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## Leaching of Iron from Hanford Tank Sludge: Results of FY 1997 Studies

G.J. Lumetta

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## **Leaching of Iron From Hanford Tank Sludge: Results of FY 1997 Studies**

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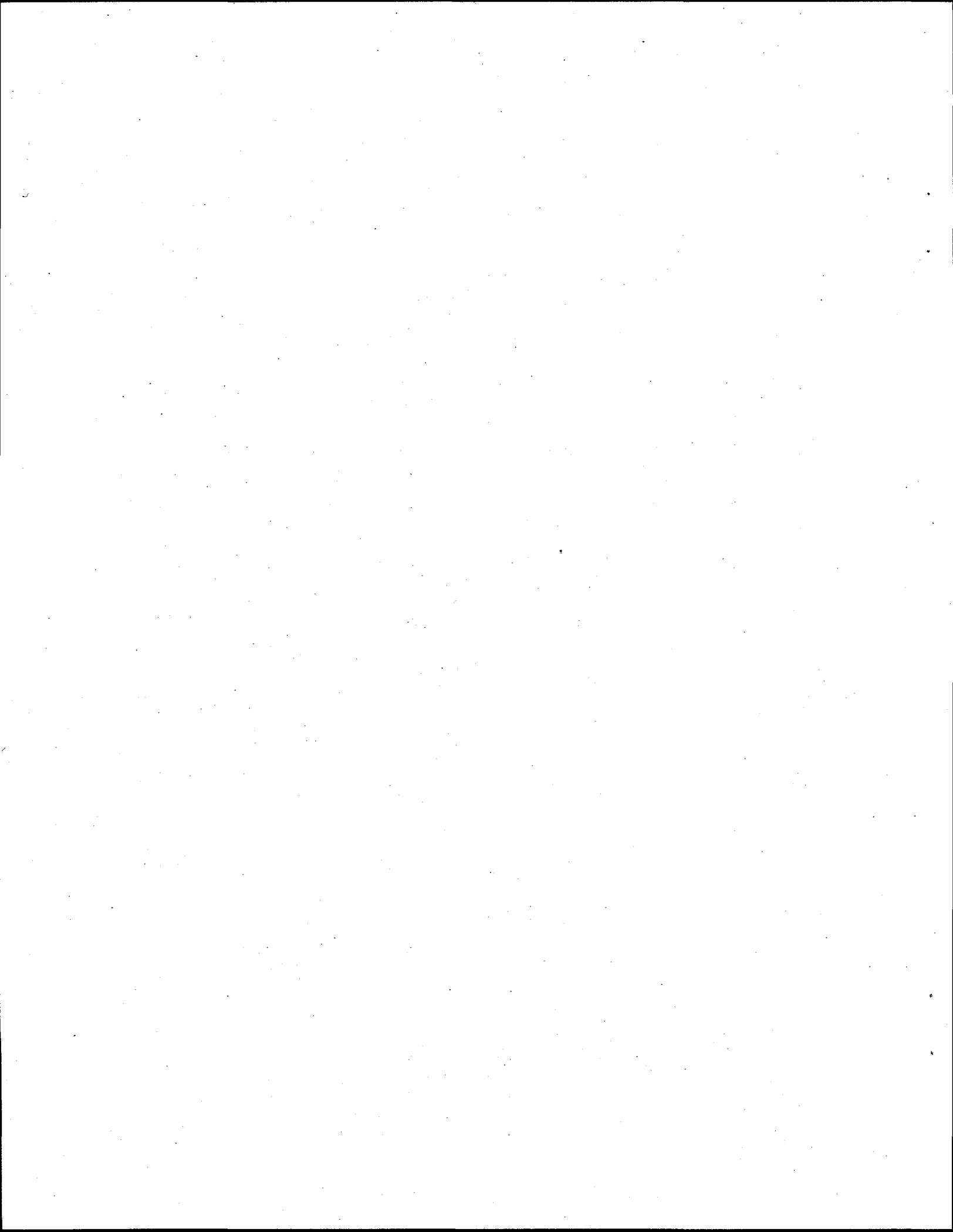
Pacific Northwest National Laboratory  
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## Summary

This report describes tests conducted at Pacific Northwest National Laboratory to investigate selective leaching of iron from Hanford tank sludges. As Fe represents a significant fraction of the sludge mass, its removal would reduce the mass of the material needed to be handled as high-level waste. Iron leaching can be viewed as an enhancement to the baseline method of caustic leaching, which primarily removes Al, P, and Cr. The U.S. Department of Energy funded the work through the Tanks Focus Area (TFA; EM-50). The results of this work can be summarized as follows.

- Acetohydroxamate (AHA) and 4,5-dihydroxy-1,3-benzenedisulfonate (Tiron®) can dissolve Fe from  $\text{Fe}(\text{OH})_3$  over a wide range of pH, but how thoroughly it dissolves appears to depend on the history of the  $\text{Fe}(\text{OH})_3$ . That is, it is more difficult to dissolve the Fe for  $\text{Fe}(\text{OH})_3$  that has been aged (as would be expected to be found in Hanford storage tanks).
- For AHA, Fe dissolution decreases upon increasing the pH from 9 to 13, but the opposite is true for Tiron®. Indeed, Tiron® at pH 13 is the most promising of the systems examined for leaching Fe from tank sludges.
- About 50% of the Fe was removed from Hanford Tank U-110 sludge by leaching with Tiron® at pH 13. A number of other elements (Bi, Cr, Mn, and Si) also dissolved to a certain extent, which is a desirable feature of the process (i.e., the mass of the HLW solids is reduced further). A combination of caustic and Tiron® leaching reduced the mass of the sludge solids by 97%. On the other hand, the TRU elements remained largely in the solids. It is estimated that the LLW form generated from immobilizing the leaching solutions would be well below the 100-nCi/g TRU limit.

It can be concluded that Tiron® leaching might be a useful enhancement to the Hanford sludge pretreatment flowsheet because it has the potential to further reduce the mass of the HLW solids requiring immobilization. Indeed, because Tiron® is more effective at higher pH, it might be possible to simply add Tiron® into the caustic leaching solution to dissolve Al and Fe (along with other elements) in a single step.

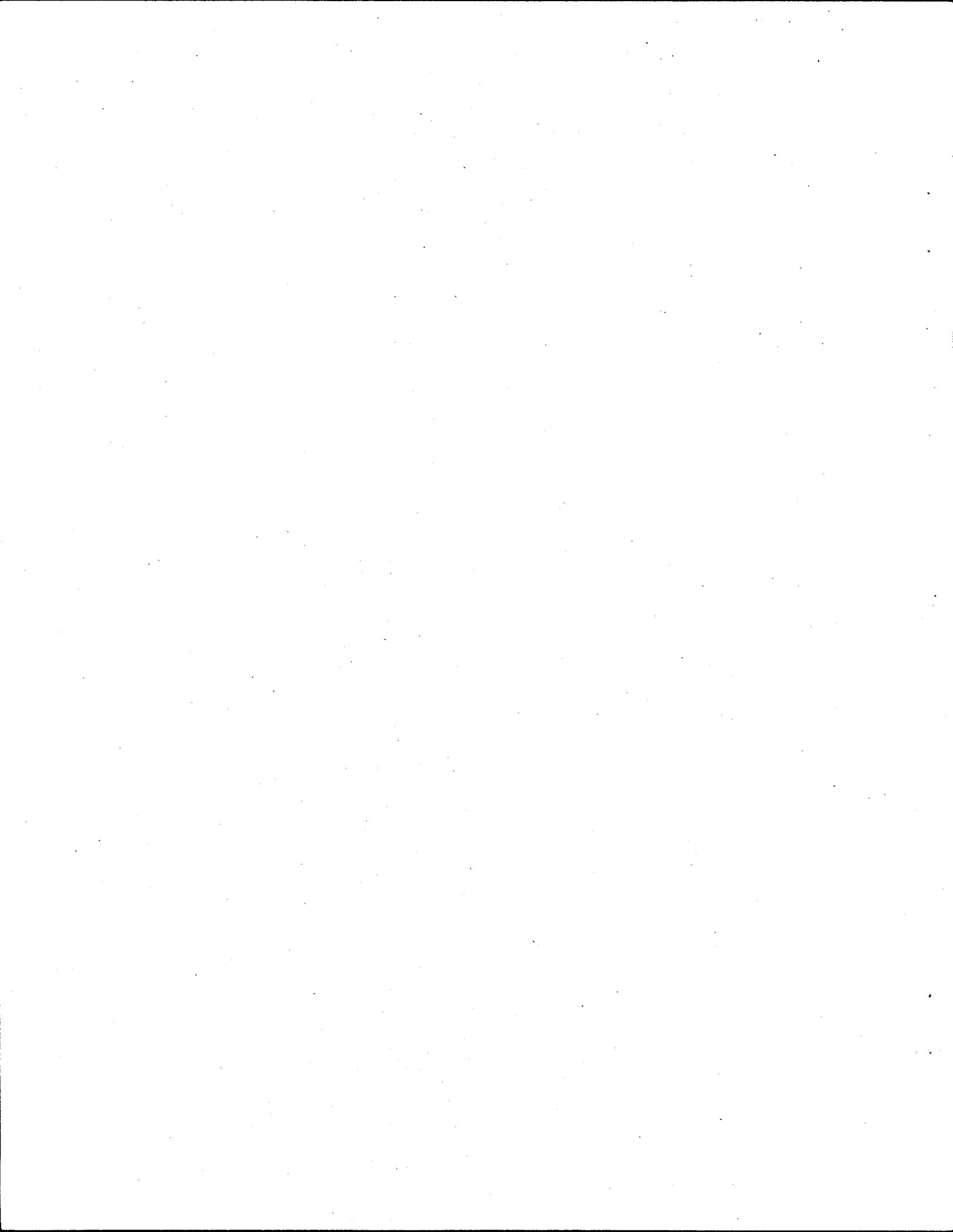


## Acknowledgments

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## 1.0 Introduction

During the past few years, the primary mission at the U.S. Department of Energy's Hanford Site has changed from producing plutonium to restoring the environment. Large volumes of high-level radioactive wastes (HLW), generated during past Pu production and other operations, are stored in underground tanks on site. The current plan for remediating the Hanford tank farms consists of waste retrieval, pretreatment, treatment (immobilization), and disposal. The tank wastes will be partitioned into high-level and low-level fractions. The low-level waste (LLW) will be processed to remove  $^{137}\text{Cs}$  (and possibly other radionuclides), and then it will be immobilized in a glass matrix and disposed of by shallow burial on site. The HLW will be immobilized in a borosilicate glass matrix; the resulting glass canisters will then be disposed of in a geologic repository (Orme et al. 1996). Because of the expected high cost of HLW vitrification and geologic disposal, pretreatment processes will be implemented to reduce the volume of immobilized high-level waste (IHLW).

Dilute hydroxide washing is the minimum pretreatment that would be performed on Hanford tank sludges. This method simply involves mixing the sludge with dilute (0.1 M or less) NaOH, then performing some sort of solid/liquid separation. This is meant to remove water-soluble sludge components (mainly sodium salts) from the HLW stream. Dilute hydroxide is used rather than water to maintain the ionic strength high enough that colloidal suspensions are avoided. And if performed in existing carbon-steel tanks, nitrite would be added for corrosion control.

Caustic leaching (sometimes referred to as enhanced sludge washing or ESW) represents the baseline method for pretreating Hanford tank sludges. Caustic leaching is expected to remove a large fraction of the Al, which is present in large quantities in Hanford tank sludges. The Al will be removed by converting aluminum oxides/hydroxides to sodium aluminate. It is also expected that water-insoluble metal phosphates and sulfates will metathesize to insoluble hydroxides and soluble  $\text{Na}_3\text{PO}_4$  and  $\text{Na}_2\text{SO}_4$ , thus removing significant portions of P and S.

Based on its known amphoteric behavior (Rai, Sass, and Moore 1987), Cr(III) is expected to be removed by caustic leaching according to the following equation:



However, recent studies conducted at the Pacific Northwest National Laboratory (PNNL) have suggested that the behavior of Cr in the caustic leaching process is more complex (Lumetta et al. 1997).

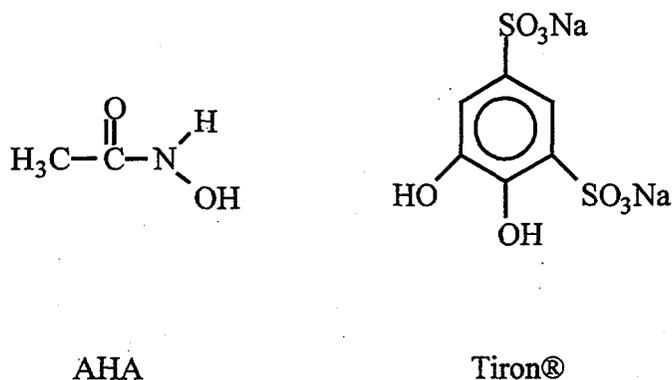
Results of testing of the baseline Hanford sludge washing and caustic leaching process have been reported (Lumetta and Rapko 1994; Rapko, Lumetta, and Wagner 1995, Lumetta et al. 1996a, 1996b, and 1997, Temer and Villarreal 1995, 1996, and 1997).

Iron is a major nonradioactive component of Hanford tank sludges. It might be beneficial to remove this element from the sludge to reduce the overall mass of the HLW solids. The baseline caustic leaching method is not capable of removing Fe. Thus, we undertook a study to determine the feasibility of

selectively leaching Fe from Hanford sludges by treatment with various Fe-complexing agents. This report describes the results of that study. The efficacy of acetohydroxamic acid (AHA) and disodium 4,5-dihydroxy-1,3-benzenedisulfonate (Tiron®) as Fe-leaching agents was investigated using nonradioactive Fe hydroxide. Tiron® was chosen as the best candidate, and a test of this leaching agent was conducted using actual waste from Hanford Tank U-110.

## 2.0 Screening of Fe Leaching Agents

Two compounds were investigated as potential Fe leaching agents: acetohydroxamic acid (AHA) and disodium 4,5-dihydroxy-1,3-benzenesulfonate (Figure 2.1). The latter compound is marketed under the trade name Tiron®. Both of these compounds were obtained from Aldrich Chemical Company (Milwaukee, Wisconsin). The  $\text{Fe}(\text{OH})_3$  used in the initial screening tests was formed by adding KOH to a solution of  $\text{FeCl}_3$ . The precipitated  $\text{Fe}(\text{OH})_3$  was washed with water and then dried at  $105^\circ\text{C}$  for several days. Analysis of the  $\text{Fe}(\text{OH})_3$  indicated it contained 55.5 wt% Fe (the theoretical for  $\text{Fe}(\text{OH})_3$  is 52 wt%). In this report, we will refer to the  $\text{Fe}(\text{OH})_3$  prepared in this manner as "aged"  $\text{Fe}(\text{OH})_3$ .



**Figure 2.1.** Fe Leaching Agents Investigated; AHA = Acetohydroxamic Acid, Tiron® = disodium 4,5-dihydroxy-1,3-benzenesulfonate.

Table 2.1 summarizes the first series of tests run. In this series, the aged  $\text{Fe}(\text{OH})_3$  was contacted with aqueous AHA solutions, and in one case, with an aqueous solution of Tiron®. Different pHs, temperatures, and ligand-to-iron ratios were examined. In these tests, an appropriate amount of the leaching agent was weighed into a 20-mL vial, then 10 mL of water was added to give the desired concentration of leaching agent. The pH was adjusted to the desired value with 10 M NaOH, except for test Fe-A3, which was adjusted with 3 M  $\text{HNO}_3$ . The aged  $\text{Fe}(\text{OH})_3$  was added and the mixtures were stirred overnight at room temperature. After centrifuging, the solutions were sampled for Fe analysis by inductively coupled plasma atomic emission spectroscopy (ICP/AES). In the cases of Fe-A4 and -A5, continued leaching was done at  $50^\circ\text{C}$  and  $100^\circ\text{C}$  as indicated in the table. The solutions were allowed to cool to ambient temperature before sampling for Fe analysis.

With the exception of test number Fe-A2, the amount of Fe dissolved ranged from 20 to 30%. All the leaching solutions had a deep red color. Thus, it appeared that both AHA and Tiron® had a propensity to dissolve Fe(III) over a wide pH range. Heating up to  $100^\circ\text{C}$  had no effect on the amount of Fe dissolved. The fact that less than 30% of the Fe dissolved, even when excess ligand was present, suggests that the

aged  $\text{Fe}(\text{OH})_3$  was composed of more than one chemical species. That is, it is possible that the aged  $\text{Fe}(\text{OH})_3$  contained 20 to 30% of a species that was easily dissolved, but the remainder was resistant to dissolution. Indeed, transmission electron microscopy analysis of the aged  $\text{Fe}(\text{OH})_3$  indicated the presence of two types of particles: needle-like particles (most likely goethite) and round particles (most likely hematite).

In the second series of tests, the  $\text{Fe}(\text{OH})_3$  was formed *in situ* by adding 0.4 mL of 1 M  $\text{Fe}(\text{NO}_3)_3$  to 10 mL of 0.5 M NaOH (this material was allowed to stand at room temperature for 3 days). The leaching agent (AHA or Tiron®) was added and the pH adjusted to the desired value with either 10 M NaOH or 10 M  $\text{HNO}_3$ . In this case, the samples were stirred at ambient temperature, with the solution being sampled at 24, 72, and 168 h. Table 2.2 summarizes the results of this series of tests. For AHA, the amount of Fe dissolved decreased with increasing pH. Figure 2.2 clearly illustrates this, with the Fe concentration  $\sim 700 \mu\text{g}/\text{mL}$  at pH 9, but only  $\sim 200 \mu\text{g}/\text{mL}$  at pH 13. Leaching with AHA for greater than 24 h did not significantly increase the amount of Fe dissolved (Figure 2.1 and Table 2.2). The AHA leaching at pH 9 dissolved 33% of the Fe.

For Tiron®, the results at pH 9 and 11 were very similar, with 32 to 33% of the Fe being dissolved. However, much improved Fe dissolution was obtained with Tiron® at pH 13. In the latter case, kinetics was clearly important in determining the amount of Fe dissolved (Table 2.2 and Figure 2.2). A total of 53% of the Fe was dissolved by Tiron® at pH 13, and the data suggest that more might have dissolved had leaching been carried out past 168 h. Thus, the latter system appeared the most promising for removing Fe from the highly alkaline Hanford sludges.

Interestingly, it was determined that both AHA at pH 9 and Tiron® at pH 13 completely dissolved *freshly* precipitated  $\text{Fe}(\text{OH})_3$ . That is, when tests Fe-A8 and Fe-T4 were repeated without letting the precipitated  $\text{Fe}(\text{OH})_3$  stand for 3 days, clear deep red solutions were obtained. This confirmed that Fe dissolution by these complexants is thermodynamically favorable, but perhaps kinetically impaired for aged iron hydroxide species.

Table 2.1. Summary of Fe Leaching Tests Using Aged Fe(OH)<sub>3</sub>

Test No.	L <sup>(e)</sup>	[L], M	Fe(OH) <sub>3</sub> , g <sup>(b)</sup>	L/Fe	Initial pH	pH <sup>(c)</sup>			Total Fe Dissolved, %		
						R.T.	50°C	100°C	R.T.	50°C	100°C
Fe-A1	AHA	0.1	0.0506	2.0	5.63	7.79	N/A	N/A	20	N/A	N/A
Fe-A2	AHA	0.1	0.0547	1.8	9.59	9.76	N/A	N/A	12	N/A	N/A
Fe-A3	AHA	0.1	0.0482	2.1	1.70	1.90	N/A	N/A	21	N/A	N/A
Fe-A4	AHA	0.25	0.0333	7.6	5.51	7.15	4.70 <sup>(d)</sup>	4.34	21	29	23
Fe-A5	AHA	0.25	0.025	10.1	9.40	9.41	8.78 <sup>(d)</sup>	5.80	20	25	20
Fe-T1	Tiron <sup>®</sup>	0.25	0.0336	7.5	11.31	11.17 <sup>(e)</sup>	N/A	N/A	21	N/A	N/A

(a) AHA =aceto hydroxamic acid, Tiron =4,5-Dihydroxy-1,3-benzenesulfonic acid, disodium salt

(b) Analysis indicated the iron hydroxide material contained 55.5 wt % Fe

(c) pH after stirring overnight at the indicated temperature

(d) Stirred for 4 days at 50°C

(e) Stirred for 6 days at room temperature

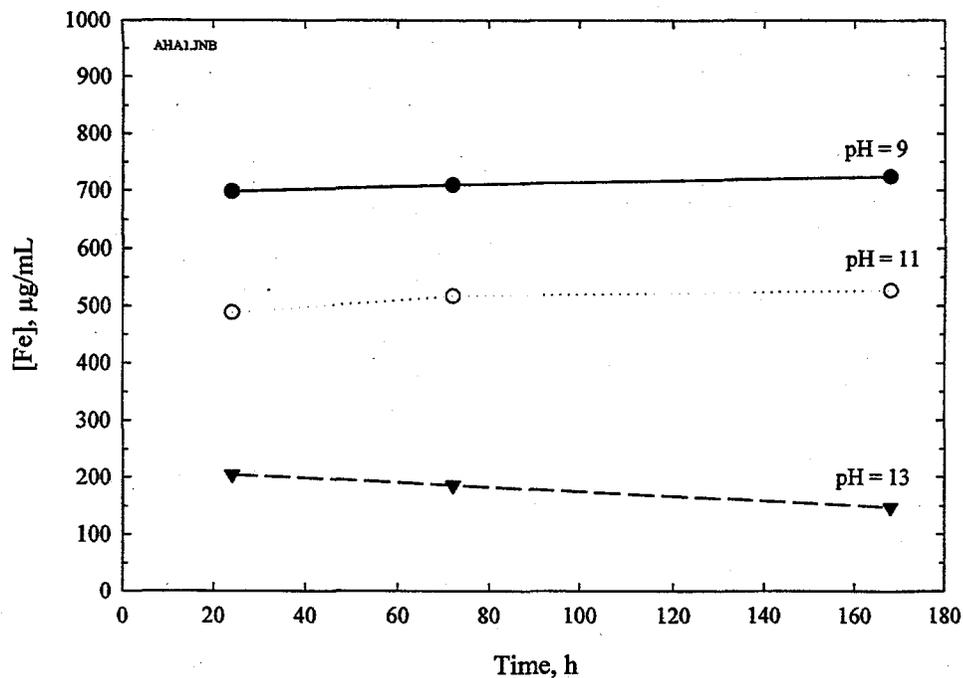
Table 2.2. Summary of Fe Leaching Tests Using Fe(OH)<sub>3</sub> Precipitated *In Situ*

Test No.	L <sup>(a)</sup>	[L], M	Fe, g <sup>(b)</sup>	L/Fe	Initial pH	Final pH <sup>(c)</sup>	Total Fe Dissolved, %		
							24 h	72 h	168 h
Fe-A8	AHA	0.25	0.0223	11.3	9.09	9.17	32	33	33
Fe-A9	AHA	0.25	0.0223	11.3	11.04	11.46	22	23	24
Fe-A10	AHA	0.25	0.0223	11.3	12.93	13.04	9	9	7
Fe-T2	Tiron <sup>®</sup>	0.25	0.0223	11.3	9.00	8.17	32	33	33
Fe-T3	Tiron <sup>®</sup>	0.25	0.0223	11.3	11.08	10.67	31	32	32
Fe-T4	Tiron <sup>®</sup>	0.25	0.0223	11.3	12.80	13.46	39	45	53

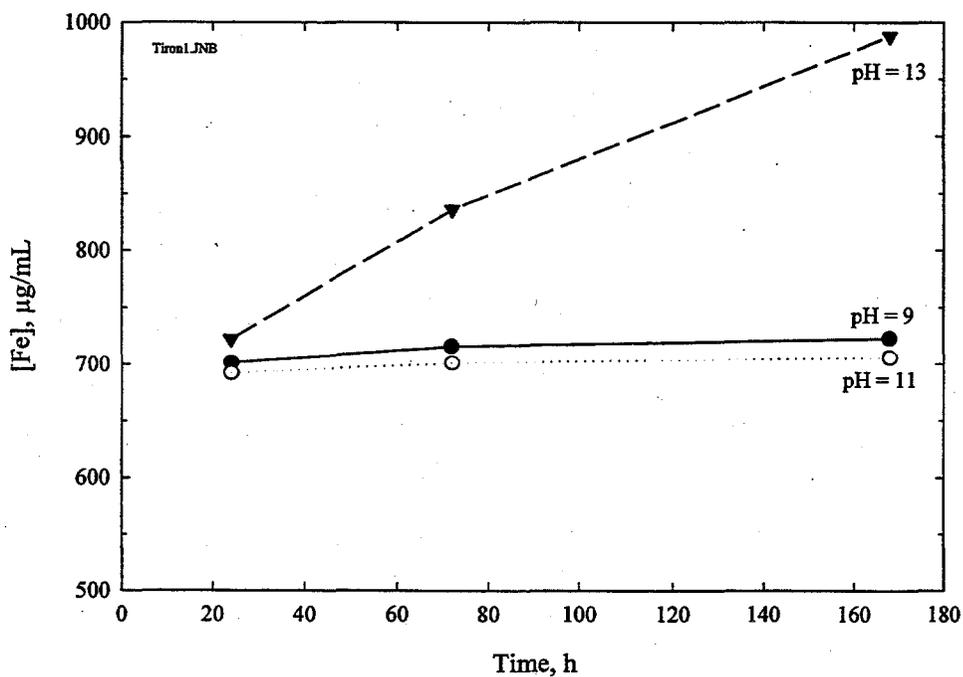
(a) AHA =aceto hydroxamic acid, Tiron =4,5-Dihydroxy-1,3-benzenesulfonic acid, disodium salt

(b) Fe(OH)<sub>3</sub> was formed *in situ* by adding 0.4 mL of Fe(NO<sub>3</sub>)<sub>3</sub> into 10 mL 0.5 M NaOH

(c) pH after stirring 168 h at the room temperature



**Figure 2.2.** Leaching of  $\text{Fe}(\text{OH})_3$  With Acetohydroxamic Acid; Fe Concentration As a Function of Time and pH.



**Figure 2.3.** Leaching of  $\text{Fe}(\text{OH})_3$  With Tiron®; Fe Concentration As a Function of Time and pH.

### 3.0 Iron Leaching From Tank U-110 Sludge

Based on the results of the screening tests (Section 2.0), we performed a test in which sludge waste from Hanford Tank U-110 was leached with Tiron® at pH 13. The U-110 sample used was a composite of material taken from seven different core samplings of the tank. The sample was thoroughly washed with 0.01 M NaOH before use to remove water-soluble components. A sample of the washed solids was dried to constant weight at 105°C, then dissolved for analysis by sequential treatment with aqua regia and 10 M HF. Table 3.1 presents the composition of the U-110 solids. Also, the dried solids contained 0.326 µCi/g transuranic (TRU) elements (as represented by the total alpha activity).

Table 3.1. Composition of the Starting U-110 Sludge Solids<sup>(a)</sup>

Component	Concentration, µg/g
Al	273000
Bi	13380
Cr	400
Fe	20070
Mn	4680
Na	2360
P	2010
Si	7470
U	6075

(a) The U-110 tank solids were washed with dilute NaOH before analysis. Concentrations are on a dry-weight basis.

Figure 3.1 summarizes the experimental procedure followed during the Fe leaching test. The dilute hydroxide-washed solids were leached twice with NaOH (~3 M) at 100°C. The first caustic leaching step was carried out for 3.5 days, while the second lasted 2.5 days. These leaching times were considered adequate for complete dissolution of boehmite from the sample (Lumetta et al. 1997). The caustic leached-sludge was washed thrice with 0.1 M NaOH. The caustic leaching and subsequent washing solution were combined and sampled for analysis.

The caustic leached-solids were treated with 2 g of Tiron® at pH 13. The molar Tiron®-to-Fe ratio was 15 in this first Fe leaching step. The leaching mixture was stirred at ambient temperature for 49 h, after which time, the mixture was centrifuged and the leaching solution decanted. The solids were washed thrice with 0.1 M NaOH. Two subsequent treatments with Tiron® were performed. Again, these steps were conducted with 2 g of Tiron® at pH 13. For both of these, the leaching mixtures were stirred at ambient temperature for 124 h. Finally, the solids were washed thrice with 0.1 M NaOH and then dried at 105°C. The residual solids were dissolved for analysis by sequential treatment with aqua regia and 10 M HF. All the solutions were analyzed for by ICP/AES and the total alpha activities were also determined.

For the key sludge components, Table 3.2 summarizes the removal during each processing step and the amount remaining in the residue. Only Al was removed to any significant extent by caustic leaching.

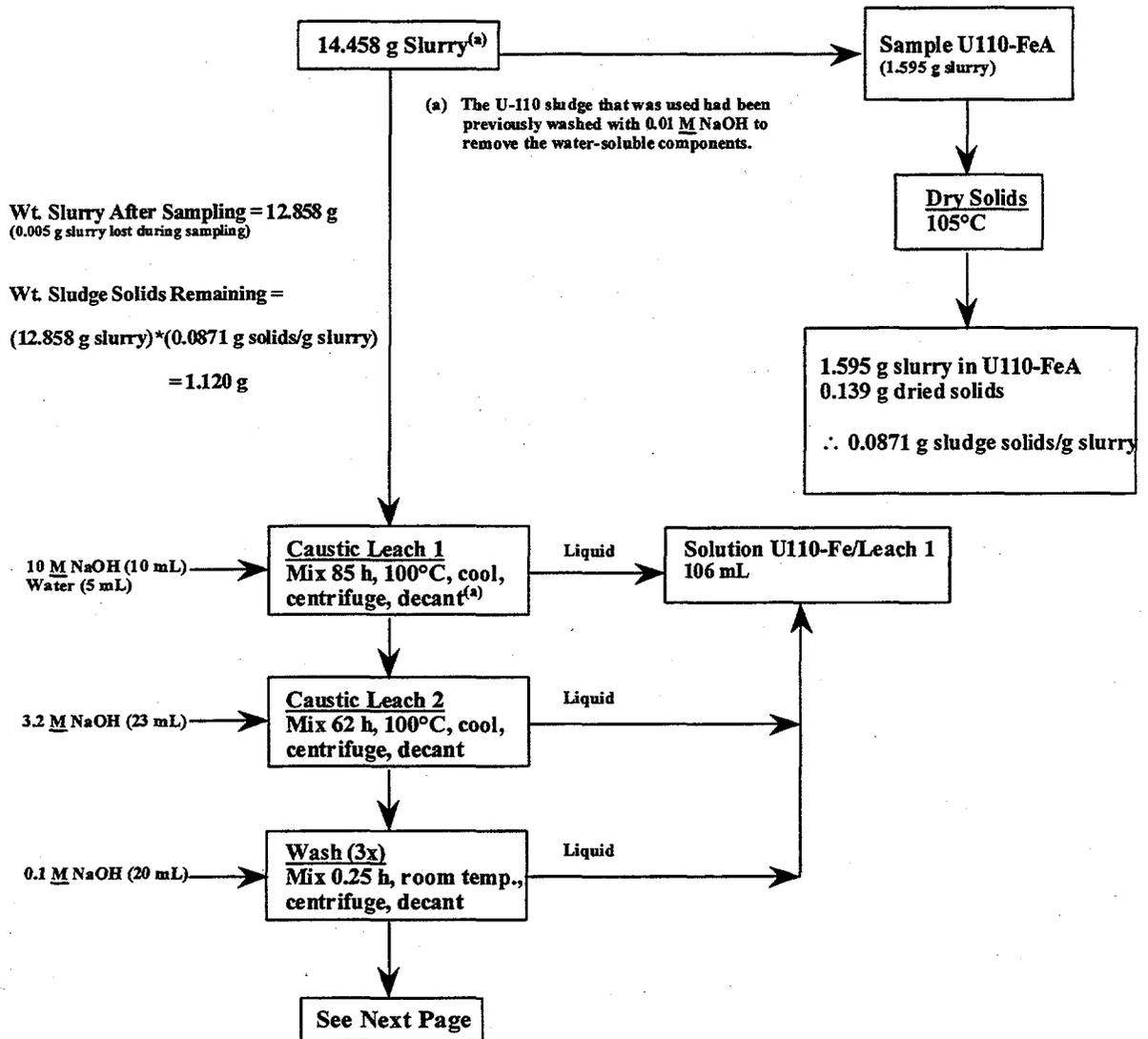
The two caustic leaching steps removed nearly all of the Al. The 96% removal of Al by caustic leaching is the best Al removal from U-110 sludge reported to date. Previous caustic leaching tests with U-110 sludge indicated ~80% Al removal (Lumetta and Rapko 1994), but the duration of those tests was usually much shorter (5 h). The improved Al removal was likely due to more complete boehmite dissolution over the longer leaching times (Lumetta et al. 1997). We have previously identified boehmite in acid-leached U-110 sludge by X-ray diffraction analysis (Lumetta et al. 1993).

Significant fractions of Bi, Cr, Fe, Mn, and Si dissolved upon leaching with Tiron®. Most of the dissolution of these elements occurred in the first Tiron® leaching step, but additional Bi, Fe, and Mn dissolved in the second and third Tiron® leaching steps. Removal of all of these elements from the HLW stream would be beneficial. Only ~50% Fe dissolution was achieved, which is similar to the result obtained in the screening study (Section 2.0).

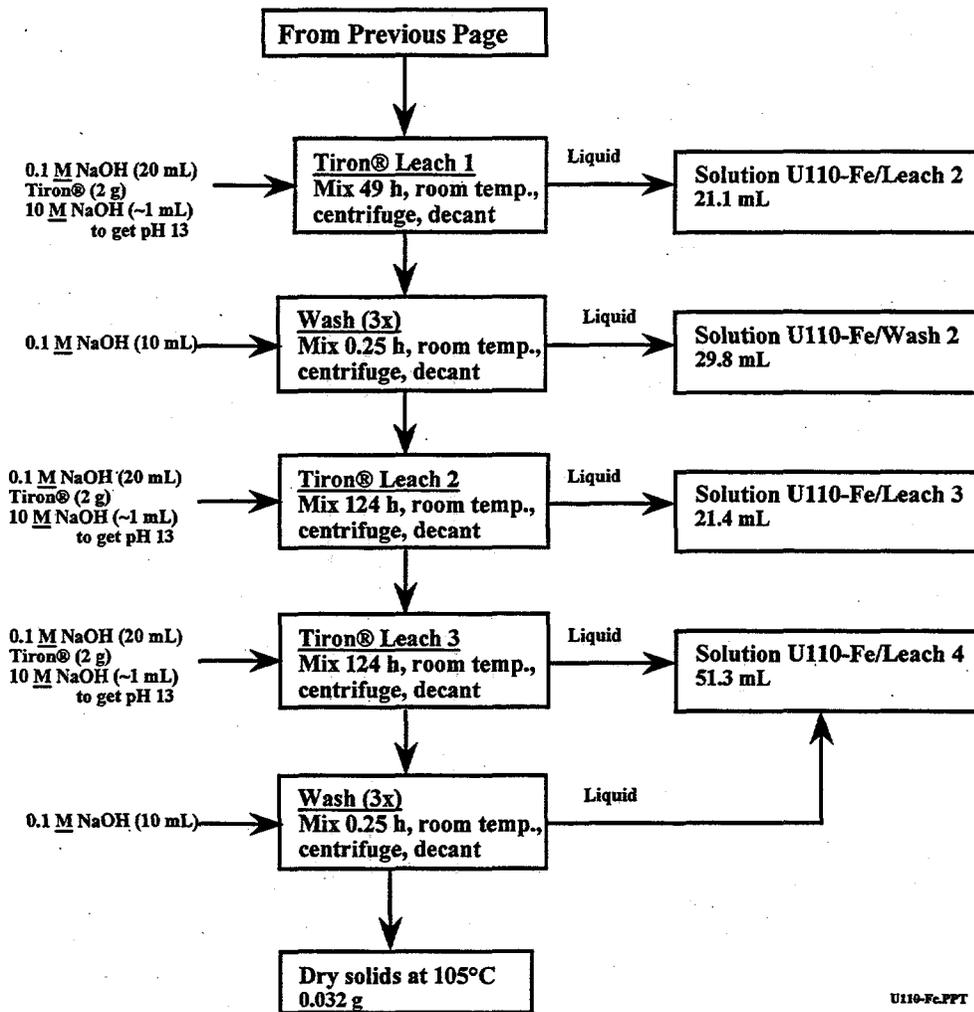
For the leaching process to be successful, it must be sufficiently selective for nonradioactive elements so that the TRU content in the LLW stream remains sufficiently low (i.e., below the 100 nCi/g limit for Class C LLW). Each of the leaching and wash solutions was analyzed for TRU content by measuring the total alpha activity. Summing the amount of TRU and Na found in each solution indicated that the TRU/Na ratio (for the specific conditions of this test) was 0.115 µCi/g Na. If the LLW glass form contains 20 wt % Na<sub>2</sub>O, then the TRU content of the LLW generated by vitrifying this mixture would be ~2 nCi/g. This value is well below the Class C limit of 100 nCi/g and is also below the Class A limit of 10 nCi/g.

Table 3.2. Results of Tiron® Leaching of Tank U-110 Sludge

Component	Removed, %				Residue, %
	Caustic Leach + Wash	First Tiron® Leach + Wash	Second Tiron® Leach	Third Tiron® Leach + Wash	
Al	96	3	1	0.2	0
Bi	0.2	57	19	11	12
Cr	<1	68 < x < 84	<19	5 < x < 6	8 < x < 9
Fe	0	25	7	7	61
Mn	0	95	3	1	1
P	3 < x < 9	32 < x < 74	<52	<13	6 < x < 17
Si	<1	60 < x < 65	<8	27 < x < 29	5
U	<1	<13	<50	<13	>23



**Figure 3.1. Schematic Representation of the U-110 Iron Leaching Test**



U110-Fe.PPT

Figure 3.1. Contd.

## 4.0 Conclusions

Acetohydroxamate and Tiron® can dissolve Fe from  $\text{Fe}(\text{OH})_3$  over a wide range of pH, but the efficacy of dissolution appears to be dependent upon the history of the  $\text{Fe}(\text{OH})_3$ . That is, it is more difficult to dissolve the Fe for  $\text{Fe}(\text{OH})_3$  that has been aged (as would be expected to be found in Hanford storage tanks). For AHA, Fe dissolution decreases upon increasing the pH from 9 to 13, but the opposite is true for Tiron®. Indeed, Tiron® at pH 13 appears to be the most promising of the systems examined for leaching Fe from tank sludges.

Consistent with the nonradioactive screening tests, ~50% of the Fe was removed from Hanford Tank U-110 sludge by leaching with Tiron® at pH 13. A number of other elements (Bi, Cr, Mn, and Si) also dissolved to a certain extent, which is a desirable feature of the process (i.e., a further reduction in mass of the HLW solids is achieved). An overall 97% reduction in the mass of the sludge solids was achieved by a combination of caustic and Tiron® leaching. On the other hand, the TRU elements remained largely in the solids. It is estimated that the LLW form generated from immobilizing the leaching solutions would be well below the 100 nCi/g TRU limit.

It can be concluded that Tiron® leaching might be a useful enhancement to the Hanford sludge pretreatment flowsheet because it has the potential to further reduce the mass of the HLW solids requiring immobilization. Indeed, because Tiron® is more effective at higher pH, it might be possible to simply add Tiron® into the caustic leaching solution to effect Al and Fe (along with other elements) dissolution in a single step.

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Richland, WA 99352

Don Temer  
Los Alamos National Laboratory  
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Los Alamos, NM 87545

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Argonne National Laboratory  
9700 South Cass Avenue, Bldg. 205  
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