Duolite™ GT-73 Resin Testing in Support of the Salt Disposition Alternatives

by

W. R. Wilmarth

Westinghouse Savannah River Company

Savannah River Site

Aiken, South Carolina 29808

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W. R. Wilmarth

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Westinghouse Savannah River Company
Savannah River Technology Center
Aiken, SC 29808

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Duolite™ GT-73 Resin Studies in Support of Salt Disposition Alternatives

W. R. Wilmarth
Waste Processing Technology Section
Savannah River Technology Center
Westinghouse Savannah River Company

Summary

This study evaluated Duolite™ GT-73 performance for removing mercury ions from several high level waste streams under consideration by the Salt Disposition Systems Engineering Team as a technical risk. Experiments conducted over an eight week period address the technical uncertainties for GT-73 performance. Conclusions from the experimental data indicate the following.

- Duolite™ GT-73 cation resin adequately removes mercury from simulated salt solutions with a K_d above $1E5$ mL/g.
- Extended exposure (i.e., eight weeks) to strongly caustic solutions does not structurally alter the Duolite™ GT-73 resin as measured by particle size analysis, scanning electron microscopy, and determination of mercury removal efficiency from simulated waste.
- Duolite™ GT-73 will bind radioactive Cobalt-60 with a K_d of 350 mL/g in these simulated waste solutions.
- Mercury does not displace Co-60 when loading the resin to less than the theoretical capacity.
- Duolite™ GT-73 resin effectively removed the mercury from a Tank 46H composite sample. Measured K_d 's agree with results previously published by Bibler and Wallace.

Introduction

Duolite™ GT-73 is a macroporous, weakly acidic polystyrene/divinylbenzene cation exchange resin with thiol functional groups that have a pronounced selectivity for mercury.^{1,2} Rohm and Haas currently manufactures the material. The GT-73 resin finds use at the Savannah River Site (SRS) in the Effluent Treatment Facility³, in the wastewater treatment facility at TNX,⁴ and for laboratory wastes.⁵ The selection of alternatives for the pretreatment of high level waste involves several possibilities (i.e., grouting, solvent extraction, tetraphenylborate precipitation, and non-elutable ion exchange).⁶ Several of these options rely on cation exchange to remove mercury. The GT-73 resin appears as the most promising candidate resin. However, insufficient understanding exists regarding the stability of the resin in strongly caustic media. The tests described below examine the stability of the GT-73 resin, the resin's affinity for Co-60, and its effectiveness in radioactive waste conditions.

As a result of these technical uncertainties, the Salt Disposition Systems Engineering Team identified the performance of Duolite™ GT-73 as a technical risk for several of the processing alternatives selected for additional evaluation in Phase III of their effort. The Flow Sheet Team requested that the Savannah River Technology Center examine the performance of the resin during simulated process alternatives.⁷

Experimental

Caustic Exposure Tests

These tests examined whether Duolite™ GT-73 cation resin would effectively remove mercury from simulated high-level waste streams containing high concentrations of free hydroxide ion. These tests added Duolite™ GT-73 cation exchange resin to a sodium salt solution containing the composition shown in Table 1. This salt solution included mercury (Hg) added at a nominal concentration of 70 mg/L. After addition of the resin (usually 0.05 g), the test protocol agitated the mixture for a period of 72 hours using a magnetic stirrer. Solution withdrawn from the sample and filtered using a 0.45 micron syringe filter (nitrocellulose) was submitted for mercury analysis using Cold Vapor Atomic Absorption.⁸

Table 1. Sodium Salt Solution Composition

Chemical	Conc. (M)	Target (g)
Al(NO ₃) ₃	0.2	75.06
KNO ₃	0.036	3.64
NaNO ₂	0.7	48.3
Na ₂ SO ₄	0.4	56.8
Na ₂ CO ₃	0.28	34.7
NaOH	3.6	144
NaCl	0.33	19.2

Co-60 Affinity Tests

Researchers conducted a set of equilibrium distribution, K_d , tests using a simulated salt solution containing radioactive Co-60. The Analytical Development Section provided the Co-60 solution (analyzed to contain $\sim 10^4$ dpm/mL). Tests added a 5-mL aliquot of this Co solution to 50 mL of the 6 M Sodium salt solution from Table 1 and equilibrated by stirring for 4 h. The Analytical Development Section analyzed duplicate initial samples by gamma spectroscopy.⁹ Tests added 30 mL of the stock solution and placed in a 35-mL polyethylene bottle containing approximately 0.1 g of Duolite™ GT-73 resin. The mixture was stirred using a magnetic stirrer for at least eight hours. Researchers removed a liquid sample by syringe, filtered using a 0.45 micron nitrocellulose syringe filter, and submitted the filtered sample for gamma counting.

A second set of experiments ascertained the affinity for Co-60 on Duolite™ GT-73 resin in competition with mercury. These tests added x mL of the Co-60 solution to x mL of the 6 M sodium stock solution described in Table 1 and repeated the K_d tests described in the previous paragraph. These tests used 15 mL of solution placed in a 35-mL polyethylene bottle containing approximately 0.05 g of resin.

Real Waste Verification Tests

Researchers analyzed several archived F- and H-Area evaporator samples for mercury content by Cold Vapor Atomic Absorption. Tank 46H contained the highest concentration (11 mg/L), although lower than the simulated waste solutions used in this study. A Waste Treatability Study¹⁰ permitted these tests of the effectiveness of Duolite™ GT-73 resin for mercury removal. The first experiment added duplicate samples (15 mL) of the Tank 46H material to separate 65-mL polyethylene bottles containing approximately 0.05 g of the Duolite™ GT-73 resin and the mixture was agitated for a period of 48 h. Another set of experiments added an aliquot (5 mL) of a mercury solution (~ 2200 mg/L) to 45 mL of the Tank 46H composite. Two tests used this mixture. Researchers submitted samples of the mercury-spiked Tank 46H solution for mercury analysis by Cold Vapor Atomic Absorption. Samples taken from the polyethylene bottles after treatment received mercury analysis by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).¹¹ The ICP-MS analysis was used because the samples were diluted (100:1) prior to removal from the Shielded Cells.

Quality Assurance

Laboratory notebooks WSRC-NB-97-61 and WSRC-NB-98-211 contain data obtained during these tests and the procedures used. Personnel used routine analytical protocol for the samples in this report.¹² The author analyzed the hazards of this experimental program and mitigated these per the protocols defined in the Conduct of Research and Development Manual.¹³

Experimental Results

Caustic Exposure Tests

The tests studied the stability and effectiveness of the Duolite™ GT-73 cation exchange resin to remove mercury from simulated high-level waste streams containing high concentrations of free hydroxide ion for a period of eight week exposure. Analyses performed on the GT-73 resin and samples included equilibrium distribution coefficient (K_d) measurements, particle size analysis, and scanning electron microscopy.

The equilibrium distribution coefficient (K_d) is determined by adding approximately 0.05 g of the sodium form of the Duolite™ GT-73 cation exchange resin to a sodium salt solution containing 70 – 80 mg/L mercury. Personnel stirred the salt solution and resin contained in polyethylene bottles for a least 72 h with the exception of first sample which received only 24 h agitation. Results from experiments with shorter agitation times indicate non-equilibrium conditions. Equilibrium distribution coefficients are calculated as follows.

$$K_d \text{ (mL/g)} = ([\text{Hg}]_i/[\text{Hg}]_f - 1)(\text{volume of solution/weight of resin})$$

Table 2 contains the data generated during these tests.

Figure 1 shows the data from Table 2. Although the data exhibit scatter, the trend suggests no significant degradation of the resin's ability to decontaminate the mercury-containing salt solution over the eight week period. The values of 0.01 mg/L in the final mercury solutions indicate the lower limit of detection of the Cold Vapor Atomic Absorption analysis. For samples with mercury concentrations at or below the detection limit, the values distribution coefficients cited in the table represent minimal values (i.e., $K_d \geq$ value shown). The K_d values for the initial sodium form of the resin measured $\geq 6.76 \text{ E } 6 \text{ mL/g}$. The K_d after seven weeks of exposure to strongly caustic solution measured $2.04 (\pm 0.74) \text{ E } 6 \text{ mL/g}$.

Previously, Bibler and Wallace¹⁴ examined ion exchange for decontamination of High Level Waste supernate. Bibler and Wallace utilized a thiol-based resin, Duolite™ ES-465, in their tests. This resin closely resembles the Duolite™ GT-73 utilized in this study. The earlier work used comparable experiments. The equilibrium distribution coefficient, K_d , calculated by Bibler and Wallace ranged from $1.84 \text{ E } 4$ to $2.45 \text{ E } 4 \text{ mL/g}$. The results of the current tests show a much higher K_d for mercury removal using GT-73 resin. Fong¹⁵ used he results of Bibler and Wallace's study in developing a Preliminary Technical Data Summary for mercury removal from supernate.

Table 2. Mercury Concentrations during Duolite™ GT-73 Caustic Exposure Tests

Week	Initial [Hg], mg/L	Wt Resin, g	Volume, mL	Final ^a [Hg], mg/L	K _d ^b , mL/g
0	75.23	0.0501	45	0.01	6.76E6
0	75.23	0.0503	45	0.01	6.73E6
0	75.23	0.0503	45	0.01	6.73E6
1 ^c	70.18	0.201	15	0.01	5.24E5
1 ^c	70.18	0.200	15	0.01	5.26E5
1 ^c	70.18	0.101	30	0.05	4.17E5
1 ^c	70.18	0.0998	30	0.01	2.11E6
2	75.23	0.0503	45	0.01	6.26E6
2	75.23	0.0458	45	0.01	7.39E5
2	75.23	0.0505	45	0.01	6.70E6
3	75.23	0.0503	45	0.044	1.53E6
3	75.23	0.0512	45	0.121	5.46E5
3	75.23	0.0489	45	0.0114	6.06E5
4	75.23	0.0488	45	0.01	6.94E6
4	75.23	0.0497	45	0.03	2.27E6
4	75.23	0.0514	45	0.01	6.58E6
5	75.23	0.0501	45	0.01	6.76E6
5	75.23	0.0504	45	0.01	6.72E6
5	75.23	0.0493	45	0.01	6.87E6
6	75.23	0.0467	45	0.191	3.79E5
6	75.23	0.0464	45	0.582	1.24E5
6	75.23	0.0494	45	0.516	1.32E5
7	75.23	0.0492	45	0.021	2.89E6
7	75.23	0.0496	45	0.040	1.50E6
7	75.23	0.0494	45	0.035	1.73E6
8	48.7	0.0467	45	0.01	4.69E6
8	48.7	0.0464	45	1.41	3.25E4
8	48.7	0.0494	45	0.01	4.44E6

^aThe Cold Vapor Atomic Absorption analysis method has a detection limit of 0.01 mg/L in this solution matrix. The table lists this value for sample analyses at the detection limit.

^bFor samples with mercury concentrations at or below the detection limit, the values distribution coefficients cited in the table represent minimal values (i.e., K_d ≥ value shown).

^cThese samples experienced 24 hours exposure to the caustic solution.

Personnel examined the physical structure of the Duolite™ GT-73 resin during these tests using particle size analysis and scanning electron microscopy. Table 3 contains the particle size data as

Figure 1. GT-73 Data from Caustic Exposure Tests

and it is also displayed in Figure 2. The macroporous resin consists of large particles initially in the range of 100 to 600 microns. Researchers examined samples taken from filtered resin after four, six, and eight weeks of exposure to the highly alkaline solution for particle size. These data show no structural breakdown of the resin during exposure to strongly caustic solution. Nearly 85% of the resin remains in the form of particles greater than 400 micron size.

Figure 3 includes scanning electron micrographs of the Duolite™ GT-73 resin. The resins come from samples of the initial resin and after exposure to caustic exposure for three and eight weeks, respectively. No significant differences exist between the appearance after exposure to strongly caustic solutions. The shape of the resin beads remains spherical with no indication of pitting or cracking due to chemical attack. The surface appearance of the GT-73 resin in the 3 week sample exhibits a slightly different surface than the original resin which could be due to dried salts on the resin surface. These dried salts potentially could cause slower kinetics for exchange experienced during these tests. These data agree with the particle size analysis and the mercury affinity (K_d) tests. Duolite™ GT-73 resin will effectively remove mercury from high level waste streams for periods of at least eight weeks. If adopted as part of the overall process used to process waste salt, the radiolytic stability of the resin should also be verified.

Table 3. Particle Size* Data for Duolite™ GT-73 Resin

Particle Size (μ)	Duration of Exposure to Caustic Solution			
	Initial	4 Week	6 Week	8 Week
52.33				
88	0.43	0.25		0.28
124.45	1.68	1.17	0.9	1.24
176	4.7	3.65	2.8	3.93
248.9	6.84	4.94	4	5.54
352	14.92	14.52	19.3	14.35
497.8	58.17	57.36	60.7	61.42

*Data presented as percent of particles.

Figure 2. Particle Size Data for Duolite™ GT-73 resin

Figure 3. SEM Micrograph of Duolite™ GT-73 Resin

Co-60 Affinity Tests

Personnel also measured the affinity of Duolite™ GT-73 cation resin to exchange for Co-60 ions in sodium salt solution and in a sodium salt solution spiked with mercury. Testing equilibrated a salt solution of targeted composition as shown in Table 1 with a spiked amount of Co-60 radiotracer obtained from the Analytical Development Section. Analysis of the solution indicated a specific activity of 57 dpm/mL. After a period of 24 h, personnel removed an aliquot using a filter syringe and submitted for gamma counting. Table 4 shows the data from this test. The aliquot measurements indicated equilibrium distribution coefficients, K_d , of 380 ± 80 mL/g, significantly below the $\sim 1E6$ mL/g for mercury.

Table 4. Duolite™ GT-73 Affinity for Co-60

Initial [Co] [*]	Wt Resin, g	Volume, mL	Final [Co] [*]	K _d , mL/g
57.7	0.1042	30	23.8	409
57.7	0.1044	30	28.8	288
57.7	0.0998	30	23.3	443
Average				380
Standard Deviation				82

[Co] _i	[Hg] _i ^{**}	Wt Resin, g	Vol., mL	[Co] _f	[Hg] _f ^{**}	K _d , mL/g	
						Co-60	Hg
57.7	70.18	0.0497	15	26.4	0.01	358	2.12E6
57.7	70.18	0.0496	15	26.7	0.01	351	2.12E6
Average						354	2.12E6
Standard Deviation						5	--

^{*}Co-60 concentrations use units of dpm/mL.

^{**}Hg concentrations use units of mg/L.

The K_d measured for Co-60 agrees with vendor information¹ that indicates a preferential affinity in the chemical series:

This chemical series indicates that Co would displace sodium from the Duolite™ GT-73 resin. A second set of tests addressed the competition between mercury and the radioactive Co-60. These tests consisted of spiking the Co-60 containing solution with 70 mg/L mercury and performing the K_d test. Table 4 also includes this data. Analysis of these data indicates the presence of mercury at 70 mg/L did not affect the affinity of the resin to absorb Co-60. The calculated equilibrium distribution coefficient, K_d, for Co-60 measured 354 ± 5 mL/g which agrees within error bounds with the measurement in the absence of mercury (380 ± 82 mL/g).

Real Waste Verification Tests

The last set of tests examined the mercury removal characteristic of Duolite™ GT-73 resin on radioactive High Level Waste. These tests used ~0.05 g of the GT-73 resin equilibrated (72 h) with Tank 46F waste that contained approximately 11.5 mg/L mercury. Additionally, a pair of tests added mercury to the Tank 46F material to reach a final concentration of 162.9 mg/L and equilibrated with the GT-73 resin. This work occurred in the SRTC Shielded Cells. Samples removed required a 100:1 dilution for radiological control. Therefore, samples treated with resin required analysis by ICP-MS to obtain mercury concentrations.

The data from these tests are shown in Table 5. The tests with the pure Tank 46F waste removed mercury from 11.5 mg/L to less than 0.2 mg/L yielding a calculated average K_d of $1.41E4 \pm 3.43E3$ mL/g. The distribution coefficient measures significantly less than nominal $1E5$ mL/g for the simulated waste tests described above. In tests when the mercury level in the waste was increased to ~170 mg/L, the Duolite™ GT-73 resin reduced the mercury concentrations to levels on the order of 1 – 2 ppm. Calculated average K_d for these tests was $2.96 \pm 0.813E4$ mL/g.

The equilibrium distribution coefficients measured in these real waste tests agree with those measured by Bibler and Wallace.¹⁴ Bibler and Wallace measured K_d 's that ranged from 1.84 to $2.45E4$ mL/g in Tank 30H supernate.

Table 5. Duolite™ GT-73 Data from Real Waste Tests

Initial [Hg]*	Wt Resin, g	Volume, mL	Final [Hg]*	K_d , mL/g
11.5	0.0500	15	0.21	1.65E4
11.5	0.0498	15	0.29	1.165E4
			Average	1.14E4
			Standard Deviation	3.43E3
162.9	0.0500	15	1.37	3.54E4
162.9	0.0501	15	2.02	2.39E4
			Average	2.96E4
			Standard Deviation	8.13E3

*Hg concentrations use units of mg/L.

There is a discrepancy between the real waste tests and the simulant tests. The equilibrium distribution coefficient measured in the simulant tests was greater by a factor of ~100 than the measured data in the real waste tests. There are several factors that may lead to this discrepancy.

Specifically, the mercury speciation may be significantly different in aged waste as compared to a freshly prepared simulant. However, this hypothesis is not substantiated by the real waste test in which mercury was spiked. This Tank 46F material was higher in sodium ion concentration that could influence the exchange process. Another possibility is that Rohm and Haas¹ indicate a hydroxide dependence for GT-73 and this may require additional testing to define the dependence on hydroxide concentration.

Conclusions

Experiments examined concerns raised by the Salt Disposition System Engineering Team concerning the ability of Duolite™ GT-73 cation exchange resin to decontaminate mercury from high level waste streams. The experiments addressed three major areas of concern. First, experiments conducted over an eight-week period determined the resin's ability to remove mercury after prolonged exposure to strongly caustic solutions. Properties examined included affinity (K_d), particle size, and scanning electron microscopy of the resin beads. The second set of experiments examined the GT-73 resin's affinity for Co-60 that has implications to shielding requirements for operational facilities. Lastly, a real waste treatability study verified the Duolite™ GT-73 resin's effectiveness on actual radioactive waste.

Results from the caustic exposure tests indicate that GT-73 resin remains stable above pH 13 over the eight weeks of testing. The equilibrium distribution coefficient, K_d , significantly exceeded previously measured data. Particle size analysis along with scanning electron microscopy did not reveal any physical degradation of the resin beads. Tests to determine the resin's affinity toward Co-60 indicate the resin will bind the radioactive species. Thus, design of operational facilities must evaluate the need for shielding. Lastly, real waste verification tests confirmed the simulant data for mercury removal. Equilibrium distribution coefficients ($\sim 2E4$ mL/g) compare to those previously measured by Bibler and Wallace. Additional testing to confirm the longer exposure times (>6 month) to caustic solution will continue at a reduced sampling frequency of once every three weeks. Additional future testing should include column experiments using real waste to verify the batch data. Adequate stability in a high radiation field may also require verification, depending on the alternative selected for salt processing.

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Approvals

Authors:

W. R. Wilmarth, Waste Processing Technology
20 Oct 98
Date

Design Review

D. T. Hobbs, per Procedure 2.40 of Manual E7
10/2/98
Date

Management

S. D. Fink, Manager, Level 4 Manager,
Waste Processing Technology
10/2/98
Date

J. R. Fowler, Lead
Cross-Cutting Issues, Flowsheet Team
10/3/98
Date

J. T. Carter, Leader
Flow Sheet Team
10/3/98
Date

K. Rueter
Salt Disposition Systems Engineering Team
10/4/98
Date

W. L. Tamosaitis, Level 3 Manager,
Waste Processing Technology
10/2/98
Date

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