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Report on Electrochemical Corrosion Testing for Tank 241-AN-106 Using 2009 Sampling Campaign Grab Samples

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Washington River Protection Solutions LLC

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Acronyms and Abbreviations

AC	Administrative Controls
AN-106	tank 241-AN-106
ASTM	ASTM International
C-110	tank 241-C-110
CPP	cyclic potentiodynamic polarization
DST	double-shell tank
E_{corr}	corrosion potential
EDS	energy dispersion spectroscopy
i_{corr}	corrosion current
LPR	linear polarization resistance
MPY	mils per year
OCP	open circuit potential
TSR	Tank Farms Technical Safety Requirements
SEM	scanning electron microscopy
XRD	X-ray Diffraction

1. INTRODUCTION

Based on an ENRAF¹ waste surface measurement taken February 1, 2009, double-shell tank (DST) 241-AN-106 (AN-106) contained approximately 278.98 inches (793 kgal) of waste. A zip cord measurement from the tank on February 1, 2009, indicated a settled solids layer of 91.7 inches in height (280 kgal). The supernatant layer in February 2009, by difference, was approximately 187 inches deep (514 kgal).

Laboratory results from AN-106 February 1, 2009 (see Table 2) grab samples indicated the supernatant was below the chemistry limit that applied at the time as identified in HNF-SD-WM-TSR-006, *Tank Farms Technical Safety Requirements*, Administrative Control (AC) 5.16, "Corrosion Mitigation Controls." (The limits have since been removed from the Technical Safety Requirements (TSR) and are captured in OSD-T-151-00007, *Operating Specifications for the Double-Shell Storage Tanks*.) Problem evaluation request WRPS-PER-2009-0218 was submitted February 9, 2009, to document the finding that the supernatant chemistry for grab samples taken from the middle and upper regions of the supernatant was noncompliant with the chemistry control limits. The lab results for the samples taken from the bottom region of the supernatant met AC 5.16 limits.

AC 5.16 included Program Key Element 5.16.2.a.1,

Maintain nitrite [NO₂⁻], nitrate [NO₃⁻], and hydroxide [OH⁻] concentrations:

a) within the limits shown in Table 5.16-1 for all DST waste except DST 241-AN-107 and DST 241-AN-102 interstitial liquid. (Note: The Table 5.16-1 limits apply to DST 241-AN-107 and DST 241-AN-102 supernatant.)

Table 5.16-1 in AC 5.16 provided the applicable OH⁻ concentration limits based on correlating NO₃⁻ ion, NO₂⁻ ion, and temperature conditions in the tank. These limits, as applicable to the AN-106 supernatant in the tank, are shown in Table 1.

Table 1. Chemistry Control Limits Applicable to Tank 241-AN-106 Supernatant.

Variable	Limits
[OH ⁻]	0.01 <u>M</u> ≤ [OH ⁻] ≤ 8 <u>M</u>
[NO ₂ ⁻]	0.011 <u>M</u> ≤ [NO ₂ ⁻] ≤ 5.5 <u>M</u>
[NO ₃ ⁻]/([OH ⁻] + [NO ₂ ⁻])	< 2.5

Notes: 1. The brackets [] denote concentration in molarity (moles/L or M).

2. The limits apply to waste with a NO₃⁻ concentration ≤ 1.0 M and waste temperatures < 167 °F. The preliminary NO₃⁻ concentrations in the tank 241-AN-106 supernatant ranged from 0.45 – 0.74 M (see Table 2). The maximum supernatant waste temperature within the last 1-year period was < 105 °F.

¹ ENRAF is a registered trademark of Honeywell International, Morris Township, New Jersey.

The AN-106 supernatant was sampled on February 1, 2009. Tank 241-AN-106 receives 241-C Tank Farm retrieval waste transfers, currently for tank 241-C-110 (C-110). The purpose of the sampling event was to investigate the tank chemistry at the midpoint of the C-110 retrieval, verify compliance with chemistry control specifications, and project the tank chemistry to the end of C-110 retrieval. Five multi-depth grab samples were obtained from the tank supernatant. The sample results and sample elevations are provided in Table 2.

Table 2. Tank 241-AN-106 Sample Data, February 2009 Sample Event^{a,b}

Sample Number	OH ⁻ , <u>M</u> ^c	NO ₂ ⁻ , <u>M</u>	NO ₃ ⁻ , <u>M</u>	Sample Elevation (inches)
6AN-09-01	0.0087	0.604	0.661	279
6AN-09-02	0.0087	0.502	0.548	278
6AN-09-03	0.0083	0.574	0.627	187
6AN-09-03DUP	0.0085	0.676	0.744	187
6AN-09-04	0.0162	0.607	0.447	102

^a Laboratory data from WRPS-0900230, "Contract Number DE-AC27-08RV14800 – Request for Approval of Recovery Plan to Restore Chemistry Control to Tank 241-AN-106 Supernatant."

^b Concentrations averaged where multiple results were available.

^c OH⁻ concentration based on pH measurements.

The OH⁻ concentration for supernatant waste surface samples (6AN-09-01, 6AN-09-02) and the supernatant middle region samples (6AN-09-03, 6AN-09-03DUP) did not meet the AC 5.16 limit of greater than or equal to 0.01 M. The OH⁻ concentration of the bottom supernatant sample met the AC 5.16 limit. Also, the NO₂⁻ concentrations and the ratio NO₃⁻ to OH⁻+NO₂⁻ concentrations for all the supernatant samples met the AC 5.16 chemistry limits of Table 1.

Tank recovery plan TF-RP-09-01, "Tank 241-AN-06 Supernatant Recovery Plan," attached to WRPS-0900230, "Contract Number DE-AC27-08RV14800 – Request for Approval of Recovery Plan to Restore Chemistry Control to Tank 241-AN-106 Supernatant," required additional sampling to be performed before and after a caustic addition. RPP-PLAN-40573, *Tank 241-AN-106 Grab Sampling and Analysis Plan to Address Recovery Plan TF-RP-09-01*, has defined four grab samples and four duplicates to be collected before the caustic addition and four grab samples and one duplicate following the addition. This plan provides details of the tank status and sample planning.

The pre-caustic addition chemistry of the tank 241-AN-106 supernatant is established by the results from the May 8, 2009, multi-depth grab sampling event, RPP-RPT-41610, *Final Report for Tank 241-AN-106 Liquid Grab Samples Collected in May 2009 in Support of the Tank Farms Recovery Plan TF-RP-09-01*. Table 3 summarizes the sample event data. The four multi-depth grab samples (primary and duplicate) have very similar compositions.

Table 3. Tank 241-AN-106 May 8, 2009 Supernatant Sample Data.

Sample Number	Elevation, Inches ¹	pH ²	[OH], <u>M</u> ³	[NO ₂], <u>M</u> ²	[NO ₃], <u>M</u> ²	Specific Gravity
6AN-09-05	279.16	11.5	0.00316	0.673	0.901	1.161
6AN-09-05DUP		11.5	0.00316	0.678	0.907	1.155
6AN-09-06	231.16	11.5	0.00316	0.683	0.915	1.162
6AN-09-06DUP		11.6	0.00398	0.685	0.918	1.159
6AN-09-07	183.16	11.5	0.00316	0.661	0.884	1.164
6AN-09-07DUP		11.5	0.00316	0.674	0.903	1.166
6AN-09-08	145.16	11.6	0.00398	0.691	0.927	1.163
6AN-09-08DUP		11.6	0.00398	0.691	0.927	1.169

Notes:

1. Sample elevation from work package TFC-WO-09-0825, 241-AN Grab Sample AN-106 (Corrosion Control).
2. Sample data from 20090322 – Letter.
3. OH⁻ concentration based on pH measurements.

The May 2009 grab sample results in Table 3 indicate that the supernatant was below the existent HNF-SD-WM-TSR-006 AC 5.16, “Corrosion Mitigation Controls,” chemistry limit. Problem evaluation request WRPS-PER-2009-0958, “AN-106 sample results confirm out of limit hydroxide concentration,” was submitted May 12, 2009, to document the finding that the chemistry for grab samples taken from the supernatant was non-compliant with chemistry control limits, as expected.

The June 2009 post-caustic addition chemistry condition of the AN-106 supernatant was established using results from the June 23, 2009, multi-depth grab sampling event (20090467 – Letter). Table 4 summarizes the sample event data.

Table 4. Tank 241-AN-106 June 23, 2009, Supernatant Sample Data

Sample Number	Elevation, Inches ¹	[OH], <u>ug/mL</u> ²	[OH], <u>M</u>	[NO ₂], <u>M</u> ²	[NO ₃], <u>M</u> ²
6AN-09-09	34'-11"/285.2"	4.10E+03	0.241	0.677	0.899
6AN-09-10	36'-6"/230.2"	4.26E+03	0.251	0.687	0.911
6AN-09-10DUP		4.31E+03	0.254	0.676	0.900
6AN-09-11	41'-1"/175.2"	4.32E+03	0.254	0.696	0.927

Note:

1. Sample elevation from work package TFC-WO-09-0825, 241-AN Grab Sample AN-106 (Appendix A), and H-14-010501 Sheet 6, *Dome Penetration Schedules (WST-WSTA) Tank 241-AN-101*.
2. Sample data from 20090467 – Letter.

Besides samples from the February 2009 sampling event, the following grab samples were made available for testing: 6AN-09-05, 6AN-09-05 DUP, 6AN-09-06, 6AN-09-06 DUP, 6AN-09-07, 6AN-09-07 DUP, 6AN-09-08, 6AN-09-08 DUP, 6AN-09-09, 6AN-09-10, 6AN-09-10 DUP, 6AN-09-11, and 6AN-09-12.

The testing reported herein was performed in accordance with RPP-PLAN-40435, *Electrochemical Corrosion Testing for Tank 241-AN-106 Using Grab Samples 6AN-09-01, 6AN-09-02, 6AN-09-03, 6AN-09-03DUP, and 6AN-09-04.*

2. MATERIALS AND METHODS

2.1 MATERIALS

The coupons used in this study were obtained from Metal Samples^{®2} and were A537 Class 1 EL410 (right cylinder configuration) with an area of approximately 5.64 cm². All coupons were prepared by a surface treatment of 600-grit wet sandpaper followed by sonication in acetone for 2 minutes. The coupon was then fixed to a type 316 stainless steel electrode rod. A new sample coupon was used for each cyclic potentiodynamic polarization (CPP) scan.

2.2 METHODS

2.2.1 Quality Assurance

As an instrument check, a scan using the ASTM G5-94, *Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements*, was carried out in a 500-mL I-CHEM^{®3} jar used as the electrochemical cell before and after the corrosion potential scans.

The data collected in this report was managed under ATS-MP-1032, *222-S Laboratory Quality Assurance Plan*. The data generated by Advanced Technologies and Laboratories International, Inc. was managed under its quality assurance program.

2.2.2 Electrochemical Methods

The potentiostat used was a VMP^{®4} with data collection and analyses software, EC-Lab^{®5} Software, Version 9.55. The potentiostat has a 20-V compliance voltage and a maximum current of up to ± 200 mA.

After surface preparations, the coupons were immersed in the sample and allowed to equilibrate at ambient hot cell temperature ($26.7^{\circ}\text{C} \pm 2^{\circ}\text{C}$) for 18 to 24 hours before a linear polarization resistance (LPR) scan was initiated at the same temperature. After the LPR scan, the electrochemical cell was allowed to re-equilibrate for 20 minutes. The CPP scan ranged from -200 mV below the open circuit potential (OCP) and reversed at 2.5 mA/cm² at a scan rate of 0.166 mV/sec.

3. RESULTS

The electrochemical results, including the corrosion potential (E_{corr}), corrosion current (i_{corr}), corrosion rate in mills per year (MPY), and OCP, are presented in Tables 5 through 10 for each of the sample events. Measurements were made in air and under inert atmospheric conditions

² Metal Samples[®] is a division of Alabama Specialty Products, Inc., Munford, Alabama.

³ I-CHEM[®] is a subsidiary of Nalge Nunc International Corporation, Rochester, New York.

⁴ VMP is a registered trademark of Bio-Logic SAS[®], 1 Rue de l'Europe, Claix, F-38640, France.

⁵ EC-Lab[®] is a registered trademark of Bio-Logic SAS[®], 1 Rue de l'Europe, Claix, F-38640, France.

using an argon purged headspace. The plots of LPR and CPP data can be found in Appendix A. The complete data for the coupons tested can be found in Appendix B.

The electrochemical experiments in the first round were interrupted by facility upgrades. During this time period, a significant portion of the supernate liquid was lost due to evaporation in sample 6AN-09-01. A precipitate was observed to have formed after evaporation. There was not sufficient volume to continue experiments on this sample beyond the first two CPP scans. Scan S09T002344 did not yield an adequate curve around the E_{corr} region for a tafel calculation.

Table 5. Electrochemical Values and Corrosion Rates for February 2009 Grab Samples from Waste Tank 241-AN-106.

Sample Coupon	i_{corr} (μA)	E_{corr} (mV)	Corrosion Rate MPY (E-3)
<i>241-AN-106 Grab Sample 6AN-09-01, S09T001177</i>			
S09T002335	0.015	-117	1.22
S09T002336	0.157	-189	12.80
<i>241-AN-106 Grab Sample 6AN-09-02, S09T001178</i>			
S09T002344	-	-	-
S09T002345	0.014	-158	1.14
S09T002345A	0.073	95.2	5.93
S09T002346 (Argon)	0.034	-18.6	2.76
S09T002347 (Argon)	0.023	-70.2	1.87
<i>241-AN-106 Grab Sample 6AN-09-03, S09T001179</i>			
S09T002349	0.017	-140	1.38
S09T002350	0.015	-72.3	1.22
S09T002351 (Argon)	0.031	25.2	2.52
<i>241-AN-106 Grab Sample 6AN-09-03DUP, S09T001180</i>			
S09T002354	0.035	-102	2.84

After sub-samples for analytical chemical analysis were taken, the remaining volume was the minimum amount necessary to conduct the electrochemical experiments on these samples. A similar evaporative loss occurred with 6AN-09-03 and 6AN-09-03DUP. After the volume was lost due to evaporation, there was not enough supernate liquid to continue experiments on this sample. Attempts to re-hydrate the samples failed as the precipitate was not readily soluble. A sub-sample of the precipitate from 6AN-09-03, sub-sample S09T003615, and 6AN-09-03DUP, sub-sample S09T003616, was collected and studied by x-ray diffraction (XRD). The resulting XRD pattern can be found in Appendix C. The diffraction patterns indicated two major phases, Dawsonite $[\text{NaAlCO}_3(\text{OH})_2]$ and Sodium Fluoride Phosphate Hydrate $[\text{NaF}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}]$. A minor phase of Sodium Strontium Phosphate $[\text{NaSr}(\text{PO}_4)(\text{H}_2\text{O})_9]$ was also identified with other minor components of Sodium Nitrate and Sodium Nitrite that are presumed to be residue from the supernate liquid when the samples were dried. Other samples from the second round of

sampling also formed precipitates during the study. The precipitate was not sub-sampled as further analysis was beyond the scope of this project.

Table 6. Electrochemical Values and Corrosion Rates for May 2009 Grab Samples from Waste Tank 241-AN-106.

Sample	i_{corr} (μA)	E_{Corr} (mV)	Corrosion Rate MPY (E-3)
<i>241-AN-106 Grab Sample 6AN-09-05, S09T007167</i>			
S09T007168	0.019	-155	1.54
S09T007169	0.035	-57.0	2.84
S09T007170 (Argon)	0.057	28.1	4.63
S09T007171 (Argon)	0.067	98.2	5.44
<i>241-AN-106 Grab Sample 6AN-09-06, S09T007173</i>			
S09T007184	0.031	22.4	2.52
S09T007185	0.023	-29.9	1.87
S09T007186 (Argon)	0.059	-29.9	4.79
S09T007187 (Argon)	0.041	26.0	3.33
<i>241-AN-106 Grab Sample 6AN-09-06DUP, S09T007174</i>			
S09T007188	0.030	-49.7	2.44
S09T007189	0.067	55.1	5.44
S09T007190 (Argon)	0.034	-30.7	2.76
S09T007191 (Argon)	0.082	105	6.66
<i>241-AN-106 Grab Sample 6AN-09-08, S09T007175</i>			
S09T007192	0.025	-4.27	2.03
S09T007193	0.036	14.9	2.94
S09T007194 (Argon)	0.057	78.1	4.63
S09T007195 (Argon)	0.067	101	5.44
<i>241-AN-106 Grab Sample 6AN-09-07, S09T007176</i>			
S09T007196	0.037	-10.5	3.01
S09T007197	0.059	77.1	4.79
S09T007198 (Argon)	0.052	81.2	4.22
S09T007199 (Argon)	0.055	-247	4.47

The CPP scans collected in this study did not indicate pitting corrosion due to the negative hysteresis loop on the reverse polarization scan. In some cases, the CPP scans conducted on the February and May samples did not reach the reverse current of 2.5 mA/cm^2 as expected. Instead the scans were reversed at an arbitrary upper bound potential setting. The CPP scans appeared to break down around 0.7 V to 0.8 V, but then became passive again before reaching either the current limit or the bounding potential setting. In the case where this effect was observed, the reverse scan failed to repassivate after establishing a negative hysteresis. The scans where this occurred are S09T002335, S09T002345, S09T002349, S09T002350, S09T002354, and S09T007185. It was then decided that the scans would be reversed at a limit near the potential at which the turnaround current should have been reached, between 0.8 V to 0.9 V. When the CPP

scans were reversed at this potential, the scan showed a repassivation potential with negative hysteresis. This is discussed in more detail in the next section.

The CPP scans that were collected on the June samples, which were the samples taken after remediation, reversed at the turnaround current of $2.5\text{mA}/\text{cm}^2$ and at potential values less than 0.8 V. This group of samples also exhibited a negative hysteresis and favorable repassivation potential with respect to corrosion prevention.

Table 7. Electrochemical Values and Corrosion Rates for June 2009 Grab Samples From Waste Tank 241-AN-106.

Sample	i_{corr} (μA)	E_{Corr} (mV)	Corrosion Rate MPY (E-3)
<i>241-AN-106 Grab Sample 6AN-09-10, S09T007177</i>			
S09T007200	0.101	-335	8.21
S09T007201	0.106	-327	8.61
S09T007202 (Argon)	2.379	-831	193
S09T007203 (Argon)	0.053	-488	4.31
<i>241-AN-106 Grab Sample 6AN-09-11, S09T007179</i>			
S09T007208	0.014	-320	11.3
S09T007209	0.104	-337	8.45
S09T007209A	0.082	-325	6.66
S09T007210 (Argon)	0.042	-461	3.41
S09T007211 (Argon)	0.068	-395	5.52

Table 8. Open Circuit Potential for February 2009 Grab Samples From Waste Tank 241-AN-106.

Sample	OCP (mV)
<i>241-AN-106 Grab Sample 6AN-09-01, S09T001177</i>	
S09T002335	-78.0
S09T002336	-6.51
<i>241-AN-106 Grab Sample 6AN-09-02, S09T001178</i>	
S09T002344	23.3
S09T002345	-119
S09T002346 (Argon)	109
S09T002347 (Argon)	1.09
<i>241-AN-106 Grab Sample 6AN-09-03, S09T001179</i>	
S09T002349	-90
S09T002350	-23.4
S09T002351	53.2
<i>241-AN-106 Grab Sample 6AN-09-03DUP, S09T001180</i>	
S09T002354	-76.0

Table 9. Open Circuit Potential for May 2009 Grab Samples From Waste Tank 241-AN-106.

Sample	OCP (mV)
<i>241-AN-106 Grab Sample 6AN-09-05, S09T007167</i>	
S09T007168	-76.9
S09T007169	-17.1
S09T007170 (Argon)	51.6
S09T007171 (Argon)	114
<i>241-AN-106 Grab Sample 6AN-09-06, S09T007173</i>	
S09T007184	51.1
S09T007185	15.9
S09T007186 (Argon)	-16.3
S09T007187 (Argon)	55.7
<i>241-AN-106 Grab Sample 6AN-09-06DUP, S09T007174</i>	
S09T007188	-1.90
S09T007189	78.5
S09T007190 (Argon)	10.5
S09T007191 (Argon)	121
<i>241-AN-106 Grab Sample 6AN-09-08, S09T007175</i>	
S09T007192	23.1
S09T007193	42.0
S09T007194 (Argon)	97.7
S09T007195 (Argon)	115
<i>241-AN-106 Grab Sample 6AN-09-07, S09T007176</i>	
S09T007196	22.2
S09T007197	96.0
S09T007198 (Argon)	99.3
S09T007199 (Argon)	-217

Table 10. Open Circuit Potential for June 2009 Grab Samples From Waste Tank 241-AN-106.

Sample	OCP (mV)
<i>241-AN-106 Grab Sample 6AN-09-10, S09T007177</i>	
S09T007200	-318
S09T007201	-304
S09T007202 (Argon)	-794
S09T007203 (Argon)	-454
<i>241-AN-106 Grab Sample 6AN-09-11, S09T007179</i>	
S09T007208	-298
S09T007209	-316
S09T007209A	-304
S09T007210 (Argon)	-419
S09T007211 (Argon)	-354

4.0 DISCUSSION

The CPP scans collected in this report showed no signs of irreversible pitting as they all show a negative hysteresis. However, they do show some abnormal results insofar as previous electrochemical corrosion studies for Hanford Tank Waste are concerned. Figure 1 shows the CPP scan from sample 6AN-09-03 for coupon S09T002349 performed at 25°C in air. The scan parameters were set to reverse the polarization at 2.5 mA/cm². Maximum current should have been attained after the break down potential (~680 mV). The scan appears to become passive again before achieving the maximum current. With increasing potential, the current decreases and then remains constant until finally reaching the current limit (14.1 mA) near 1.7 V. The scan reverses at this point and yields a negative hysteresis. The scan never achieves repassivation cleanly during the reverse scan. This could be an indication that the electronic double layer simply broke down around 300 mV. The scan then appears to repassivate at or below the corrosion potential. Coupon S09T002335 was examined using optical microscopy and scanning electron microscopy (SEM). There were no signs of pitting using these techniques. The interface of the coupon and the Teflon^{®6} bullet produced an iron oxide ring. The ring was analyzed by energy dispersion spectroscopy (EDS). The results are presented in Appendix D with the SEM results.

The second CPP scan on the 6AN-09-03 using sample coupon S09T002350 is shown in Figure 2. The CPP scan was collected under the same conditions as the CPP scan for sample coupon S09T002349 and has similar features. In the scans of both of these coupons, the reverse current was not met when expected. However, the S09T002350 scan then failed to display a repassivation potential at all, as is observed in Figure 2. Upon inspection by microscopy, the coupon was not found to pit. The only sign of corrosion was found at the interface of the coupon and the Teflon[®] bullet. This was determined to be iron oxide by EDS. The results are also given

⁶ Teflon[®] is a registered trademark of I. E. du Pont de Nemours and Company, Wilmington, Delaware.

in Appendix D. Optical images of coupons S09T002349 and S09T002350 can be found in Appendix E.

CPP scan using coupon S09T002351 was performed on the same grab sample, 6AN-09-03, and is plotted in Figure 3. This scan was set to reverse at 900 mV or 14.1 mA. The reverse scan was what was to be expected for a sample that does not show signs of irreversible pitting. The sample had a repassivation potential near 200mV and displayed a negative hysteresis. The remaining CPP scans were reversed at 800 to 900 mV instead of 2.5 mA/cm^2 .

Figure 1. Cyclic Potentiodynamic Polarization Scan of Sample 6AN-09-03, Sample Coupon S09T002349.

**Figure 2. Cyclic Potentiodynamic Polarization Scan of Sample 6AN-09-03,
Sample Coupon S09T002350.**

**Figure 3. Cyclic Potentiodynamic Polarization Scan of Sample 6AN-09-03,
Sample Coupon S09T002351.**

The samples that were collected in February and May of 2009 appear to be inconsistent from scan to scan including the in-air scans and the scans where the headspace is purged with argon. There is no clear explanation for this phenomenon. It was discovered that a small amount of precipitate settled in the electrochemical jar during the experiments. The precipitate was not analyzed. It is well known that a phase transition occurs between carbonate and bicarbonate, as well as in the phosphate ion chemistry around pH 12 (Poubaix, *Atlas of Electrochemical Equilibria in Aqueous Solutions*; Rayner-Canham, *Descriptive Inorganic Chemistry*). The February samples were around pH 11.9 and the May samples had pH around 11.6. Instantaneous fluctuations of the pH around the working electrode or the counter electrode could account for the formation of the precipitate. Based on the precipitate analyzed in the 6AN-09-03 and 6AN-09-03DUP samples, Dawsonite $[\text{NaAlCO}_3(\text{OH})_2]$ and Sodium Fluoride Phosphate Hydrate $[\text{NaF}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}]$ are the major constituents in the precipitate. The following would account for the formation of the Dawsonite in relation to pH:

After the Dawsonite precipitates, this would then affect other species in the grab sample. This could explain the inconsistencies from scan to scan. The June grab samples were more consistent in the duplicate scans than the earlier samples as shown in Figure 4. The pH in these samples are >13. The CPP and LP scans performed on the June samples show no pitting corrosion and low general corrosion rates. Scan S09T007202 yielded a higher corrosion rate, and very low E_{corr} value and a high i_{corr} value. The scan is an irregularity within the data set.

Figure 4. Cyclic Potentiodynamic Polarization Scan of Sample 6AN-09-10, Sample Coupon S09T007200.

From the data collected for this study, there do not appear to be any aggressive corrosion conditions even for the out-of-specification samples collected in February and May. There is some abnormality in the conditions that may be attributed to the specific chemistry of this tank, but again it does not appear to be of concern from a corrosion chemistry point of view.

5. REFERENCES

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APPENDIX A

Electrochemical Scans

APPENDIX B

Electrochemical Data

Table B-1. Electrochemical Data (2 Sheets)

CPP Data							
Sample	OCP (mV)	I _{corr} (μ A)	E _{RP} (mV)	E _{pass} (mV)	E _{Corr} (mV)	MPY (E-3)	Date Complete
<i>241-AN-106 Grab Sample 6AN-09-01, S09T001177</i>							
S09T002335	-78.0	0.015		374	-117	1.22	30-Jun-2009
S09T002336	-6.51	0.157	411	-91.4	-189	12.8	14-Jul-2009
<i>241-AN-106 Grab Sample 6AN-09-02, S09T001178</i>							
S09T002344	23.3	N/D	390	-117	N/D	N/D	30-Jun-2009
S09T002345	-119	0.014	N/D	330	-158	1.14	14-Jul-2009
S09T002345A	109	0.073	203	308	95.2	5.93	15-Jul-2009
S09T002346	28.1	0.034	205	104	-18.6	2.76	16-Jul-2009
S09T002347	1.09	0.023	196	20.1	-70.2	1.87	22-Jul-2009
<i>241-AN-106 Grab Sample 6AN-09-03, S09T001179</i>							
S09T002349	-90	0.017	-144	56.8	-140	1.38	30-Mar-2009
S09T002350	-23.4	0.015	N/D	109	-72.3	1.22	1-Apr-2009
S09T002351	53.2	0.031	205	609	25.2	2.52	16-Apr-2009
<i>241-AN-106 Grab Sample 6AN-09-03DUP, S09T001180</i>							
S09T002354	-76.0	0.035	-186	307	-102	2.84	31-Mar-2009
<i>241-AN-106 Grab Sample 6AN-09-05, S09T007167</i>							
S09T007168	-76.9	0.019	221	-0.993	-155	1.54	28-Aug-2009
S09T007169	-17.1	0.035	223	93.5	-57.0	2.84	1-Sep-2009
S09T007170	51.6	0.057	221	194	28.1	4.63	2-Sep-2009
S09T007171	114	0.067	224	327	98.2	5.44	3-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-06, S09T007173</i>							
S09T007184	51.1	0.031	227	253	22.4	2.52	3-Sep-2009
S09T007185	15.9	0.023	N/D	269	-29.9	1.87	9-Sep-2009
S09T007186	-16.3	0.059	N/D	291	-29.9	4.79	16-Sep-2009
S09T007187	55.7	0.041	239	271	26	3.33	22-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-06DUP, S09T007174</i>							
S09T007188	-1.90	0.030	235	212	-49.7	2.44	27-Aug-2009
S09T007189	78.5	0.067	234	237	55.1	5.44	28-Aug-2009
S09T007190	10.5	0.034	243	240	-30.7	2.76	1-Sep-2009
S09T007191	121	0.082	238	292	105	6.66	2-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-08, S09T007175</i>							
S09T007192	23.1	0.025	N/D	111	-4.27	2.03	3-Sep-2009

Table B-1. Electrochemical Data (2 Sheets)

CPP Data							
Sample	OCP (mV)	I _{corr} (μA)	E _{RP} (mV)	E _{pass} (mV)	E _{Corr} (mV)	MPY (E-3)	Date Complete
S09T007193	42.0	0.036	255	287	14.9	2.94	22-Sep-2009
S09T007194	97.7	0.057	251	286	78.1	4.63	23-Sep-2009
S09T007195	115	0.067	260	360	101	5.44	24-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-07, S09T007176</i>							
S09T007196	22.2	0.037	247	380	-10.5	3.01	23-Sep-2009
S09T007197	96	0.059	237	351	77.1	4.79	24-Sep-2009
S09T007198	99.3	0.052	248	342	81.2	4.22	25-Sep-2009
S09T007199	-217	0.055	-13.2	361	-247	4.47	30-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-10, S09T007177</i>							
S09T007200	-318	0.101	220	85.3	-335	8.21	25-Aug-2009
S09T007201	-304	0.106	176	117	-327	8.61	26-Aug-2009
S09T007202	-794	2.379	20.1	-702	-831	193	27-Aug-2009
S09T007203	-454	0.053	-57.4	-360	-488	4.31	1-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-11, S09T007178</i>							
S09T007208	-298	0.014	237	-177	-320	11.3	25-Sep-2009
S09T007209	-316	0.104	197	59.4	-337	8.45	30-Sep-2009
S09T007209A	-304	0.082	202	98.6	-325	6.66	6-Oct-2009
S09T007210	-419	0.042	158	-368	-461	3.41	7-Oct-2009
S09T007211	-354	0.068	202	-369	-395	5.52	8-Oct-2009

Table B-2. Electrochemical Data (2 sheets)

LPR Data					
Sample	OCP (mV)	I _{corr} (μA)	E _{Corr} (mV)	MPY (E-3)	Date Complete
<i>241-AN-106 Grab Sample 6AN-09-02, S09T001178</i>					
S09T002346	109	0.047	105	3.82	15-Jul-2009
<i>241-AN-106 Grab Sample 6AN-09-05, S09T007167</i>					
S09T007168	-69.6	0.018	-87.2	1.46	28-Aug-2009
S09T007169	-13.0	0.090	-26.5	0.731	1-Sep-2009
S09T007170	51.2	0.006	43.7	0.487	2-Sep-2009
S09T007171	114	0.020	108	1.62	3-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-06, S09T007173</i>					
S09T007184	45.4	0.011	37.1	0.894	3-Sep-2009

Table B-2. Electrochemical Data (2 sheets)

LPR Data					
Sample	OCP (mV)	I _{corr} (μA)	E _{Corr} (mV)	MPY (E-3)	Date Complete
S09T007185	19.4	0.006	8.07	0.487	9-Sep-2009
S09T007186	-25.4	0.008	-32.5	0.650	16-Sep-2009
S09T007187	57.7	0.014	49.2	1.14	22-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-06DUP, S09T007174</i>					
S09T007188	-1.65	0.009	-13.6	0.731	27-Aug-2009
S09T007189	77.6	0.016	70.6	1.30	28-Aug-2009
S09T007190	12.3	0.005	1.51	0.406	1-Sep-2009
S09T007191	120	0.004	115	0.325	2-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-08, S09T007175</i>					
S09T007192	24.1	0.009	14.3	0.731	3-Sep-2009
S09T007193	41.9	0.013	31.0	1.06	22-Sep-2009
S09T007194	98.5	0.016	90.9	1.30	23-Sep-2009
S09T007195	116	0.024	110	1.95	24-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-07, S09T007176</i>					
S09T007196	22.2	0.013	14.1	1.06	23-Sep-2009
S09T007197	101	0.025	93.9	2.03	24-Sep-2009
S09T007198	98.6	0.016	92.2	1.30	25-Sep-2009
S09T007199	-206				30-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-10, S09T007177</i>					
S09T007200	-318	0.025	-325	2.03	25-Aug-2009
S09T007201	-303	0.023	-310	1.87	26-Aug-2009
S09T007202	-828	1.285	-828	1.04	27-Aug-2009
S09T007203	-454	0.015	-464	1.22	1-Sep-2009
<i>241-AN-106 Grab Sample 6AN-09-11, S09T007179</i>					
S09T007208	-299	0.028	-307	2.27	25-Sep-2009
S09T007209	-315	0.035	-320	2.84	30-Sep-2009
S09T007209A	-306	0.026	-312	0.487	6-Oct-2009
S09T007210	-422	0.006	-432	1.87	7-Oct-2009
S09T007211	-348	0.023	-356	2.11	8-Oct-2009

APPENDIX C

X-Ray Diffraction Analysis

Table C-1. X-Ray Diffraction Pattern of Precipitate of 6AN-09-03**Sample Number: S09T003615**

Sample S09T003615. The major phases identified were Sodium Fluoride Phosphate Hydrate $[\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}]$ and Dawsonite $[\text{NaAlCO}_3(\text{OH})_2]$. A minor phase of $[\text{NaSr}(\text{PO}_4)(\text{H}_2\text{O})_9]$ was also identified as a possible phase. Sodium Nitrate $[\text{NaNO}_3]$ and Sodium Nitrite $[\text{NaNO}_2]$ were identified; but were probably residues from the supernatant liquid when the sample dried.

Table C-2. X-Ray Diffraction Pattern of Precipitate of 6AN-09-03DUP**Sample Number: S09T003616**

Sample S09T003616. The major phases identified were Dawsonite [$\text{NaAlCO}_3(\text{OH})_2$] as the major phase and Sodium Fluoride Phosphate Hydrate [$\text{Na}_7\text{F}(\text{PO}_4)_2 \cdot 19\text{H}_2\text{O}$] as a minor phase.

APPENDIX D

Energy Dispersion Spectroscopy

S09T002349-1.TIF

4/21/2009

11:24:37 AM

15.049 ×

accelerating voltage = 20.0 kV working distance = 25.9 mm spot size = 25.0% live time = 30.00

S09T002349-2.TIF

4/21/2009

11:29:49 AM

263.01 ×

accelerating voltage = 20.0 kV

working distance = 22.4 mm

spot size = 25.0%

live time = 30.00

S09T002349-3.TIF

4/21/2009

11:36:05 AM

234.08 ×

accelerating voltage = 20.0 kV working distance = 22.4 mm spot size = 20.0% live time = 15.00

S09T002350-1.TIF

4/21/2009

11:39:38 AM

13.883 ×

accelerating voltage = 20.0 kV working distance = 22.4 mm spot size = 20.0% live time = 15.00

S09T002350-2.TIF

4/22/2009

11:42:21 AM

46.789 ×

accelerating voltage = 20.0 kV working distance = 22.4 mm spot size = 20.0% live time = 80.00

S09T002354-1.TIF

4/22/2009

12:12:38 PM

13.140 ×

accelerating voltage = 20.0 kV working distance = 24.1 mm spot size = 20.0% live time = 30.00

S09T002354-2.TIF

4/22/2009

12:14:57 PM

13.452 ×

accelerating voltage = 20.0 kV working distance = 23.4 mm spot size = 20.0% live time = 15.00

S09T002354-3.TIF

4/22/2009

12:15:59 PM

13.452 ×

accelerating voltage = 20.0 kV

working distance = 23.4 mm

spot size = 20.0%

live time = 20.00

S09T002354-4.TIF

4/22/2009

12:16:49 PM

13.452 ×

accelerating voltage = 20.0 kV working distance = 23.4 mm spot size = 20.0% live time = 15.00

S09T002354-5.TIF

4/22/2009

12:19:53 PM

13.452 ×

accelerating voltage = 20.0 kV

working distance = 23.4 mm

spot size = 20.0%

live time = 30.00

APPENDIX E

Optical Images

S09T002349

10X

20X

40X

60X

60X

20X

20X

20X

S09T002350

10X

10X

20x

20X

20X

40X

60X

60X

UserName: Wyrwas, Richard (h3390899)

Title: APD Chemist

Date: Wednesday, 14 April 2010, 12:48 PM Pacific Daylight Time

Meaning: Approved by the author or delegate

UserName: Mabrouki, Ridha (h1335536)

Title:

Date: Wednesday, 14 April 2010, 01:33 PM Pacific Daylight Time

Meaning: Additional approval obtained

UserName: Carothers, Kelly (h0072896)

Title: Process Engineering

Date: Wednesday, 14 April 2010, 01:45 PM Pacific Daylight Time

UserName: Frye, Jann (h0049995)

Title: APD Chemist

Date: Wednesday, 14 April 2010, 03:01 PM Pacific Daylight Time

Meaning: Approved by the APD Manager or delegate