Hanford Tank Waste – What is in it? Where is it going? - 10277

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ABSTRACT

The Hanford Tank Waste Treatment and Immobilization Plant currently under construction for treating high-level waste (HLW) at the Hanford Site near Richland, Washington, will process HLW to reduce the quantity of HLW material that must be immobilized. Recently, an extensive testing program was undertaken to characterize the composition of some of the major sources of HLW in the Hanford tank farm system. The focus of this report has led to an increased understanding of the chemical form and the underlying dissolution chemistry for much of the waste.

These tests focused on identifying the primary species for selected components of interest. The tests were able to identify the primary make up of aluminum, phosphate, and chromium in the HLW. For example, the aluminum-bearing components can be broken into four major groups: water soluble components (such as sodium aluminate), easy-to-treat components (such as gibbsite), difficult-to-treat components (such as boehmite), and intractable components (such as sodium aluminosilicates). Each of the key components will be assessed with respect to these four main groupings, and the primary sources for these components will be identified.

INTRODUCTION

A large fraction of the tank waste types present at Hanford was analyzed in an attempt to understand the composition of the feeds to the Waste Treatment Plant and to assess the expected performance of those feeds during planned operations. Specific points of interest were leaching as well as filtration data for the primary purpose to better characterize actual waste processing results and to provide input into simulant development.

A basic premise of the simulant development approach is based on the development of components that can be blended to form a wide variety of more comprehensive simulants. The components will include:

- Boehmite (for Al)
- Gibbsite (for Al)
- Chromium compounds
- Filtration components
- Other components such as phosphate, oxalate, or sulfate.

Leaching Groups

The tank waste complexity and history does not lead to easy-discernable targeted groupings. Hill and Simpson [1] created a Sort on Radioactive Waste Type (SORWT) model that groups the single-shell tank wastes into broad sub-groups according to waste type. Agnew [2] presented waste forms in tanks based on process history and modeling. The tank waste Best Basis Inventory (BBI) delineates tank waste source identifications as well as specific analytical results according to tank, core, and segment sample. These sources were consulted in an attempt to establish appropriate groupings from which tank waste samples could be selected for testing.

The BBI categorizes waste in three phases: supernate, saltcake, and sludge. Table I provides a summary of the quantities of each of the components of interest in each primary waste phase. The BBI also provides wash and leach factors for each element. These were applied to each waste phase on a tank-by-tank basis to provide a breakdown of the fate of each component during Hanford Tank Waste Treatment and Immobilization Plant (WTP) processing.

Table I. Summary of Waste Phases.

Waste Phase		Al (MT)	Cr (MT)	PO_4 (MT)
Supernate	Total	1320	60	259
Saltcake	Total	3085	416	2826
	After Washing	1270	281	414
	After Leaching	579	226	260
Sludge	Total	4405	126	2063
	After Washing	3620	74	871
	After Leaching	1490	58	191

The water-soluble components of aluminum, chromium, and phosphate are likely present as sodium salts of aluminate, chromate, and phosphate and will likely dissolve or remain dissolved during waste retrieval or feed blending. As such, these will not be considered in the development of the waste groupings.

In BBI, saltcake is generally divided into six main groupings as a function waste source, A, B, BY, R, S, and T. Note that these designations are generally associated with the tank farm from which these wastes originate. For sludge, there is a much larger number of groupings. However, 80% of the sludge can be represented by four main groups: 1) bismuth phosphate wastes (1st and 2nd cycle), 2) cladding waste, 3) reduction oxidation (REDOX) waste, 4) tributyl phosphate (TBP) waste, and 5) and ferrocyanide (FeCN) waste.

Table II provides a summary of the quantities (defined by BBI) of the components Al, Cr and phosphate from each of these waste groups. These are the three components that will require pretreatment to minimize the quantity of High Level Waste (HLW) glass produced. The S-type saltcake clearly accounts for the largest single source of chromium.

Table II. Water-Insoluble Waste Type Summary.

	Al (MT)	Cr (MT)	PO_4 (MT)
Sludge	4405	126	2063
Bi phosphate	241	18	1042
CWP	901	7	124
CWR	577	6	23
Redox	1916	44	70
TBP	52	4	404
FeCN	113	7	203
Balance—sludge	606	42	197
Saltcake	3085	416	2826
A	580	44	129
B	83	4	256
BY	702	84	709
R	240	23	6
S	831	204	600
T	622	52	1051
Balance—saltcake	28	5	75

Table III shows groupings of saltcake based on Al, Cr, and PO_4. The S-type saltcake clearly accounts for the largest single source of chromium (approximately 38% of the insoluble chromium).

Table III. Saltcake Groupings.

Saltcake Group	**Components**	**Al (MT)**	**Cr (MT)**	**PO_4 (MT)**
Bi-Phosphate saltcake	BY and T	620	64	294
S – salt cake	S	354	165	57
Balance	A, B and R	288	48	59

Thus, for leaching performance, eight groups account for the majority of the material to be processed. The groupings and component distributions are provided Table IV. The ferrocyanide waste grouping was further included because of its high Fe content. Iron hydroxide is a particularly difficult matrix for cross-flow filtration, and the extent that the FeCN wastes behave as $Fe(OH)_3$ is not known. The FeCN sludge has not yet been tested in the Cell Unit Filter (CUF) operations, so it was added to this test matrix.

Table IV. Tank Waste Grouping Basis for Sample Selection.

Group ID	Type	Al	Cr	PO_4
1	Bi Phosphate sludge	3%	3%	21%
2	Bi Phosphate saltcake (BY, T)	18%	25%	36%
3	PUREX Cladding Waste sludge	12%	1%	3%
4	REDOX Cladding Waste sludge	8%	1%	0%
5	REDOX sludge	26%	8%	1%
6	S-saltcake (S)	11%	38%	12%
7	TBP waste sludge	1%	1%	8%
8	FeCN wastes	2%	1%	4%
	Balance	21%	22%	14%
Rounding leads to total Al, Cr, and PO_4 values other than 100%				

As seen in Table V, these eight groups cover ~80% of the material of interest in the expected feeds to the waste treatment plant with respect to leaching. Note that the processing history is expected to have the most significant impact of waste properties. For example, REDOX waste contains primarily boehmite crystalline phase aluminum [3] while cladding waste contains primarily gibbsite crystalline phase aluminum[4].

Further analysis identified fluoride, oxalate, and sulfate as additional anions of interest. Table V indicates that more than 60% of these anions will be represented by the groupings chosen. The most significant source of these materials will be the saltcake, and in particular, the bi-phosphate saltcake. Further note that the anion concentrations in the solid and liquid phases will be determined to the extent possible. Also, the major compounds/minerals for these anions (in the solid phase) along with their washing and leaching factors will be determined.

Table V. Anion Inventory in Selected Waste Groups.

Group	Type	Oxalate	Sulfate	F
1	Bi phosphate Sludge	2%	6%	12%
2	Bi phosphate Saltcake	36%	43%	36%
3	PUREX cladding	1%	1%	3%
4	REDOX cladding	0%	0%	2%
5	REDOX	3%	1%	2%
6	S-Saltcake	24%	14%	3%
7	TBP Sludge	0%	2%	1%
8	FeCN	1%	1%	1%
	Other saltcake	17%	21%	18%
	Balance	17%	12%	23%
Rounding leads to total Oxalate, Sulfate, and F values other than 100%				

Further, note that the two primary chromium-containing waste types are S-saltcake and bi-phosphate saltcake. One of the objectives of the work for oxidative leaching was to perform a prototypic filtration trial with one of these samples. Based on prior testing, a small amount of residual solids remain after leaching, making necessary to blend solids from one of the saltcake composites with sludge from an alternative source. Currently, a significant quantity of Tank AY-102 sludge is in inventory in the 325 Shielded Analytical Laboratory hot cells. This material will be blended with the dissolved saltcake from the S-saltcake composite to provide the feed for the oxidative leaching filtration demonstration. This provides the added benefit of providing additional cross reference for low solids filtration performance for the Tank AY-102 waste material [5,6]. Note that there may be some impact of the saltcake solids on filtration performance.

EXPERIMENTAL

The primary goals of this work are to obtain leaching as well as filtration data to better characterize actual waste processing results and to provide input into the simulant development. No single sample from a given tank that typifies a waste grouping is present (in the 222S sample archive) in sufficient volume for testing. The proposed sample selection approach composites samples within each waste grouping from multiple tanks. Thus, no single tank waste will be tested; instead, a blend of tank wastes representative of the grouping will be tested. To this end, sample selection criteria were developed to identify the specific waste samples within each category for testing as follows.

- Waste type identified by BBI to specific waste tanks.
- Sample availability—samples already available at 222S (based on the inventory provided 9/1/06 at ≥5 g.
- Analysis information available—samples with known compositions were selected most representative of the challenges associated with the waste grouping.
- Bi-phosphate Sludge (Group 1)
  - The sludge in the waste tank must be made up of at least 95% either 1st cycle or 2nd cycle waste according to BBI.
  - Only samples from 222S listed with a sludge/solid matrix will be used.
  - The Tank Waste INventory System (TWINS)[1] database confirms that the sample core/segment was a sludge or solid (as opposed to saltcake).
- Bi-phosphate Saltcake (Group 2)
  - The saltcake waste composition must be made up of 100% BY, T1, or T2 saltcake as identified in BBI.
  - The phosphate concentration must contain 20 mg/g phosphate.
  - Only samples listed as saltcake matrix will be used.
- PUREX Cladding Waste (Group 3)
  - The samples must contain at least 70 mg/g of aluminum.
  - The sludge must be defined by BBI to contain >50% CWP.
- REDOX Cladding Waste (Group 4)
  - Samples must contain (or be estimated to contain) at least 100 mg/g of aluminum.

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[1] The TWINS database is a U.S. Department of Energy (DOE) owned resource. It is a web-based interface providing access to information about a wide variety of Hanford tank waste information. It is available at URL http://twins/twins3/twins.htm.

- REDOX Sludge (Group 5)
    - Samples must contain (or be estimated to contain) at least 100 mg/g of aluminum.
    - The tank contents must have boiled during the waste storage period (better possibility of boehmite phase aluminum).
- S-Saltcake (Group 6)
    - The saltcake in the waste tank must be 100 % S-saltcake as defined by BBI.
    - The samples must contain at least 7.5 mg/g of chromium as defined by the Tank Waste Information Network System (TWINS) sample data for specific core segments.
    - The tank waste history indicates the contents did not boil.
- TBP Sludge (Group 7)
    - The sludge in the waste tank must be made up of at least 95% TBP sludge as defined by BBI.
    - Only samples from 222S listed with a sludge/solid matrix will be used.
- FeCN Sludge (Group 8)
    - Waste must be at least 70% FeCN sludge.
    - Only samples listed with a sludge/solid matrix will be used.

Aliquots were taken from a composite sub-sample(s) and analyzed according to the scheme shown in Figure 1. The key information to be obtained includes:

- Slurry density
- Slurry rheology, flow curve, and shear strength
- Solids settling rate, settled solids fraction, centrifuged solids fraction
- Composition by Inductively Coupled Plasma (ICP), Total Inorganic Carbon/Total Organic Carbon (TIC/TOC), Plutonium (Pu), Strontium (Sr) and Gamma Energy Analysis (GEA) and density of the liquid phase
- Weight percent total dissolved solids (wt% total dissolved solids (TDS)) and weight percent undissolved solids (wt% undissolved solids (UDS))
- Composition of the water-insoluble solids (note that the solids were washed with a 3:1 volume ratio of deionized (DI) water.
- Particle size distribution (PSD) of the water-insoluble solids
- Phase characterization of the water-insoluble solids—including mineralogy by X-ray diffraction (XRD) and crystal shape, size, and habit by scanning electron microscopy/transmission electron microscopy (SEM/TEM).
- Surface area by Brunnauer-Emmett-Teller (BET) surface adsorption analysisof the water-insoluble solids.

Note also that a 5 to 10 mL of sample will be archived for potential particle density measurement by sedimentation testing.

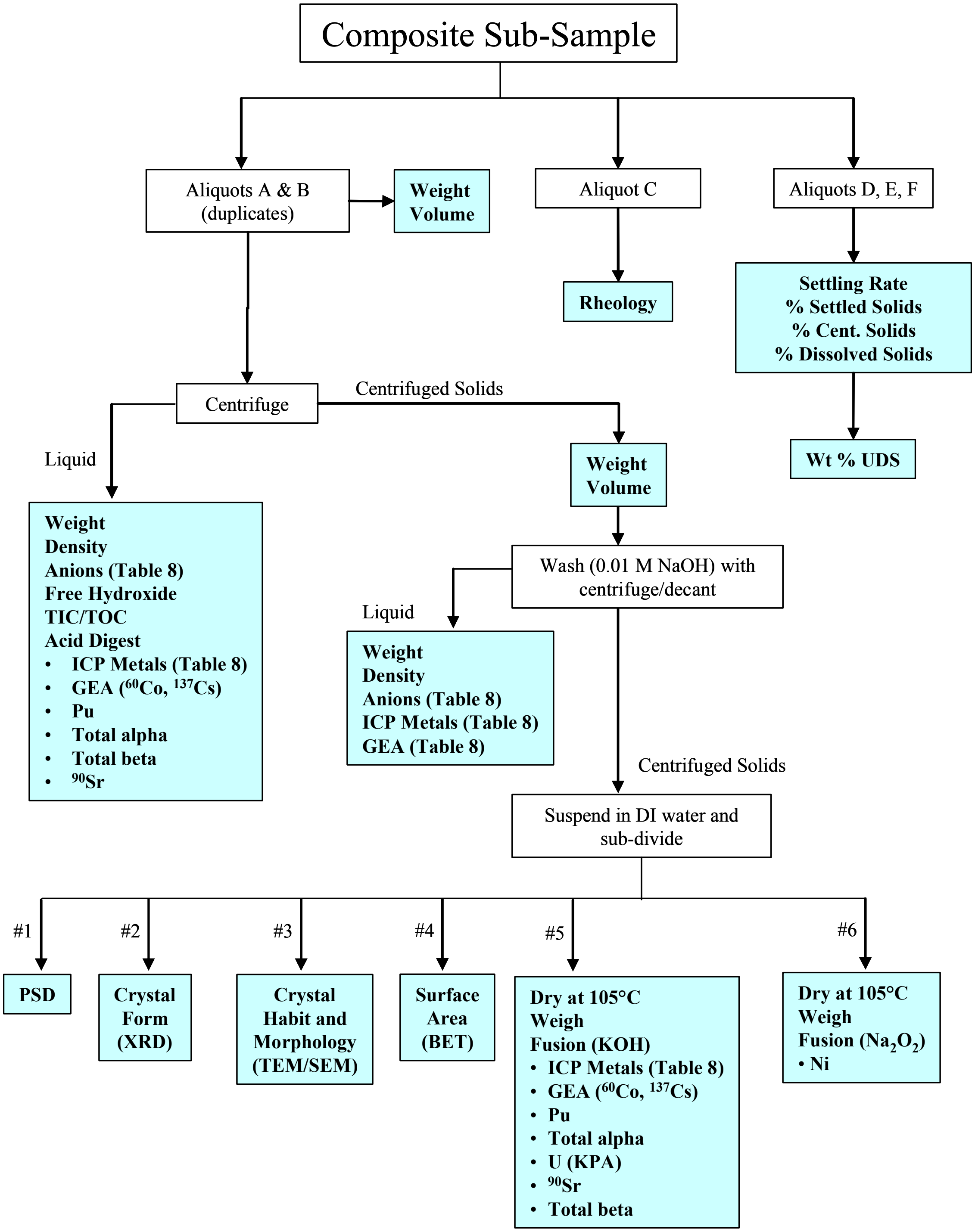

Fig. 1. Analytical protocol for actual waste samples.

Additional tests will include determining 1) dissolution kinetics for aluminum and phosphate during caustic leaching, 2) the stability of aluminum in the caustic leachate during wash dilutions, and 3) chromium dissolution during oxidative leaching. The protocols for these tests are provided by Snow [4]. The test matrices were statistically designed to provide an estimate of the uncertainty associated with the measurements. The appropriate process conditions for the parametric testing are contingent on the actual waste chemistry. The waste chemistry was defined after characterization analyses of key components (e.g., Al and free hydroxide).

The leach kinetics testing for caustic leaching of gibbsite, boehmite, and phosphates were to provide data on the rate of dissolution of these constituents under varying conditions, such as temperature, free [OH$^-$] concentration, and waste composition. In addition to this, information on the physical and chemical characteristics of selected leached and washed solids were available from the characterization efforts

RESULTS

Table VI contains a summary of the physical properties for the as received solids. The median particle size appears to be between 3 and 20 microns as indicated by the range in d50 values. Note that the d values for particle size represent the size at which that percentage of the particle are smaller than the value given. These samples settled to between 14 and 63 wt% insoluble solids. The two samples with the highest settled solids concentrations were the two cladding waste samples. This high degree of settling is likely due to the highly crystalline nature of these solids. Two samples that were below 20 wt% (Group 5 and Group 7) exhibited non-newtonian behavior. Group 5 was strongly non-newtonian with a yield stress of 57 Pa. Note that two different model were used to assess the rheolgical behavior of the non-newtonian materials.

Table VI. Initial Solids Physical Parameters.

		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
PSD—Pre Sonication Volume Count	d10 (µm)	1.234	0.864	1.169	3.184	1.062	0.774	1	2.8
	d50 (µm)	6.931	3.169	7.658	20.156	4.25	3.141	5.5	9.1
	d90 (µm)	31.466	11.095	21.502	82.574	15.989	39.952	17	20
Wt. % Undissolved Solids in Total Sample		9.0%	37%	29%	30%	19%	15%	10%	11%
Wt.% Undissolved Solids in Settled Sludge		14%	40%	50%	63%	22%	25%	11%	26%
Wt% Undissolved Solids in Centrifuged Sludge		22%	49%	58%	75%	34%	41%	20%	29%
Flow Behavior Classification									
	Newtonian	x		x	x		x		x
	Non-Newtonian		x			x		x	
Newtonian Results									
Viscosity	[cP]	6.4	n/a	3.3	2.3	n/a	6.5	n/a	3.3
Non-Newtonian Results									
Yield Stress [Pa]	Bingham	n/a	1.1	n/a	n/a	57	n/a	4.1	n/a
	Herschel-Bulkley	n/a	1.1	n/a	n/a	56	n/a	1.9	n/a
Consistency [mPa·s^n]	Bingham	n/a	13	n/a	n/a	13	n/a	11	n/a
	Herschel-Bulkley	n/a	13	n/a	n/a	44	n/a	6.6	n/a
Flow Index	Bingham	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	Herschel-Bulkley	n/a	1.00	n/a	n/a	0.83	n/a	n/a	n/a
h_{app} (33 s^{-1}) [cP]	Bingham	n/a	45	n/a	n/a	1800	n/a	41	n/a
	Herschel-Bulkley	n/a	46	n/a	n/a	1700	n/a	35	n/a
BET Surface Area	m^2/g	93	46	4	3	26	44–95	66	70.7

Table VII provides a summary of the supernate concentrations for the various groups. Table 8 indicates that the Na concentrations ranged between ~ 3 M and 5 M in the solutions. As expected, the dominant anion was nitrate. Hydroxide concentrations were relatively low, accounting for less than 15% of the total anion concentration in all cases. This relatively low hydroxide concentration accounts for the relatively low soluble Al concentrations observed. As expected, the highest phosphorous concentrations were observed for the bi-phosphate sludge and the TBP sludge.

Table VII. Initial Supernate Concentration.

		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
Al	µg/mL	<3.77	2,030	2,035	505	2,595	7,590	<0.73	1,430
B	µg/mL	[9.4]	98.6	[6.15]	160	45.8	30.3	30	128
Bi	µg/mL	<2.29	<2.27	<3.61	<2.3	<2.E+0	<2.E+0	<3.65	<3.67
Cd	µg/mL	<0.24	<0.24	<0.50	5.74	<3.E-1	<2.E-1	<0.41	<0.42
Cr	µg/mL	26.0	798	261	221	1,225	535	57.8	180
Fe	µg/mL	<2.05	[7.45]	[1.35]	[3.05]	<2.E+0	<2.E+0	[1.15]	45.6
K	µg/mL	[85]	979	1,055	278	487	1,140	[86]	1,110
Mn	µg/mL	<0.21	<0.20	[0.37]	[0.81]	<2.E-1	<2.E-1	[0.16]	[0.18]
Na	µg/mL	89,300	112,000	73,700	65,300	73,700	117,500	92,300	79,900
Ni	µg/mL	<0.58	[0.72]	3.27	37.5	<6.E-1	[2.25]	<0.42	125
S	µg/mL	5,355	3,845	7,260	3,755	[235]	2,615	6,260	2,290
Si	µg/mL	12.6	[8.25]	[3.80]	551	54	90	<0.68	25.6
Sr	µg/mL	<0.017	<0.02	[0.043]	[6.2]	[0.039]	<2.E-2	[0.050]	2.2
U	µg/mL	<8.41	<8.33	[17]	[0.101]	<9.E+0	<9.E+0	162	[7.3]
Zn	µg/mL	[2.51]	[3.5]	[1.35]	[65]	<6.E-1	<6.E-1	[.99]	[1.1]
Zr	µg/mL	<0.81	<0.81	<0.13	<0.6	<8.E-1	<8.E-1	<0.13	<0.13
nitrite	µg/mL	2,820	11,600	19,350	10,400	24,500	37,650	19,000	18,500
nitrate	µg/mL	198,500	177,000	46,800	100,150	89,600	119,500	193,000	70,900
phosphate	µg/mL	14,800	2,805	3,635	12,250	1,165	8,355	11,300	7,420
sulfate	µg/mL	14,800	11,550	20,400	1,925	702	7,965	17,000	6,240
oxalate	µg/mL	[36]	1,305	3,040	3,675	873	<5.8	<5	2,910
free OH	M	<0.001	0.295		0.104	0.235	0.715	0.35	0.295

Analyte uncertainties were typically within ±15%; results in brackets and red indicate that the analyte concentrations were greater than the minimum detection limit (MDL) and less than the estimated quantitation limit (EQL), and uncertainties were >15%. Results in blue and preceeded by "<" indicate less than MDL.

The chemical analysis of the washed solids for each of the groups is provided in Table VIII. Inspection of Table VIII indicates that 5 waste groups contained quantities of aluminum in excess of 100,000 ppm. XRD analysis of Group 3 and 4 indicated that the aluminum in these waste types is predominately gibbsite. Analysis of Group 6 indicates that approximately 80% of the aluminum was gibbsite, while 20% was boehmite. Analysis of Group 5 indicates that this contained approximately 90% boehmite and 10% gibbsite. The Group 2 solids contained mostly gibbsite with some alumino silicate (as indicated by the presence of a relatively large Si concentration. As expected, the FeCN wastes (Group 8) contained a large quantity of Fe, as did the Group 7 and Group 1 wastes. Groups 7 and 8 contain very high concentrations of U. Groups 1, 2, and 7 contain very high concentrations of Na that was not removed through washing.

Table VIII. Initial Washed Solids Characterization.

	Group 1	**Group 2**	**Group 3**	**Group 4**	**Group 5**	**Group 6**	**Group 7**	**Group 8**
Al	26,350	112,500	297,500	296,500	323,000	165,500	17,275	88,350
B	[130]	[105]	<27	<72	[78]	<95	[115]	
Bi	98,200	[895]	[730]	[1,100]	<65	[395]	6,093	5,600
Cd	<7	90.2	[24]	[7.9]	<3	114	<8.4	
Cr	4,260	7,485	314	1,610	2,110	78,950	772	2,145
Fe	85,550	21,150	14,000	5,090	6,800	13,400	148,000	103,000
K	na		na	na			[315]	
Mn	373	1,034	1,009*	1,545	4,450	3,950	905	1,295
Na	146,000	177,000	[14,500]	[11,000]	55,200	[83,500]	141,000	[50,500]
Ni	na		na	na			517	
S	[3,250]	[1,450]	<554	[815]	<317	<777	[1,088]	[4,400]
Si	42,850	31,650	8,980*	5,985	8,390	15,500	7,285	[17,000]
Sr	888	4,005	83.6	[24]	1,160	[44]	4,183	41,300
U	[7,800]	14,650	12,750	[3,400]	19,500	[4,000]	119,000	121,000
Zn	[380]	[325]	184	723	[56]	639	771	[580]
Zr	[205]	<59	4,810	<48	[140]	[65]	[23]	98

Analyte uncertainties were typically within ±15%; results in brackets and red indicate that the analyte concentrations were greater than the minimum detection limit (MDL) and less than the estimated quantitation limit (EQL), and uncertainties were >15%. Results in blue and preceeded by "<" indicate less than MDL.

Table IX contains a summary of the residual solids after proposed waste processes, generally caustic leaching, or in the cases of Group 6 and 1-2, caustic leaching and oxidative leaching. The highlighted elements represent the dominant species present in each of these leached solids. Note that Group 8 was not leached as it was determined that leaching would not improve the glass loading characteristics. As expected, Group 1 is a mix of Fe and Bi. Groups 5 and 7 contain mostly U after leaching, while Group 6 contains a significant quantity of Cr after caustic leaching. After oxidative leaching of Group 6, as one would expect, a large quantity of Mn is present (residual from the permanganate oxidant) as well as a marked decrease in Cr. Interestingly, the Group 2 and Group 3 solids contain a large quantity of Al after caustic leaching. However, this Al is in nearly stoichiometric ratio to Si, suggesting that it is present as an aluminosilicate.

Table IX. Leached and Oxidatively Leached Solids Composition

		Caustic Leaching							Oxidative Leaching	
		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 6	Group 1-2
Al	microg/g	[12,000]	91,200	83,400	[38,000]	86,200	73,500	[6,550]	26,800	82,000
B	microg/g	[210]	--	[57]	[410]	[490]	[330]	<31.452	--	[54]
Bi	microg/g	317,000	3,670	85,900	33,000	[1,100]	[1,400]	13,950	[1,500]	110,000
Cd	microg/g		325	[120]	[240]	[45]	425	[66]	449	159
Cr	microg/g	13,300	10,300	13,600	16,500	9,940	238,000	1,370	6,250	5,850
Fe	microg/g	306,000	83,600	111,000	232,000	74,700	42,700	331,000	45,000	139,000
Mn	microg/g	1,230	4,180	1,730	97,200	64,400	13,700	1,975	313,000	15,100
Na	microg/g	[14,000]	[100,000]	138,000	[38,000]	[80,000]	[41,000]	[32,500]	[96,000]	88,500
Ni	microg/g			na	na	na	na	18,400	na	na
S	microg/g	[1,000]	[3,900]	--	[5,300]	[4,600]	--	<1347.953	--	--
Si	microg/g	20,200	95,800	84,400	56,000	18,700	21,800	[8,250]	[7,400]	86,200
Sr	microg/g	3,120	17,500	6,250	1,500	15,300	[180]	9,165	[150]	7,690
U	microg/g	8,470	59,800	27,900	151,000	295,000	[11,000]	217,500	[15,000]	34,500
Zn	microg/g	207	2,290	596	2,840	[280]	1,690	749	[380]	790
Zr	microg/g	583	--	190	[330]	[1,500]	[920]	[120]	--	[85]

Analyte uncertainties were typically within ±15%; results in brackets and red indicate that the analyte concentrations were greater than the minimum detection limit (MDL) and less than the estimated quantitation limit (EQL), and uncertainties were >15%. Results in blue and preceeded by "<" indicate less than MDL.

CONCLUSIONS

A large fraction of the tank waste types present at Hanford was analyzed in an attempt to understand the composition of the feeds to the Waste Treatment Plant and the expected performance of those feeds during planned operations. The data suggest that there is a variety of phases present for the species of interest. Three distinct insoluble Al phases have been identified, gibbsite, boehmite and aluminosilicates. In addition, a wide range of compositions for the wastes has been identified. Several wastes have been identified that are predominately U. In addition, the physical characteristics of the waste have been measured, indicating that, as one might expect, there is a wide range in both settling behavior and in rheology behavior. These results can be used to provide insight into the eventual types of feeds and leaching behaviors as a result of operations of the Waste Treatment Plant.

REFERENCES

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