*Effects of Aqueous-Soluble Organic Compounds
on the Removal of Selected Radionuclides
from High-Level Waste*

*Part I: Distribution of Sr, Cs, and Tc onto 18 Absorbers
from an Irradiated, Organic-Containing Leachate Simulant
for Hanford Tank 101-SY*

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EFFECTS OF AQUEOUS-SOLUBLE ORGANIC COMPOUNDS ON THE REMOVAL OF SELECTED RADIONUCLIDES FROM HIGH-LEVEL WASTE

Part I: Distributions of Sr, Cs, and Tc onto 18 Absorbers from an Irradiated, Organic-Containing Leachate Simulant for Hanford Tank 101-SY

by

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ABSTRACT

Many of the radioactive waste storage tanks at U.S. Department of Energy facilities contain organic compounds that have been degraded by radiolysis and chemical reactions. In this investigation, we measured the effect of some aqueous-soluble organic compounds on the sorption of strontium, cesium, and technetium onto 18 absorbers that offer high sorption of strontium from organic-free solutions. For our test solution we used a leachate from a simulated slurry for Hanford Tank 101-SY that initially contained ethylenediaminetetraacetic acid (EDTA) and then was gamma-irradiated to 34 Mrads. We measured distribution coefficients (K_d s) for each element/absorber combination for dynamic contact periods of 30 min, 2 h, and 6 h to obtain information about sorption kinetics. To facilitate comparisons, we include K_d values for these same element/absorber combinations from three organic-free simulant solutions. The K_d values for strontium sorption from the simulant that contained the degraded organics usually decreased by large factors, whereas the K_d values for cesium and technetium sorption were relatively unaffected.

I. INTRODUCTION

The Hanford Reservation near Richland, Washington, incorporates 177 underground tanks that store more than 65 million gallons of radioactive waste containing some 165 million curies. These high-level wastes (HLWs) are a byproduct of the production of nuclear materials for national defense needs during the past half-century. Because Hanford operating contractors used numerous chemical processes during this period, many different reagents and waste streams were combined in these underground tanks. The resulting wastes consist of complex and sometimes unstable mixtures of sludges,

saltcakes, slurries, and supernates. Adding significantly to the stored-waste problem is the fact that 67 of these underground waste storage tanks are known, or are presumed, to have leaked.

The U.S. Department of Energy (DOE) has directed Los Alamos National Laboratory (LANL) and Sandia National Laboratories/New Mexico (SNL) to support the Hanford Tank Waste Remediation System (TWRS) mission, which is to store, treat, and dispose of all tank waste in a safe, cost-effective, and environmentally sound manner. An essential prerequisite for achieving this goal is the identification of suitable partitioning agents and technologies.

II. OBJECTIVE

In support of the TWRS mission, we have previously completed three absorber screening studies, which collectively provide measured distribution of 16 elements onto 100 commercially available and experimental absorber materials from five simulated Hanford waste solutions. The individual simulants tested were acid-dissolved sludge, acidified supernate, and alkaline supernate solutions for Hanford Tank 102-SY;¹ Hanford double-shell slurry feed (DSSF);² and Hanford neutralized current acid waste (NCAW).³ However, none of these simulants contained any of the degraded organic compounds known to be present in many Hanford waste tanks.

Because many of the organic compounds in Hanford waste tanks have a strong affinity for multivalent cations, these organic compounds could interfere by forming aqueous-soluble complexes with metal ions that would otherwise be sorbed.

The objective of this study was to quantify the effects of ethylenediaminetetraacetic acid (EDTA) and its radiolysis products on the sorption of strontium, cesium, and technetium by 18 absorbers that showed high sorption of strontium in our previous screening studies.¹⁻³ Although strontium was the element of primary concern, we included cesium and technetium tracers in our study to obtain comparable data for the effects of degraded organic compounds on these two key elements.

III. EXPERIMENTAL PARAMETERS

A. Simulant Solutions

The procedure of Herting⁴ had earlier been used by Sandia/NM personnel to prepare a simplified simulant for Hanford Tank 101-SY slurry, whose preirradiation composition is shown in Table I. This slurry subsequently was gamma-irradiated at 285 krad/h to a total of 34.2 Mrads with ⁶⁰Co in the Low-Intensity Cobalt Facility of Sandia/NM. Additional degradation of organic compounds very likely occurred in the concentrated alkaline medium during the 2 years between the preparation of the slurry and when Norman Brown of Sandia/NM provided it to us for our study.

Table I. Composition of Preirradiated Simulant for Hanford Tank 101-SY Slurry

Constituent	Concentration (M)
Na	13.8
Al	2.2
NO ₃ ⁻	3.7
NO ₂ ⁻	3.2
CO ₃ ⁼	0.6
EDTA	0.3

In accord with instructions,⁴ we leached the simulated 101-SY slurry by contacting it with four volumes of distilled water and stirred this mixture at room temperature for 18 h. (Dissolution was far from complete; in fact, most of the initial solids appeared to remain after this treatment.) We centrifuged and filtered the resulting leach solution through a 0.45- μ m filter to remove suspended solids. Because this leachate simulant for Hanford Tank 101-SY included no strontium or anions other than nitrate and nitrite, we added realistic levels of fluoride, chloride, sulfate, phosphate, and strontium.⁵ The composition of the final leach solution, as determined by the LANL Analytical Chemistry Group, is shown in Table II. The compositions of three organic-free simulant solutions, whose distribution coefficients (Kds) we present for comparison, also are listed in Table II.

B. Radiotracers

Because gamma spectrometry had been selected as the technique for measuring the Kd values of the three elements, we added appropriate quantities of radiotracers for strontium, cesium, and technetium to the 101-SY leach solution. The three radiotracers used and their gamma energies, branching ratios, and concentrations are shown in Table III. The simulant solution and added radiotracers were stirred continuously for 48 h and then allowed to remain undisturbed for 2 months to ensure that there was sufficient time for soluble complexes or insoluble compounds to form. The aged solution was again filtered through a 0.45- μ m filter just before the absorber-contact phase of this study.

Table II. Compositions of Leachate from Irradiated Tank 101-SY (Organic-Containing) Simulant and of Three Other Alkaline Hanford HLW (Organic-Free) Simulant Solutions Whose Kd Values Are Included in This Study

Constituent	Concentration (M)			
	102-SY	DSSF	NCAW	101-SY ^a
Cations				
Na	2.2	7.0	5.0	1.94
K	not determined	0.945	0.12	none
Rb	none	none	5.0×10^{-5}	none
Cs	4.5×10^{-8}	7.0×10^{-5}	5.0×10^{-4}	1.1×10^{-7}
Al	0.16	0.72	0.43	0.93
Si	0.0024	none	none	none
Ca	0.0069	none	none	none
Cr(VI)	0.0051	none	none	none
Fe	0.0061	none	none	none
Sr	3.4×10^{-8}	3.4×10^{-8}	3.4×10^{-8}	2.9×10^{-6}
Anions				
F	0.21	none	0.89	0.015
Cl	0.102	0.102	none	0.104
NO ₃	1.31	3.23	1.67	0.59
NO ₂	none	1.56	0.43	0.60
PO ₄	0.061	0.0073	0.025	0.068
SO ₄	0.022	0.008	none	0.017
CO ₃	none	0.147	0.23	0.20
Oxalate	none	none	none	0.006
Total organic carbon	none	none	none	8475 ppm
Free OH ⁻	0.7	1.75	1.68	0.3

^aThe 101-SY composition shown includes anions we intentionally added to the leach solution.

Table III. Radiotracers Used in This Study

Radiotracer	Gamma Energy (MeV)	Gamma Branching (%) ^a	Estimated Concentration
⁸⁵ Sr	0.514	100	3 µg/L
¹³⁷ Cs	0.662	85	6 µg/L
^{95m} Tc	0.204	100	2 pg/L

^aGamma branching information is provided to indicate the assay sensitivity.

C. Absorbers

The 18 absorbers selected were among those that offered high sorption of strontium from simulated alkaline supernate solution.¹ The selected absorbers are listed in Table IV. Their suppliers or preparation details are included in the referenced report.

We intentionally air-dried all moist resins and composites before testing to put all absorbers on a directly comparable basis. We also sought to avoid diluting the simulant solutions by adding as-received wet resins, which in the worst case would have diluted the simulant solution by 23%. However, the concern expressed by a few colleagues about the adverse effects of air-drying absorbers appears overstated because all air-dried absorbers performed well in our earlier studies.¹⁻³

D. Solution/Absorber Contacts

In most cases, a 250-mg portion of each air-dried absorber was contacted with a measured 6-mL volume of the simulant solution in a specially modified 20-mL disposable hypodermic syringe. We modified these syringes by inserting cylindrical plugs cut from 1/4-in.-thick porous KynarTM, obtained from Porex Technologies, Fairburn, Georgia, into the tapered tips as filters, which permitted only liquid to pass through.

For each set of experiments, we selected six syringes, each containing a different absorber. A measured volume of the simulant solution was transferred by pipet into a disposable 15-mL plastic beaker and then drawn into a syringe through the KynarTM filter. We then sealed the tip of each syringe with a solid Luer cap and placed the syringes on a 48-rpm tube rotator for dynamic contact periods of 30 min, 2 h, and 6 h.

Table IV. Absorbers Evaluated in This Study

Absorber Type/Source	Trademark ^a or Other Identification
Commercial Absorbers	Bone char absorber Diphonix TM cation resin (polyfunctional) Duolite TM C-467 cation resin (phosphonic acid) Ionsiv TM TIE-96 (TiO ₂ -loaded zeolite) Tannin absorber
Composite Absorbers	Ba(Ca)SO ₄ -PAN barium/calcium sulfate
F. Sebesta, Czech Republic (polyacrylonitrile matrix)	MgO-PAN magnesium oxide M315-PAN synthetic mordenite NaTiO-PAN sodium titanate NiFC-PAN nickel hexacyanoferrate TiO-PAN titanium dioxide
J. Narbutt, Poland (phenolsulfonic-formaldehyde matrix)	CoFC-PSF titanium hexacyanoferrate TiFC-PSF cobalt hexacyanoferrate
LANL-Prepared Extractant Beads	LIX TM -54 (a beta diketone)
Sandia/NM Absorbers	SNL/CST 120 crystalline silico-titanate SNL/CST 141 crystalline silico-titanate SNL/CST 149 crystalline silico-titanate SNL/HTO amorphous hydrous titanium oxide

At the end of each contact period, approximately 25% of the total solution volume was expelled through the attached 0.45- μ m filter into a tared counting vial. We then weighed the dispensed solution and used that figure in the calculation of the Kd values.

The described filter-tip syringes were unsuitable for the crystalline silico-titanate (CST) and hydrous titanium oxide (HTO) absorbers from Sandia/NM because the small particles often plugged the Kynar™ filters. For these finely divided materials, we pipetted 6 mL of solution directly into a 30-mL centrifuge tube that contained 250 mg of absorber and mixed each absorber/solution on the rotator for the prescribed time. At the end of each contact period, the tubes were centrifuged at high speed for 10 min. We then transferred approximately 1.5 mL of the centrifuged solution into a 3-mL hypodermic syringe with a 0.45- μ m filter attached to its Luer-lock tip. The centrifuged solution was expelled through this filter into a tared counting vial, from which we determined the solution weight.

E. Calculation of Kd Values

Distribution coefficients (Kds) normally are calculated in terms of the dry absorber weight;⁶ optionally, Kds can be calculated in terms of the wet absorber volume. Because most of the absorbers we tested were received in dry form, we tested all absorbers on a comparable, dry basis to conform to a consistent Kd convention. We recognize that air-drying moist absorbers to constant weight at room temperature causes some polymers to temporarily shrink as they lose physically sorbed water; however, such polymers swell again when they recontact aqueous solution. Therefore, the only adverse effect of air-drying might be a temporary decrease in polymer porosity that could result in slower kinetics until the polymer regains its lost water. To protect the air-dried absorbers from any lasting damage, we never applied heat during the drying cycles.

Each portion of postcontact solution was assayed by gamma spectrometry using the characteristic gamma energies of the added radionuclides. The fraction of each element sorbed was determined indirectly from the difference in the measured gamma activity of the selected radionuclide before and after contact with each specified absorber.

All Kd values were calculated as follows:

$$K_d = \frac{Pr - Po}{Po} \frac{S}{A},$$

where Pr = measured precontact activity per milliliter,
Po = measured postcontact activity per milliliter,
S = milliliters of solution contacted, and
A = grams of dry absorber contacted.

F. Other Experimental Parameters

Other experimental parameters, such as data transfer and processing, applied corrections, uncertainty estimates, sources of error, and detection limits were identical to those described previously.¹⁻³

IV. RESULTS AND DISCUSSION

Tables V through VII present our measured Kd values for strontium, cesium, and technetium on 18 selected absorbers from the organic-containing 101-SY leachate simulant solution and from three organic-free simulant solutions. Kd values were measured for three different contact times to obtain information about sorption kinetics. Data for these three elements from the irradiated 101-SY simulant are published here for the first time, whereas the Kd values from 102-SY supernate, DSSF, and NCAW simulants are excerpted from our previous, more-comprehensive studies.¹⁻³ The Kd values for the three organic-free simulants having the compositions given in Table II are included to provide a basis for comparing the performance of the 18 selected absorbers in the presence and absence of organic compounds.

A. Strontium

As expected, the sorption of strontium on these 18 absorbers is dramatically affected by the organic compounds in the irradiated 101-SY simulant. The general order for sorbing strontium from the other three

simulants of NCAW > DSSF > 102-SY does not appear to correlate with any of the composition information in Table II.

Table V. Kd Values for Strontium onto Various Absorbers from Four Alkaline Simulant Solutions

Absorber	102-SY (Organic-Free)			DSSF (Organic-Free)			NCAW (Organic-Free)			Irradiated 101-SY (Organic-Containing)		
	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h
SNL/CST 149	>9K	>9K	>9K	2047	3808	4692	2950	6K	8K	2.9	4.8	7.3
Bone char	5K	>11K	>11K	713	2378	4836	825	2812	7K	45	77	125
SNL/CST 120	>4400	>4500	>4600	13K	>32K	>32K	18K	21K	23K	93	113	119
SNL/HTO	>4000	>4100	>4200	23K	10K	>24K	>34K	>35K	>35K	801	1090	1097
SNL/CST 141	2755	>4300	>4400	1319	1839	2343	1581	2082	3001	3.0	5.2	8.0
NiFC-PAN ^a	1121	3736	4651	1750	5K	12K	1952	5K	11K	3.3	328	1587
Duolite™ C-467	1051	2734	5K	324	604	777	361	706	1010	0.5	0.5	0.6
TiO-PAN	917	1627	2856	6K	24K	>29K	>5K	17K	>42K	118	157	173
Diphonix™	546	696	918	1930	4921	7K	3338	8K	17K	0.8	1.2	1.3
MgO-PAN	499	768	689	751	1214	1690	808	1276	1458	0.8	0.9	1.0
NaTiO-PAN	314	410	296	2415	9K	25K	3487	9K	14K	30	92	168
Ionsiv™ TIE-96	286	405	421	222	578	1053	407	965	1696	3.3	5.2	6.3
TiFC-PSF	144	260	254	147	1076	1716	1216	4402	7K	2.8	8.2	14
CoFC-PSF ^a	84	293	1270	34	140	1222	76	531	1199	1.0	6.2	25
Ba(Ca)SO ₄ -PAN	76	116	146	17	22	25	68	119	166	0.5	0.4	0.4
Tannin	50	58	102	203	476	761	324	672	1081	<0.1	<0.1	<0.1
LIX™-54	37	67	84	32	108	185	37	101	178	0.2	0.4	0.3
M315-PAN	22	165	432	5.7	8.7	15	8.2	17	37	<0.1	<0.1	<0.1

^aThe characteristic color of the metal ion in these absorbers appeared in the irradiated 101-SY simulant within minutes, which indicated that dissolution was enhanced by the degraded organics.

B. Cesium

The sorption of cesium on these 18 absorbers appears unaffected by the degraded organic compounds in the irradiated 101-SY simulant. The observed order for cesium Kd values of 101-SY > 102-SY > NCAW > DSSF appears to be inversely related to the potassium concentration in

these four simulants (see Table II). The unusual decrease in the Kd values for cesium with increasing time seen for NiFC-PAN and CoFC-PAN may reflect enhanced dissolution of these two absorbers in the 101-SY simulant.

Table VI. Kd Values for Cesium onto Various Absorbers from Four Alkaline Simulant Solutions

Absorber	102-SY (Organic-Free)			DSSF (Organic-Free)			NCAW (Organic-Free)			Irradiated 101-SY (Organic-Containing)		
	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h
SNL/CST 141	2521	2521	2312	530	649	736	1812	1990	2071	7K	9K	9K
NiFC-PAN ^a	2090	943	76	16	2.6	2.4	261	158	115	752	939	72
SNL/CST 149	1866	2402	3084	436	570	612	1301	1759	1829	5K	6K	6K
CoFC-PSF ^a	1154	3793	3575	55	213	354	237	1072	1488	1925	360	90
SNL/CST 120	570	786	898	20	26	27	71	95	101	1132	1767	2125
M315-PAN	161	177	177	29	39	29	94	99	91	317	398	408
Tannin	64	66	66	12	12	12	28	29	29	106	127	125
TiFC-PSF	59	102	102	14	20	23	35	44	45	123	208	218
Ionsiv TM TIE-96	49	86	107	8.2	11	12	23	38	45	86	163	218
Diphonix TM	3.0	1.6	1.7	0.8	0.3	0.3	1.0	1.0	0.8	1.5	1.4	1.5
NaTiO-PAN	1.9	1.6	1.5	2.3	1.6	1.0	0.6	0.4	0.4	3.3	3.1	3.1
SNL/HTO	1.5	1.4	1.4	0.5	0.3	0.3	0.2	0.3	0.3	1.1	1.3	1.0
Bone char	1.1	3.1	2.9	<0.1	<0.1	<0.1	0.2	0.1	0.3	0.3	0.3	0.6
TiO-PAN	0.5	0.5	0.4	<0.1	<0.1	<0.1	0.1	0.2	0.2	0.4	0.5	0.5
Duolite TM C-467	0.4	1.5	2.0	6.6	4.8	2.2	0.4	0.3	0.3	0.3	<0.1	0.2
LIX TM -54	<0.1	<0.1	<0.1	3.5	2.3	1.2	<0.1	<0.1	<0.1	0.3	0.2	0.2
MgO-PAN	<0.1	<0.1	<0.1	2.2	1.1	0.7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Ba(Ca)SO ₄ -PAN	<0.1	<0.1	<0.1	1.1	0.4	0.3	<0.1	0.2	0.1	0.3	<0.1	0.1

^aThe characteristic color of the metal ion in these absorbers appeared in the irradiated 101-SY simulant within minutes, which indicated that dissolution was enhanced by the degraded organics.

8 C. Technetium

The sorption of technetium on these absorbers appears unaffected by the degraded organic compounds in the irradiated 101-SY simulant. Because the 18 absorbers were selected from those that sorbed strontium well from organic-free simulants, it is not surprising that technetium sorption is

generally low. In the few instances where technetium is sorbed with double-digit Kd values, these values often increase sharply with increased contact time.

Table VII. Kd Values for Technetium onto Various Absorbers from Four Alkaline Simulant Solutions

Absorber	102-SY (Organic-Free)			DSSF (Organic-Free)			NCAW (Organic-Free)			Irradiated 101-SY (Organic-Containing)		
	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h	30 min	2 h	6 h
Tannin	4.3	5.4	4.0	4.5	14	85	4.7	15	62	6.2	9.7	17
Bone char	2.9	5.1	4.9	0.7	1.0	1.2	1.1	1.3	1.3	2.6	5.8	3.6
Duolite™ C-467	2.5	3.7	4.7	10	9.0	6.4	3.8	5.1	6.4	3.7	6.3	10
Diphonix™	2.4	1.1	1.2	2.6	2.5	2.5	2.7	2.4	2.0	0.6	0.6	0.7
SNL/CST 149	1.5	1.5	1.4	0.4	0.1	0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
MgO-PAN	1.3	1.3	1.6	3.8	2.6	2.4	2.1	1.7	1.9	2.1	1.9	2.2
TiO-PAN	0.8	0.7	0.8	0.4	0.7	0.9	1.0	1.1	1.2	0.8	0.8	0.9
NaTiO-PAN	0.6	0.3	0.4	2.1	1.7	1.3	0.7	0.2	0.5	0.6	1.2	1.1
M315-PAN	0.4	0.4	0.3	1.0	0.6	0.7	0.7	0.4	0.4	0.6	0.3	0.6
Ba(Ca)SO ₄ -PAN	0.3	0.9	1.1	1.6	1.0	1.1	1.2	1.2	1.5	1.3	1.6	1.8
NiFC-PAN ^a	0.2	0.2	1.6	0.1	0.2	2.6	0.9	0.8	0.9	<0.1	0.4	0.5
Ionsiv™ TIE-96	0.2	0.2	0.2	<0.1	<0.1	<0.1	0.3	<0.1	0.1	0.4	0.3	0.3
TiFC-PSF	<0.1	<0.1	<0.1	6.1	1.6	1.4	0.6	0.6	0.6	0.4	0.1	0.2
CoFC-PSF ^a	<0.1	<0.1	<0.1	5.9	1.2	1.3	0.8	0.5	0.6	<0.1	0.5	0.5
LIX™-54	<0.1	0.1	0.2	3.6	2.6	1.6	0.5	0.5	0.7	0.3	0.8	0.6
SNL/CST 120	<0.1	0.1	<0.1	<0.1	0.4	0.1	0.2	<0.1	<0.1	<0.1	<0.1	<0.1
SNL/CST 141	<0.1	0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
SNL/HTO	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

^aThe characteristic color of the metal ion in these absorbers appeared in the irradiated 101-SY simulant within minutes, which indicated that dissolution was enhanced by the degraded organics.

V. FUTURE STUDIES

This brief investigation demonstrates that degraded organic compounds seriously interfere with the sorption of strontium onto absorbers that perform well in the absence of such organics. Moreover, these organic compounds would be expected to interfere in the sorption of other targeted multivalent cations, such as plutonium and americium. Therefore, we plan to remeasure the distribution of strontium, cesium, technetium, americium, and plutonium (if these actinides are sufficiently soluble) from organic-containing simulants onto the best identified absorbers from more-realistic simulants containing a representative mixture of initial organic compounds that have been degraded by radiolysis and hydrolysis.

VI. CONCLUSIONS

Degraded organic compounds seriously interfere with the sorption of strontium on absorbers that have previously been demonstrated to efficiently sorb strontium from organic-free simulant solutions. These organic compounds could also interfere with the sorption of other targeted multivalent cations, such as plutonium and americium.

On the basis of solution coloration, specific absorbers appear to dissolve rapidly in the presence of degraded organic compounds. The effect of aqueous-soluble organic compounds on absorber stability should be evaluated as a part of future absorber-stability studies.

Additional studies that measure the distribution of other important multivalent cations, such as actinides, from simulants that contain a realistic mixture of degraded organic compounds are needed. Similar tests with organic-containing Hanford waste solutions should be performed as soon as such waste samples are available.

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