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MEMORANDUM

ACC. NO. 105206

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TO: M. A. EBRA

FROM: B. A. HAMM, R. E. EIBLING, J. R. FOWLER
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DEMONSTRATION OF IN-TANK SLUDGE PROCESSING
PART I. ALUMINUM DISSOLUTION, SLUDGE WASHING AND SETTLING RESULTS

INTRODUCTION AND SUMMARY

In-tank sludge processing will reduce the scope and cost of the Defense Waste Processing Facility (DWPF).[1,2] A full-scale demonstration of this process has recently been completed. This test established that aluminum dissolution and sludge washing can be accomplished in a high level waste tank. The Savannah River Laboratory had the responsibility of characterizing (physically and chemically) samples from the demonstration. This memo presents physical properties, aluminum concentration, and supernate inhibitor levels. Later memos will address the concentration of the radionuclides and other chemical species followed during the test.

Using a 1.3 million gallon, Type IIIA waste tank as a process vessel, the in-tank demonstration:

- Removed 79% of the insoluble aluminum from high level waste sludge using caustic dissolution (Goal: 75% of Al^{+3} removed).
- Reduced soluble salts in the liquid associated with the sludge to 0.37 wt% of the liquid by washing the residual sludge three times with inhibited water (Goal: <0.47 wt% to yield a 19 wt%

solids slurry with <2 wt% residual salts on a dry basis). The washes achieved 2.3 wt% residual salt on a 14 wt% total solids basis.

DISCUSSION

The actual sample results are presented in the following sections. A brief review of sample procedure is given first. The test results are given in chronological order.

A simple schedule of events during the test, a record of tank fill volumes and a review of technical standards on inhibitor levels are given in the Appendix.

I. Experimental Procedure

A. Methods of Sampling

Small samples were taken by a dip or pump method and received at SRL in 100-ml stainless steel bottles. Dip samples were collected at the liquid surface by submerging the steel bottle in the slurry. Pump samples were collected at any depth in the slurry by pumping slurry up a polyethylene tube to the sample bottle. Safe transport was insured, in part, by placing the bottles in lead or uranium containers (doorstops) to provide shielding. The samples were transferred from the doorstops to the shielded cells for processing. Required sample volume depended on the analyses to be requested. Bottles were generally filled with about 80 ml of sample. If more sample was required, two bottles were provided and the contents combined in the shielded cells.

Upon receipt in the shielded cells, samples were transferred to polyethylene bottles and the stainless steel bottles were discarded. Samples were shaken thoroughly before they were transferred from the stainless steel bottles, and shaken again before any portion was removed from the polyethylene bottles.

Large samples were obtained by using SRL's 25 liter sampler. The sampler was placed in the cells through the roof of the facility. After positioning the sample over a receiving tank, the sampler was opened by hydraulic pressure. Deionized water was then added through a pressure washer to remove any sludge remaining inside the sampler.

After decanting the sludge and flush water to the receiving tank, the mixture was stirred for one to three hours based on the thickness of the sludge. Samples of the blended slurry were removed by a peristaltic pump and placed in polyethylene bottles. Samples were then processed like the smaller samples.

B. Samples with Visible Insoluble Solids

All weights were determined on a top-loading, digital balance (Mettler or Arbor). Slurry density was determined by weighing 50 ml of slurry in a graduated cylinder. Thirty-five ml of the slurry was then transferred to a centrifuge tube and centrifuged in a HN-S-II centrifuge for one hour. The volume of insoluble solids was recorded. The supernate was decanted and either sampled for analyses or dried to determine the soluble solids. The remaining insoluble solids were washed with 20 ml portions of deionized water. The insoluble and soluble solids were dried overnight (16 hours minimum) in a Blue M oven at 115°C. The dried insoluble solids were weighed, broken up, and transferred to vials for analyses.

C. Dip or Pump Samples Without Visible Solids

The density of the solution was determined as described above. The concentration of insoluble solids was determimined by centrifuging 10 ml portions in a DeLaval gyro-tester centrifuge until sufficient solids were present in the capillary tip of the tube to provide accurate readings. Samples of the solution were taken for analyses.

II. Experimental Results

A. Tank 15H Slurrying and Transfer

To demonstrate in-tank sludge processing, 125,000 gallons of Tank 15H sludge were slurried with sufficient inhibited water and supernate to yield 704,000 gallons of sludge slurry. Samples were taken during the slurrying process to measure the efficiency of mixing and to establish the quantity of sludge transferred to Tank 42H. Both dip and pump samples were taken during this phase of the test. Physical properties of these samples are shown in Table II.A.1. The samples showed that an adequate quantity of sludge had been transferred to Tank 42H. Results from separate determinations of soluble and insoluble solids, compared to a single "total solids" determination, were more consistent. Inhibitor levels were monitored during these operations to assure that technical standards were maintained.[2] They are shown in Table II.A.2.

B. Initial Decanting, Settling, and Blending in Tank 42H

Samples were taken during initial sludge settling to confirm interface detector results. Excellent mixing was achieved as evidenced by the good agreement among samples taken at various depths. After the sludge settled below 67 inches in Tank 42H, clear supernate (approximately 640,000 gallons) was decanted from above the sludge to the evaporator feed tank. The remaining sludge supernate were slurried to test the uniformity of mixing in Tank 42H. After decanting, the sludge layer was blended to establish the weight percent of insoluble solids achieved during initial settling. Based on dip samples and 25 liter samples, the sludge settled to 12.7±0.5 wt% insoluble solids (Table II.B.1). Inhibitors were also monitored during this phase of the test (Table II.B.2).

C. **Aluminum Dissolution**

To dissolve insoluble aluminum salts from the sludge, 104,000 gallons of 50 wt% NaOH and 118,000 gallons of Tank 24H supernate were added to Tank 42H. After this addition, the free hydroxide concentration in the supernate was approximately 3.6 M. At the end of the dissolution and settling cycle, the free hydroxide was approximately 2.9 M (goal = 3.0 M). Soluble and insoluble aluminum was monitored during the 30 day period of caustic addition and heating to establish the efficiency of the dissolution step.

Physical properties of samples taken during the dissolution phase are shown in Table II.C.1. Inhibitor analyses are shown in Table II.C.2. Soluble and insoluble aluminum analyses are shown in Table II.C.3. A plot of mole% of soluble and insoluble aluminum against time shows the effectiveness of aluminum dissolution. At the end of the dissolution cycle, approximately 79% of the total aluminum in Tank 42H was soluble (see Figure II.C.1).

D. **Washing and Settling**

Three washing and settling operations were required to yield the desired soluble salt level in the liquid remaining in the settled sludge. Sodium nitrite inhibitor was added during the second and third wash to maintain Technical Standards for waste storage in a carbon steel tank.

Physical properties of samples from the washing operation are shown in Table II.D.1. Inhibitor levels are shown in Table II.D.2. Final soluble salt concentration in the liquid remaining in the settled sludge was 0.37 wt%. This level of soluble salts corresponds to 1.7 wt% on a dry basis for a slurry containing 19wt% total solids or 2.3 wt% on a dry basis for a slurry containing 14 wt% total solids. The desired concentration is <2 wt% on a dry basis at 19 wt% total solids. The quantity of sodium, determined by atomic absorption, remaining in Tank 42H after each wash is shown in Figure II.D.1. Sodium was tracked to illustrate the efficiency of in-tank washing. At final sludge blending, the contents of Tank 42H were still within Technical Standards.

QUALITY ASSURANCE

The Quality Assurance Program that covers the in-tank demonstration support is described in DPSTQA-82-2-59. Throughout the demonstration, results were recorded on written procedure sheets or in technical notebooks. To improve the quality of the data, chemical analyses were done on multiple samples and the results averaged. In some cases different methods of analysis were applied. Technical notebooks used during the demonstration were DPSTN-3381, 4009, and 4024.

BAH,REE,JRF:pmc
Att
#5

1. JONES, D. W., "Preliminary Technical data Summary for In-Tank Sludge Processing," DPST-80-40, July 1980

2. SNELL, E. B., "In-tank Sludge Processing," Test Authorization NO. 2-1008, January 25, 1982.

3. Memo, R. A. Ator to G. M. Johnson, "Sampling Schedule for In-Tank Processing Demonstration, December 3, 1981.

TABLE II.A.1

SLURRYING AND TRANSFER PHYSICAL PROPERTIES

Sample ID	Date	Type	Volume Percent Solids	Density (g/ml)	wt% Insoluble Solids	wt% Dissolved Solids	wt% Total Solids
Step 1. Slurrying of Tank 15H							
1A	2/11	Dip	11.4	1.18	3.1	20.5	22.6
1B	2/12	Dip	12.1	1.19	4.3	23.6	23.8
1B-P	2/12	Pump	15.1	1.19	3.8	17.3	25.6
1C	2/17	Dip	12.9	1.18	3.8	20.4	30.3
1D	2/18	Dip	13.4	1.18	4.3	21.0	32.6
1D-P	2/18	Pump	17.9	1.19	4.7	21.1	28.9
1E	2/19	Dip	14.3	1.18	4.6	21.4	33.3
1E-P	2/19	Pump	17.1	1.15	4.6	22.9	40.1
1F	2/20	Dip	13.4	1.19	4.6	24.2	34.1
1G	2/21	Dip	14.5	1.18	5.1	22.9	33.6
1H	2/22	Dip	14.3	1.18	4.9	22.9	29.5
1H-P	2/22	Pump	14.7	1.19	4.5	22.2	31.2
1H-R	2/22	Riser	14.7	1.17	4.5	22.5	35.0
1I	2/23	Dip	14.3	1.17	4.7	21.1	31.7
1J	2/24	Dip	14.3	1.18	4.2	20.5	28.8
1J-P	2/24	Pump	15.7	1.18	4.4	20.5	37.9
1K	2/25	Dip	14.3	1.18	4.6	21.1	31.6
1L	2/26	Dip	14.3	1.16	4.0	19.7	24.6
Step 2. Transfer from Tank 15H to Tank 42H							
2A	2/27	Dip	14.3	1.18	4.3	19.2	23.8
2B	2/28	Dip	14.0	1.17	4.3	18.3	22.4
2C	3/1	Dip	14.3	1.16	4.6	18.9	22.7
2D	3/2	Dip	14.3	1.15	4.4	18.0	24.2
2E	3/3	Dip	14.3	1.16	4.3	17.2	22.9
2F	3/4	Dip	17.1	1.13	4.0	15.1	22.4
2G	3/5	Dip	18.6	1.03	4.8	18.3	24.7
2H	3/6	Dip	20.0	1.15	4.7	18.0	25.9
2I	3/7	Dip	18.6	1.16	4.3	18.8	31.9
2J	3/8	Dip	18.2	1.15	4.7	18.4	26.9
2K	3/9	Dip	15.9	1.15	4.2	16.9	21.3
2L	3/10	Dip	15.7	1.15	4.4	16.2	21.0
2M	3/11	Dip	17.1	1.14	4.7	15.7	21.3
2N	3/12	Dip	25.7	1.14	5.6	15.2	24.5
2O	3/13	Dip	45.6	1.17	13.7	5.3	21.7

TABLE II.A.2

SLURRYING AND TRANSFER INHIBITOR AND ALUMINUM LEVELS

Sample ID	Total OH^- (M)	Free OH^- (M)	$Al(OH)_4^-$ (M)	$CO_3^=$ (M)	NO_3^- (M)	NO_2^- (M)	$SO_4^=$ (M)	Al(l) (M)	Al(s) (wt%)
Step 1. Slurrying of Tank 15H									
1A	9.03E-1	8.03E-1	1.73E-1	1.34E-1	1.77	4.90E-1	4.20E-2	1.75E-1	32.4
1B	9.64E-1	7.17E-1	1.30E-1	1.00E-1	1.66	5.70E-1	---	1.88E-1	30.7
1C	8.55E-1	6.93E-1	1.54E-1	1.19E-1	1.71	6.20E-1	4.50E-2	1.69E-1	32.3
1D	8.55E-1	6.92E-1	1.53E-1	1.23E-1	1.74	6.10E-1	4.40E-2	1.82E-1	28.5
1E	8.60E-1	6.83E-1	1.54E-1	1.14E-1	1.67	6.40E-1	4.00E-2	1.70E-1	29.1
1F	9.50E-1	6.70E-1	1.40E-1	1.50E-1	1.66	6.00E-1	4.30E-2	---	28.7
1G	9.70E-1	6.50E-1	1.50E-1	9.00E-2	1.66	6.10E-1	4.30E-2	1.44E-1	25.7
1H	9.52E-1	6.70E-1	1.43E-1	1.50E-1	1.66	6.00E-1	4.30E-2	1.65E-1	---
Step 2. Transfer from Tank 15H to Tank 42H									
2B	8.35E-1	6.37E-1	1.40E-1	1.12E-1	1.62	6.20E-1	4.90E-2	1.56E-1	16.8
2E	8.40E-1	5.76E-1	1.70E-1	1.53E-1	1.44	5.80E-1	3.70E-2	1.35E-1	--
2J	9.06E-1	5.71E-1	9.00E-2	2.30E-1	1.32	5.60E-1	4.40E-2	1.25E-1	29.8
2M	6.26E-1	4.16E-1	1.34E-1	2.09E-1	1.24	4.80E-1	3.80E-2	1.14E-1	24.9

TABLE II.B.1

TANK 42H INITIAL SETTLING, DECANT AND BLENDING PHYSICAL PROPERTIES

Sample ID	Date	Type	Volume Percent Solids	Density (g/ml)	wt% Insoluble Solids	wt% Dissolved Solids	wt% Total Solids
Step 3. Initial Settling							
3A, 3B	3/31,4/1	Samples not used, identification mix-up.					
3C	4/7	Dip	12.9	1.13	3.6	16.9	21.3
3C-55	4/7	Pump	12.9	1.14	3.5	16.8	22.1
3C-110	4/7	Pump	12.9	1.14	3.6	16.9	22.5
3D	4/30	Dip	—	1.10	—	—	—
3D-P	4/30	Pump	6E-3	1.11	—	—	--
3E-130	5/5	Pump	1E-2	1.10	—	—	—
3E-61	5/5	Pump	2.8E-2	1.10	—	—	--
3E-37	5/5	Pump	5.3E-1	1.07	—	—	—
3C	5/22	Pump	6E-3	1.11	—	—	--
	—	Composite	7.4E-3	1.10	—	16.84	—
Step 4. Sludge Blending before Aluminum Dissolution							
4-1	6/3	25 1	39.5	1.15	13.4	13.0	26.3
4-2	6/3	25 1	39.6	1.17	12.9	13.1	25.9
4A1	6/4	Dip	41.4	1.19	12.3	11.2	24.8
4A2	6/4	Dip	37.1	1.15	12.0	11.8	23.1
4A3	6/4	Dip	40.0	1.25	12.6	11.8	24.3

TABLE II.B.2

TANK 42H INITIAL SETTLING, DECANT AND BLENDING INHIBITOR AND ALUMINUM LEVELS

Sample ID	Total OH^- (M)	Free OH^- (M)	$Al(OH)_4^-$ (M)	$CO_3^=$ (M)	NO_3^- (M)	NO_2^- (M)	SO_4^- (M)	Al(l) (M)	Al(s) (wt%)
Step 3. Initial Settling									
3C	—	—	—	—	—	—	—	—	31.9
3C-55	—	—	—	—	1.35	4.20E-1	2.80E-2	1.53E-1	54.0
3C-110	—	—	—	—	Samples were combined				36.5
3D	6.31E-1	5.06E-1	9.27E-2	—	1.33	4.80E-1	3.00E-2	1.32E-1	
3E-130	6.64E-1	5.25E-1	9.81E-2	1.11E-1	1.29	4.70E-1	2.40E-2	—	
3F	6.16E-1	4.91E-1	1.03E-1	5.40E-2	1.14	6.80E-1	1.20E-2	--	
COMP.	9.63E-1	7.87E-1	9.34E-2	3.98E-2	1.20	5.00E-1	3.00E-2	1.62E-1 1.58E-1	
Step 4. Sludge Blending before Aluminum Dissolution									
4A1	6.74E-1	5.43E-1	8.90E-2	9.62E-2	1.12	3.70E-1	<5.00E-2	—	30.2
4-1	8.35E-1	6.90E-1	5.05E-2	1.20E-1	1.53	6.56E-1	4.86E-2	1.65E-1 1.75E-1	27.1
4-2	8.99E-1	6.87E-1	5.39E-2	<2.4E-4	1.53	6.31E-1	4.86E-2	1.55E-1 1.70E-1	25.9
4A2	—	—	—	—	—	—	—	—	27.1
4A3	—	—	—	—	—	—	—	—	22.5
OTHER	—	—	—	—	—	—	—	—	26.9 30.2 32.3

TABLE II.C.1

PHYSICAL PROPERTIES DURING ALUMINUM DISSOLUTION

Sample ID	Date	Type	Volume Percent Solids	Density g/ml	wt% Insoluble Solids	wt% Dissolved Solids	wt% Total Solids
Step 5. Aluminum Dissolution							
5A-1	6/8	Dip	40.0	1.17	10.6	17.0	26.2
5A-2	6/8	Dip	37.1	1.20	10.1	17.0	26.7
5B	6/11	Dip	----	----	----	----	----
5C	6/15	Dip	24.3	1.23	7.8	----	28.7
5D	6/22	Dip	18.4	1.23	6.8	25.7	26.8
5E	6/25	Dip	14.6	1.25	4.1	27.8	28.3
5F	6/26	Dip	15.0	1.25	4.3	28.9	32.4
5G	6/28	Dip	15.7	1.25	4.0	26.9	28.4
5H	6/30	Dip	14.3	1.26	3.3	28.9	28.6
5I	7/1	Dip	12.1	1.22	2.8	28.0	27.5
5J	7/2	Dip	10.0	1.23	3.2	28.4	27.6
5K	7/3	Dip	10.0	1.22	2.7	28.6	27.9
5L	7/4	Dip	8.6	1.24	2.7	28.0	26.8
5M	7/5	Dip	9.9	1.24	2.4	28.5	29.0
5N	7/6	Dip	8.5	1.24	2.1	25.3	27.2
Step 6. Dissolution Settling							
6	7/13	Dip	6.0E-2	1.20	—	—	—
6A-60	7/13	Pump	6.67	1.18	—	—	--
6A-100	7/13	Pump	3.0E-1	1.21	—	—	—
6B-42	7/20	Pump	8.0E-2	1.20	—	—	--
6B-66	7/20	Pump	6.0E-2	1.21	—	—	—
6B	7/20	Dip	4.0E-2	1.22	—	—	—
Step 7. Blended Sludge After Aluminum Dissolution							
7-1	7/30	25 1	19.6	1.20	—	20.4	34.2
7-2	7/30	25 1	21.0	1.24	5.4	23.2	28.2

TABLE II.C.2

INHIBITOR AND ALUMINUM LEVELS DURING ALUMINUM DISSOLUTION

Sample ID	Total OH^- (M)	Free OH^- (M)	$Al(OH)_4^-$ (M)	$CO_3^=$ (M)	NO_3^- (M)	NO_2^- (M)	$SO_4^=$ (M)	Al(l) (M)	Al(s) (wt%)
Step 5. Aluminum Dissolution									
5A-1	2.95	2.52	1.91E-1	4.78E-2	1.01	4.05E-1	4.00E-2	--	27.4
5A-2	2.88	2.44	2.01E-1	8.88E-2	9.35E-1	3.80E-1	2.50E-2	—	25.7
5B	3.21	2.07	3.19E-1	1.04E-1	9.10E-1	3.70E-1	2.40E-2	4.92E-1	22.1
5C	2.89	2.66	3.03E-1	4.60E-2	1.08	2.60E-1	4.00E-2	3.68E-1	
5D	3.49	2.99	4.46E-1	9.39E-2	1.23	2.90E-1	4.00E-2	4.86E-1 5.60E-1	21.3
5E	4.20	3.66	4.27E-1	2.11E-1	1.21	3.20E-1	5.00E-2	5.20E-1 5.20E-1	24.1
5F	3.95	3.40	4.31E-1	1.53E-1	1.44	2.80E-1	6.00E-2	5.20E-1 5.40E-1	24.1
5G	4.07	3.64	4.30E-1	1.00E-1	1.30	2.80E-1	6.00E-2	5.40E-1 5.00E-1	24.9
5H	3.93	3.40	4.19E-1	2.28E-1	1.26	2.70E-1	4.00E-2	5.00E-1 5.10E-1	21.4
5I	3.85	3.32	4.99E-1	1.70E-1	1.17	2.50E-1	3.00E-2	5.90E-1 5.20E-1	23.0
5J	3.85	3.29	5.20E-1	5.80E-2	1.15	2.60E-1	5.00E-2	6.30E-1 6.40E-1	20.9
5K	3.68	3.10	5.70E-1	4.70E-2	1.14	2.50E-1	5.00E-2	6.50E-1 6.70E-1	16.5
5L	3.81	3.01	5.96E-1	1.28E-1	1.19	2.40E-1	5.00E-2	6.90E-1 6.90E-1	19.8
5M	3.82	3.00	5.59E-1	7.53E-2	1.19	2.60E-1	5.00E-2	6.96E-1 6.90E-1	16.1
5N	3.60	3.00	6.00E-1	9.40E-2	1.17	2.50E-1	5.00E-2	7.10E-1 7.00E-1	14.1
Step 6. Dissolutions Settling									
6A	3.56	2.94	5.82E-1	6.40E-2	1.30	2.40E-1	4.00E-2	6.30E-1	
6B	3.60	2.87	5.66E-1	8.46E-2	1.10	2.30E-1	5.00E-2	6.20E-1	
Step 7. Blended Sludge after Aluminum Dissolution									
7-1	3.71	2.77	4.62E-1	2.51E-1	1.24	2.89E-1	4.07E-2	7.29E-1 7.95E-1	14.9 13.2
7-2	3.69	2.97	6.19E-1	9.37E-2	1.32	2.65E-1	4.69E-2	7.70E-1 7.76E-1	
7-3	3.73	2.87	6.66E-1	9.78E-2	1.30	2.65E-1	4.69E-2	7.87E-1 7.40E-1	11.7
OTHER									16.5 17.5

TABLE II.C.3

NORMALIZED ALUMINUM CONCENTRATIONS

Date	Liquid Moles (mole %)	Solid Moles (mole %)	Total Moles
Before (avg)	3.50E5 (18.9)	1.50E6 (81.2)	1.85E6
Caustic Addition			
6/11	3.91E5 (25.4)	1.15E6 (74.7)	1.54E6
6/22	7.17E5 (39.4)	1.10E6 (60.4)	1.82E6
6/25	8.70E5 (49.4)	8.92E5 (50.7)	1.76E6
6/26	9.61E5 (49.0)	1.00E6 (51.0)	1.96E6
6/29	9.61E5 (48.5)	1.02E6 (51.5)	1.98E6
Heating			
6/30	9.92E5 (57.0)	7.47E5 (42.9)	1.74E6
7/1	1.14E6 (62.3)	6.87E5 (37.5)	1.83E6
7/2	1.36E6 (65.7)	7.16E5 (34.6)	2.07E6
7/3	1.42E6 (74.7)	4.78E5 (25.2)	1.90E6
7/4	1.53E6 (71.8)	6.03E5 (28.3)	2.13E6
7/5	1.53E6 (78.1)	4.37E5 (22.3)	1.96E6
7/6	1.59E6 (82.4)	3.39E5 (17.6)	1.93E6
After (avg)	1.53E6 (82.8)	3.18E5 (17.2)	1.85E6

TABLE II.D.1

Sample ID	Date	Type	Volume Percent Solids	Density (g/ml)	wt% Insoluble Solids	wt% Dissolved Solids	wt% Total Solids	wt% Soluble Solids
Step 8. First Water Wash								
8A1	8/6	Dip	7.1	1.07	—	—	—	
8A2	8/6	Pump	7.1	1.08	—	—	—	
8B1	8/9	Pump	5.7	1.04	—	—	—	
8B2	8/9	Dip	4.3	1.04	—	—	—	
8C	8/10	Dip	4.9	1.05	—	—	—	
8D1	8/11	Pump	5.1	1.02	—	6.3	—	
8D2	8/11	Dip	4.5	1.07	—	6.2	—	
8E1	8/12	Dip	4.9	1.03	—	—	—	
8E2	8/12	Pump	4.3	1.04	—	—	—	
Step 9. First Wash Settling								
9A1	8/18	Dip	8.0E-3	1.03	—	—	—	
9A2	8/18	Pump	5.9E-3	1.03	—	—	—	
9B	8/21	Pump	4.0E-3	1.02	—	—	—	
Step 10. Blended Sludge								
10	9/10	Dip	18.4	1.06	4.9	4.4	10.5	
Step 11. Second Water Wash								
11A	9/23	Dip	5.7E-3	0.98	—	—	—	
11B	9/23	Pump	6.0	1.03	1.0	0.6	2.4	
11C	9/24	Dip	2.6	0.99	0.3	1.4	1.8	
11D	9/24	Pump	5.9	0.99	0.8	1.0	2.3	
Step 12. Second Wash Settling								
12A	10/4	Pump	2.0E-3	1.01	—	—	—	
12B	10/4	Dip	2.6E-3	1.00	—	—	—	
Step 13. Blended Sludge								
13	10/21	Dip	24.1	1.02	5.2	0.8	6.0	1.1
Step 14. Third Water Wash								
14A	10/26	Dip	12.9	1.02	—	—	—	—
14B	10/30	Dip	5.7	0.99	1.0	0.5	1.3	0.5
14C	10/30	Pump	10.4	0.99	1.8	0.5	2.3	0.6
Step 15. Third Wash Settling								
15	11/24	Pump	—	0.99	—	—	—	—
Step 16. Blended Sludge								
16	1/4/83	Dip	16.4	1.02	4.8	0.3	5.2	0.35
16-1	1/5	25 l	17.3	1.00	4.9	0.3	5.4	0.38
16-2	1/5	25 l	17.4	1.03	4.8	0.3	5.4	0.39

TABLE II.D.2

INHIBITOR AND ALUMINUM LEVELS DURING ALUMINUM DISSOLUTION

Sample ID	Total OH^- (M)	Free OH^- (M)	$Al(OH)_4^-$ (M)	$CO_3^=$ (M)	NO_3^- (M)	NO_2^- (M)	$SO_4^=$ (M)	Al(l) (M)	Al(s) (wt%)
Step 8. First Water Wash									
8A1	1.00	9.16E-1	8.08E-2	4.28E-2	3.20E-1	6.00E-2	<1.00E-2	2.24E-1	
8A2	1.06	8.50E-1	1.72E-1	<1.00E-2	3.70E-1	7.00E-2	<1.00E-2	2.53E-1	
8B1	8.24E-1	6.90E-1	1.24E-1	—	2.70E-1	5.00E-2	<1.00E-2	1.50E-1	
								1.58E-1	
8B2	8.26E-1	6.84E-1	1.18E-1	—	2.90E-1	5.00E-2	<1.00E-2	1.55E-1	
8C	8.77E-1	7.73E-1	1.33E-1	7.60E-2	2.60E-1	5.00E-2	<1.00E-2	1.50E-1	
8D1	6.49E-1	4.67E-1	1.15E-1	3.80E-2	2.10E-1	2.80E-2	<1.00E-2	1.30E-1	
8D2	6.88E-1	5.23E-1	1.20E-1	2.20E-2	2.10E-1	3.80E-2	<1.00E-2	1.30E-1	
8E1	6.49E-1	5.30E-1	1.17E-1	2.00E-2	2.30E-1	5.60E-2	4.00E-3	1.30E-1	
8E2	6.49E-1	5.22E-1	1.23E-1	1.90E-2	2.40E-1	5.00E-2	4.00E-3	1.30E-1	
Step 9. First Wash Settling									
9A1	7.51E-1	4.87E-1	1.07E-1	8.80E-2	2.40E-1	5.00E-2	3.00E-3	—	
9A2	6.29E-1	4.64E-1	1.15E-1	4.20E-2	2.40E-1	5.00E-2	3.00E-3	—	
9B	8.90E-1	5.07E-1	8.70E-2	9.30E-2	2.40E-1	5.00E-2	3.00E-3	—	
Step 10. Blended Sludge									
10	7.84E-1	5.36E-1	8.80E-2	1.10E-2	2.35E-1	3.60E-2	<1.00E-2	1.18E-1	14.9
								1.29E-1	
Step 11. Second Water Wash									
11A	1.20E-1	—	—	—	5.00E-2	2.00E-2	<1.00E-3		
11B	1.21E-1	—	—	—	5.00E-2	2.00E-2	<1.00E-3		
11C	1.31E-1	—	—	—	5.00E-2	2.00E-2	<1.00E-3	2.24E-2	
								2.16E-2	
11D	9.27E-2	—	—	—	4.00E-2	1.00E-2	<1.00E-3	1.76E-2	
								1.68E-2	
Step 12. Second Wash Settling									
12A	1.12E-1	7.20E-2	3.10E-2	3.60E-2	4.40E-2	1.60E-2	2.00E-3	—	
12B	1.18E-1	7.18E-2	3.24E-2	4.16E-2	4.50E-2	1.60E-2	2.00E-3	—	
Step 13. Blended Sludge									
13	1.17E-1	8.75E-2	1.57E-2	3.92E-2	1.17E-2	5.33E-2	2.26E-5	1.95E-2	16.1
								1.61E-2	17.4

TABLE II.D.2, cont'd

INHIBITOR AND ALUMINUM LEVELS DURING ALUMINUM DISSOLUTION

Sample ID	Total OH^- (M)	Free OH^- (M)	$Al(OH)_4^-$ (M)	$CO_3^=$ (M)	NO_3^- (M)	NO_2^- (M)	$SO_4^=$ (M)	Al(l) (M)	Al(s) (wt%)
Step 14. Third Water Wash									
14a	4.55E-2	3.53E-2	1.20E-2	1.40E-2	2.58E-2	1.95E-2	8.64E-4		
14B	3.80E-2	3.20E-2	5.47E-3	1.50E-3	1.02E-2	2.48E-2	6.68E-4	6.77E-3 6.50E-3	22.6
14C	3.80E-2	2.90E-2	5.41E-3	1.76E-3	1.02E-2	2.57E-2	6.94E-4	6.62E-3 6.62E-3	18.6
Step 15. Third Wash Settling									
None									
Step 16. Blended Sludge									
16	2.29E-2	3.75E-3	4.55E-3	1.68E-2	7.92E-3	2.44E-2	8.22E-4	4.48E-3 2.03E-3	18.1
16-1	2.86E-2	3.073-3	1.19E-2	2.73E-2	6.57E-3	2.10E-2	7.19E-4	3.43E-3	20.9
16-2	2.86E-2	7.01E-3	1.59E-2	4.00E-2	6.86E-3	2.16E-2	1.10E-3	5.11E-3	20.3

FIGURE II.C.1
ALUMINUM CONCENTRATION DURING DISSOLUTION STEP

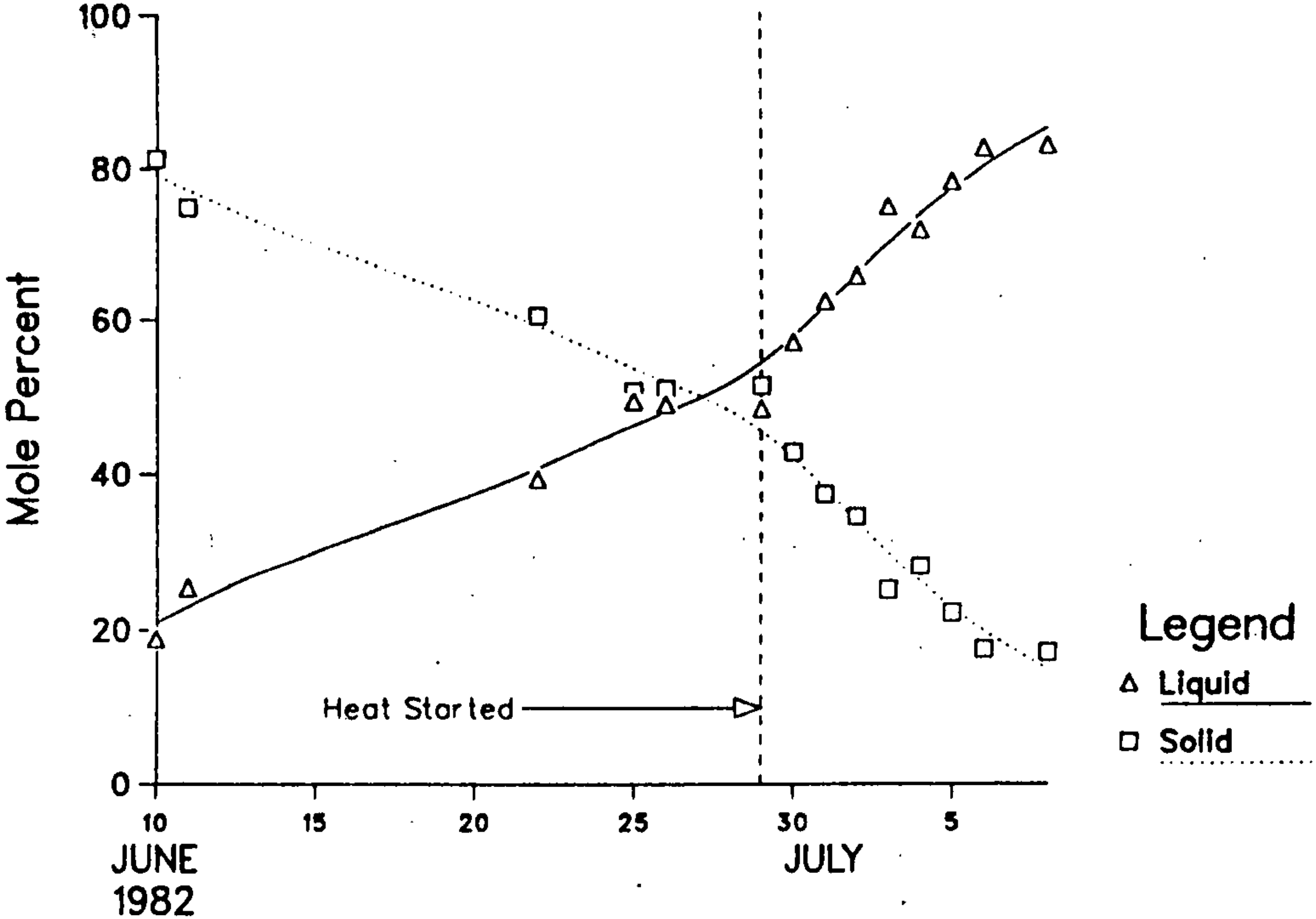
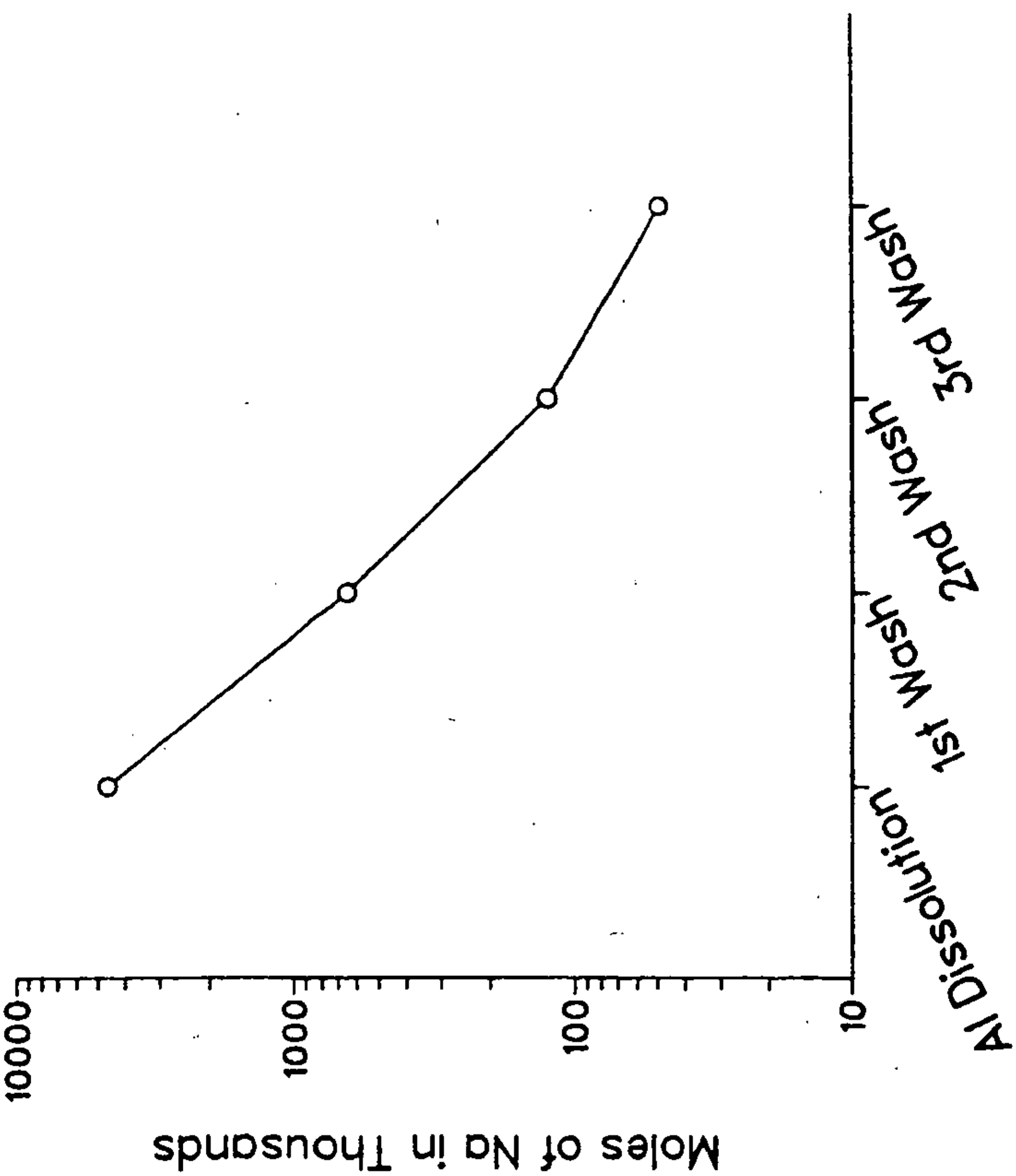


FIGURE II.D.1
SODIUM DURING WASH STEPS



APPENDIX

TABLE 1

TEST SCHEDULE OF EVENTS

Step 1.	**Slurrying of Tank 15H**
2/10	Begin Tk15H slurrying
2/12	Slurry pumps lowered, pumps down for repairs
2/16	Slurry pumps restarted
2/26	Begin transfer to Tk42H
Step 2.	**Transfer from Tk15H to Tk42H**
3/3	Transfer stopped, slurrying to increase solids
3/9	Transfer restarted
3/13	Transfer stopped, completed
Step 3.	**Initial Settling**
3/31 to 4/7	Doing pump mixing tests
4/7	Start settling
5/25	Start supernate decant
6/1	Finish decant
Step 4.	**Sludge Blending**
6/4	Samples of blended sludge
Step 5.	**Aluminum Dissolution**
6/7	Start caustic add'n (add off & on for days)
6/11-6/14	Tk24 add'n
6/18-6/23	Down with pump problems
6/27-6/29	Tk24 add'n
6/29	Start heating
7/6	Dissolution completed, start settling
Step 6.	**Dissolution Settling**
7/6-7/22	Settling
7/22	Start supernate decant
Step 7.	**Blended Sludge**
7/30	Samples of blended sludge
Step 8.	**First Wash**
8/2	Start water addition
8/11	Finish water add'n. Had pump problems
8/13	Start settling

Table 1, cont'd

Step 9.	**First Wash Settling**
8/13-8/24	Settling
8/24-29	Start decant
8/29-9/3	Waiting for space
9/3-9/7	Finish decant
Step 10.	**Blended Sludge**
9/10	Sample of blended sludge
Step 11.	**Second Wash**
9/10-9/22	Water addition, making videotapes
9/24	Sample
9/27	Start settling
Step 12.	**Second Wash Settling**
9/27-10/4	Settling
10/4	Sample
10/6-10/20	Supernate decant, slow due to pump problems, waiting for room
Step 13.	**Blended Sludge**
10/22	Sample of blended sludge
Step 14.	**Third Wash**
10/22-10/30	Water addn
Step 15.	**Third Wash Settling**
10/30	Start settling
11/30-12/3	Decant 1st
12/29-1/2	Decant 2nd
Step 16.	**Blended Sludge**
1/4	Sample
1/5	

TABLE 3
TECHNICAL STANDARDS FOR INHIBITOR CONCENTRATIONS[2]

CONDITION	MINIMUM CONCENTRATION
NO_3^- 3.0 TO 5.5 M	OH^- = 0.3 M
	$OH^- + NO_2^-$ = 1.2 M
NO_3^- = 1.0 TO 3.0 M	OH^- = 0.1 NO_3^-
	$OH^- + NO_2^-$ = 0.4 NO_3^-
NO_3^- <1.0 M	OH^- = 0.01 M (pH 12)

NO_2^- $\geq$500 PPM ALWAYS

TABLE 2

TANK FILLS AT DEMONSTRATION STEPS

Date	Tank Fill (inches)
Step 1	
2/11	251.7
2/12	252.6
2/17	257.1
2/18	258.4
2/19	259.7
2/20	261.1
2/21	262.6
2/22	264
2/23	265.6
2/24	266.9
2/25	269.6
2/26	271.2
Step 2	
2/27	241.8
2/28	220.3
3/1	200.5
3/2	180.1
3/3	167
3/4	168.7
3/5	170.3
3/6	171.9
3/7	173.6
3/8	175.2
3/9	166.4
3/10	145.9
3/11	117.7
3/12	96.8
3/13	91.5
Step 3	
4/7	200
4/30	225
5/5	230
5/25	250
Step 4	
6/4	67
6/8	80.1
6/11	87.4
6/15	119.3
6/22	126.2
6/25	147.4
6/26	160.0
6/29	164.8
6/30	172.0
7/1	175.8
7/2	178.0
7/3	179.9
7/4	181.5
7/5	183.5
7/6	185.4
Step 6	
7/13	186.2
7/20	187.9
Step 7	
7/30	67.0
Step 8	
8/6	211.0
8/9	276.0
8/10	297.0
8/11	328.0
8/12	329.8
Step 9	
8/19	332.0
8/21	334.0
Step 10	
9/10	67.0
Step 11	
9/23	330.0
9/24	331.5
Step 12	
10/4	339.3
Step 13	
10/22	71.0
Step 14	
10/27	146.0
10/30	220.0
Step 15	
11/24	225.5
Step 16	
1/4	75.5

TABLE 3

TECHNICAL STANDARDS FOR INHIBITOR CONCENTRATIONS[2]

CONDITION	MINIMUM CONCENTRATION
NO_3^- 3.0 TO 5.5 M	OH^- = 0.3 M
	$OH^- + NO_2^-$ = 1.2 M
NO_3^- = 1.0 TO 3.0 M	OH^- = 0.1 NO_3^-
	$OH^- + NO_2^-$ = 0.4 NO_3^-
NO_3^- <1.0 M	OH^- = 0.01 M (pH 12)

NO_2^- $\geq$500 PPM ALWAYS