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Silicon Analysis of Tank 8H and Tank 40H Turbidity Samples

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Summary

The need for silicon measurements in the field exists and can enhance the scheduling of waste transfers in both F- and H-Area Tank Farms. This report examines the use of field turbidity measurements as an at-line method to ensure that entrainment of silicon-bearing sludge materials are minimized. A total of twelve samples taken from two risers in two high level waste tanks have been analyzed for soluble and insoluble silicon. The results indicate the following conclusions.

- Samples taken from Tank 40H at heights of 5, 10, and 15 inches above the sludge interface layer (58.3") exhibited total silicon concentration of 9.6 mg/L to 21.1 mg/L.
- Samples taken from the C3 riser in Tank 40H exhibited silicon concentrations approximately double those silicon concentrations taken from the B5 riser at equivalent heights.
- Samples taken from the two risers in Tank 8F had silicon concentrations that were very low (< 7 mg/L) with no detectable differences between riser locations.
- The data supports the use of the turbidity field measurement as a method for ensuring limited entrainment of silicon-bearing sludge materials.

Keywords: Silicon, Sludge Washing, Evaporators

Introduction

The second batch of High Level Waste sludge to be processed in the Defense Waste Processing Facility (DWPF) consists of a mixture of sludge in Tanks 8H and 40H. The sludge mixture will be washed with inhibited water (0.01 M

sodium hydroxide) to lower the sodium content. Previous studies[1] showed that silicon would not be dissolved from the sludge during this process. However, the work showed that sludge settled very slowly and that elevated silicon concentrations could be encountered during this process. These elevated silicon levels need to be avoided to ensure that aluminosilicate deposits are not accumulated in the 2F Evaporator or 3H Evaporator.[2]

Due to the laborious nature of the silicon analysis at SRTC and the associated delays in analytical processing of samples, a more rapid technique to assess the silicon concentration is desired. One method proposed was to utilize turbidity measurements.[3] Therefore, several samples were taken from Tank 8H and 40H after turbidity measurements were taken and analyzed for silicon and other species.

Experimental

The SRTC received variable-depth samples from Tank 8H[4] and Tank 40H. The samples entered A-block of the Shielded Cells upon receipt. Personnel determined the density of the liquid samples by emptying the contents of the dip bottles into pre-weighed 100-mL graduated cylinders with the accuracy of the graduated cylinders verified to ± 0.5% at 100 mL prior to use. The samples were put through a filtering protocol to determine the total amount of silicon in a given volume of liquid waste. The protocol included sampling as-received waste, sequential filtration through at least 0.2 micron and 0.02 micron filters, and analyzing filtrates and filters for aluminum and silicon. Tables of data are presented with the following information: Si concentration (mg/L) in diluted sample, corrected Si concentration (mg/L, Si diluted x dilution factor), Si concentration (mg/L) from digested filter, Si amount (mg) on filter (Si concentration * 0.05 L volume) and Si concentration total (mg/L, $[Si]_{0.02\ filtrate}$ + S Si_{filter}/Sample volume).

The Analytical Development Section used Inductively Coupled Plasma – Emission Spectroscopy[5] to determine the concentration of silicon. Sodium peroxide fusion[6] was used to digest the filter and associated solids.

Results and Discussion

Table 1 contains the results from the analysis of the samples taken from Tank 40H shortly after (~24 h) turbidity measurements. Tank 40H was sampled through two risers (C3 and B5). Samples were taken at 5, 10 and 15 inches above the sludge interface layer (58.3" in the C3 riser and 58.5" in the B5 riser). Additionally, these samples were taken approximately 96 days after discontinuing slurry pump operation. The total of insoluble and soluble silicon concentrations is very low and ranged from 9.6 mg/L to 21.1 mg/L. Using the previously reported aluminum concentration of 0.204 M, the caluclated equilibrium solubility product, K_{sp}, would range from $6.9 \times 10^{-5}\ M^2$ to $1.5 \times 10^{-4}\ M^2$. The calculated K_{sp}'s for Tank 40 H are well below the expected K_{sp} in similar systems from the literature indicating the system is slightly below saturation in silicon.[7]

Table 2 contains the same information obtained from analyzing samples taken from Tank 8F. These samples were taken approximately roughly 8 and 16 inches above the respective sludge interface layer and at least 10 hours after the turbidity measurement. As with the Tank 40H measurements the silicon concentrations are very low. The Si concentrations ranged from 3.9 mg/L to 6.8 mg/L. These concentrations are very near the detection limit and do not pose a significant operational concern. The samples were taken from 174 hours to 294 hours after the slurry pump operation ceased.[8]

Table 1. Silicon Concentration in Tank 40 H Samples

TF-Test-45 C3 Riser Elevation:63.3"	ESP-266 Diluted Filtrate (mg/L)	ESP-266 Filtrate (mg/L)	ESP-266 Filter Measured (mg/L)	Si on ESP-266 Filter (mg)	ESP-266 Total (mg/L)
As-Received	0.179	8.7			
0.2	0.149	7.6	4.308	0.22	14.7
0.02	0.228	11.6	3.674	0.18	17.3
TF-Test-46 C3 Riser Elevation:68.3"	ESP-267 Diluted Filtrate (mg/L)	ESP-267 Filtrate (mg/L)	ESP-267 Filter Measured (mg/L)	Si on ESP-267 Filter (mg)	ESP-267 Total (mg/L)
As-Received	0.199	10.0			
0.2	0.192	9.7	6.018	0.30	15.5
0.02	0.218	11.0	7.501	0.38	21.1
TF-Test-47 C3 Riser Elevation:73.3"	ESP-268 Diluted Filtrate (mg/L)	ESP-268 Filtrate (mg/L)	ESP-268 Filter Measured (mg/L)	Si on ESP-268 Filter (mg)	ESP-268 Total (mg/L)
As-Received	0.205	10.3			
0.2	0.157	7.8	7.713	0.39	17.4
0.02	0.242	11.8	4.163	0.21	20.4
TF-Test-48 B5 Riser Elevation:63.5"	ESP-269 Diluted Filtrate (mg/L)	ESP-269 Filtrate (mg/L)	ESP-269 Filter Measured (mg/L)	Si on ESP-269 Filter (mg)	ESP-269 Total (mg/L)
As-Received	0.156	7.8			
0.2	0.145	7.3	2.258	0.11	11.5
0.02	0.2	9.8	2.581	0.13	13.5
TF-Test-49 B5 Riser Elevation:68.5"	ESP-270 Diluted Filtrate (mg/L)	ESP-270 Filtrate (mg/L)	ESP-270 Filter Measured (mg/L)	Si on ESP-270 Filter (mg)	ESP-270 Total (mg/L)
As-Received	0.227	11.5			
0.2	0.236	11.8	1.876	0.09	8.4
0.02	0.174	7.0	1.615	0.08	9.6
TF-Test-50 B5 Riser Elevation:73.5"	ESP-271 Diluted Filtrate (mg/L)	ESP-271 Filtrate (mg/L)	ESP-271 Filter Measured (mg/L)	Si on ESP-271 Filter (mg)	ESP-271 Total (mg/L)
As-Received	0.212	11.0			
0.2	0.245	12.2	6.223	0.31	12.2
0.02	0.138	7.1	2.25	0.11	14.1

Conclusions

The need for silicon measurements in the field exists and can enhance the scheduling of waste transfers in both F- and H-Area Tank Farms. This report examines the use of field turbidity measurements as an at-line method to ensure that entrainment of silicon-bearing sludge materials are minimized. A total of twelve samples taken from two risers in two high level waste tanks have been analyzed for soluble and insoluble silicon. The results from samples taken at three heights from the turbidity interface show very low silicon concentrations. These results support the use of the turbidity probe to determine if silicon-bearing solids have settled.

Table 2. Silicon Concentrations in Tank 8F

TF-Test-68 Elevation: 91.6"	FTF-008 Diluted Filtrate (mg/L)	FTF-008 Filtrate (mg/L)	FTF-008 Filter Measured (mg/L)	Si on FTF-008 Filter (mg)	FTF-008 Total (mg/L)
As-Received	0.086	4.2			
0.2	0.061	3.1	2.053	0.10	5.0
0.02	0.07	3.5	1.7	0.09	6.2
TF-Test-69 Elevation: 91.6"	**FTF-009 Diluted Filtrate (mg/L)**	**FTF-009 Filtrate (mg/L)**	**FTF-009 Filter Measured (mg/L)**	**Si on FTF-009 Filter (mg)**	**FTF-009 Total (mg/L)**
As-Received	0.088	4.4			
0.2	0.051	2.5	1.996	0.10	5.2
0.02	0.074	3.8	2.199	0.11	6.8
TF-Test-70 Elevation: 83.6"	**FTF-010 Diluted Filtrate (mg/L)**	**FTF-010 Filtrate (mg/L)**	**FTF-010 Filter Measured (mg/L)**	**Si on FTF-010 Filter (mg)**	**FTF-010 Total (mg/L)**
As-Received					
0.2	0.043	2.3	No Filter		
0.02	0.042	2.3	2.176	0.11	3.9
TF-Test-71 Elevation: 90.36"	**FTF-011 Diluted Filtrate (mg/L)**	**FTF-011 Filtrate (mg/L)**	**FTF-011 Filter Measured (mg/L)**	**Si on FTF-011 Filter (mg)**	**FTF-011 Total (mg/L)**
As-Received					
0.2	0.061	3.4	No Filter		
0.02	0.052	2.8	2.356	0.12	4.5
TF-Test-72 Elevation: 82.36"	**FTF-012 Diluted Filtrate (mg/L)**	**FTF-012 Filtrate (mg/L)**	**FTF-012 Filter Measured (mg/L)**	**Si on FTF-012 Filter (mg)**	**FTF-012 Total (mg/L)**
As-Received					
0.2	<0.016	1.1	2.873	0.14	3.7
0.02	0.028	1.6	2.421	0.12	5.5
TF-Test-73 Elevation: 82.36"	**FTF-013 Diluted Filtrate (mg/L)**	**FTF-013 Filtrate (mg/L)**	**FTF-013 Filter Measured (mg/L)**	**Si on FTF-019 Filter (mg)**	**FTF-013 Total (mg/L)**
As-Received					
0.2	0.036	1.8	3.129	0.16	3.6
0.02	<0.016	0.8	2.506	0.13	5.9

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