WSRC-RP-92-1027

**LATE WASHING FILTER CLEANING CYCLE
DEMONSTRATION (U)**

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Late Washing Filter Cleaning Cycle Demonstration (U)

SUMMARY

The DWPF Late Washing Facility will filter cesium and potassium tetraphenyl borate (TPB) solids using a Mott sintered metal filter, identical to the filter now used in the In-tank Precipitation Facility. The purpose of the late wash step is primarily to remove the nitrite salts from the slurry prior to delivery to DWPF. Periodic chemical cleaning of the filter will be required, presumably after each batch although the actual required frequency could not be determined on the lab-scale. Minimization of chemical cleaning solution volumes is key to maximizing the attainment of the Late Wash facility. This report summarizes work completed in experiments designed to identify minimum cleaning solution requirements. It was observed that 4 wt% oxalic acid and 4 wt% caustic were adequate for restoration of filter flux. It was demonstrated that the cleaning solutions could be reused numerous times. Depletion rates of the caustic and oxalic acid solution strengths, and source terms for the cleaning solutions were calculated. The calculated data indicate that the solutions could meet tank 22 activity limits. Simulated slurry flux rates were comparable to previous results. Recommended cleaning procedures and requirements were elucidated from the results. Based on the results of this experiment, it is concluded that the design should allow for the cleaning cycle to be conducted as follows:

1. Backpulse the filter.
2. Drain the filter shell side and tube side.
3. Flush the filter tube side with 250 gallons of water.
4. Recirculate 1 wt% oxalic acid for 1 hour at 6-8 ft/sec while backpulsing for 1-2 seconds every 5 minutes, drain filter shell side and tube side.
5. Flush the filter with 250 gallons of water.
6. Recirculate 1 wt% sodium hydroxide for 1 hour at 0.5 to 1.0 ft/sec while backpulsing for 1-2 seconds every 5 min, drain filter shell side and tube side.
7. Flush filter with 250 gallons of water.

INTRODUCTION

The DWPF Late Washing Facility (LWF) is designed to remove soluble species from the reprecipitated potassium and cesium tetraphenyl borate solids. According to the current plan, removal is accomplished by filtration of the solids using a Mott sintered metal filter similar to that used in In-tank Precipitation (ITP). Periodic chemical cleaning of the filter will be required. It was assumed for this work that chemical cleaning would be required once per batch. Minimization of the volume of rinse water and cleaning solutions required for chemical cleaning and the reuse of these solutions will permit a smaller, more economical design.

Previous filtration studies have been conducted on simulated slurry solutions with a composition similar to that expected after ITP processing and irradiation with a dose equivalent to two years' storage. It was observed in those experiments that oxalic acid and sodium hydroxide solutions are necessary to restore filter performance. Minimizing the volume of rinse solutions necessary, examination of the reuse of cleaning chemicals, and composition of the solutions, have not been studied.

Laboratory-scale experiments on simulant solution will not allow accurate prediction of the absolute flux or the frequency of cleaning on the plant-scale system. Approximate cleaning reagent depletion rates and chemical compositions can be obtained from a lab-scale apparatus. The composition of the material which predominates in a fouled filter can also be identified. It should be noted that this material may or may not be the actual cause of filter fouling. An approximation of the source term in the cleaning chemicals can also be obtained. Determining a material balance may not be feasible due to the ability to obtain only single samples of several of the solutions which will not permit statistical evaluation of the variability. Clean water flux tests allow absolute determination of the effectiveness of the cleaning cycles. Reuse of the cleaning chemicals to the point where they were no longer effective was considered impractical, and three recycles was considered adequate for the end-point approximation.

EXPERIMENTAL

The Experimental Laboratory Filter (ELF) with a 1/2-inch diameter 0.5-micron sintered metal Mott filter set in the vertical configuration has been described elsewhere (WSRC-RP-92-359 rev.1). The simulant solution used for fouling the filters was provided by the DWPT organization. The simulant

contained potassium and cesium tetraphenyl borate solids, simulated sludge solids, mercury, and was irradiated to a 2-year equivalent dose (at 24 Ci Cs-137/gal). The material was reprecipitated with sodium tetraphenylborate and concentrated to 9-10 wt% insoluble solids prior to use. Analytical results of the material before and after reprecipitation are presented in Table 1. Surfynol™ was added (200 mg/Kg) to mitigate foaming of the simulant.

Deionized water was utilized to rinse the filter and to prepare caustic and oxalic acid (OA) cleaning solutions. Samples of all solutions were collected and submitted to DWPT analytical laboratories at TNX for analysis. Filter flux measurements were determined by calibrating the flow meters with the simulant solution so that no variations due to density or viscosity would be encountered. A regression analysis was used to obtain the calibration curve for the flow meter (Figure 1).

Cycle Sequence

The entire cycle sequence was completed three times. The same slurry was used for each cycle. The same oxalic acid and caustic cleaning solutions were reused for each cycle. The temperature of the cleaning solutions was room temperature (25-30 °C). The filter was initially cleaned, and a clean water baseline was established. Sample designations are given in parentheses following each step. Backpulsing was conducted with air to force draining of the filter shell side. Each cycle sequence involved the following steps:

1. Conduct standard slurry flux measurement (~11 hours)
2. Shut off pump, drain filter
3. Backpulse ELF with air, return backpulsed slurry to tank
4. Rinse filter tube-side with 80 mL water (1 filter internal volume) (#1 WF 80)
5. Repeat filter tube-side rinse with 80 mL water (#1 WF 160)
6. Recycle ~ 2 L of 4.0 wt% OA at 1.6 ft/s, 20 psi (1 hr)
7. Shut off pump, drain filter (sample the OA, #OA 1R)
8. Backpulse ELF with air, return backpulsed liquid to tank
9. Rinse filter tube-side with 80 mL water (#OA1RWF 80)
10. Rinse filter tube-side with 80 mL water (#OA1RWF 160)
11. Recycle ~2 L of 4.0 wt% NaOH @ 0.49 ft/s, 10 psi (1 hr)
12. Shut off pump, drain filter (sample the NaOH, #NaOH 1R)
13. Backpulse ELF with air, return backpulsed liquid to tank

14. Rinse filter tube-side with 80 mL water (#C1RWF 80)
15. Rinse filter tube-side with 80 mL water (#C1RWF 160)
16. Determine the closed-end clean water flux rate

Filter Operation

The filtration cycle was operated under the standard experimental regime for this type of study. A statistically-designed experiment allowed variation of cross-flow velocity (1.9-10 ft/sec) and transmembrane pressure (15-45 psi) over time. The filter was operated for 1 hour at each of 11 combinations of flow and pressure parameters and 25 data points were collected at each combination. The filter was operated for 11 hours between each cleaning cycle. The filter was backpulsed for one second every 15 minutes of operation. Manual backpulsing was also conducted during the cleaning cycle at 15 minute intervals with an approximately 1-second duration.

RESULTS

Analytical Analyses

Analytical results (in triplicate) of the simulant solution composition after reprecipitation are consistent with that expected for irradiated slurry containing both simulated sludge and mercury (Table 1). The measured quantity of soluble potassium is very close to the minimum detection limit of the method (0.1 mg/L) and thus may not be reliable. The concentrations of various organic constituents derived from the radiolysis of tetraphenyl borate (TPB) anion vary due to the inhomogeneity of the sample and sampling technique.

Analytical analyses of all of the flush, rinse, and cleaning solutions were completed for the predominant constituents of interest but were not characterized in triplicate or as extensively as the initial slurry due to the limited sample size (Table 2). The organic and inorganic constituents were quantified by instrumental techniques, and the molarity of the base or acid was determined by titration. From these data, base and acid depletion rates, cross-contamination (i.e. residual oxalic acid reaction with caustic cleaning solution), and expected source terms can be estimated.

Flush Solutions

The filters were flushed with two filter tube volumes (80 mL) of water, and each volume was analyzed. It appears that, for most of the constituents, 80-90% of the material which can be removed by water flushing is removed in the first flushing. The average amount of potassium removed in the first flushing

is 920 mg/L. This corresponds to 74 mg of total potassium removed per flush (80 mL flush). Based on the surface area of the filter (0.262 ft²), this corresponds to 280 mg of potassium collecting per square foot of filter surface area. The average potassium present in the second flush is 125 mg/L or 38 mg/ft² per flush. Similarly, the average titanium content in the first flush is 65 mg/L, which is 5 mg of titanium per flush, or 19 mg/ft². The average second flush yielded 3 mg/ft² of titanium.

Cleaning Solutions

The cleaning solution composition increases for most constituents in a fairly consistent manner (Table 2, Figures 2 and 3). The initial amount of cleaning solution was 2.0 L, which would correspond to 460 gallons of solution in the full-scale filter, based on the filter surface area (2 gal/ft²). Before cleaning was initiated, 125 mL was withdrawn for analysis, and an additional 125 mL was withdrawn at the completion of each cleaning cycle. The oxalic acid cleaning solution contains most of the constituents as it was used first, and because many of the compounds are broken down or are soluble in OA. The depletion rate of acid was negligible, dropping from 0.25 M to 0.23 M after three cleaning cycles. Similarly, the caustic was reduced from 1.09 M to 0.96 M. The final quantity of cleaning solutions remaining after the test was 1.5 L, which was used for the compositional calculations.

The composition of the oxalic acid solution suggests that much of the potassium and boron are removed in the water flushes. Only 14 mg/L of potassium were present after three cycles of OA cleaning, which corresponds to 21 mg total potassium and 80 mg/ft². By comparison, the average amount of potassium removed in each of the first flushes was 280 mg/ft², and 38 mg/ft² in each of the second flushes. The potassium content did not increase linearly as expected. The titanium content is 13.4 mg/L after three cycles and corresponds to 20 mg total (77 mg/ft²). The mercury content at the end of three cleaning cycles was 6.0 mg/L, which is 9 mg total Hg, and 34 mg/ft². Only a small increase in the amount of sodium was observed after three OA cleaning cycles. The increase of 171 mg/L of sodium was expected and is due to a combination of sodium hydroxide, sodium nitrate, and other sodium-containing compounds remaining in the filter after the water flushes. The TPB was not detected in the titration method, presumably due to decomposition of the TPB by the OA, and, according to the potassium content, would be below the detection limit.

The caustic cleaning solution was relatively clean even after three cleaning cycles. Cross-contamination of oxalate ion

from the oxalic acid cleaning solution would account for consumption of only 0.01 moles of the caustic. The potassium content (10 mg/L) corresponds to 15 mg total potassium and 57 mg/ft². The titanium content is approximately 1 mg/L in the final caustic solution, which is approximately 6 mg/ft². The mercury content in this solution increased with each cycle up to 1.31 mg/L after three cycles. The TPB content was below the detection limit of the titration method.

The solutions used for rinsing between oxalic acid and caustic cleaning solutions, and the rinse solutions after the caustic cleaning solution were relatively clean. Approximately 80% of the flushable constituents are removed in the first flush.

Flux measurements collected during the cleaning cycles indicated that cleaning is rapid. In all cases, the flux did not increase further after 30 minutes of cleaning solution circulation. The flux during the OA cleaning cycles decreased each time the solution was reused. The initial OA flux was 96 gpm/ft², the second recirculation flux was 92 gpm/ft², and the third was 39 gpm/ft² at 20 psi of transmembrane pressure. The caustic recirculation at 10 psi transmembrane pressure was similar with 116, 111, and 68 gpm/ft² for the first, second, and third cleaning cycles, respectively.

FLUX MEASUREMENTS

Clean Water Flux

The results from the closed-end, clean water fluxes are shown in Figures 4 and 5. The closed-end flux is the filter flux obtained with the concentrate line valve closed, i.e. ~0 ft/sec crossflow velocity. Historically, a line with a regression coefficient greater than 0.9 and a slope between 0.3-0.5 gpm/ft²/psi is indicative of a clean filter and is consistent with vendor literature. After the first cleaning cycle, a slightly lower slope was observed. Continued recirculation of the deionized water restored the clean-water flux. Following the second cleaning, the caustic cleaning was followed by a weak (0.11 M) oxalic acid flush. This acid flush returned the water flux to an acceptable level. After the third cleaning cycle deionized water was recirculated for 30 minutes and an acceptable flux was restored (See Figure 5).

Simulant Flux

The flux of the simulated slurry during the three 11-hour flux tests is summarized in Table 3. The data includes the

wide range of cross-flow velocities (1.9-10 ft/sec) and transmembrane pressures (15-45 psi).

Table 3
Simulated Slurry Flux

Description	Average Flux	Minimum Flux	Maximum Flux
1st Recirc.	0.037	0.021	0.052
2nd Recirc.	0.044	0.031	0.054
3rd Recirc.	0.048	0.034	0.063

All units are in GPM/ft²

Surfynol™ had been added to the simulant (200 mg/Kg) solution to mitigate foaming in the vigorously-agitated feed tank. Analysis of the filter permeate indicated that Surfynol™ had passed through the filter. As the analytical method development for this sample matrix has not been completed, quantification of the Surfynol™ was not feasible.

DISCUSSION

Cleaning Reagent Depletion

On the laboratory-scale filter, a single water flush after fouling appears to be adequate for removal of 80-90% the flushable species. Similarly, after cleaning with oxalic acid and caustic, a single rinse appears to be adequate. On a full-scale system, rinsing may not be quite as effective due to preferential flow through some tubes relative to others. It would be beneficial to allow for two rinses in each step in order to get a more thorough flush of the filter. Additionally, there would be a concurrent increase in the radioactivity uptake and cleaning reagent depletion rates with a single rinse.

The cleaning solutions, both caustic and oxalic acid, could be reused. The depletion rate of the oxalic acid suggests that it could be used many times. Previous experiments have indicated that a 1 wt% OA solution is adequate for cleaning (WSRC-RP-92-925). The 0.25 M OA solution was determined to still contain 0.23 M OA after three cleaning cycles. The depletion rate observed was only ~0.01 M per cycle, suggesting that it could be used numerous times before depletion. The caustic was similarly shown to decrease only marginally in base strength after three cycles, 1.09 M to 0.96 M (0.04 M per cycle). The caustic could be used numerous times before depletion below 1 wt% (0.25 M), which has also been shown to be effective. (The aliquot removed

after each cycle was not accounted for in the calculations as each was only 8% of the total.)

The flux obtained during the cleaning solution recirculation decreased progressively through the three cleaning cycles. Although the cleaning was effective, as evidenced by the clean water flux, the flux during the cleaning cycle became lower as the solution was reused. This was presumably due to undissolved solids present in the cleaning solution. Although the total insoluble solids measurement does not indicate that large amounts of insoluble solids were present in the cleaning solutions, the solutions were quite cloudy and it was concluded that the lower flux was due to the solids. This conclusion suggests that extended reuse of the cleaning solutions may eventually impact the cleaning effectiveness.

Radioactivity Uptake

The uptake of radioactivity by the cleaning solutions can be estimated from the data obtained in this experiment. The measured amount of potassium can be used to calculate the amount of radioactive cesium expected in the cleaning solutions. It is assumed that the potassium:radioactive cesium ratio is $1.0 \text{ g K} = 1.0 \text{ Ci Cs-137}$. (A 10 wt% solution of KTPB would contain 10.9 g of potassium (0.28 M). The highest projected activity at LWF is 41 Ci/gal, which is 10.8 Ci/L, assuming all activity is due to Cs-137) It is also assumed that the potassium and cesium collect on the filter at the same rate. The amount of potassium present in the oxalic acid solution after one cleaning cycle (14 mg/L) corresponds to 80 mg/ft^2 of filter surface area. This would yield an estimate of 18.4 Ci Cs-137 in the cleaning solution after one cleaning cycle of a 230 ft^2 filter. Recycling the cleaning solution three times would yield 55 Ci of Cs-137 in the solution.

A linear increase in the potassium content in the oxalic acid solution was not observed in this experiment, although the analysis was repeated, and checked with another analytical method. A linear increase in potassium content had been observed in the OA solution in a nearly identical previous experiment. In that experiment, the amount of potassium contained in the cleaning solution after three recycles was 120 mg/L of potassium. The amount of potassium in the flush solutions was also higher, although more variable. The reason for the variability between the experiments has not been determined. As a result, it must be assumed that at least three times as much activity could be in the cleaning solution (i.e. 166 Ci Cs-137 after 3 cycles).

The amount of titanium present in the OA cleaning solution (13 mg/L) can also be used to estimate the activity from the

absorbed sodium titanate. The total amount of titanium was 77 mg/ft², which would be 17.7 g of Ti after three cycles of OA cleaning solution on a 230 ft² filter (=37 g NaTi₂O₅H). The activity associated with this amount of titanium would be dependent on the activity loading on sodium titanate used in the process. The maximum expected amount of activity of Sr-90 is 0.16 Ci/g Ti (WSRC-RP-92-93). The 17.7 g of Ti corresponds to 2.8 Ci of Sr-90 activity in the solution. The amount of Ti in the final OA solution in the previous experiment was 50 ppm, which would predict 68 g Ti (11 Ci of Sr-90) in solution after triple cleaning of the full-scale unit. The contribution from alpha-emitting isotopes or any other isotopes is assumed to be small.

The activity in the caustic cleaning solution was lower than in the oxalic acid solution. The potassium content in the caustic solution after three recycles corresponded to 57 mg/ft², which would predict 13 Ci of Cs-137 to be present. The titanium content was also lower, with a predicted value of 6 mg/ft² (0.2 Ci of Sr-90). In the previous experiment, the potassium content after three recycles was 81 mg/ft² (18.6 Ci Cs-137) and the titanium was 16 mg/ft² (0.6 Ci total Sr-90).

Application to Full-Scale Unit

The volume and concentration of reagents used in chemical cleaning in the full-scale unit is somewhat flexible. A lower concentration of oxalic acid and caustic could be utilized in the full-scale unit. This has the advantage of adding smaller amounts of chemicals but the disadvantage of more frequent make-up and disposal. The pH of the oxalic acid solution must remain below 2 pH units to effectively dissolve sodium titanate. The radioactivity uptake may dominate the concentration and disposal rate. The estimates from this and previous experiments suggest that Ci of Cs-137 may be present after one cleaning cycle. This would yield 0.037 Ci/gal in a 500-gallon OA tank (assuming all activity is due to Cs-137), which is below the 0.6 Ci/gal tank 22 limit. The tank size used for solution recirculation will also impact the concentration and disposal rate. If only one flush of water is conducted prior to chemical cleaning, the increase in radioactivity can be 50% per cleaning cycle (38 mg/ft² of potassium was removed in the second flush, compared to 80 mg/ft² from the first OA cleaning cycle). The volume of water used in flushes and rinses was one internal tube volume (80 mL), this would correspond to 23 gallons in the full-scale unit. However, practical considerations for the feasibility of this in the full-scale system may require a larger volume. At a minimum, 250 gallons of flush water is recommended if a once-through flush can be utilized. More

liquid will be required if the shell and tube side must be filled and the filter backpulsed to initiate solids removal.

Clean Water Flux

It was observed that the clean water flux was obtained only after recirculating deionized water for 30 minutes, or by flushing with a small amount of oxalic acid (Figures 4-5). Even without this step, the filter was still quite clean after each cleaning cycle, compared to the simulated slurry flux. It is therefore not considered crucial for plant operation.

Simulant Flux

The simulant flux was restored after each cleaning cycle (Figure 6). No significant impact on simulated slurry flux was observed by reusing the cleaning solutions. The flux appears to have increased slightly over the course of the experiment. This observation is probably due to the error in the flux measurement and variability in the experiment. The simulant slurry flux is significantly lower than the clean water flux, consistent with previous results (Figure 7).

CONCLUSIONS and RECOMMENDATIONS

Based on the results of this experiment, it is concluded that the design should allow for the cleaning cycle to be conducted as follows:

1. Backpulse the filter.
2. Drain the filter shell side and tube side.
3. Flush the filter tube side with 250 gallons of water.
4. Recirculate 1 wt% oxalic acid for 1 hour at 6-8 ft/sec while backpulsing for 1-2 seconds every 5 minutes, drain filter shell side and tube side.
5. Flush the filter with 250 gallons of water.
6. Recirculate 1 wt% sodium hydroxide for 1 hour at 0.5 to 1.0 ft/sec while backpulsing for 1-2 seconds every 5 min, drain filter shell side and tube side.
7. Flush filter with 250 gallons of water.

Although the laboratory-scale unit was cleaned in 30 minutes at lower cross-flow velocities, the full-scale unit is expected to be more difficult to clean as it may be run longer between cleanings. Shorter cleaning cycles would only be advisable if operational data in the plant suggest that it is acceptable. Cleaning at a higher velocity is recommended as it is expected to be more efficient on a full-scale unit. Fewer water flushes are not recommended because of an increased source term and a more rapid oxalic acid depletion rate. The technical basis for Late Wash (WSRC-RP-92-793) will be updated to reflect the recommended modifications.

QUALITY ASSURANCE

All work was conducted according to the SRTC Quality Assurance Program. This task was described in WSRC-RP-92-359 Rev.1. The sample analyses performed by the Analytical Laboratory of DWPT and were controlled by their QA Program as identified in SRL-PMC-91-0075, Rev.2, June 15, 1992. All data for this test are recorded in Laboratory Notebook WSRC-NB-92-150.

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Table 1. Initial Slurry Composition

	1	2	3	4	5
1		units	REPPT-01	REPPT-02	REPPT-03
2	2-PHENYLPHENOL	MG/L	163	536	81
3	4-PHENYLPHENOL	MG/L	<1	<1	103
4	ANILINE	MG/L	2	14	3
5	BIPHENYL	MG/L	1360	1316	1215
6	DIPHENYLAMINE	MG/L	68	55	104
7	M-TERPHENYL	MG/L	114	107	106
8	NITROBENZENE	MG/L	<1	<1	<1
9	NITROSOBENZENE	MG/L	<1	<1	<1
10	O-TERPHENYL	MG/L	18	19	19
11	P-TERPHENYL	MG/L	28	26	25
12	PHENOL	MG/L	1026	1036	962
13	PHENYLBORONIC ACID	MG/L	528	540	506
14	TOTAL CARBON	PPM	78101	76912	73604
15	TOT INOR+ORG C	PPM	66402	62692	62362
16	TOT INOR CARBON	PPM	2151	2112	2141
17	TOTAL ORG CARBON	PPM	64251	60580	60221
18	CHLORIDE	MG/L	298	290	251
19	FLUORIDE	MG/L	23	109	<94
20	FORMATE	MG/L	315	371	314
21	NITRATE	MG/L	616	19	20
22	NITRITE	MG/L	644	528	439
23	OXALATE	MG/L	435	759	271
24	PHOSPHATE	MG/L	211	95	83
25	SULFATE	MG/L	700	30	81
26	PH	PH	13	13	12
27	AMMONIUM (soluble)	MG/L	<1	<1	<1
28	SODIUM (soluble)	PPM	8177	8007	7036
29	POTASSIUM (soluble)	PPM	0.15	0.12	0.19
30	MERCURY (total)	PPM	1848	1680	1634
31	DIPHENYLMERCURY	MG/L	1634	1349	1154
32	TETRAPHENYLBORATE	WT %	9	9	9
33	BORON	PPM	2747	3172	2161
34	CHROMIUM	PPM	4	4	3
35	COPPER	PPM	1	1	1
36	IRON	PPM	564	557	432
37	MANGANESE	PPM	181	180	137
38	POTASSIUM	PPM	5958	7545	5306
39	SODIUM	PPM	9455	9561	7135
40	TITANIUM	PPM	986	958	735
41	CARBONATE	m/L	0	0	0
42	BORON (soluble)	PPM	913	803	874
43	CHROMIUM (soluble)	PPM	1	1	1
44	TOTAL BASE	m/L	0.29	0.28	0.26

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1			J4507	J4508	J5606	J6706	J4503	J4504	J5603	J6703	J4501	J4502	J5601	J5602	J6701	J6702
2			NaOH Init	NaOH 1R	NaOH 3R	NaOH 3R	OA Init	OA 1R	OA 2R	OA 3R	1WF80	1WF160	2WF80	2WF160	3WF80	3WF160
3	2-PHENYLPHENOL	MGL	2	3	<1	<1	7	2	2	<1	nm	nm	<1	3	3	3
4	4-PHENYLPHENOL	MGL	1	2	<1	<1	<1	16	<1	1	nm	nm	<1	<1	<1	<1
5	ANILINE	MGL	<1	<1	<1	<1	<1	<1	<1	33	nm	nm	<1	<1	<1	14
6	BIPHENYL	MGL	<1	4	5	3	2	2	4	5	75	7	112	10	130	10
7	DIPHENYLAMINE	MGL	2	<1	2	36	7	4	5	8	4	5	<1	<1	7	20
8	M-TERPHENYL	MGL	<1	<1	<1	2	2	<1	2	<1	9	1	9	1	35	<1
9	NITROBENZENE	MGL	23	<1	<1	<1	<1	<1	<1	<1	nm	nm	nm	nm	<1	<1
10	NITROSOBENZENE	MGL	<1	5	6	<1	14	17	4	5	nm	nm	<1	<1	<1	1
11	O-TERPHENYL	MGL	<1	<1	<1	2	3	<1	<1	<1	2	2	2	<1	14	<1
12	P-TERPHENYL	MGL	<1	<1	<1	5	<1	<1	5	<1	1	<1	<1	<1	23	<1
13	PHENOL	MGL	26	<1	<1	1	<1	11	15	73	125	20	80	14	80	26
14	PHENYLBORONIC ACID	MGL	<1	<1	<1	<1	<1	<1	<1	26	65	2	79	8	53	2
15	CHLORIDE	MGL	<1	<1	<1	<1	ND.	ND.	ND.	ND.	23	<1	19	<4	16	<2
16	FLUORIDE	MGL	<1	<1	<1	<1	ND.	ND.	ND.	ND.	<6	<1	<5	<1	<5	<1
17	FORMATE	MGL	<1	<1	<1	<1	ND.	ND.	ND.	ND.	29	<2	25	<5	23	<2
18	NITRATE	MGL	<9	10	12	13	ND.	ND.	ND.	ND.	<5	<1	<5	<2	<4	<1
19	NITRITE	MGL	<1	<1	<1	<1	ND.	ND.	ND.	ND.	12	<2	10	<2	<9	<2
20	OXALATE	MGL	<2	298	741	1072	23985	23139	22037	21583	82	<3	71	14	67	<6
21	PHOSPHATE	MGL	<1	<1	<1	<1	ND.	ND.	ND.	ND.	<2	<1	<4	<1	<2	<2
22	SULFATE	MGL	<6	<12	<12	<11	ND.	ND.	ND.	ND.	18	<1	15	<4	14	<1
23	PH	PH	13.17	13.18	13.08	13.13	1.5	1.47	1.6	1.44	11.18	10.36	11.1	10.5	11.04	9.86
24	MERCURY	PPM	0.18	0.51	1.17	1.31	2.2	6.7	4	6	35.2	11.9	97.1	17.8	64.5	16.3
25	INSOL SOLIDS	WT %	0	0.04	0.13	0.01	0.04	0.02	0.06	0.02	0.79	0.07	0.94	0.03	1.13	0.02
26	SOLUBLE SOLIDS	WT %	4.91	4.78	4.69	4.58	3.13	3.12	3.26	3.1	0.3	0	0.23	0	0.22	0
27	TOTAL SOLIDS	WT %	4.91	4.82	4.82	4.59	3.17	3.14	3.2	3.12	1.09	0.07	1.17	.03	1.35	0.02
28	TETRAETHYLENEDIBORATE	WT %	ND.	ND.	ND.	ND.	ND.	ND.	ND.	ND.	0.62	ND.	1.01	<2	0.9	ND.
29	TOT BASE/ACID	m/L	1.09	1.05	0.99	0.96	0.5	0.48	0.47	0.47	0.02	0.002	0.018	0.002	0.013	ND.
30	BORON	PPM	<0.1	0.89	1.92	3.2										

[illegible]

Table 2 continued

Code	Description
NaOH Ini	Initial Caustic Cleaning Solution
NaOH 1R	Caustic Cleaning Solution after 1 Recirculation
NaOH 2R	Caustic Cleaning Solution after 2 Recirculation
NaOH 3R	Caustic Cleaning Solution after 3 Recirculation
OA Init	Initial Oxalic Acid Cleaning Solution
OA 1R	Oxalic Acid Cleaning Solution after 1 Recirculation
OA 2R	Oxalic Acid Cleaning Solution after 2 Recirculation
OA 3R	Oxalic Acid Cleaning Solution after 3 Recirculation
1WF80	First Water Flush, first 80 mL
1WF160	First Water Flush, second 80 mL
2WF80	Second Water Flush, first 80 mL
2WF160	Second Water Flush, second 80 mL
3WF80	Third Water Flush, first 80 mL
3WF160	Third Water Flush, second 80 mL
OA1RWF80	Water Flush after first OA cycle, first flush
OA1RWF160	Water Flush after first OA cycle, second flush
OA2RWF80	Water Flush after second OA cycle, first flush
OA2RWF160	Water Flush after second OA cycle, second flush
OA3RWF80	Water Flush after third OA cycle, first flush
OA3RWF160	Water Flush after third OA cycle, second flush
C1RWF80	Water Flush after first caustic cycle, first flush
C1RWF160	Water Flush after first caustic cycle, second flush
C2RWF80	Water Flush after second caustic cycle, first flush
C2RWF160	Water Flush after second caustic cycle, second flush
C3RWF80	Water Flush after third caustic cycle, first flush
C3RWF160	Water Flush after third caustic cycle, second flush

Figure 1. Regression Analysis Generated
Flow Meter Calibration

Figure 2. Oxalic Acid Cleaning Cycle
Analytical Results

Figure 3. Caustic Cleaning Cycle
Analytical Results

Figure 4. Closed-End Clean Water Flux -
First Recirculation (Tests J45-J77)

Figure 5. Closed-End Clean Water Flux -
Second & Third Recirculation (Tests J45-J77)

Figure 6. Slurry Flux Rates

Figure 7. Slurry Flux vs. Clean Water Flux

END

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