eluate heel in the demineralizer. The values of V_T remaining in the DA and DB vessels after transfers were 145 and 175 gal, respectively.

Samples from the neutralizer tank were analyzed after each batch elution. Routine analyses included determination of 137Cs by γ spectrometry, 90Sr by β -counting, and sodium by atomic absorbtion. The progress of the batch elutions were monitored from the determination of the concentrations and volumes in the neutralizer tanks before and after transfer of batch eluates.

Laboratory Development Studies

Laboratory-scale experiments on the development of the process flowsheet included: (1) batch elution tests to determine the feasibility of Cs elution by sodium ion displacement; (2) tests with small-zeolite beds to indicate the feasibility of satisfactorily processing eluates in the SDS; (3) studies of eluate clarification by settling and by filtration through sintered metal filters. The results of these experimental studies provided the technical basis for the process flowsheet (Figure 1) for the cleanup of the demineralizers. The DB sample was available about 1 y before a satisfactory DA sample was obtained and therefore most of the elution flowsheet conditions were developed using the DB resin.

Description and Analysis of Demineralizer Samples. Samples from the DA and DB vessels consisted of resin mixed with liquid. The DA sample contained about ~20 ml of resin and ~50 ml of liquid and the DB sample contained about 40 ml of resin and 80 ml of liquid. The resin was separated from the liquid by settling. Most of the resin settled very rapidly. The resin was then dried in air at ambient conditions (25-30°C). In some cases the separated liquid was further clarified by centrifugation before analysis. The resin and liquid were analyzed by chemical and radiochemical methods. The resin was also examined by visual microscopy at ~20X magnification to assess its physical characterization.

Chemical and radionuclide analyses of the resin and liquid phases of the DA and DB samples are given in Table II. Only the principal constituents have been listed in Table II. Small quantities (10-1000 ppm) of many other metal cations were also present in the resin but are not relevant to the work reported here.

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	D	A	DB		
Analyses	Liquid ^a	Resin	Liquid ^b	Resin	
Radionuclides, µCi/g	<u></u>				
137 _{Cs}	209	5,520	1,480	21.800	
134Cs	11	285	101	1.458	
90Sr	6.72	3,060	9.46	890	
Chemical, ppm					
C	164		9 50		
В	1,000	20	2,000	>200	
Na	500	4,900	8,500	>1,000	
S04c	900	29,000	7,700	15,000	
II II	<1	2,400	10	200	
Fe	4	2,400	10	200	
Ca	3	970	15	30	
Ba	-	240		<1	

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Table II. Radionuclide and Chemical Analyses of the Demineralizer Samples

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apH = 7.1. bpH = 5.7. CSulfur was determined as sulfate.

Microscopic examinations revealed that the DA resin was more severely damaged than the DB resin (Figure 2). The DA resin contained large angular particles and clustered resin beads in significant amounts, perhaps 5 to 10 vol % of the sample. However, the remainder of the particles were clearly distinguishable at individual resin beads with colors ranging from black (non-transparent) to amber (transparent). The angular particles had an appearance that is typical pyrolytic carbon derived from tars, pitches, and polymeric resins. The angular particles were not observed in the DB resin.

The DB resin principally contained only resin beads which were black or amber colored. Very few bead clusters were observed. In some cases, there was partial spalling of the surface layer of the black beads and the blackening only appeared to occur to a depth of a few micrometers.

Liquids separated from the demineralizer samples were visually of good clarity but yellowish brown in color. It is later shown in further liquid clarification tests that the solutions were slightly turbid even after settling times of 1 to 4 days.

<u>Multistage Batch Elution Tests</u>. Results from multistage batch tests which demonstrated a satisfactory method for Cs elution are shown in Table III and Figure 3. The elution process consisted of 17 contact stages in which a sodium ion concentration gradient was employed to limit the cesium activities of the eluates to a maximum of ~1000 μ Ci/ml (Table III). The resin was initially rinsed with 0.18 M H₃BO₃ in the

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ORNL Photo CYN 5747 DA Resin

Figure 2. Photomicrographs of the Demineralizer Resins (20X Magnification)

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Table III. Elution of ¹³⁷Cs in Multistage Batch Elution Tests with the DA and DB Resins

Conditions: 15 ml of eluent solution contacted with ~10 ml of resin for 30 min; air dried resin weights were 6.56 and 7.22 g for the DA and DB resin, respectively.

	ب هم به المراجع ا	¹³⁷ Cs c	onc. in	$K_{d}, \frac{\mu Ci/g}{\mu Ci/ml}$		
Stage		Eluate,	μCi/ml			
Stage Number 	Eluent Solution	DA	DB	DA	DB	
1	0.14 M H ₃ BO ₃	96.8	1090	54.8	17.9	
2	0.035 M NaH2 BO3-0.32 M H3BO3	136	340	36.7	55.4	
3		135	420	34.7	42.8	
Ļ	0.26 M NaH2BO2-0.09 M H3BO3	245	1230	16.8	12.5	
5		185	1140	20.0	11.4	
6		122	1050	28.0	10.3	
7		78.1	870	41.5	10.5	
8		67.3	757	45.9	10.0	
9		51.9	632	57.2	9.9	
10		37.0	516	78.0	10.1	
11		25.5	405	111	10.8	
12		18.1	343	154	10.9	
13	1 M NaOH	64.3	432	41.0	6.6	
14	-	42.7	395	59.5	5.1	
15		25.7	264	96.5	5.6	
16		16.0	175	153	6.4	
17		21.1	97	200	9.7	

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Fig 3

first stage and then the sodium ion concentration of the eluent was increased in the succeeding elution stages from 0.035 to 1 M through the use of partially neutralized boric acid solutions (NaH₂BO₃), and finally 1 M NaOH.

The elution of 137 Cs was 96% from the DB resin after 17 stages but was only about 56% from the more degraded DA resin (Figure 3). The residual specific activities of 137 Cs remaining on the DA and DB resins was 2.4 and 0.9 mCi/g, respectively. Partial elution of the 90 Sr also occurred and about 25% was eluted from either resin. The distribution coefficient (Kd) values of 137 Cs for the DB resin were relatively constant throughout the stage-wise contacts using the same sodium concentration whereas a significant increase in Kd values for the DA resin occurred. The difference in elution characteristics of 137 Cs can be attributed to the greater damage suffered by the DA resin.

To determine if the large clustered and carbonized particles in the DA resin sample contained most of the uneluted 137 Cs, the eluted resin bed was air dried and classified into three fractions by screening which were analyzed. Only about 12 to 14 particles were present in the >2000 µm fraction, about 10 vol % in the 850-2000 µm fraction, and about 90 vol % in the <850 µm fraction. The specific activities of 137 Cs were 4.1, 4.7, and 2.4 mCi/g for the large-, intermediate-, small-size fractions, respectively. Although the larger particles are of higher specific activity, most of the uneluted 137 Cs (~80%) is associated with the smallest size fraction.

Organic compounds were solubilized throughout the 17 stages of batch contacts (Figure 4). However, the carbon compounds were only

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solubilized to an extent of about 8 mg of C/g of resin in 17 stages of contact. Eluates from the elution of the DA and DB showed no significant difference in dissolved organics. No attempt was made to identify these compounds.

The clarity of eluates appeared generally good by visual observations. However, some cloudiness was observed in the first stages of elution with the DB resin and during the final treatment with 1 <u>M</u> NaOH with either the DA or DB resin. The eluates from stages 1 and 4 of the DB resin elution were filtered through a 0.5 M-rated nylon filter and the filter was scanned by γ spectroscopy (Table IV). Only small quantities of radioactivity were observed that was principally due to the 137Cs and with much lesser amounts of ¹²⁵Sb, ¹⁴⁴Ce, and ⁶⁰Co). The observed activity of ¹³⁷Cs was about 0.01% that of the filtered solution (~400 MBq).

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Radionuclide	Activity, Bq
60 _{Co}	27
125 Sb	310
134 Cs	1.66×10^3
137 _{Cs}	2.69×10^4
144Ce	52

Table IV. Gamma Scan of Filter Solids from a DB Resin Eluate

Zeolite Bed Tests. Decontamination factors (DF's) for 137 Cs and 90 Sr were determined in the zeolite beds tests with resin eluate feeds and with the liquid separated from the as received DA sample (DA liquid). Tests consisted of passing 70-100 bed volumes of feed through a 2 ml bed of mixed zeolites (60 vol % Ionsiv 96 and 40 vol % Linde A-51). The

superficial bed residence time was maintained constant at 8.4 min throughout each test, a value which is comparable to that (~10 min.) used in SDS processing at TMI-2.

Results of the tests are shown in Table V. The cumulative DF's for 137 Cs ranged from about 5 \times 10³ to 3 \times 10⁵ and for 90 Sr from about 200 to 500. The values of the DF's and their variation with the feed concentrations of 137 Cs and 90 Sr are consistent with previous studies (<u>3,4</u>) on the performance of the SDS zeolites. The DF of 137 Cs for the DA liquid feed was slightly improved by filtering it with a 10- μ rated filter frit, perhaps indicating a very small percentage of the 137 Cs (0.01%) in the feed was associated with the nonsorbable and finely dispersed solids that were present. No evidence of bed plugging was noted in any of the tests though the feeds for Runs Z-1, Z-2, and Z-3 were not filtered.

Some sorbtion of the soluble organic compounds in the eluate feeds on the zeolite bed occurred. The cumulative breakthrough for the organic compounds for the DA and DB resin were 45 and 75%, respectively. However, there was no significant change in the DF for 137 Cs as the bed loading of organics increased in a near liuear fashion during the tests.

Liquid Clarification. Clarification of the DA liquid was investigated by filtration and by settling. Filtration tests were conducted with sintered metal (stainless steel) filter disk with ratings of 0.5 and of 10 μ m at filtration rates of 2.2 ml/cm²/min. This flow rate was the design basis for the filtration system to be used at TMI-2 which also provided for periodic backflushing when the filter pressure drop reached

Table V. Cumulative Decontamination Factors for ¹³⁷Cs and ⁹⁰Sr in Zeolite Bed Tests

Conditions: 2 ml mixed zeolite bed (60 vol % Ionsix 96-40 vol % Linde A-51); residence time = 8.4 min

	Feed	Solution	8	Effluent	:				
Test	Source	¹³⁷ Cs, μCi/ml	⁹⁰ Sr, µCi/ml	Total C, mg/ml	bed volumes	C137C	umula s	90Sr	DF
Z-2	DB resin eluates; ^a Stages 1-13	346	0.14	80	80	1.50	E2p	5.11	E2
Z-3	DA resin eluates; ^a Stages 1-13	97	32.5	130	70	>3.48	E 5	4.96	E2
Z-4	DA liquid	10	0.26	8	100	4.34	E3	2.60	E2
Z-5	DA liquid; filtered through 20 μ metal	8	0.18	12	100	1.99	E 4	2.03	E2

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^aFrom elutions described in Table III. ^bRead as 1.5×10^5 .

20 psi. The liquid used for the filtration and settling tests was separated from the DA sample after 8 h of settling of the initially well stirred sample and was subsequently diluted 20-to-1 with 0.18 \underline{M} boric acid (2000 ppm B).

The filtration test results (Table VI) showed that good clarification was obtained using either the 0.5 or the 10 μ -rated filters. The turbidity was decreased from 12.6 to about 2 nephleometric turbidity units (NTU's) using either filter. The 0.5 μ m-rated filter rapidly plugged (16 to 19 min) whereas the 10 μ m rated filter was more slowly plugged (170 min). On stream filtration times of 170 min were judged marginal for the clarification operations at TMI-2 with backflushing and a 20 μ -rated filter was installed at TMI-2 as an additional precaution. However, it remained to be demonstrated in the actual operations at TMI-2 that the filter could be satisfactorily restored by backflushing. The volume of samples available was too small to permit investigation of filter backflushing or of extensive studies of filtration.

Settling tests (Table VII) showed that clarification could be significantly improved by using longer settling times. Therefore, relative long settling times after batch contacts of resin and eluents could be employed to reduce filter loadings in eluate clarifications and to increase liquid throughputs before filter pluggage occurred. Relatively long settling times (24 to 72 h) were used in the processing of the demineralizer B eluates at TMI-2 which is discussed later.

An attempt was made to increase the settling rate by using fresh anion exchange resin (Amberlyte IRN-400; 20-40 mesh) as a flocculation

Table VI. Clarification Tests with Sinter Metal Disk Filters using DA Liquid

Run Number	Filter rating (µm)	Time (min)	Final pressure drop (ps1)	Volume	Turbidity ^a NTU's
I	0.5	19	20	30	2.1
2	0.5	16	20	25	2.3
3	10	170	14 ^b	270	2.2

Conditions: 9.5 mm-diam filter disk, filtration rate = 2.2 ml/cm²/min; DA liquid diluted 20-to-1 with 0.18 <u>M</u> H₃BO₃

^aTurbidity of feed was 12.6 NTU's.

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^bFeed supply was used up. Extrapolation of pressure drop vs. time curve indicated that 20 psi would be reached after 190 min and a filtrate volume of ~300 ml.

	Turbidity	, NTU's
Settling Time ^d	With flocculant addition	Raw liquid
0	12.6	12.6
1	5.2	6.1
2	4.4	5.5
3	3.5	4.6
4	2.8	4.0

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Table VII. Effect of Settling Time on DA Liquid Turbidity

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aid in one test. However, it did not significantly improve the rate. In that test, 1 g of the resin was added each day to the same 35 ml aliquot of liquid.

Processing Results at TMI-2

After 15 stages of batch elutions, approximately 750 and 3300 curies of 137 Cs have been eluted from DA and DB, respectively (Table VIII). — Process flowsheet operations have been generally satisfactory with some difficulty encountered because of frequent filter plugging when eluting DB with sodium concentrations above about 0.25 <u>M</u> (8000 ppm). The filter plugging was minimized and satisfactory filtration rates were obtained by increasing the settling time of the eluates from the nominal 24 h to 72 h and by addition of boric acid to reduce solution alkalinity. It is believed that the filter plugging is due to degraded resin product particles that are more effectively dispersed at the highly alkaline conditions. This phenomenon was not observed in the DA elution. Results from the processing of the filtered eluates through the SDS system has been completely satisfactory.

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Based on the NDA estimates of the 137Cs demineralizer loadings in Table I, about 22 and 46% of the 137Cs has been eluted from DA and DB, respectively (Figure 5). The removal of 137Cs was about one-half of that expected on the basis of the hot-cell tests at ORNL if it is assumed that the samples tested were representative of the bulk resin from each demineralizer resin bed. The liquid-resin contact was no doubt better in the hot-cell tests because the resin was slurried with the liquid via shaking. Nevertheless, the trends in 137Cs elution behavior with increasing sodium

	Demineraliz	er A	Demineralizer B			
Stage Number	Cumulative ¹³⁷ Cs removal, Ci	Sodium conc. of eluent, <u>M</u>	Cumulative ¹³⁷ Cs removal, Ci	Sodium conc. of eluent, M		
1	119	0 ^a	811	0 ^a		
2	201	0 ^a	932	0 ^a		
3	206	0.07	1190	0.07		
4	395	0.34	1510	0.07		
5	464	0p	194 0	0.34		
6	+99	0,23	2000	0.34		
7	532	Ор	2180	0р		
8	562	0,23	2420	0.30		
9	608	0.74	264 0	0.30		
10	630	0.38	284 0	0.23		
11	646	0р	2920	0.30		
12	706	0.38	3070	0.51		
13	711	0 .9 0	3170	0.51		
14	745	1.0	3240	0.59		
15	745	1.0	326 0	0.59		

Table	VIII.	Cesi	um	Remov	vals	Ace	compli	shed	after	15	Stages	of
	1	Batch	Elu	tion	of	the	TMI-2	Make	eup and	1		
		Р	hri	ficat	tion	Der	ninera	lize	rs			

^aProcessed water containing 0.18 M H₃BO₃. ^bRemoval of filter backflush liquid.

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Figure 5. Percentage ¹³⁷Cs Eluted in the Multistage Batch Elution of the TMI-2 Demineralizers.

concentration are expected to be the same. The in-plant results from the DB resin elution show the expected trend but the LA results do not, particularly at the higher sodium concentrations approaching 1 M. The results to date indicate little additional 137 Cs can be removed by continuing the DA elutions with 1 M sodium concentrations. Thus it appears that either the original curie loading estimates for DA (Table I) are high or the resin sample used in the hot-cell elution tests is not representative of DA resin bed.

In resin sampling operations with the demineralizers, it was found by quartz fiber optical examination of the DA resin bed that it was dry which is contrary to the wet resin bed model used to interpret the NDA data. The possil lity that the original estimate of the 137 Cs loading on DA is high is currently being explored in calculations using a revised source term model to interpret the NDA data. No attempts at further elution of DA are planned pending outcome of the reassessment of the 137 Cs loading estimate and radiation level surveys of the DA vessel. Elutions will continue for DB at higher sodium concentrations to further reduce the cesium activity. Following the completion of the cesium elution operations, dose rate surveys of the demineralizer cubicles will be performed to confirm final resin elution efficiencies and the dose rate reductions that were achieved.

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