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COMPOSITION AND CORROSIVENESS OF LOW-ACTIVITY WASTE SUPERNATES STORED AT THE SAVANNAH RIVER PLANT

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PREPARED FOR THE U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION UNDER CONTRACT AT(07-2)-1

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ABSTRACT

Compositions of low activity radioactive waste (LAW) supernates stored at the Savannah River Plant were determined in conjunction with a study of the possibility of stress corrosion of storage tanks by the supernates.

Stress corrosion appears unlikely on the bases of 1) a statistical correlation between mild steel elongation and supernate composition and 2) measured polarization of steel in actual LAW. According to the statistical correlation, the tanks should not crack. Furthermore, the polarization data shows the steel acts as an active-passive metal, and the open-circuit potentials are outside the range of those reported for pure sodium nitrate.

The LAW supernates were analyzed in October 1975 from four F-Area tanks and three H-Area tanks. Concentrations of major constituents (Na^+, OH^-, NO_3^-, NO_2^-, and $Al(OH)_4^-$) are much higher in F-Area supernates because F-Area LAW appears to be in equilibrium with salt in the tanks. Minimum concentrations of major constituents in H-Area LAW are low because one tank (23H) contains water from a fuel storage basin and wastes from regenerating ion exchange resins. Other nonradioactive constituents that were determined include SO_4^{2-}, CO_3^{2-}, PO_4^{3-}, CrO_4^{2-}, Cl^-, F^-, U, Fe, K^+, and Ca^{2+}.

Radionuclides determined included ^{137}Cs, ^{90}Sr, and $^{238-239}Pu$. Cesium-137 was the major gamma emitter; its activity was as high as 10^9 dis/(min)(ml). Activities of ^{90}Sr (beta) and $^{238-239}Pu$ (alpha) were all very low [10^3-10^4 dis/(min)(ml)].

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INTRODUCTION

Low activity radioactive waste (LAW) generated at the Savannah River Plant (SRP) is stored in uncooled, 1.3-million-gallon (4.9-million-liter) tanks. Eight of these tanks were constructed, four in each of the F and H chemical separations areas. The tanks are of concrete construction with steel liners to contain the waste. Figure 1 shows a typical LAW tank. The top of the tank is a poured concrete dome. The bottom concrete support has radial grooves that act as collectors of waste that might leak through the steel liner. Piping runs from the center bottom of the concrete to a sampling sump. Cooling coils are not required to maintain a low waste temperature. Decay heat produces only 0.003 to 0.03 watts per liter initially and gradually decreases.

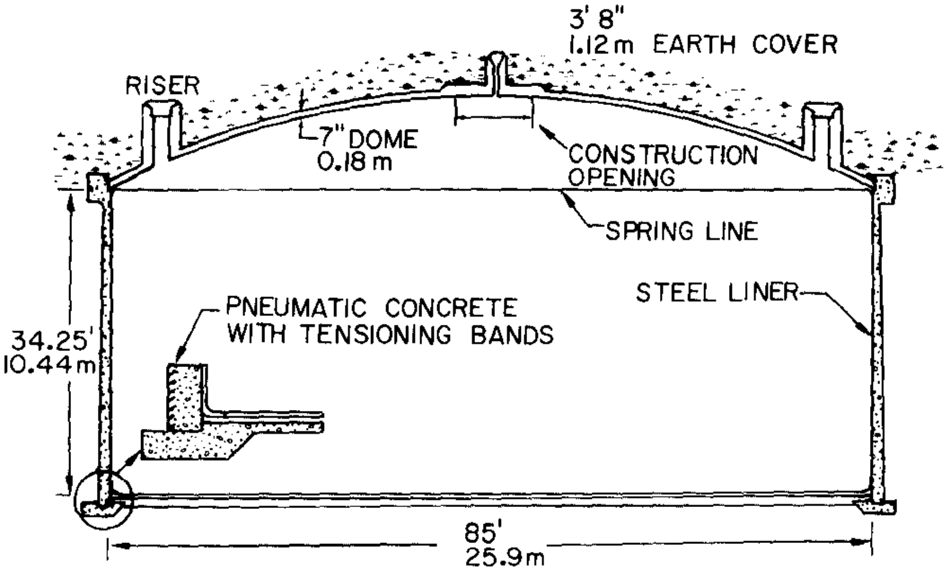

FIGURE 1. Low Activity Waste Tank

Most of the LAW at SRP is produced in the Purex process in F Area and the enriched uranium process in H Area. The Purex process recovers plutonium and uranium from irradiated natural uranium; the enriched uranium (HM) process recovers uranium from irradiated uranium-aluminum alloy. Other processes and facilities such as ion exchange, decontamination, and laboratories produce significant quantities of LAW. Mixing of these various wastes, precipitation of insoluble components, evaporation, and crystallization make a meaningful generalization of waste composition based on flowsheet or process analyses impossible.

The LAW wastes in the uncooled tanks are in three forms: a supernate that is an aqueous solution, a sludge that is a gel containing the insoluble components that settle to the bottom of the tank with trapped supernate, and a salt that is a mixture of crystals formed during cooling after evaporation of waste supernate.

The tank liners are made of mild steel, A285 Grade B, which is susceptible to stress-corrosion cracking in nitrate and caustic solutions. Stress corrosion has not been observed in LAW tank liners, but has been observed in high activity waste (HAW) tanks made of the same steel. A similar study of the compositions of HAW is reported in DP-1347.[1] The aggressiveness of corrosive attack by an environment in contact with a metal depends on the composition and temperature of the environment, on the metal composition, and on metal fabrication variables.

Compositions of LAW had been determined previously, but only for a limited number of samples; tank-to-tank variations were not determined. More detailed and more accurate analyses were made possible by the use of new methods, some of which were specially developed for analyzing HAW. Analytical results were used as the basis for evaluating the potential of LAW for causing stress-corrosion cracking of waste tanks.

SAMPLING AND ANALYSIS TECHNIQUES

The LAW supernates in seven of the eight uncooled waste tanks were sampled in October 1975, one sample per tank. Tank 21H, an evaporator feed tank, was not sampled because it contained HAW at the time. Sample volumes of 250 ml were taken by dipping the sampler about one foot below the liquid surface. The analytical techniques are outlined in Appendix A.

WASTE COMPOSITIONS

Nonradioactive Constituents

Compositions of LAW produced by the Purex and HM processes are varied. LAW also comes from other sources. Major sources of these wastes are listed in Table 1.

TABLE 1

Sources of Low Activity Wastes

F Area

- Purex Process
    - Dejacketing Al-clad targets
    - Second stage solvent extraction
    - Plutonium finishing
    - Solvent recovery washes
- Decontamination
- F-Area Miscellaneous Materials

H Area

- HM Process
    - Second stage solvent extraction
    - Solvent recovery washes
- Neptunium Target Processing
- Waste from Receiving Basin for Offsite Fuel (RBOF)
- H-Area Miscellaneous Materials

The primary differences between the compositions of F- and H-Area LAW's, as summarized in Table 2, are the high concentrations of Na^+, OH^-, NO_3^-, NO_2^-, and $Al(OH)_4^-$ in F Area and low concentrations of all major ions in H Area. Detailed results on the analyzed concentrations of each constituent in each tank are listed in Appendix B.

TABLE 2

Concentration Ranges of Major Constituents of LAW Supernates

	Concentration, M	
Constituent	*F Area*	*H Area*
Na^+	3.2 - 11.0	0.2 - 4.0
OH^-	1.4 - 7.9	0.06 - 1.5
NO_3^-	1.9 - 2.6	0.2 - 2.8
NO_2^-	<0.05 - 1.6	<0.05 - 0.25
$Al(OH)_4^-$	0.10 - 1.1	<0.01 - 0.06

The high concentrations in F-Area LAW result from the supernate probably being in equilibrium with the salt. The low concentrations in H Area result from the very dilute contaminated wastes from the Receiving Basin for Offsite Fuel (RBOF) and from the Resin Regeneration Facility (RRF) for reactor moderator deionizers. These dilute wastes are stored in Tank 23H. Generally, however, the major ions in the LAW are the same as the major ions in the HAW.

All wastes are stored as basic solutions in carbon steel tanks. Before storage, most waste solutions are acid, and NaOH is added to make them basic. Base addition is one source of Na^+ and OH^-. Dejacketing of aluminum-clad uranium with NaOH and $NaNO_3$ not only adds more Na^+ and OH^-, but also $Al(OH)_4^-$ and NO_2^-, to F-Area wastes. NO_2^- comes from chemical reduction of NO_3^-. Nitrite ingrowth by radiolysis of nitrate is very low. Because of the low level of radioactivity contained in most LAW tanks, nitrite would be expected to increase only about 0.04 moles/yr based on HAW-nitrite ingrowth data. The concentrations of Na^+, NO_3^-, NO_2^-, $Al(OH)_4^-$, and OH^- increase as the waste solutions are processed through the waste evaporator. After it is returned to the storage tanks, the evaporated solution cools, and then $NaNO_3$ and $NaNO_2$ crystallizes out preferentially to form the major salt cake components. The evaporation-precipitation process leads to higher and higher hydroxyl ion concentrations with some increase in aluminate concentration in the supernates.

The minor components of the supernates come from a variety of sources. Table 3 shows the concentration ranges observed.

TABLE 3

Concentration Ranges of Minor Constituents of LAW Supernates

	Concentration[a]	
Constituent	*F Area*	*H Area*
K^+	0.023 - 0.13	0.006 - 0.05
SO_4^{2-}	0.05 - 0.08	0.005 - 0.18
PO_4^{3-}	<0.02 - 0.09	<0.02
CO_3^{2-}	0.008 - 0.05	<0.005 - 0.02
Cl^-	0.005 - 0.02	0.004 - 0.007
F^-	0.004 - 0.04	<0.001 - 0.002
CrO_4^{2-}	$<5\times10^{-4}$ - 6×10^{-3}	$<5\times10^{-4}$ - 5×10^{-4}
Ca^{2+}	$<6.7\times10^{-5}$	5.0×10^{-5} - 1.0×10^{-4}
Fe	7.3×10^{-5} - 8.4×10^{-5}	1.0×10^{-5} - 8.4×10^{-5}
U	$<1\times10^{-6}$ - 1.3×10^{-5}	1×10^{-8} - 2.1×10^{-6}
Density	1.16 - 1.45 g/ml	1.00 - 1.20 g/ml

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[a] All unspecified ranges are M

The exact sources of the minor constituents of LAW are difficult to define. Decontamination wastes, flushes of equipment or sumps, impurities in reagents, and laboratory wastes combine to give a large variety of possible ions at low concentrations.

Radioactive Constituents

In addition to analyses of nonradioactive constituents of LAW supernates, the major radioactive constituents were determined. Radioisotopes of most concern are those of cesium, strontium, and plutonium. The ranges of concentrations of these isotopes are listed in Table 4.

TABLE 4

Concentration Ranges of Radioactive Constituents of LAW Supernates

	Concentration, dis/(min)(ml)			
	F Area		*H Area*	
Constituent	*Minimum*	*Maximum*	*Minimum*	*Maximum*
^{134}Cs	$7.5\text{x}10^4$	$7.6\text{x}10^6$	$<3.6\text{x}10^3$	$2.3\text{x}10^6$
^{137}Cs	$2.4\text{x}10^6$	$1.3\text{x}10^9$	$3.0\text{x}10^4$	$4.0\text{x}10^8$
^{144}Ce	$<4.8\text{x}10^4$	$<9.1\text{x}10^6$	$<9.7\text{x}10^3$	$<5.1\text{x}10^6$
^{103}Ru	$<9.3\text{x}10^3$	$<4.1\text{x}10^6$	$<1.5\text{x}10^3$	$6.7\text{x}10^6$
^{106}Ru	$8.9\text{x}10^5$	$<1.3\text{x}10^7$	$<2.8\text{x}10^4$	$2.2\text{x}10^7$
^{90}Sr	$2.9\text{x}10^3$	$9.9\text{x}10^3$	$0.5\text{x}10^3$	$3.6\text{x}10^3$
Gross α	$1.1\text{x}10^4$	$14\text{x}10^4$	$0.9\text{x}10^4$	$38\text{x}10^4$
Pu[a]	$1.1\text{x}10^4$	$4.9\text{x}10^4$	$0.4\text{x}10^4$	$3.3\text{x}10^4$

[a] Pu alpha activity >90% ^{238}Pu.

The major sources of gamma activity in the supernate were ^{137}Cs and ^{134}Cs. Maximum ^{90}Sr beta activity was about 10^4 dis/(min)(ml). Plutonium* alpha activity was about 5×10^4 dis/(min)(ml). Strontium and plutonium would be expected to concentrate in the sludge, rather than in the supernate; however, colloidal suspension of sludge in the supernate is the probable cause for their presence. The highest plutonium concentrations were found in those tanks that contained sludge (Tanks 18F and 22H). Very low concentrations of radioactive constituents were found in Tank 23H, as would be expected, since this tank receives RBOF water and RRF wastes.

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* Total alpha activity of LAW plutonium is mainly (>90%) from ^{238}Pu.

POLARIZATION CURVES

Polarization experiments were conducted by varying the potential between a mild steel test electrode and an inert counter electrode; the potential was measured against a reference electrode. As the potential was varied, the current flow was then measured.

Each polarization curve has passivation features which limit the amount of corrosion that should occur. Therefore, nitrate stress-corrosion cracking would not be expected because the initiating step, corrosion, is unlikely. Six waste supernates produced the two typical curves shown in Figure 2.

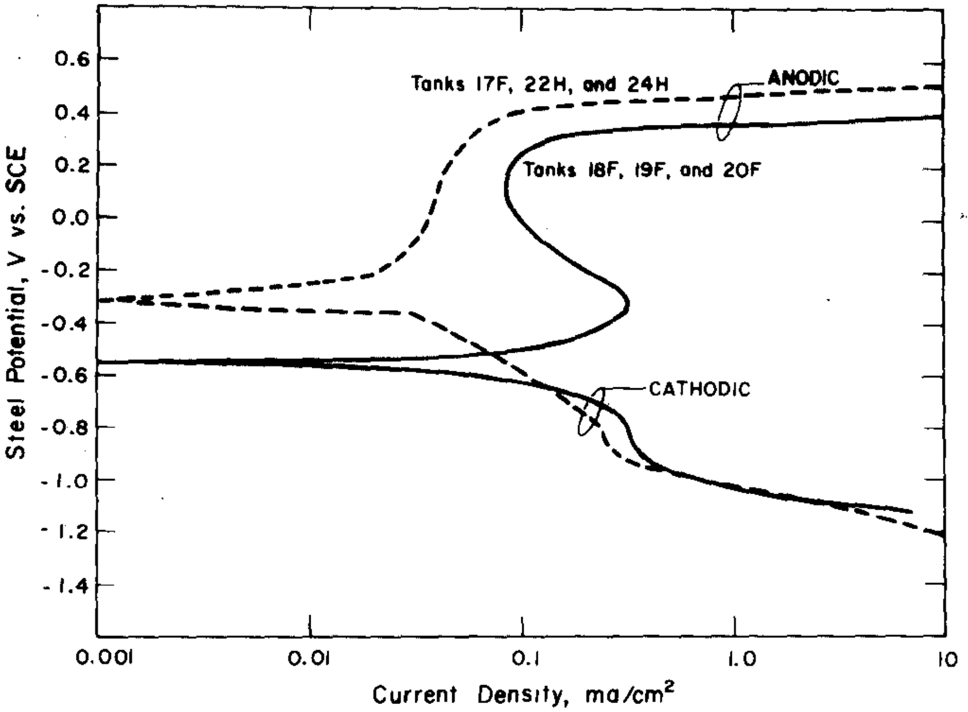

FIGURE 2. Low Activity Waste Polarzation Curves

In LAW from Tanks 17F, 22H, and 24H, the steel acted as an active-passive metal with the passive state stable. This state is ideal for a metal container because the metal cannot be activated in the solution, and corrosion will always be low. This type of behavior has been reported for steel in <1M pure hydroxide solutions.[2] Hydroxide concentrations in Tanks 17F, 22H, and 24H range from 1 to 1.5M. In LAW from Tanks 18F, 19F, and 20F, the steel behaved as an active-passive metal with the active state stable. This type of behavior has been reported for >6M pure hydroxide solutions.[2] Hydroxide concentrations in Tanks 18F, 19F, and 20F range from 5 to 8M. The curve for Tank 23H indicated a very low possible corrosion rate; at 0.2M NO_3^- and 0.06M OH^-, this solution contained so small a concentration of dissolved solids that it would be expected to produce the almost zero corrosion rate characteristic of dilute basic solutions. Nitrate cracking would not be expected to occur because of the low NO_3^- concentrations.

Corrosion potentials were either slightly or far below the -0.30 V vs. standard calomel electrode (SCE) value reported as the minimum for nitrate stress-corrosion cracking in pure nitrate solutions.[3] For Tanks 17F, 22F, and 24H, the corrosion potentials were about -0.32 V, and for Tanks 18F, 19F, and 20F, potentials were about -0.56 V vs. SCE.

QUANTITATIVE EVALUATION OF NITRATE STRESS-CORROSION CRACKING

In a related investigation on HAW tanks,[1] a rapid screening test was developed to evaluate the potential of waste supernates for producing stress-corrosion cracking. In this test, standard tensile specimens (as shown in Figure 3) are fractured in the supernate in question while the specimen is maintained as the anode in an electrochemical cell. If the observed elongation is less than the uniform elongation of the steel when tested in air (about 13% for A 285-B steel at 100°C), the solution is considered to be an aggressive one that may cause stress corrosion. A statistical correlation of variables in the ranges 50 to 100°C, 1.5 to 5.5M NO_3^-, 0 to 3.5M NO_2^-, and 0 to 5.0M OH^- was developed from which the aggressiveness of any composition within these ranges can be estimated.

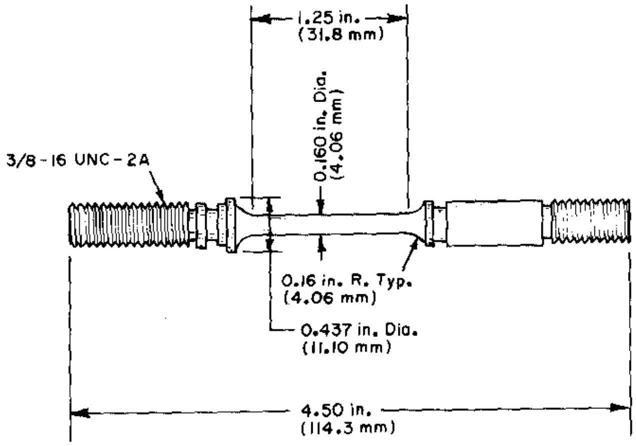

FIGURE 3. Tensile Test Specimen

On the basis of the electrochemical tensile test and wedge-opening loaded specimens[4] some technical standards were proposed for waste tank supernate compositions. These standards specified temperature <70°C, NO_3^- <5.5M (OH^- + NO_2^-) >1.2M for all LAW tanks except 23H. These standards have not been approved; however, if they were, Tank 22H would not have met them at the time of sampling (10/75). As shown in Table B-2, (OH^- + NO_2^-) = 1.0M. The present method of operation has been changed with respect to Tank 22H; some evaporator bottoms are now being added to it. This has the primary effects of increasing the OH^- concentration, the temperature, and crystallizing salt on the bottom of the tank. If Tank 22H were sampled at present, the supernate would probably meet the 1.2M (OH^- + NO_2^-) minimum.

Thus, nitrate stress-corrosion cracking appears unlikely in any of the SRP LAW tanks as long as compositions approximate those reported here. On the basis of the electrochemical tensile test (a test intended for measuring crack initiation), even a 5.5M $NaNO_3$ solution will not initiate the stress-corrosion cracking sequence at 50°C or lower. This does not mean that pre-existing cracks will not propagate under these circumstances.

APPENDIX A. METHODS OF ANALYSIS

Constituent	*Method*
NO_3^- NO_2^- SO_4^{2-} PO_4^{3-} CrO_4^{2-}	Laser Raman spectroscopy with 448 nm argon ion excitation. Nitrate peak was used as an internal standard after independent nitrate determination with perchlorate standard. The line frequencies (wave no./cm) used in the constituent order were 1050, 818, 985, 935, and 847, respectively.[5]
CO_3^{2-}	Precipitation of $BaCO_3$, dissolution of precipitate in excess acid, and titration of excess acid with hydroxide.[6]
$Al(OH)_4^-$ OH^-	The difference between two titrations to pH 7.0, the first with standard acid to neutralize the base and precipitate the aluminum. Precipitated aluminum is complexed with oxalate, and the released hydroxide is again titrated with standard acid to determine aluminum concentration.[6]
Cl^-	Chloride specific ion electrode.
Na^+	Atomic absorption using 589- or 330-nm illumination.
F^-	Fluoride specific ion electrode.[7]
Fe	Visible absorption spectrophotometry of Fe-orthophenanthroline complex measured at 510 nm.
U	Spark source mass spectrometry.
Density	Weight of a known volume.
^{144}Ce ^{134}Cs ^{137}Cs ^{103}Ru ^{106}Ru	Gamma pulse height analysis. Instrumentation included a Ge(Li) detector and 4096-channel analyzer. Gamma activities of ^{134}Cs and ^{137}Cs in diluted samples were measured directly. Other activities were measured in samples from which cesium was first removed by absorption on "Linde"* AW-500 zeolite pretreated with synthetic waste solution.[8]

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* Trademark of Union Carbide.

APPENDIX A (contd)

Constituent	*Method*
^{90}Sr	Measurement of beta activity of a dilute nitric acid solution by liquid scintillation counting. Strontium was separated and purified by $Sr(NO_3)_2$ precipitation in fuming nitric acid, water dissolution, homogeneous precipitation of $SrSO_4$ with ammonium persulfate, and conversion to $SrCO_3$ by 4M K_2CO_3 wash.
Gross alpha	Measurement with a windowless, gas-flow proportional alpha counter with dilution of solution by a factor of 500 and standard alpha mounting.

APPENDIX B. TABULAR SUMMARY OF ANALYTICAL DATA

TABLE B-1

Analyses of Supernates in F-Area LAW Tanks

	Tank 17F[a]	*Tank 18F*	*Tank 19F*[a]	*Tank 20F*[a]
Date Sampled	*10/20/75*	*10/14/75*	*10/14/75*	*10/14/75*
Concentration, M				
Na^+	3.2	8.9	11.0	11.0
K^+	0.023	0.076	0.20	0.13
NO_3^-	1.9	2.6	2.4	1.9
NO_2^-	<0.1	1.6	0.74	1.3
OH^-	1.4	4.8	6.9	7.9
$Al(OH)_4^-$	0.10	0.70	0.66	1.1
CO_3^{2-}	0.008	0.009	0.020	0.048
SO_4^{2-}	0.07	0.08	0.07	0.05
PO_4^{3-}	<0.03	0.05	<0.02	0.09
Cl^-	0.005	0.013	0.015	0.019
F^-	0.04	0.009	0.005	0.004
CrO_4^{2-}	$<5.1x10^{-4}$	$5.6x10^{-3}$	$<3.4x10^{-4}$	$1.8x10^{-3}$
Ca^{2+}	$<6x10^{-5}$	$<6x10^{-5}$	$<6x10^{-5}$	$<6x10^{-5}$
Fe	$8.4x10^{-5}$	$7.3x10^{-5}$	$7.3x10^{-5}$	$8.4x10^{-5}$
U	$1.3x10^{-5}$	$<1.3x10^{-6}$	$<3.8x10^{-6}$	$2.5x10^{-6}$
Density, g/ml	1.164	1.364	1.430	1.446
Radioactivity, dis/(min)(ml)				
Gross α	$1.1x10^4$	$1.4x10^5$	$2.7x10^4$	$2.7x10^4$
Pu	$1.1x10^4$	$4.9x10^4$	$1.5x10^4$	$2.2x10^4$
^{137}Cs	$2.4x10^6$	$8.9x10^8$	$7.4x10^8$	$1.3x10^9$
^{134}Cs	$7.5x10^4$	$7.6x10^6$	$5.0x10^6$	$6.6x10^6$
^{103}Ru	$<9x10^3$	$<1.4x10^6$	$<4.1x10^6$	$<1.7x10^6$
^{106}Ru	$<8.9x10^5$	$<1.3x10^7$	$<1.3x10^7$	$<7.4x10^6$
^{144}Ce	$<4.8x10^4$	$<8.4x10^6$	$<9.1x10^6$	$<6.4x10^6$
^{90}Sr	$9.9x10^3$	$7.7x10^3$	$2.9x10^3$	$6.4x10^3$

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a. Salt receiver tanks. Tank 17F supernate does not appear to be at equilibrium with salt; Tanks 19F and 20 do.

TABLE B-2

Analyses of Supernates in H-Area LAW Tanks[a]

Date Sampled	*Tank 22H* *10/24/75*	*Tank 23H* *10/24/75*	*Tank 24H*[b] *10/24/75*
Concentration, M			
Na^+	4.0	0.22	2.3
K^+	0.015	0.0006	0.011
NO_3^-	2.8	0.20	0.67
NO_2^-	<0.05	ND[c]	0.25
OH^-	1.0	0.06	1.5
$Al(OH)_4^-$	0.023	<0.01	0.059
CO_3^{2-}	0.010	<0.005	0.024
SO_4^{2-}	0.18	0.005	0.027
PO_4^{3-}	<0.01	ND	<0.02
Cl^-	0.005	0.007	0.004
F^-	0.002	<0.001	<0.001
CrO_4^{2-}	$<5.1 \times 10^{-4}$	ND	5.1×10^{-4}
Ca^{2+}	1.00×10^{-4}	5.0×10^{-5}	6.7×10^{-5}
Fe	6.4×10^{-5}	1.1×10^{-5}	8.4×10^{-5}
U	2.1×10^{-6}	$<1.3 \times 10^{-8}$	$<1.3 \times 10^{-6}$
Density, g/ml	1.202	1.005	1.113
Radioactivity, dis/(min)(ml)			
Gross α	3.8×10^{5}	8.6×10^{3}	2.5×10^{4}
Pu	3.3×10^{4}	3.5×10^{3}	1.8×10^{4}
^{137}Cs	1.6×10^{7}	3×10^{4}	4.0×10^{8}
^{134}Cs	2.3×10^{6}	$<4 \times 10^{3}$	$<2.2 \times 10^{6}$
^{103}Ru	6.7×10^{6}	$<2 \times 10^{3}$	$<9.8 \times 10^{5}$
^{106}Ru	2.2×10^{7}	$<3 \times 10^{4}$	1.1×10^{7}
^{144}Ce	$<1.4 \times 10^{5}$	$<1 \times 10^{4}$	$<5.1 \times 10^{6}$
^{90}Sr	5.3×10^{2}	3.6×10^{3}	1.3×10^{3}

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[a] Tank 21H, evaporator feed tank, contained high activity waste at time other tanks were sampled; therefore, Tank 21H was not sampled.

[b] Salt-receiver tank solution does not appear to be in equilibrium with salt because of addition of cesium-removal column flushes. See analyses for Tanks 19F and 20F for ranges of equilibrium concentrations.

[c] Not detected.

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