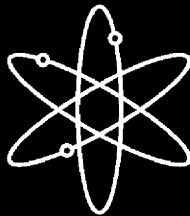




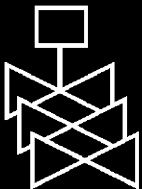
# **Integrated Chemical Effects Test Project: Test #3 Data Report**



**Los Alamos National Laboratory**



**U.S. Nuclear Regulatory Commission  
Office of Nuclear Regulatory Research  
Washington, DC 20555-0001**



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# Integrated Chemical Effects Test Project: Test #3 Data Report

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# INTEGRATED CHEMICAL EFFECTS TEST PROJECT: TEST #3 DATA REPORT

## ABSTRACT

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized water reactor containment water pool after a loss-of-coolant-accident. The initial chemical environment contained 14.54 kg of boric acid and 0.663 g of lithium hydroxide. Trisodium phosphate (3.786 kg), hydrochloric acid (211 mL), and additional boric acid (600 g) were added beginning at 30 minutes and lasting until 4 hours into the test. The test was conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within this environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples (80% calcium silicate and 20% fiberglass). Representative amounts of concrete dust and latent debris were also added to the test solution. Water was circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens. The test solution reached a pH of 7.9 by Day 3, and the test solution turbidity decreased to less than 1 NTU after 24 hours. During the introduction of trisodium phosphate at the beginning of the test, a white flocculence was observed through the submerged observation window. This flocculence was accompanied by a rise in turbidity and total suspended solids (TSS). Turbidity and TSS dropped after chemical addition was complete, and the white flocculence was no longer visible in the water after the first day. Observations of the test solution indicated similar behavior of the solution at both room temperature and test temperature. After the initial flocculence had settled out of solution, no chemical byproducts were visible in the water and no precipitation occurred as samples cooled from test temperature to room temperature. Large amounts of white deposits of varying size were observed on the submerged galvanized steel, aluminum, copper, and inorganic zinc coated steel coupons. The bottom of the tank was filled with sediment that had a pinkish-white deposit on top. The test solution remained clearly Newtonian for the entire test. Aluminum was detectable in the solution, but only in trace amounts.

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# CONTENTS

ABSTRACT.....	iii
EXECUTIVE SUMMARY .....	xiii
ACKNOWLEDGMENTS .....	xv
ACRONYMS AND ABBREVIATIONS .....	xvii
1. INTRODUCTION .....	1
1.1. Objective and Test Conditions.....	1
1.2. Information Presented in This Report.....	2
2. TEST PROCEDURES .....	3
2.1. Chemical Test Apparatus Functional Description .....	3
2.2. Pre-Test Preparation.....	4
2.2.1. Test Loop Cleaning.....	4
2.2.2. Test Coupons and Samples .....	4
2.2.3. Quality Assurance Program .....	10
2.2.4. Test Parameters .....	10
2.3. Test Operation.....	12
2.3.1. Description.....	12
2.3.2. Process Control .....	13
2.4. Analytical Methods.....	13
2.4.1. Data Compilation and Nomenclature.....	13
3. TEST RESULTS.....	21
3.1. General Observations.....	21
3.1.1. Control of Test Parameters .....	22
3.1.2. Hydrogen Generation.....	24
3.2. Water Samples .....	25
3.2.1. Wet Chemistry .....	25
3.2.2. Metal Ion Concentration .....	28
3.2.3. Optical/TEM Images from Filtered and Unfiltered Samples.....	33
3.3. Insulation.....	33
3.3.1. Deposits in Fiberglass Samples .....	33
3.3.2. Calcium Silicate Samples .....	70
3.4. Metallic and Concrete Samples .....	70
3.4.1. Weights and Visual Descriptions.....	70
3.4.2. SEM Analyses.....	79
3.5. Sedimentation .....	80
3.6. Precipitates.....	86
3.7. Corrosion Products.....	86
3.8. Gel Analysis.....	88
3.8.1. Visual Description .....	88
3.8.2. ESEM and SEM/EDS Analyses.....	89
4. SUMMARY OF KEY OBSERVATIONS .....	97
REFERENCES .....	98

# APPENDICES

Preface to Appendices.....	Preface-1
Appendix A. SEM and ESEM/EDS Data for Test #3, Day-4 Fiberglass in Low-Flow Zone .....	A-i
Appendix B1. ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in High-Flow Zones .....	B1-i
Appendix B2. ESEM/EDS and SEM/EDS Data for Test #3, Day-15 Fiberglass in Low-Flow Zones.....	B2-i
Appendix C1. ESEM/EDS Data for Test #3, Day-30 Fiberglass in High-Flow Zones .....	C1-i
Appendix C2. ESEM Data for Test #3, Day-30 Fiberglass in Low-Flow Zones.....	C2-i
Appendix C3. ESEM/EDS Data for Test #3, Day-30 Drain Collar Fiberglass.....	C3-i
Appendix C4. SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Birdcage Fiberglass .....	C4-i
Appendix D. ESEM and SEM/EDS Data for Test #3, Day-30 Corrosion Products .....	D-i
Appendix E1. SEM/EDS Data for Test #3, Day-30 Aluminum Coupons .....	E1-i
Appendix E2. SEM/EDS Data for Test #3, Day-30 Copper Coupons.....	E2-i
Appendix E3. SEM/EDS Data for Test #3, Day-30 Galvanized Steel Coupons .....	E3-i
Appendix E4. SEM/EDS Data for Test #3, Day-30 Steel Coupons .....	E4-i
Appendix F. SEM/EDS Data for Test #3, Day-30 Flow Meter.....	F-i
Appendix G. SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Gel.....	G-i
Appendix H. SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Cal-Sil .....	H-i
Appendix I. ESEM/EDS Data for Test #3, Day-30 Sediment.....	I-i
Appendix J. TEM Data for Test #3 Solution Samples.....	J-i
Appendix K. UV Absorbance Spectrum—Day-30 Solution Samples .....	K-i
Appendix L. ICET Test #3: Pre-Test, Test, and Post-Test Project Instructions .....	L-i



## FIGURES

Figure 2-1.	Test loop process flow diagram. ....	4
Figure 2-2.	Coupon rack configuration in the ICET tank. The blue line represents the surface of the test solution. ....	6
Figure 2-3.	A loaded coupon rack in the ICET tank.....	7
Figure 2-4.	Fiberglass samples attached to coupon rack. ....	8
Figure 2-5.	Solid pieces of cal-sil samples in SS mesh. ....	9
Figure 2-6.	Cal-sil dust. ....	9
Figure 3-1.	In-line pH measurements. ....	23
Figure 3-2.	Bench-top pH meter results. ....	24
Figure 3-3.	Hydrogen generation.....	24
Figure 3-4.	Day-1 turbidity results. ....	25
Figure 3-5.	Daily turbidity results. ....	26
Figure 3-6.	Test #3 TSS results. ....	27
Figure 3-7.	Viscosity at 60°C and 23°C. ....	28
Figure 3-8.	Aluminum concentration. ....	29
Figure 3-9.	Calcium concentration. ....	29
Figure 3-10.	Copper concentration. ....	30
Figure 3-11.	Iron concentration. ....	30
Figure 3-12.	Magnesium concentration. ....	31
Figure 3-13.	Silica concentration.....	31
Figure 3-14.	Sodium concentration. ....	32
Figure 3-15.	Zinc concentration. ....	32
Figure 3-16.	Phosphate concentration. ....	33
Figure 3-17.	ESEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 150 times. (T3D4FX1, 4/12/05).....	36
Figure 3-18.	ESEM image for a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 1000 times. (T3D4FX2, 4/12/05).....	36
Figure 3-19.	EDS counting spectrum (after calibration) for the deposits between the fibers on the ESEM image shown in Figure 3-18. (T3D4FX4, 4/12/05).....	37
Figure 3-20.	SEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 150 times. (T3D4FibGlsEX001, 4/12/05) .....	37
Figure 3-21.	SEM image of a Test #3, Day-4 low-flow exterior fiberglass sample, magnified 300 times. (T3D4FibGlsEX003, 4/12/05) .....	38
Figure 3-22.	ESEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 150 times. (T3D4FI6, 4/12/05) .....	38
Figure 3-23.	ESEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 1000 times. (T3D4FI7, 4/12/05).....	39

Figure 3-24.	EDS counting spectrum (after calibration) for the deposits between the fibers on the ESEM image shown in Figure 3-23. (T3D4FI8, 4/12/05) .....	39
Figure 3-25.	SEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 50 times. (T3D4FibGlsIN001, 4/12/05) .....	40
Figure 3-26.	SEM image for a Test #3, Day-4 low-flow interior fiberglass sample, magnified 150 times. (T3D4FibGlsIN002, 4/12/05).....	40
Figure 3-27.	SEM image of a Test #3, Day-4 low-flow interior fiberglass sample, magnified 400 times. (T3D4FibGlsIN003, 4/12/05).....	41
Figure 3-28.	ESEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 110 times. (T3D15LX9).....	42
Figure 3-29.	ESEM image of a Test #3, Day15 low-flow exterior fiberglass sample, magnified 1000 times. (T3D15LX0).....	42
Figure 3-30.	EDS counting spectrum for the deposits between the fibers on the ESEM image shown in Figure 3-29. (T3D15LXD) .....	43
Figure 3-31.	SEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 100 times. (T3D15LowFlowExt011).....	43
Figure 3-32.	SEM image of a Test #3, Day-15 low-flow exterior fiberglass sample, magnified 1000 times. (T3D15LowFlowExt013).....	44
Figure 3-33.	EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-32. (T3D15LowFlowExtEDS3).....	44
Figure 3-34.	ESEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 100 times. (T3D15LI6) .....	45
Figure 3-35.	ESEM image of a Test# 3, Day-15 low-flow interior fiberglass sample, magnified 1000 times. (T3D15LI8) .....	45
Figure 3-36.	EDS counting spectrum for the flocculent deposits between the fibers on the ESEM image shown in Figure 3-35. (T3D15LIC) .....	46
Figure 3-37.	SEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 100 times. (T3D15LowFlowInt008).....	46
Figure 3-38.	SEM image of a Test #3, Day-15 low-flow interior fiberglass sample, magnified 1000 times. (T3D15LowFlowInt010).....	47
Figure 3-39.	ESEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 110 times. (T3D15HX4, 4/22/05).....	48
Figure 3-40.	ESEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 1000 times. (T3D15HX5, 4/22/05).....	48
Figure 3-41.	EDS counting spectrum for the deposits between the fibers on the ESEM image shown in Figure 3-40. (T3D15HIB, 4/22/05) .....	49
Figure 3-42.	SEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 100 times. (T3D15HiFlowExt005, 4/22/05).....	49
Figure 3-43.	SEM image of a Test #3, Day-15 high-flow exterior fiberglass sample, magnified 500 times. (T3D15HiFlowExt007, 4/22/05).....	50
Figure 3-44.	EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-43. (T3D15HiFlowExtEDS2, 4/22/05).....	50

Figure 3-45.	ESEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 100 times. (T3D15HI1, 4/22/05) .....	51
Figure 3-46.	ESEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 2000 times. (T3D15HI3, 4/22/05) .....	51
Figure 3-47.	EDS counting spectrum for the flocculent deposits between the fibers on the ESEM image shown in Figure 3-46. (T3D15HIA, 4/22/05) .....	52
Figure 3-48.	SEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 100 times. (T3D15HIFlowInt002, 4/22/05).....	52
Figure 3-49.	SEM image of a Test #3, Day-15 high-flow interior fiberglass sample, magnified 1000 times. (T3D15HIFlowInt004, 4/22/05).....	53
Figure 3-50.	EDS counting spectrum for the flocculent deposits between the fibers on the SEM image shown in Figure 3-49. (T3D15HiFlowIntEDS1, 4/22/05).....	53
Figure 3-51.	ESEM image of a Test #3, Day-30 exterior low-flow fiberglass sample, magnified 70 times. (t3lfex09, 5/6/05).....	54
Figure 3-52.	ESEM image of a Test #3, Day-30 exterior low-low fiberglass sample, magnified 1000 times. (t3lfex11, 5/6/05).....	54
Figure 3-53.	ESEM image of a Test #3, Day-30 interior low-flow fiberglass sample, magnified 70 times. (t3lfin12, 5/6/05) .....	55
Figure 3-54.	ESEM image of a Test #3, Day-30 interior low-flow fiberglass sample, magnified 1000 times. (t3lfin13, 5/6/05) .....	55
Figure 3-55.	ESEM image of a Test #3, Day-30 exterior high- flow fiberglass sample, magnified 100 times. (t3hifx33, 5/11/05) .....	56
Figure 3-56.	EDS counting spectrum for the large masses of particulate deposits shown in Figure 3-55. (t3hifx34, 5/11/05) .....	57
Figure 3-57.	ESEM image of a Test #3, Day-30 exterior high-flow fiberglass sample, magnified 600 times. (t3hifx35, 5/11/05) .....	57
Figure 3-58.	ESEM image of a Test #3, Day-30 interior high-flow fiberglass sample, magnified 100 times. (T3HiFI36, 5/11/05).....	58
Figure 3-59.	ESEM image of a Test #3, Day-30 interior high-flow fiberglass sample, magnified 1000 times. (t3hifi37, 5/11/05) .....	58
Figure 3-60.	Drain screen collar removed from the tank.....	59
Figure 3-61.	ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen), magnified 100 times. (t3dcex25, 5/6/05).....	60
Figure 3-62.	ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (away from the drain screen), magnified 1000 times. (t3dcex21, 5/6/05).....	60
Figure 3-63.	EDS counting spectrum for the light particulate deposits (EDS1) shown in Figure 3-62. (t3dcex22, 5/6/05).....	61
Figure 3-64.	EDS counting spectrum for the dark deposits (EDS2) shown in Figure 3-62. (t3dcex23, 5/6/05).....	61
Figure 3-65.	Comparison of EDS counting spectra between Figure 3-63 (yellow) and Figure 3-64 (red). (t3dcex24, 5/6/05) .....	62

Figure 3-66.	ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen), magnified 100 times. (t3DCSC16, 5/6/05).....	62
Figure 3-67.	ESEM image of a Test #3, Day-30 exterior fiberglass sample on the drain collar (adjacent to the drain screen), magnified 1000 times. (t3dcsc17, 5/6/05) .....	63
Figure 3-68.	EDS counting spectrum for the particulate deposits shown in Figure 3-67. (t3dcsc18, 5/6/05) .....	63
Figure 3-69.	ESEM image of a Test #3, Day-30 interior fiberglass sample on the drain collar, magnified 100 times. (t3dcin28, 5/6/05).....	64
Figure 3-70.	ESEM image of a Test #3, Day-30 interior fiberglass sample on the drain collar, magnified 1000 times. (t3dcin27, 5/6/05).....	64
Figure 3-71.	Tank bottom after the test fluid was drained. ....	65
Figure 3-72.	ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 80 times. (T3BCEX01, 5/6/05).....	66
Figure 3-73.	EDS counting spectrum for the large deposits shown in Figure 3-72. (t3bcexe2, 5/6/05).....	66
Figure 3-74.	ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 80 times. (t3bcex02, 5/6/05) .....	67
Figure 3-75.	ESEM image of a Test #3, Day-30 exterior fiberglass sample within the birdcage, magnified 500 times. (t3bcex03, 5/6/05) .....	67
Figure 3-76.	EDS counting spectrum for the deposits shown in Figure 3-75. (T3BCExE1, 5/6/05).....	68
Figure 3-77.	Comparison of EDS counting spectra of Figure 3-76 (red) and Figure 3-73 (yellow). (t3bcexe3, 5/6/05).....	68
Figure 3-78.	ESEM image of a Test #3, Day-30 interior fiberglass sample within the birdcage, magnified 80 times. (t3bcin05, 5/6/05).....	69
Figure 3-79.	ESEM image of a Test #3, Day-30 interior fiberglass sample within the birdcage, magnified 1000 times. (t3bcin07, 5/6/05).....	69
Figure 3-80.	Al-155, submerged, pre-test.....	71
Figure 3-81.	Al-155, submerged, post-test. ....	71
Figure 3-82.	Al-156, submerged, pre-test.....	71
Figure 3-83.	Al-156 submerged, post-test. ....	71
Figure 3-84.	Al-157, submerged, pre-test. ....	72
Figure 3-85.	Al-157, submerged, post-test. ....	72
Figure 3-86.	GS-468, submerged, pre-test.....	72
Figure 3-87.	GS-468, submerged, post-test. ....	72
Figure 3-88.	IOZ-156, submerged, pre-test. ....	73
Figure 3-89.	IOZ-156, submerged, post-test. ....	73
Figure 3-90.	CU-207 submerged, pre-test. ....	73
Figure 3-91.	CU-207, submerged, post-test.....	73
Figure 3-92.	CU-225, submerged, pre-test. ....	74
Figure 3-93.	CU-225, submerged, post-test.....	74

Figure 3-94.	US-11, submerged, pre-test.....	74
Figure 3-95.	US-11, submerged, post-test.....	74
Figure 3-96.	Conc-005, submerged, pre-test.....	75
Figure 3-97.	Conc-005, submerged, post-test.....	75
Figure 3-98.	Al-159, unsubmerged, pre-test.....	76
Figure 3-99.	Al-159, unsubmerged, post-test.....	76
Figure 3-100.	GS-503, unsubmerged, pre-test.....	76
Figure 3-101.	GS-503, unsubmerged, post-test.....	76
Figure 3-102.	CU-228, unsubmerged, pre-test.....	77
Figure 3-103.	CU-228 unsubmerged, post-test.....	77
Figure 3-104.	IOZ-187, unsubmerged, pre-test.....	77
Figure 3-105.	IOZ-187, unsubmerged, post-test.....	77
Figure 3-106.	US-13, unsubmerged, pre-test.....	78
Figure 3-107.	US-13, unsubmerged, post-test.....	78
Figure 3-108.	Sediment removed from the tank. Some gel appears on the top.....	81
Figure 3-109.	Samples removed from the tank bottom.....	81
Figure 3-110.	ESEM image of a Test #3, Day-30 pink sediment, magnified 100 times. (t3pnkp31, 5/6/05).....	82
Table 3-111.	ESEM image of a Test #3, Day-30 pink sediment, magnified 1000 times. (t3PNKP29, 5/6/05).....	82
Table 3-112.	EDS counting spectrum for the sediment shown in Figure 3-111. (t3pnkp30, 5/6/05).....	83
Figure 3-113.	ESEM image of a Test #3, Day-30 yellow sediment, magnified 100 times. (t3ylwp33, 5/6/05).....	83
Figure 3-114.	ESEM image of a Test #3, Day-30 yellow sediment, magnified 100 times. (t3ylwp34, 5/6/05).....	84
Figure 3-115.	EDS counting spectrum for the particles shown in Figure 3-114. (t3ylwp35, 5/6/05).....	84
Figure 3-116.	Comparison of EDS counting spectra for pink sediment (yellow, t3pnkp30) and yellow sediment (red, t3ylwp35). (t3ylwp36, 5/6/05).....	85
Figure 3-117.	XRD results for Test #3, Day-30 mixed sediment.....	85
Figure 3-118.	Gel-like material covering SS mesh on the bottom of the test tank.....	88
Figure 3-119.	Gel-like material recovered from the bottom of the test tank.....	89
Figure 3-120.	SEM image of a Test #3, Day-30 white gel-like material from the top of the birdcage, magnified 100 times. (T3D30GelMaterial003, 5/9/05).....	90
Figure 3-121.	SEM image of a Test #3, Day-30 white gel-like material from the top of the birdcage, magnified 1000 times. (T3D30GelMaterial004, 5/9/05).....	91
Figure 3-122.	EDS counting spectrum for the white, gel-like material (whole image) shown in Figure 3-121. (T3D30Gel02, 5/9/05).....	91

Figure 3-123. ESEM image of a Test #3, Day-30 white gel-like material from the top of the birdcage, magnified 1000 times. (t3Gel08, 5/6/05) .....	93
Figure 3-124. EDS counting spectrum for the white, gel-like material shown in Figure 3-123. (t3geled6, 5/6/05).....	93
Figure 3-125. Comparison of EDS counting spectra for Figure 3-124 (yellow: the gel-like materials shown in Figure 3-123) and Figure 3-73 (red: the large deposits taken from the birdcage exterior shown in Figure 3-72). (t3geled7, 5/6/05) .....	94
Figure 3-126. XRD results for a Test #3, Day-30 white, gel-like sample.....	94

## TABLES

Table 1-1. Test Series Parameters .....	2
Table 2-1. Quantity of Each Coupon Type in Test #3 .....	5
Table 2-2. Material Quantity/Sump Water Volume Ratios for the ICET Tests.....	11
Table 3-1. ICP Results for Selected Elements .....	28
Table 3-2. Weight Data for Submerged Coupons .....	75
Table 3-3. Mean Weight Gain/Loss (g) Data for Submerged Coupons.....	75
Table 3-4. Weight Data for Unsubmerged Coupons.....	78
Table 3-5. Mean Gain/Loss (g) Data for Unsubmerged Coupons .....	78
Table 3-6. Dry Mass Compositions of Test #3, Day-30 Sediment by XRF Analysis.....	86
Table 3-7. The Chemical Compositions for Figure 3-122 .....	92
Table 3-8. Dry Mass Composition Results for a Test #3, Day-30 White Gel-like Sample by XRF Analysis.....	95

# INTEGRATED CHEMICAL EFFECTS TEST PROJECT: TEST #3 DATA REPORT

## EXECUTIVE SUMMARY

The U.S. Nuclear Regulatory Commission (NRC) Office of Nuclear Regulatory Research has developed a comprehensive research program to support resolution of Generic Safety Issue (GSI)-191. GSI-191 addresses the potential for debris accumulation on pressurized-water-reactor (PWR) sump screens with the consequent loss of net-positive-suction-head margin in the emergency-core-cooling-system (ECCS) pump. Among the GSI-191 research program tasks is the experimental investigation of chemical effects that may exacerbate sump-screen clogging.

The Integrated Chemical Effects Test (ICET) project represents a joint effort by the U.S. NRC and the nuclear utility industry, undertaken through the Memorandum of Understanding on Cooperative Nuclear Safety between NRC and Electrical Power Research Institute, Addendum on Integral Chemical Effects Testing for PWR ECCS Recirculation. The ICET project simulates the chemical environment present inside a containment water pool after a loss-of-coolant accident (LOCA) and monitors the chemical system for an extended time to identify the presence, composition, and physical characteristics of chemical products that form during the test. The ICET test series is being conducted by Los Alamos National Laboratory at the University of New Mexico, with the assistance of professors and students in the civil engineering department.

This report describes the ICET experimental apparatus and surveys the principal findings of Test #3. As an interim data report compiled during preparation for subsequent ICET tests, this description summarizes both primary and representative findings that were available at the time the report was prepared. The NRC and the nuclear power industry may conduct additional analyses to enhance the understandings obtained from this test.

All of the ICET tests are being conducted in an environment that simulates expected containment pool conditions during recirculation. The initial chemical environment contains 2800 mg/L of boron, 100 mg/L of hydrochloric acid, and 0.7 mg/L of lithium hydroxide. (The hydrochloric acid and a small portion of the boron were added during the spray phase.) The tests are conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within this environment include representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples. Representative amounts of concrete dust and latent debris are also added to the test solution. Tests consist of an initial 4-hour spray phase to simulate containment spray interaction with the unsubmerged samples. Water is circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens.

ICET Test #3 was conducted using trisodium phosphate (TSP) to control pH, with a target pH of 7. (TSP was added during the spray phase.) Insulation samples consisted of scaled amounts of NUKON™ fiberglass and calcium silicate (cal-sil) material. In addition, the test apparatus contained 373 metal coupon samples and 1 concrete sample. Process control consisted of

monitoring online measurements of recirculation flow rate, test solution temperature, and pH. Flow rate and temperature were controlled to maintain the desired values of 25 gpm and 140°F. Daily water samples were obtained for measurements of pH, turbidity, total suspended solids, kinematic viscosity, and shear-dependent viscosity and for analytical laboratory evaluations of the chemical elements present. In addition, microscopic evaluations were conducted on water sample filtrates, fiberglass, cal-sil, coupons, and sediment.

Before time zero, 14.54 kg of boric acid and 0.663 g of LiOH were dissolved into the ICET tank. The measured in-line probe pH was 4.2, which was the expected value obtained from analytical predictions. TSP Batch 1 (300 g of boric acid, 1893 g of TSP) and Batch 2 (300 g of boric acid, 1893 g of TSP, 211 mL of 12.29 N HCl) were added into the recirculation line from 30 minutes until 4 hours into the test. When these prepared batches were added, the pH began to rise until it reached a pH of 7.32 (bench-top reading) 4 hours after the initiation of TSP addition. The test ran uninterrupted for 30 days, and the conditions were maintained within the accepted flow and temperature ranges.

During the introduction of TSP at the beginning of the test, a white flocculence was observed through the submerged observation window. This flocculence was accompanied by a rise in turbidity and total suspended solids (TSS). Turbidity and TSS dropped after chemical addition was complete, and the white flocculence was no longer visible in the water after the first day. Observations of the test solution indicated similar behavior of the solution at both room temperature and test temperature. After the initial flocculence had settled out of solution, no chemical byproducts were visible in the water and no precipitation occurred as samples cooled from test temperature to room temperature.

Analyses of the test solution revealed aluminum in the solution, but only at trace amounts. Calcium, silica, and sodium were prevalent in the solution.

Examinations of fiberglass taken from the test apparatus after 15 days revealed chemical products and a web-like material that spanned individual fibers. After 30 days of testing, the web-like material was more prevalent, and contiguous webbing appeared to span multiple fibers.

Daily measurements of the constant-shear kinematic viscosity revealed an approximately constant value at both test temperature and room temperature for both filtered and unfiltered samples. Shear-dependent viscosity measurements indicated that the test solution was representative of a Newtonian fluid.

The ICET series is being conducted under an approved quality assurance (QA) program, and QA procedures and project instructions were reviewed and approved by the project sponsors. Analytical laboratory results are generated under a quality control program approved by the Environmental Protection Agency, and other laboratory analyses were performed using standard practices as referenced in the body of this report.



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## ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing of Materials
cal-sil	Calcium Silicate
CFR	Code of Federal Regulations
CPVC	Chlorinated Polyvinyl Chloride
CS	Coated Steel
DAS	Data Acquisition System
DHR	Decay Heat Removal
ECCS	Emergency Core-Cooling System
EDS	Energy-Dispersive Spectroscopy
EPA	Environmental Protection Agency
ESEM	Environmental Scanning Electron Microscopy
F	Filtered
gpm	Gallons per Minute
GS	Galvanized Steel
GSI	Generic Safety Issue
HCl	Hydrochloric Acid
HI	High-Flow Interior Fiberglass Sample
HX	High-Flow Exterior Fiberglass Sample
ICET	Integrated Chemical Effects Tests
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma—Atomic Emission Spectroscopy
IOZ	Inorganic Zinc
LANL	Los Alamos National Laboratory
LCS	Laboratory Control Sample
LI	Low-Flow Interior Fiberglass Sample
LiOH	Lithium Hydroxide
LOCA	Loss-of-Coolant Accident
LX	Low-Flow Exterior Fiberglass Sample
MSDS	Material Safety Data Sheets
NIST	National Institute of Standards and Technology
NRC	Nuclear Regulatory Commission
NTU	Nephelometric Turbidity Unit
PI	Project Instruction

PVC	Polyvinyl Chloride
PWR	Pressurized Water Reactor
QA	Quality Assurance
RO	Reverse Osmosis
SEM	Scanning Electron Microscopy
SS	Stainless Steel
T1	ICET Test #1
T2	ICET Test #2
T3	ICET Test #3
TEM	Transmissive Electron Microscopy
TOC	Total Organic Carbon
TSP	Trisodium Phosphate
TSS	Total Suspended Solids
U	Unfiltered
UNM	University of New Mexico
US	Uncoated Steel
UV	Ultraviolet
VFD	Variable Frequency Drive
WD	Working Distance
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

# 1. INTRODUCTION

The Integrated Chemical Effects Test (ICET) project represents a joint effort by the United States Nuclear Regulatory Commission (NRC) and the nuclear utility industry to simulate the chemical environment present inside a containment structure after a loss-of-coolant accident (LOCA) and to monitor the chemical system for an extended time to identify the presence, composition, and physical characteristics of chemical products that may form. The ICET series is being conducted by Los Alamos National Laboratory (LANL) at the University of New Mexico (UNM), with the assistance of professors and students in the civil engineering department.

## 1.1. Objective and Test Conditions

Containment buildings of pressurized water reactors (PWRs) are designed to accommodate the energy release following a postulated accident. They also permit recirculation of reactor coolant and emergency-core-cooling-system (ECCS) water to the decay heat removal (DHR) heat exchangers. The water collected in the sump from the reactor coolant system, the safety injection system, and the containment spray system is recirculated to the reactor core to remove residual heat. The sump contains a screen to protect system structures and components in the containment spray and ECCS flow paths from the effects of debris that could be transported to the sump. Concerns have been raised that fibrous insulation material could form a mat on the screen, obstructing flow, and that chemical reaction products such as gelatinous or crystalline precipitants could migrate to the screen, causing further blockage and increased pressure-head losses across the debris bed. Another potential adverse chemical effect includes increased bulk fluid viscosity that could also increase head losses through a debris bed.

The primary objectives for the ICET series are (1) to determine, characterize, and quantify chemical reaction products that may develop in the containment sump under a representative post-LOCA environment and (2) to determine and quantify any gelatinous material that could be produced during the post-LOCA recirculation phase.

The ICET series was conceived as a limited-scope suite of five different 30-day tests with different constituents. The conditions selected for each test are shown in Table 1-1. As shown in this table, Test #3 of the ICET series was to be operated at a low pH level (~7) and included substantial quantities of calcium silicate insulation material as well as fiberglass. All tests in the series included metal coupons whose surface areas were scaled to those in representative PWR containment and sump systems.

A complete rationale for the selection of these test conditions is provided in Ref. 1, but in brief, the ICET apparatus consists of a large stainless-steel (SS) tank with heating elements, spray nozzles, and associated recirculation pump and piping to simulate the post-LOCA chemical environment. Samples of structural metals, concrete, and insulation debris are scaled in proportion to their relative surface areas found in containment and in proportion to a maximum test dilution volume of 250 gal. of circulating fluid.

**Table 1-1. Test Series Parameters**

Run	Temp	TSP <sup>a</sup>	NaOH	pH	Boron	Note
	(°C)	Na <sub>3</sub> PO <sub>4</sub> ·12H <sub>2</sub> O			(mg/L)	
1	60	N/A	Yes	10	2800	100% fiberglass insulation test. High pH, NaOH concentration as required by pH
2	60	Yes	N/A	7	2800	100% fiberglass insulation test. Low pH, TSP concentration as required by pH.
3	60	Yes	N/A	7	2800	80% calcium silicate/20% fiberglass insulation test. Low pH, TSP concentration as required by pH
4	60	N/A	Yes	10	2800	80% calcium silicate/20% fiberglass insulation test. High pH, NaOH concentration, as required by pH.
5	60	TBD	TBD	TBD	TBD	Confirmatory test; one of the above four tests will be repeated.

<sup>a</sup>TSP = trisodium phosphate.

Representative chemical additives, temperature, and material combinations are established in each test; the system is then monitored while corrosion and fluid circulation occur for a duration comparable to the ECCS recirculation mission time.

## 1.2. Information Presented in This Report

This report surveys the principal findings of ICET Test #3. As an interim data report compiled during preparation for subsequent ICET tests, this exposition summarizes both primary and representative findings, but it cannot be considered comprehensive. For example, only a small selection of photographs out of several hundred is presented here. In addition, this report presents observations and data without in-depth analyses or interpretations. However, trends and typical behaviors are noted where appropriate. Section 2 of this report reviews the test procedures followed for Test #3. Analytical techniques used in evaluating test results are also briefly reviewed in Section 2. Section 3 presents key test results for Test #3, including representative and noteworthy results of water sampling, insulation samples (cal-sil and fiberglass), metallic and concrete coupon samples, tank sediment, corrosion products, and gel. The results for Test #3 are presented in both graphical and narrative form. Section 4 presents a summary of key observations for Test #3. This report also includes several appendices that capture additional Test #3 images and information. The data presented in the appendices are largely qualitative, consisting primarily of environmental scanning electron microscopy (ESEM), scanning electron microscopy (SEM) and transmissive electron microscopy (TEM) micrographs and energy-dispersive spectroscopy (EDS) spectra.

## 2. TEST PROCEDURES

The functional description and physical attributes of the ICET apparatus were presented in detail in the ICET Test #1 report (Ref. 2). The experimental apparatus is briefly described below, followed by a review of the test operation and analytical techniques used to evaluate the test results.

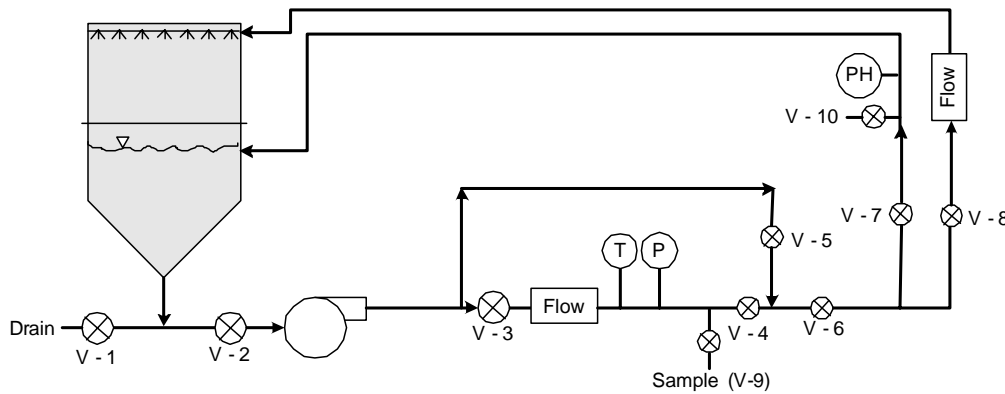
### 2.1. Chemical Test Apparatus Functional Description

The test apparatus was designed to meet the functional requirements of the Project Test Plan (Ref. 1). Functional aspects of the test apparatus are as follows:

1. The central component of the system is a test tank. The test apparatus was designed to prevent solids from settling in the test piping.
2. The test tank can maintain both a liquid and vapor environment, as would be expected in post-LOCA containment.
3. The test loop controls the liquid temperature at 140°F ( $\pm 5^\circ\text{F}$ ).
4. The system circulates water at flow rates that simulate spray flow rates per unit area of containment cross section.
5. The test tank provides for water flow over submerged test coupons that is representative of containment pool fluid velocities expected at plants.
6. Piping and related isolation valves are provided such that a section of piping can be isolated without interrupting the test.
7. The pump discharge line is split in two, one branch directing the spray header in the tank's vapor space and the other returning to the liquid side of the tank. Each branch is provided with an isolation valve, and the spray line includes a flow meter.
8. The recirculation piping includes a flow meter.
9. The pump circulation flow rate is controlled at the pump discharge to be within  $\pm 5\%$  of the flow required to simulate fluid velocities in the tank. Flow is controlled manually.
10. The tank accommodates a rack of immersed sample coupons, including the potential reaction constituents identified in the test plan.
11. The tank also accommodates six racks of sample coupons that are exposed to a spray of liquid that simulates the chemistry of a containment spray system. Provision is made for these racks to be visually inspected.

12. The coupon racks provide sufficient space between the test coupons to preclude galvanic interactions among the coupons. The different metallic test coupons are also electrically isolated from each other and the test stand to prevent galvanic effects resulting from metal-to-metal contact between specimens or between the test tank and the specimens.
13. The fluid volumes and sample surface areas are based on scaling considerations that relate the test conditions to actual plant conditions.
14. All components of the test loop are made of corrosion-resistant material (for example, SS for metallic components).

The as-built test loop consists of a test tank, a recirculation pump, 2 flow meters, 10 isolation valves, and pipes that connect the major components, as shown schematically in Figure 2-1. P, T, and pH represent pressure, temperature, and pH probes, respectively.



**Figure 2-1. Test loop process flow diagram.**

## 2.2. Pre-Test Preparation

### 2.2.1. Test Loop Cleaning

In preparation for Test #3, the experiment test loop was thoroughly cleaned to remove all Test #2 deposits and residues. In addition to visual inspections, the test apparatus was flushed and cleaned per the written direction given in the pre-test operations project instruction (PI) (Ref. 3). The system was flushed with ammonium hydroxide followed by ethanol until it was visually clean and the water conductivity was  $<50 \mu\text{S}/\text{cm}$ .

### 2.2.2. Test Coupons and Samples

Each ICET experiment exposes metallic and concrete coupons to anticipated post-LOCA environments. Each coupon is approximately 12 in. square. The metallic coupons are approximately 1/16 in. thick, except for the inorganic-coated steel coupons, which are approximately 3/32 in. thick. The concrete coupons (one per test) are approximately



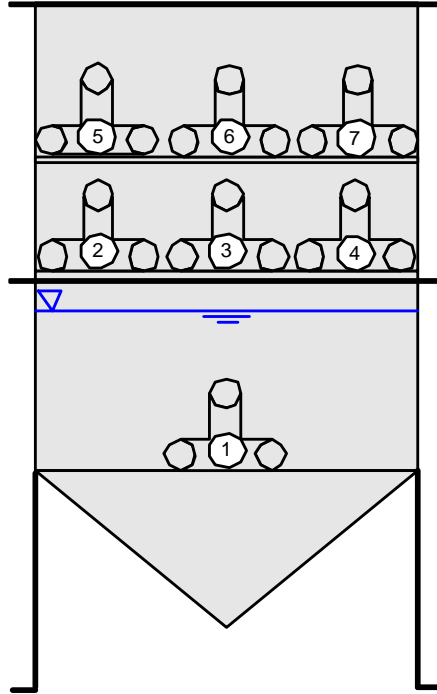
1-1/2 in. thick. Insulation materials are also exposed. For Test #3, fiberglass insulation samples and calcium silicate samples were included in the test. As with Tests #1 and #2, Test #3 subjected seven racks of coupon to the specified environment, with one being submerged in the test tank and the remaining six being held in the tank's gas/vapor space. The Test #3 coupons of each type were as shown in Table 2-1.

**Table 2-1. Quantity of Each Coupon Type in Test #3**

<b>Material</b>	<b>No. of Coupons</b>
Coated Steel (CS)	77
Aluminum (Al)	59
Galvanized Steel (GS)	134
Copper (Cu)	100
Uncoated Steel (US)	3
Concrete	1

Note: Inorganic zinc (IOZ) coated steel and CS are the same coupon type.

The arrangement of the coupon racks in the test tank is schematically illustrated in Figure 2-2. The figure shows a side view of the ICET tank, with the ends of the seven CPVC racks illustrated. The normal water level is shown with the blue line in the figure. Rack #1 is the only submerged rack, and it sits on angle iron. It is centered in the tank, so that flow from the two headers reaches it equally. Racks #2–#4 are positioned above the water line, supported by angle iron in the tank. Racks #5–#7 are positioned at a higher level, also supported by angle iron. Racks #2–#7 are exposed to spray. In the figure, north is to the right and south is to the left. Directions are used only to identify such items as rack locations and sediment locations.



**Figure 2-2. Coupon rack configuration in the ICET tank. The blue line represents the surface of the test solution.**

Figure 2-3 shows the configuration of an unsubmerged coupon rack loaded with metal coupons in the ICET tank. The loading pattern of the racks was nearly identical, varying by only one or two coupons. Shown in the figure from left to right, the coupons are arranged as follows: 4 Cu, 4 Al, 4 inorganic zinc (IOZ), 7 GS, 4 Cu, 3 Al, 4 IOZ, 7 GS, 4 Cu, 3 Al, 4 IOZ, and 7 GS.



**Figure 2-3. A loaded coupon rack in the ICET tank.**

Several fiberglass samples were placed in the ICET tank. Samples were either submerged or held above the water level. The unsubmerged fiberglass samples were positioned so they would be exposed to sprays. The fiberglass samples were contained in SS wire mesh that allowed water flow while confining the fiberglass material. Both loosely-packed and more tightly-packed samples were used. In addition, some submerged fiberglass samples were located where they would be exposed to relatively high-flow conditions, and others were located in quiescent regions of the tank. Figure 2-4 shows the so-called “sacrificial” fiberglass samples in wire mesh pouches attached to the submerged coupon rack (rack 1 in Figure 2-2). Each pouch contains approximately 5 g of fiberglass. Those samples were attached with SS wire and removed from the tank on Days 4, 15, and 30, and examined. As shown in the figure, bigger insulation bags were wrapped around the sacrificial specimens during the test. See Subsection 2.4.1.1 for descriptions of other fiberglass samples.



**Figure 2-4. Fiberglass samples attached to coupon rack.**

Several cal-sil insulation samples were also placed in the ICET tank. Samples were either submerged or held above the water level. The unsubmerged cal-sil samples were positioned so they would be exposed to sprays. As with the fiberglass samples, all of the cal-sil samples (except for the dust) were contained in SS wire mesh that allowed water flow while confining the cal-sil material. In addition to solid pieces of cal-sil, cal-sil dust (approximately 43.5 lb) was placed in the tank solution before the test began. Both types of samples are shown in Figures 2-5 and 2-6.



**Figure 2-5. Solid pieces of cal-sil samples in SS mesh.**



**Figure 2-6. Cal-sil dust.**

### **2.2.3. Quality Assurance (QA) Program**

A project QA manual was developed to satisfy the contractual requirements that apply to the ICET project. Specifically, those requirements were to provide credible results by maintaining an appropriate level of QA in the areas of test loop design, sampling, chemicals, operation, and analysis. These requirements were summarized in the contract requirement that QA was to be consistent with the intent of the appropriate sections of 10CFR50, Appendix B.

The 18 criteria of 10CFR50, Appendix B, were addressed separately in the QA manual, and the extents to which they apply to the ICET project were delineated. A resultant set of QA procedures was developed. In addition, project-specific instructions were written to address specific operational topics that required detailed step-by-step guidance. PIs generally applicable to all tests were written for the following topics and were followed for Test #3:

- Data Acquisition System (DAS)
- Coupon Receipt, Preparation, Inspection, and Storage
- DAS Alarm Response
- Chemical Sampling and Analysis
- TEM Examination of Test Samples
- SEM Characterization of Test Samples
- Viscosity Measurements

Project instructions specific to Test #3 were written for the following:

- Pre-Test Operations
- Test Operations, Test #3 (cal-sil and fiberglass, with TSP)
- Post-Test Operations

The pre-test, test, and post-test operations PIs that were used in Test #3 are included in Appendix L.

### **2.2.4. Test Parameters**

ICET test parameters were selected based on literature surveys and the results of surveys of United States nuclear power plants. Quantities of test materials were selected to preserve the scaling of representative ratios between material surface areas and total cooling-water volumes. Chemical additives also simulate the post-LOCA sump environment. The Project Test Plan (Ref. 1) is the basis for the following information in this section.

The materials included in the tests are zinc, aluminum, copper, carbon steel, concrete, and insulation materials such as fiberglass and calcium silicate. The amounts of each material are given in Table 2-2 in the form of ratios (material surface area to water volume), with three exceptions: concrete dust, which is presented as a ratio of mass to water volume, and fiberglass and calcium silicate, which are presented as a ratio of insulation volume to water volume. Also shown in the table are the percentages of material that are submerged and unsubmerged in the test chamber.

**Table 2-2. Material Quantity/Sump Water Volume Ratios for the ICET Tests**

Material	Value of Ratio for the Test (ratio units)	Percentage of Material Submerged (%)	Percentage of Material Unsubmerged (%)
Zinc in Galvanized Steel	8.0 (ft <sup>2</sup> /ft <sup>3</sup> )	5	95
Inorganic Zinc Primer Coating (non-top coated)	4.6 (ft <sup>2</sup> /ft <sup>3</sup> )	4	96
Inorganic Zinc Primer Coating (top coated)	0.0 (ft <sup>2</sup> /ft <sup>3</sup> )	–	–
Aluminum	3.5 (ft <sup>2</sup> /ft <sup>3</sup> )	5	95
Copper (including Cu-Ni alloys)	6.0 (ft <sup>2</sup> /ft <sup>3</sup> )	25	75
Carbon Steel	0.15 (ft <sup>2</sup> /ft <sup>3</sup> )	34	66
Concrete (surface)	0.045 (ft <sup>2</sup> /ft <sup>3</sup> )	34	66
Concrete (particulate)	0.0014 (lbm/ft <sup>3</sup> )	100	0
Insulation Material (fiberglass or calcium silicate)	0.137 (ft <sup>3</sup> /ft <sup>3</sup> )	75	25

The physical and chemical parameters which are critical for defining the tank environment and which have a significant effect on sump-flow blockage potential and gel formation, have been identified in Ref. 1. These physical and chemical parameters are summarized as follows:

**Physical Parameters**

• Water volume in the test tank	949 L	250 gal.
• Circulation flow	0–200 L/min	0–50 gpm
• Spray flow	0–20 L/min	0–5 gpm
• Sump temperature	60°C	140°F

## Chemistry Parameters

• $\text{H}_3\text{BO}_3$ concentration	2800 mg/L as boron
• $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ concentration	As required to reach pH 7 in the simulated sump fluid
• NaOH concentration	As required to reach pH 10 in the simulated sump fluid
• HCl concentration	100 mg/L
• LiOH concentration	0.7 mg/L as Li

Specific parameters planned for each ICET test run were described in Table 1-1.

## 2.3. Test Operation

### 2.3.1. Description

Preparation of ICET Test #3 (Run 3 in Table 1-1) began with 240 gal. of reverse osmosis (RO) water being heated to 65°C. (Adding the metal coupons and insulation samples reduces the water temperature by approximately 5°C, so the water was heated initially to 65°C.) With 25 gpm circulating through the loop, the predetermined quantities of boric acid (14.54 kg) and LiOH (0.66 g) were added and dissolved in the ICET tank solution. After the chemicals were added and observed to be well mixed, a baseline grab sample and measurements of the test solution were taken. Then the premeasured latent debris and concrete dust were added to the tank solution. After the solution circulated for 10 minutes, the pump was stopped and the coupon racks and insulation samples were put into the tank (see Section 2.2.2).

The test commenced with initiation of the tank sprays. The sprays lasted for 4 hours, and they resulted in 3.5 gpm of solution being sprayed on the unsubmerged coupons, through the 4 flow nozzles. Beginning at 30 minutes into the test, Batch 1 of TSP ( $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ ) solution was metered into the recirculation line. That batch consisted of 300 g of boric acid and 1893 g of TSP in 5 gal. of RO water. When that batch had been injected, after approximately 90 minutes, injection of Batch 2 began. Batch 2 consisted of 300 g of boric acid, 1893 g of TSP, and 211 mL of 12.29 N HCl in 5 gal. of RO water. That batch was injected over the remainder of the spray cycle. The sprays were terminated after 4 hours, and the test continued uninterrupted for the next 30 days.

The experiment commenced at 3:30 p.m. on Tuesday, April 5, 2005, and it ended on May 5, 2005. During the test, grab samples were taken daily for wet chemistry and inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analyses. Water loss due to water sample removals and evaporation was made up with RO water. Water samples, insulation, and metal coupons were analyzed after the test. Sampling and analyses were conducted in accordance with approved project instructions (Refs. 3, 4, and 5).



### **2.3.2. Process Control**

During the test, critical process control parameters were monitored to ensure that the test conditions met the functional test requirements. Recirculation flow rate and temperature were controlled throughout the test. The solution pH was initially targeted to reach the prescribed value of 7 after the spray phase ended. The pre-determined amounts of chemicals were added, and pH was not controlled.

Recirculation flow in the test loop was controlled by adjusting the pump speed. Fine tuning was achieved by manually adjusting a valve located downstream of the recirculation pump. In-line flow meters were used to measure the flow rate in the recirculation line and the spray line.

Titanium-jacketed immersion electric heaters controlled the water temperature. The heaters were thermostatically controlled to automatically maintain the desired temperature.

## **2.4. Analytical Methods**

Data collected during Test #3 included the in-line measurements of temperature, pH, and loop flow rate. During the daily water grab sample analysis, bench-top measurements were obtained for temperature, pH, turbidity, total suspended solids (TSS), and kinematic viscosity. The concentration of hydrogen in the tank atmosphere was also measured and could be used as an indicator of chemical reactions taking place. Water, fiberglass, and metal coupon samples were taken to other laboratory locations for additional analyses. These analyses included strain-rate viscosity, ESEM, SEM, EDS, TEM, ICP-AES, x-ray fluorescence (XRF), and x-ray diffraction (XRD). EDS provided a semi-quantitative elemental analysis after calibration of the instrument's x-ray signal using an internal element standard. Descriptions of the principles of operation and limitations of these analytical methods were provided in the Test #1 report (Ref. 2).

### **2.4.1. Data Compilation and Nomenclature**

This section provides a brief guide to assist the reader in interpreting the ICET Test #3 information and data presented in the following sections and in the appendices. Standardized nomenclature is defined first to clarify the origin of samples that are described in the data sets. The appendices are listed, and a description is provided of how they were compiled.

#### **2.4.1.1. Nomenclature**

Many spatially unique but physically similar sample types were collected in ICET Test #3. To ensure that consistent interpretations and comparisons of data sets are made, it is imperative that a standardized nomenclature be adopted when referring to each sample type. Many different qualitative descriptions of these samples might be equally suitable, but different adjectives convey different connotations to each observer.

Therefore, the following definitions establish the convention used in this report when making generic references to sample type.

White Precipitate	The behavior of test solution at temperature and upon cooling is observed during testing. Precipitates and their prominence indicate chemical interactions occurring in the solution. White precipitate formed in Test #1 (T1) water solution samples drawn from the test loop. Upon cooling below the test temperature, T1 daily water samples extracted from the tank formed a visible white material that is referred to as a precipitate. This precipitate was absent in Test #3 (T3) water samples. One probable cause of the differences is the pH levels of the test solutions and their effects on the aluminum samples. T1 had a high pH of about 9.5, whereas the pH for T3 was about 8.0. Aluminum solubility is highly dependent on pH levels. There may be other causes that effect the corrosion of aluminum and cause precipitates to form.
Latent Debris	Commercial power plant containments gradually accumulate dust, dirt, and fibrous lint that are generically referred to as latent debris. This classification distinguishes resident material from debris generated during an accident scenario. At the beginning of T3, measured quantities of crushed concrete and soil were added to simulate the latent debris present in containment. These materials were examined via SEM/EDS to establish a baseline composition for comparison with sediment samples (see “Sediment” below).
Sediment	Surrogate latent debris particulates and fugitive fiberglass fragments that were initially suspended in water at the beginning of T3 gradually settled to the bottom of the tank. In addition, a large amount of cal-sil dust was added to the tank solution. Most of that dust ended up on the tank bottom. A deep layer of sediment formed, which was 8-in. deep in some places. During the course of the test, additional material may have been deposited in this layer. At the conclusion of the test, the sediment layer was recovered as completely as possible.
White Residue	At the conclusion of T3, all water was drained slowly from the tank. Exposed metal surfaces that cooled rapidly collected a thin deposit of white residue or scale. Some of this material was scraped from internal piping surfaces and tank walls for comparison with other sample types. A similar residue was observed after Tests #1 and #2.
Powder	At the conclusion of T3, fine particulate deposits were found on the submerged CPVC coupon rack. They were referred to as powder and examined by SEM/EDS.

Fiberglass	<p>One of the principal debris types introduced to T3 was shredded fiberglass insulation. This debris was bundled in 3-in.-thick bags (or blankets) of fiberglass confined in SS mesh to prevent ingestion through the pump and to better control the placement of debris in various flow regimes. Fiberglass samples are designated by their placement in high-flow and low-flow areas of the tank. Additional 4-in.-square envelopes of fiberglass were also prepared for extraction during the course of the test. These samples are referred to as “sacrificial” samples. One sample, called the “birdcage,” was constructed so that the fiberglass within was loose and not compacted. The birdcage fiberglass sat on the tank bottom and was removed on Day 30. Some amount of fiber, especially short-fiber fragments, escaped the mesh bags and was deposited in other locations within the tank. This material is referred to as “fugitive” fiberglass.</p>
Cal-sil	<p>The insulation samples used in T3 were 80% cal-sil. That amount was divided into 4 size categories, in pieces that were roughly cubes. Of the total cal-sil, 14% was over 3 in., 19% was 1–3 in., 5% was less than 1 in., and 62% was “dust.” The dust consisted of cal-sil pieces that were ground into a fine powder. With the exception of the dust, the cal-sil pieces were contained within SS mesh and apportioned into submerged and unsubmerged samples. The dust was put into the tank solution before the test started, and it became the primary constituent of the sediment.</p>
Drain Screen	<p>A 12-in.-tall screen made of coarse SS mesh (1/8-in. holes) wrapped into a 2-in.-diam cylinder was inserted into the outlet drain at the bottom of the tank to protect the pump from ingesting large debris items. Two inches of the screen were inserted into the tank outlet to provide a solid base and stability. A 6-in.-tall drain collar was installed around the drain screen. This drain collar was a cylinder of fiberglass held in SS mesh. The drain collar was exposed to higher-velocity water flow than other samples in the tank. The drain collar fiberglass was examined as a separate debris location to identify any apparent differences with other sample locations.</p>
Gelatinous Material	<p>This term generically refers to any observed sample constituent with amorphous, hydrated, or noncrystalline physical characteristics. When Test #3 was shut down, deposits of pinkish-white gel-like precipitates were observed on the top of the birdcage and on other objects on the tank’s bottom.</p>

Water Sample	Daily water samples were extracted from the ICET tank for elemental concentration analyses. After the sample line was properly flushed, some of this water was extracted directly from the tap. An equal amount of water was also generally collected through a micropore filter. Thus, daily water samples were designated as filtered (F) and unfiltered (U), and a corresponding filter paper exists in the sample archive for each daily sample that was collected.
High-Volume Filter	If white precipitates are observed in the tests, larger quantities of test solution are periodically extracted for filtration to determine whether suspended chemical products are present in the test liquid under in situ conditions. The intent of this exercise is to maintain the liquid temperature while forcing the liquid through a micropore filter under vacuum. Because the precipitates were not present in T3, these high-volume filter samples were not obtained.
Filter Paper	Many different samples of tank solution were fractionated by micropore filtration into a liquid supernate and a solid filtrate that existed at the time and temperature conditions of the filtering process. These samples included (1) daily water samples filtered during extraction, (2) daily water samples filtered after cooling to room temperature, and (3) high-volume water samples.
Chemical Deposits	Sacrificial fiberglass samples that were extracted at Day 4, Day 15, and Day 30 showed evidence of chemical products forming on and between fiber strands. These products are referred to as “deposits,” although the exact physical mechanism of formation is not well understood. The physical appearance suggests growth, agglomeration, or crystallization on and around the fiber strands over time rather than capture or impaction of particles from the bulk solution. This observation is supported by the fact that the small sacrificial fiberglass samples were located in a region of lower-velocity water flow (i.e., in the interior of larger blankets).
Concrete Sample	Several chips of concrete (1/4–3/4 in. diam) were broken from the primary slab of submerged concrete and introduced to the tank in a small SS envelope at the start of the test. Examinations of these chips were conducted to determine if concrete surfaces provide a preferential site for gel formation.

Although these terms have been defined, the reader may note minor inconsistencies in the caption labels used in this document. The caption labels use the same descriptions that were applied in laboratory notebooks to improve traceability of the data.

### 2.4.1.2. Usage

The twelve appendices listed below are provided to present data collected for the sample types and analysis methods listed below. In addition, an appendix is provided with pertinent Test #3 project instructions.

- A SEM and ESEM/EDS Data for Test #3, Day-4 Fiberglass in Low-Flow Zone
- B ESEM and SEM Day-15 Fiberglass
- C ESEM and SEM Day-30 Fiberglass
- D ESEM and SEM/EDS Data for Test #3, Day-30 Corrosion Products
- E SEM Day-30 Coupons
- F SEM/EDS Data for Test #3, Day-30 Flow Meter
- G SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Gel
- H SEM/EDS and ESEM/EDS Data for Test #3, Day-30 Cal-sil
- I ESEM/EDS Data for Test #3, Day-30 Sediment
- J TEM Data for Test #3 Solution Samples
- K UV Absorbance Spectrum—Day-30 Solution Samples
- L ICET Test #3: Pre-Test, Test, and Post-Test Project Instructions

These data are largely qualitative in nature, consisting primarily of ESEM, SEM, TEM micrographs, and EDS spectra. Each appendix represents a separate session of laboratory work that can be traced to a batch of samples that were processed in chronological order. This organizational scheme preserves the connection with laboratory notebooks and timelines that naturally developed during operation; however, in a few cases, results for a given sample type may be mixed across two or more appendices because of the order in which the individual samples were analyzed.

ESEM analyses were added to the ICET diagnostic suite for the first time during Test #2 as a means of examining hydrated chemical products. This equipment operates as an electron microscope, but it does not require a high-vacuum condition in the sample chamber. Thus, a sample need only be thoroughly drained of free water content before examination rather than fully desiccated, making the ESEM ideal for examinations of biological and environmental specimens. The complementary EDS capability that is often found with equipment of this type is not presently functional at UNM, so duplicate examinations are often performed on the same ICET sample using ESEM to obtain images of hydrated structural details and SEM/EDS to obtain representative elemental compositions. Throughout the report, ESEM analyses are also indicated by the descriptions of “hydrated” and “low-vacuum” findings.

Transcriptions of the logbooks are provided for each appendix to better document commonalities that existed among the samples at the time of analysis. Interpretation and understanding of the images and their accompanying EDS spectra will be greatly improved by frequent reference to the logbook sample descriptions and sequences. Typically, a relatively large quantity of a test sample was delivered for SEM or TEM

analysis, and then several small sub-samples of each item were examined. Note that each sub-sample was assigned a sequential reference number during the laboratory session. These reference numbers have been cited in the figure captions whenever possible to preserve the connection between the micrographs and the notebook descriptions. Electronic file names have also been stamped on the images to permit retrieval of the original data files that are archived elsewhere. Individual data sets for a given sample item have been collated into a typical sequence of (1) visual image, (2) EDS spectra, and (3) semi-quantitative mass composition.

For most of the EDS spectra, semi-quantitative mass compositions are also presented. These results are obtained from a commercial algorithm that decomposes the spectra into the separate contributions of each element. Several caveats should be considered when interpreting the numeric compositions thus obtained; however, despite these caveats, semi-quantitative EDS analysis offers a natural complement to micrographic examination as a survey technique for identifying trends in composition.

1. The spectral deconvolution algorithm is based on a library of unique signatures of each element that were obtained for pure samples using a standard beam setting that may not identically match the conditions applied for the test item.
2. The operator must select a limited number of elements to be used in the proportional mass balance. These candidates are chosen from among the peaks that are observed in the spectrum; however, the composition percentages can vary, depending on which elements are included in the list. In a few cases, two or more alternative compositions have been generated by selecting a different set of elements from the same spectrum to illustrate the sensitivity of this technique to operator input.
3. The spectral unfolding algorithm is a statistical technique having a precision that depends on the relative quality of the data in each peak. Compositions with high  $R^2$  correlation coefficients and total-mass normalization factors closer to unity represent the more-reliable estimates. The precision obtained in the fit depends on the duration of the scan and the number of counts received in each energy bin.
4. All sub-samples examined in the SEM microprobe facility are coated with a thin layer of either carbon or gold/palladium alloy to prevent the sub-samples from accumulating a charge from the impinging electron beam. Spectral peaks visible for gold (Au) and palladium (Pd) are not indigenous to the samples.
5. The EDS spectral analysis software contains a peak-recognition algorithm and an automated cursor that scans across the spectrum to locate each peak. An accompanying library of elemental energy signatures is also provided to suggest what constituents might be contributing to a given energy bin, but the operator must judge what label to assign to the spectral image. It is possible that some peaks near closely neighboring elements have been mislabeled in these images. However, every effort was made to choose from candidate elements that were most likely to be present in the test material. In a few cases, the spectral peaks were not labeled by the SEM operator. These spectra should be viewed as

corroborating evidence for similar samples that are definitively labeled. Careful comparisons of the energy scales in combination with a library of electron-scattering energies can also be used to infer the origin of the more-prominent peaks that are present in unlabeled spectra.

6. Unless an obvious spatial heterogeneity is being examined, the exact location of an EDS spectrum is not always relevant because the operator chooses arbitrary sites that are visually judged to be representative. It is not possible to sample a surface comprehensively on a microscopic basis and compute average compositions. In many cases, two or three replicate spectra are provided for this purpose, but SEM/EDS is most effective as a survey diagnostic.
7. EDS analysis is not particularly sensitive to the presence of boron for several reasons: (a) boron has a low atomic mass that does not interact well with electrons in the beam, (b) the emission lines are very close to those of carbon, and (c) the beam-port material has a high absorption cross section for these emission energies. Therefore, the correction factors used in the semi-quantitative composition analysis are quite large, as are the uncertainties in the estimated percentage of total composition for this element. There may be spectra presented in the appendices in which the lowest energy peaks are labeled as either B (boron) and/or C (carbon).

EDS locations were chosen manually at regions of specific interest. In many cases, multiple spectra were collected from a single sample and an annotated image is provided to identify the specific location. These annotated images are not generally noted in the laboratory logbook entries, but they are provided in proper sequence within the appendices.

Appendix J presents TEM data for water samples extracted from the ICET solution at Day 4, Day 15, and Day 30. The purpose of this examination was to determine whether the physical structure of any suspended products exhibits crystalline or amorphous characteristics. These data are also qualitative in nature, consisting generally of a set of high-resolution micrographs followed by companion electron diffraction images. The TEM sample holder consists of a carbon grid that is “lacey,” or filamentary, in nature. This grid is visible as a relatively large-scale structure in the background of most images. Surface tension in a droplet of liquid suspends the particulates of interest across the grid so that the electron beam can illuminate the sample through the holes without interference from a substrate. Crystalline material will exhibit diffraction patterns unique to the molecular arrangement. Amorphous material that is diffuse or disorganized in structure will not exhibit regular diffraction patterns that can be identified.

Water samples submitted for TEM analysis are not temperature controlled because the temperature cannot be maintained during the examination.

In a few cases, data file names that were noted by the operator in the laboratory log were not successfully saved in electronic form. These cases are noted in the transcribed log sheets, but the corresponding images are unavailable and therefore cannot be presented in the data sequence.

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### 3 TEST RESULTS

This section describes the results obtained from Test #3. An overview is first presented in the form of general observations. This overview is followed by more-detailed information organized by the type of samples/data collected. Data and photographs are provided here for the (1) water samples, (2) insulation (NUKON™ fiberglass samples and cal-sil materials) (3) metallic and concrete samples, (4) sediment, and (5) gel. Precipitates and corrosion products are also discussed.

#### 3.1. General Observations

Twenty minutes after Batch 1 chemicals were introduced, white flocculence was observed through the submerged observation window. The entire tank solution, as viewed through the window, exhibited a large secondary circulating flow, and the particulates appeared to be neutrally buoyant. Approximately 2-1/2 hours later, the white flocculence was smaller than before but were very fine and dense. Flow was not evident through the view window at that time.

At the 24-hour test point, a white deposit was observed on the submerged SS insulation mesh. This deposit was also observed on the submerged GS coupons. On Day 2, a very light scale was observed on the lower observation view window. Irregular vertical patterns were also observed on the lower view window. These appeared to be related to observed Day-1 bubble formation. (On Day 1, bubbles were observed to be released from the deposits on the interior of the lower view window.)

Five gal. of RO make-up water were added to the tank on Day 3. This caused some agitation of the particulates on the bottom of the tank and resulted in an increase in water cloudiness. Less than 1 day after the make-up water was added, the water clarity returned. From then on, the observed conditions of the coupons, insulation baskets, and other submerged items remained largely unchanged.

On Day 8, 5 gal. of RO water were added to the tank through the valve on the bottom of the tank. Even though the RO water was added in a fashion that should not have disturbed the settled particulates, the water became cloudy. Again, in less than 1 day's time, the water returned to normal test clarity.

On Day 9, about 3 gal. of test solution were lost when the flow meter was removed and then replaced after being cleaned. On Day 13, 5 gal. more of RO water had to be added. Again, the water was added through the bottom of the tank. This time, the particulate matter did not appear to be disturbed, and the tank solution did not appear cloudy.

The water clarity on Day 15 decreased somewhat. This also corresponded to an increase in TSS (Figure 5) but was 2 days after RO water was added.

On Day 22, 5 gal. of RO water were added to the tank through the totalizing flow meter that was attached just upstream of the valve at the bottom of the tank. Despite this fact,

water clarity was unaffected on this day and on Day 23. This trend continued through Day 27.

On Day 27, 4 gal. more of RO water were added to the tank. Agitation of the deposits on the tank bottom was not observed.

Although the conditions in the tank remained relatively steady beginning on Day 3, a few exceptions have been observed. What appeared to be dissolution of white particulate was observed from Day 7 through Day 9. The white particulate that was observed on the bottom of the tank was nearly absent on Day 9. In addition to the perturbations caused by the addition of water, that could be an indication of continuing chemical interactions.

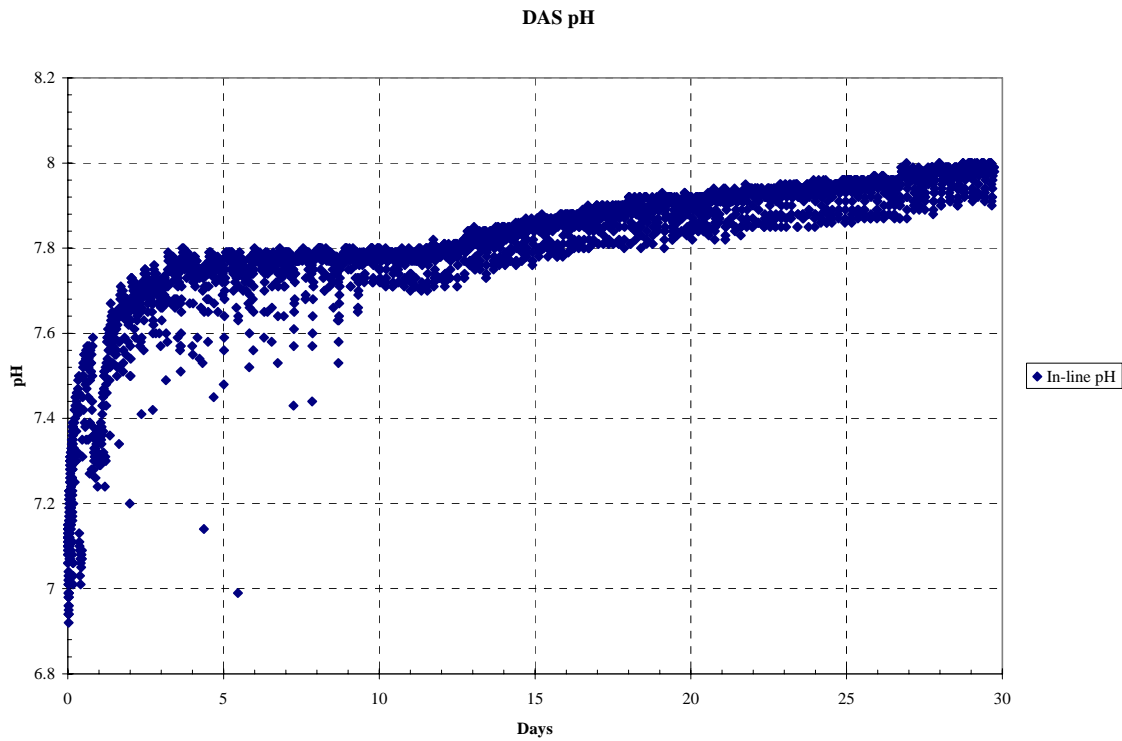
### 3.1.1. Control of Test Parameters

**Recirculation flow rate:** On Day 8 of the test, the flow meter output was lost. The problem appeared to be with the internals of the instrument. The following day, the flow meter was taken out of service and cleaned. Scale and precipitation deposits had accumulated on the turbine inside the flow meter, preventing the turbine from moving. The deposits were white and were observed to be coating the inside of the removed piping as well. They were solid, but could be removed easily with light scraping. The flow meter was flushed with RO water, which removed the scale on the turbine. After cleaning, the flow meter was re-installed and the measured flow rate was steady as before. While the flow meter was not operating, regular observations of the line pressure indicated that no flow perturbations occurred. Excluding the time that the flow meter was out of service and during the spray cycle, the average recirculation flow rate was 95.9 L/min (25.3 gpm). The recirculation flow rate had a standard deviation of 1.1 L/min, with a range of 93.3 to 99.1 L/min (24.6 to 26.2 gpm).

**Temperature:** Temperature is recorded at three submerged locations in the ICET tank. On Day 12 of Test #3, one of the three thermocouples began giving an erroneous reading. To reset the high-temperature alarm, the bad thermocouple within the DAS was replaced with a thermocouple that measured room temperature. Thus, temperature 1 on the DAS display panel became the room temperature. This temperature was factored into the average temperature displayed on the DAS panel. Neglecting the deactivated thermocouple, the average recorded temperature at the two valid locations was 60.4°C and 60.6°C (140.8°F and 141.0°F). The standard deviation in temperature recorded by the two thermocouples was within  $\pm 0.27^\circ\text{C}$  ( $\pm 0.49^\circ\text{F}$ ), with a maximum range of 58.0°C to 61.7°C (136.3°F to 143°F).

**pH:** Before time zero, 15.45 kg of boric acid and 0.663 g of LiOH were dissolved in the ICET tank. The measured bench-top pH was 4.24, which was similar to the expected value obtained from analytical predictions. Following the addition of the premixed TSP batches, the pH was 7.32. The pH then continued to rise through Day 3 of testing to 7.9. After Day 3, the pH varied in the range of 7.8–8.0.

The in-line pH probe tracked the overall bench-top pH readings, but the results have noise and a slight offset. Due to the noise within the raw data, the bench-top pH readings are more reliable. Figure 3-1 shows results from the in-line pH probe.



**Figure 3-1. In-line pH measurements.**

Four hours into the test, the in-line pH measurement was 7.37, and at Day 30 the pH measurement was 7.92. Figure 3-2 presents the bench-top pH readings. Four hours into the test, the bench-top pH measurement was 7.32, and at Day 30 the pH measurement was 8.05.

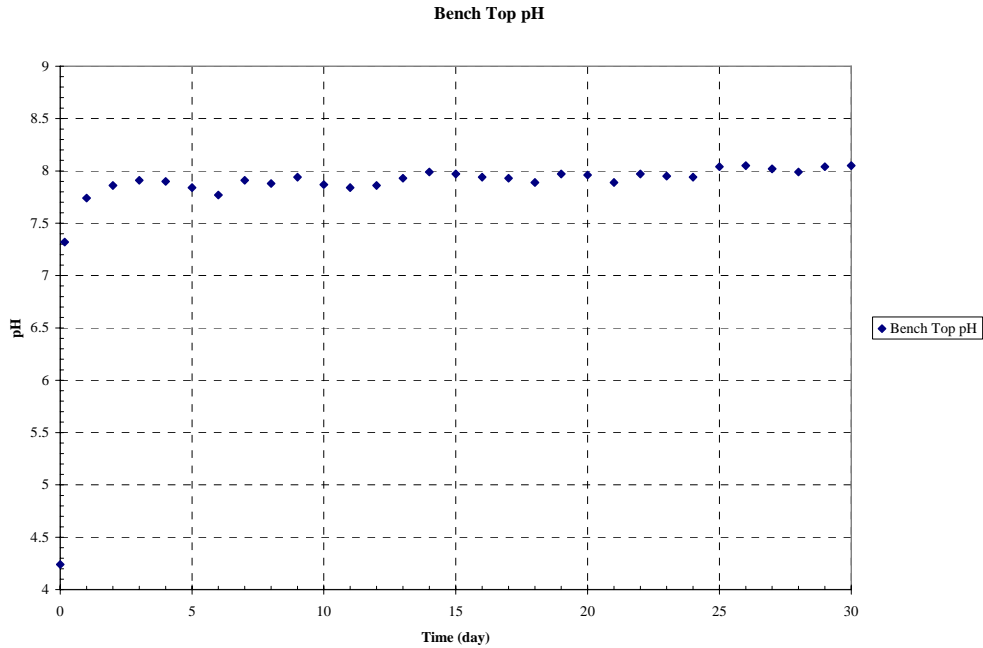


Figure 3-2. Bench-top pH meter results.

### 3.1.2. Hydrogen Generation

Hydrogen remained at or below 0.05% for Test #3. Figure 3-3 displays the evolution of hydrogen generation throughout the test. A general trend line has been superimposed upon the hydrogen generation data set. Each of the measured values is well below the hydrogen safety action threshold of 0.4%.

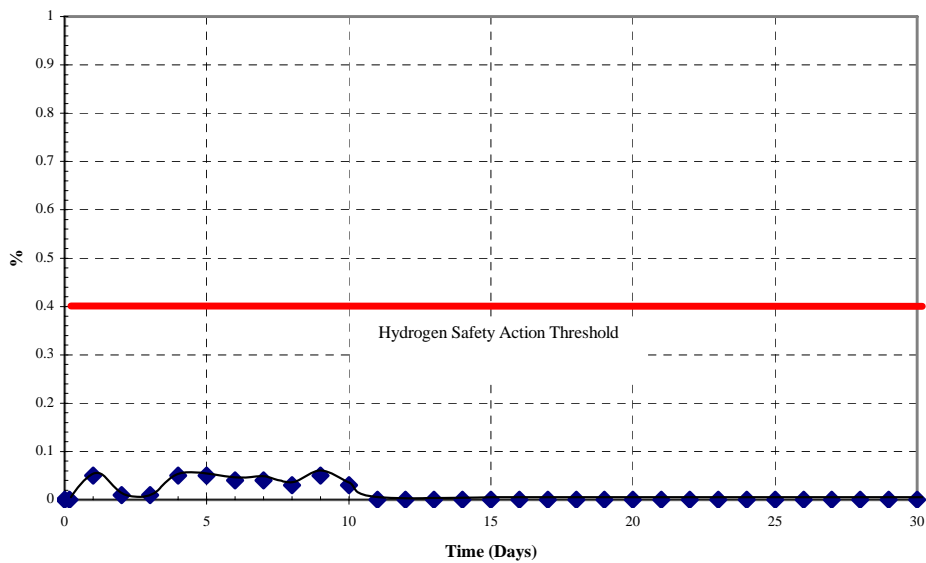


Figure 3-3. Hydrogen generation.

## 3.2. Water Samples

### 3.2.1. Wet Chemistry

Wet chemistry analyses included turbidity, TSS, and kinematic viscosity.

**Turbidity:** For the 23°C and 60°C water samples, the baseline turbidity values, which were taken after the latent debris and crushed concrete were added, were 7.3 NTU and 7.5 NTU, respectively. After the addition of cal-sil, the tank solution was observed to become much more turbid. Then with time, the turbidity decreased. Upon the addition of Batch 1 and Batch 2 chemicals, particulates were suspended in solution, and the solution was noticeably cloudier. Figure 3-4 presents the daily turbidity results.

Due to the cloudy nature of the water in the tank after the circulation pump was turned on, additional turbidity values were measured at 60°C over a 9-hour time period, in addition to the regular daily monitoring. Figure 3-4 depicts the evolution of turbidity during that time period. The x-axis on the graph represents the time in hours after the circulation pump was turned on, following cal-sil addition and while the system was heating up. As can be seen, the turbidity gradually decreased from 192 NTU at the 7-minute point to 54 NTU at the 5.9-hour point. Note that the 5.23-hour point on the graph corresponds to the time-zero test point. The spray nozzles were activated at that time. At the 6.4-hour point, the turbidity peaked above 200 NTU. The turbidity decreased to 79 NTU by the end of the spray phase. (Note that the Batch 1 addition began at approximately 5.8 hours on Figure 3-4, and Batch 2 addition began at 7.5 hours.)

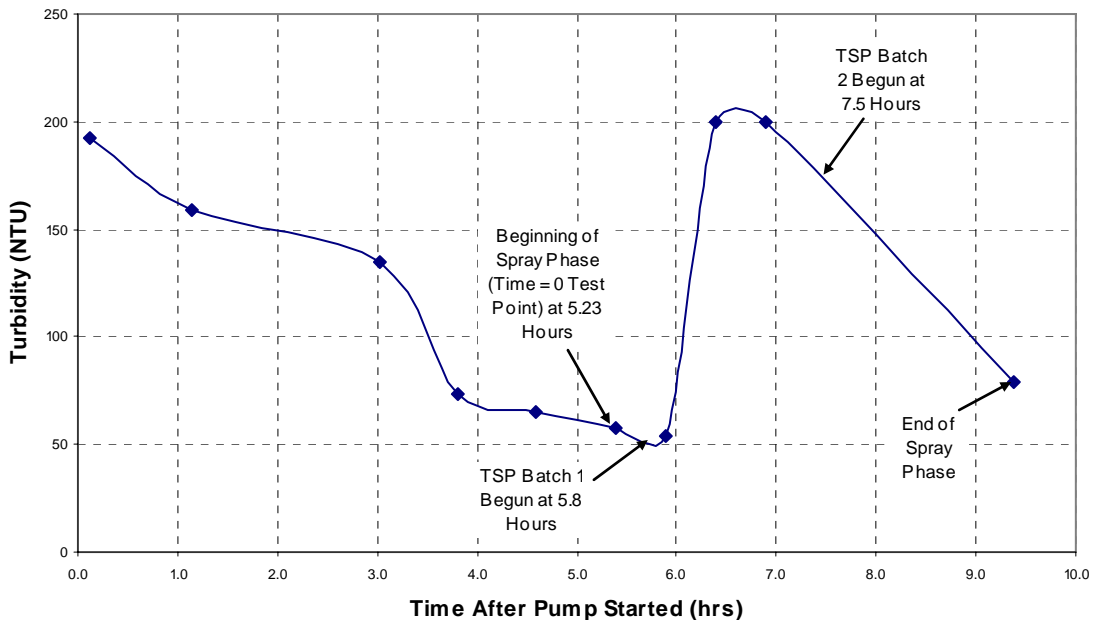
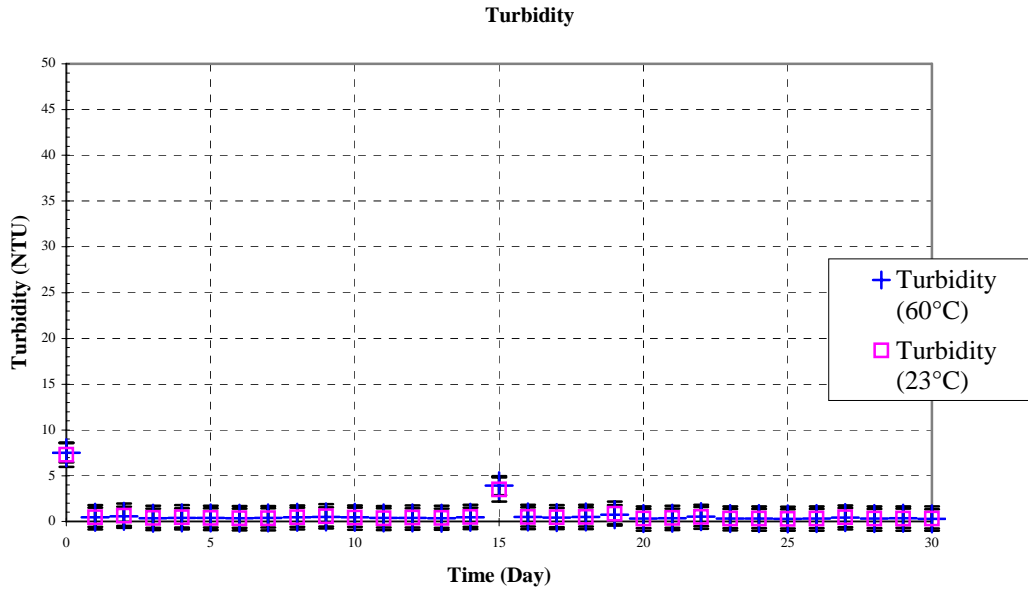


Figure 3-4. Day-1 turbidity results.

Figure 3-5 depicts the evolution of turbidity at 23°C and 60°C throughout Test #3. The trend of each curve exhibits an initial spike during the addition of Batch 1 chemicals, followed by a sharp decline to the Day-1 test point and steady behavior from then on. The turbidity values on Day 15 were slightly higher, but no direct cause has been identified. The Day-15 values were 3.9 NTU and 3.5 NTU for the 60°C and 23°C samples, respectively. Thereafter, the turbidity values decreased and remained steady.



**Figure 3-5. Daily turbidity results.**

**Total Suspended Solids:** TSS are measured by filtering a volume of approximately 500 mL through an in-line filter directly at the sample tap. The selected equipment ensures that TSS measurements are not affected by temperature-dependent or time-dependent precipitation reactions that may occur once the process solution is removed from the tank. Figure 3-6 presents Test #3 TSS data as the experiment progressed. The baseline TSS measurement was taken after the addition of latent debris but before the Batches 1 and 2 chemical additions. After the addition of the Batch 1 chemicals but before the addition of Batch 2 chemicals, a second TSS measurement (not shown on the graph) was taken, and the value was 268 mg/L. Although very high, it appeared to be a reasonable measurement because a large amount of particles was suspended in solution at that time. Four hours into the test, another TSS measurement was taken, and that value was 73.2 mg/L. At that time, fewer particles were seen suspended in solution. After 24 hours, the TSS measurement dropped to 13.9 mg/L, where it leveled out for the rest the test.

Although TSS remained fairly stable over the entirety of the test, TSS measurements from six different days deviated from the trend. The Day-6 TSS measurement was slightly higher at 20.6 mg/L, which was not unexpected because the sample was taken right after the return of excess test solution to the tank. The return of the excess test solution caused some of the particles that were sitting at the bottom of the tank to be

become resuspended in the solution, a situation that was reflected in the final measurement. The Day-15 and Days-24–27 measurements were higher than the test average of approximately 13.4 mg/L that was observed for the majority of the test. The TSS filter weights for these days were confirmed, and there is no direct explanation for these higher values.

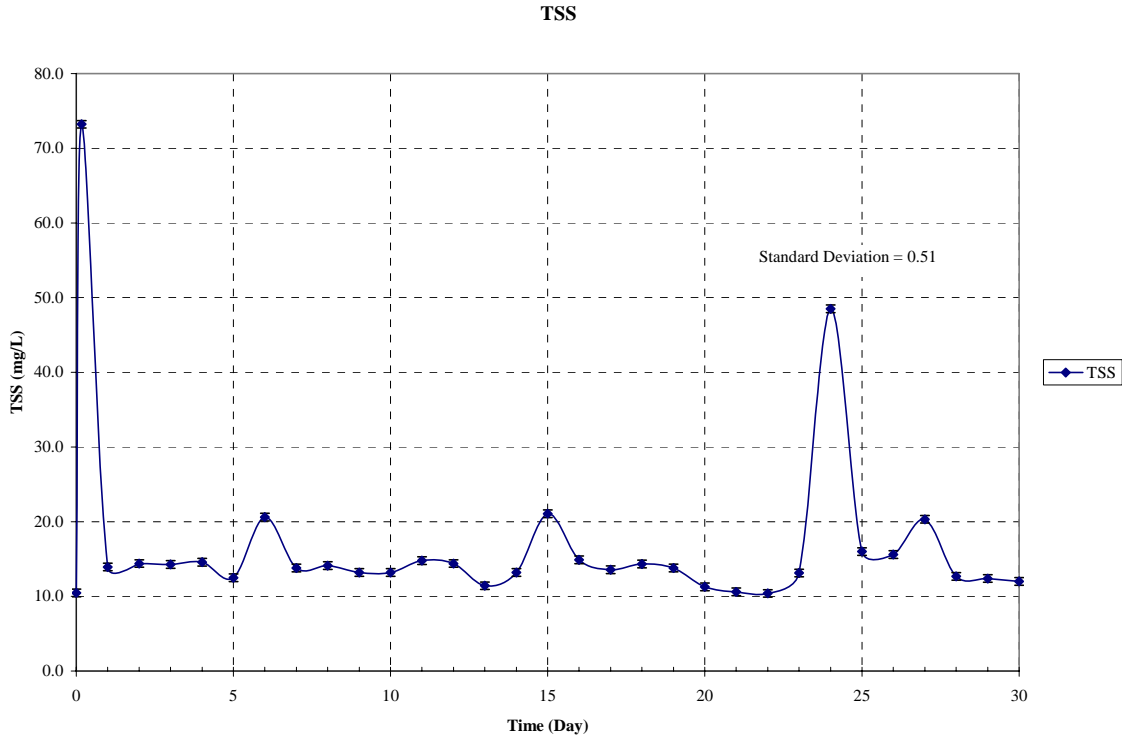


Figure 3-6. Test #3 TSS results.

**Kinematic Viscosity:** Kinematic viscosity was measured with a Cannon-Fenske capillary viscometer. Viscosity was measured on unfiltered samples, each at a temperature of 60°C ± 1.0°C (140°F ± 1.8°F) and 23°C ± 2.0°C (73.4°F ± 3.6°F). Figure 3-7 shows the viscosity results. Water’s viscosity is highly sensitive to temperature, and the allowed temperature range results in a variation of viscosity of 2.9% between 59°C (138.2°F) and 60°C (141.8°F) and 9.3% between 21°C (69.8°F) and 25°C (77.0°F). For this reason, temperature was measured to a 0.1°C accuracy with a National Institute of Standards and Technology (NIST)-traceable thermometer for all viscosity measurements. Also, the measured viscosity values were corrected to common temperatures to facilitate comparisons. The corrected temperatures were 60.0°C (140°F) and 23.0°C (73.4°F).

Viscosity remained relatively constant throughout Test #3. The viscosity at 60.0°C (140°F) remained at a value of approximately 0.49 mm<sup>2</sup>/s, and the 23.0°C (73.4°F) measurements remained around 0.95 mm<sup>2</sup>/s. These constant viscosity values are consistent with the steady water conditions in the test. Figure 3-7 shows the viscosity values through the course of the test.

Viscosity 60°C and 23°C

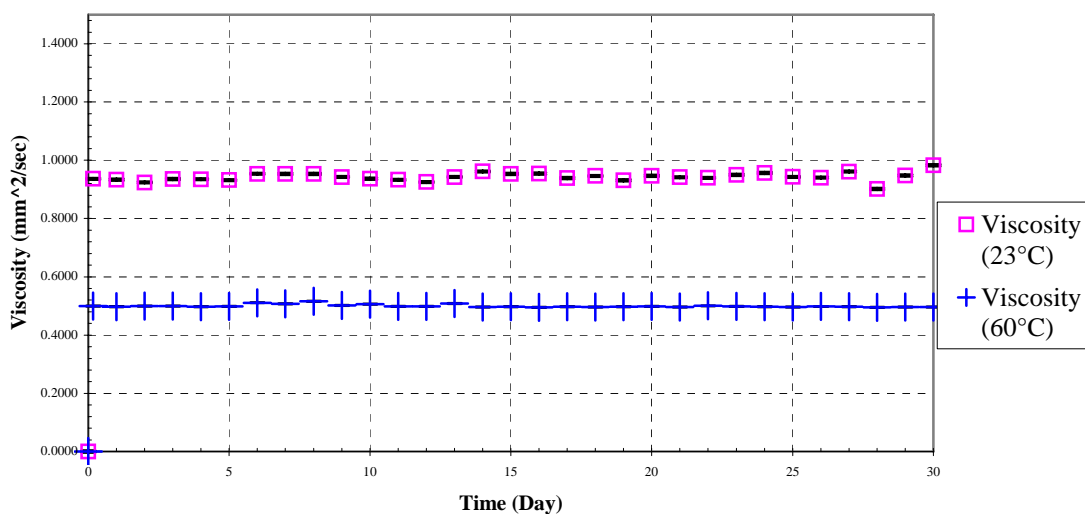


Figure 3-7. Viscosity at 60°C and 23°C.

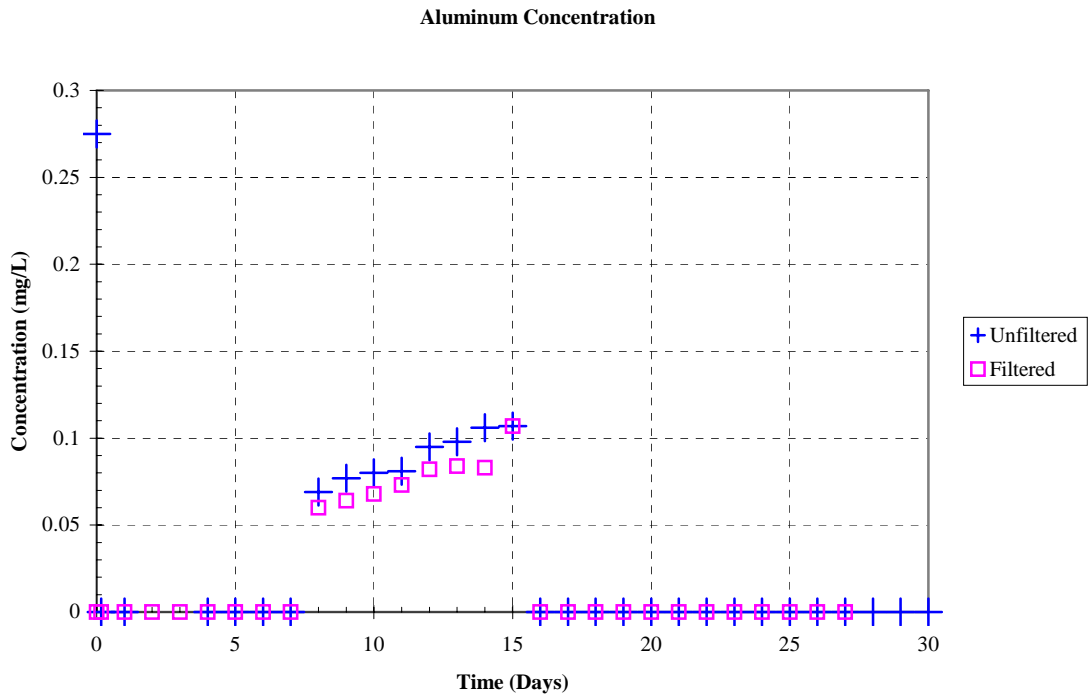
### 3.2.2. Metal Ion Concentration

ICP results for Test #3 are displayed in Figures 3-8 through 3-16, which are daily sample results. Table 3-1 contains ICP results for elements that were analyzed on Days 1, 4, 15, and 30. Table 3-1 shows that chloride, boron, lead, lithium, and potassium concentrations remained relatively constant throughout the test. An examination of the figures reveals that phosphate, copper, iron, aluminum, and zinc are present in trace amounts, below 1 mg/L. It also can be seen that calcium, silica, sodium, and magnesium are present in higher concentrations.

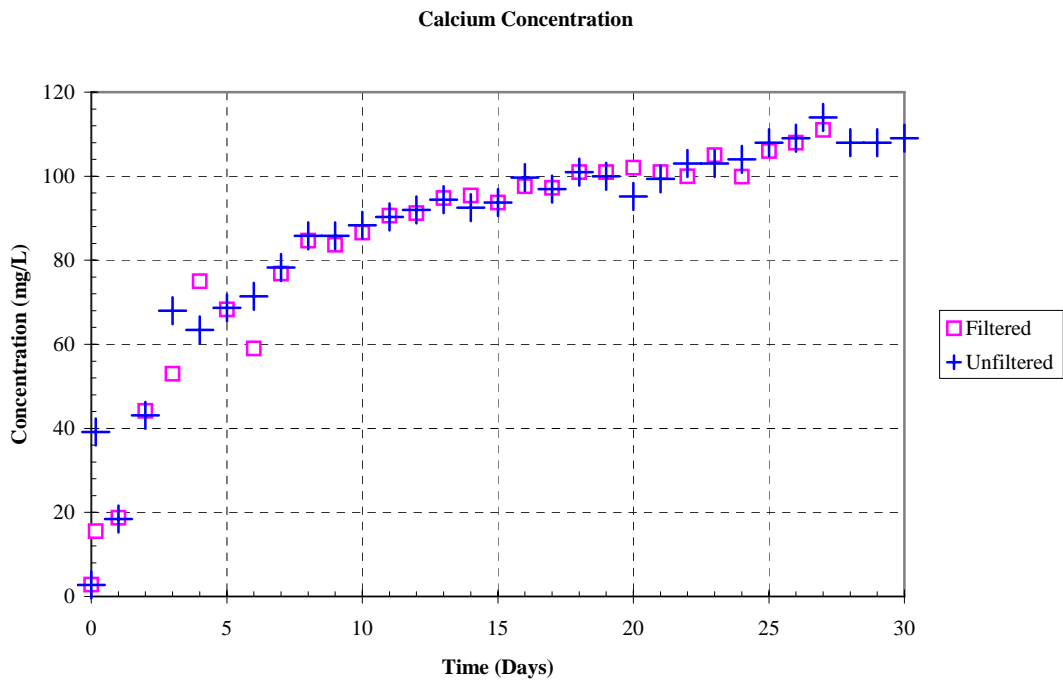
Table 3-1. ICP Results for Selected Elements

Unfiltered Samples					
Sample Time	Chloride	Boron	Lead	Lithium	Potassium
	mg/L				
Baseline	No data	2090	0.05	0.22	No data
4 Hours	111	2180	No data	0.26	17.3
Day 1	105	2160	0.04	0.250	19.8
Day 15	113	No data	0.03	0.23	27.1
Day 30	110	2550	0.03	0.18	25.7





**Figure 3-8. Aluminum concentration.**



**Figure 3-9. Calcium concentration.**

### Copper Concentration

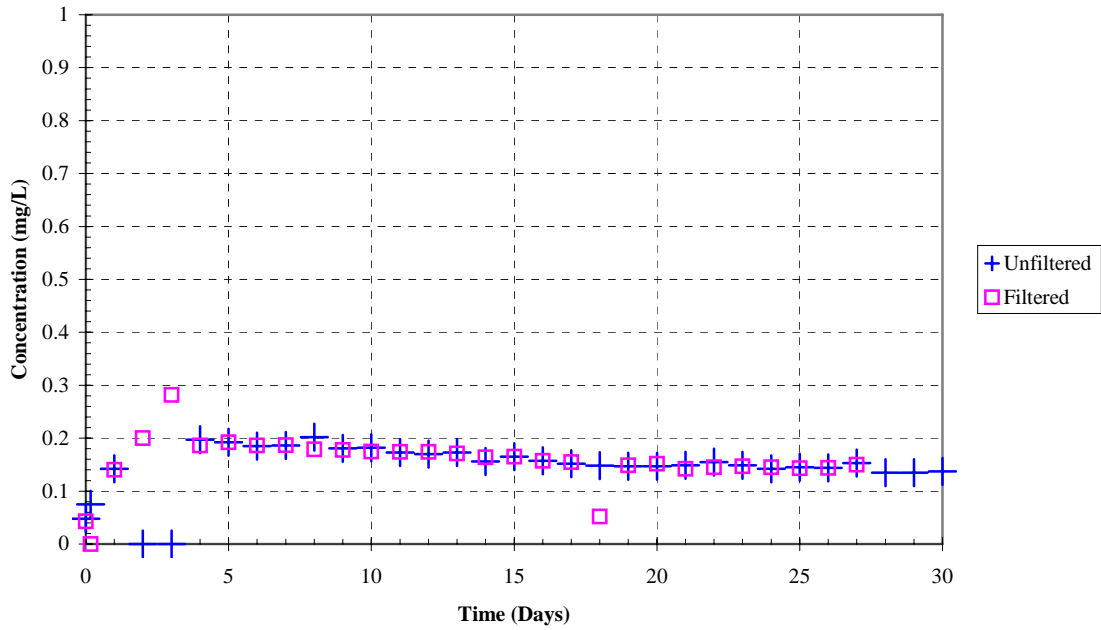


Figure 3-10. Copper concentration.

### Iron Concentration

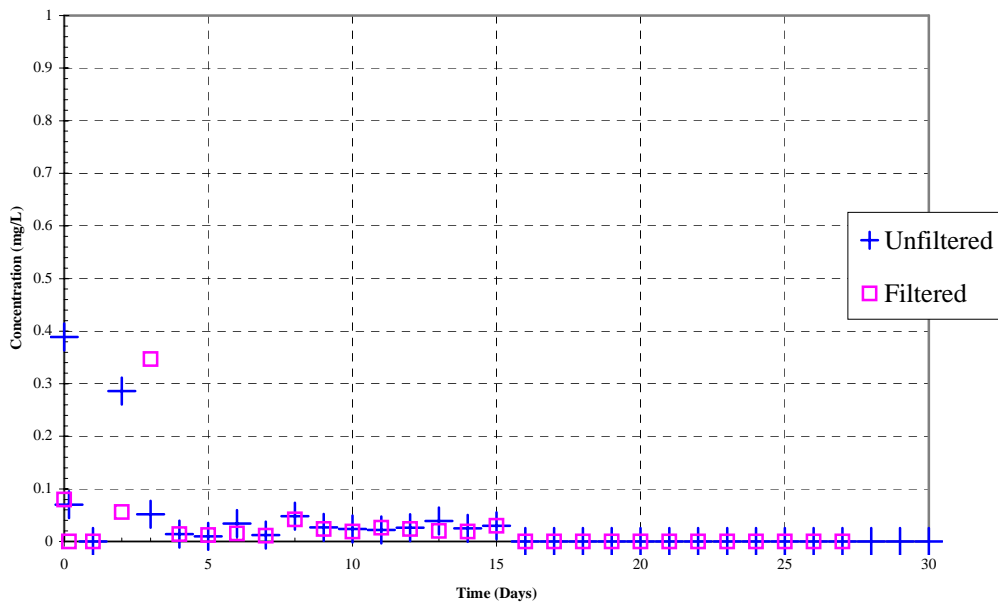
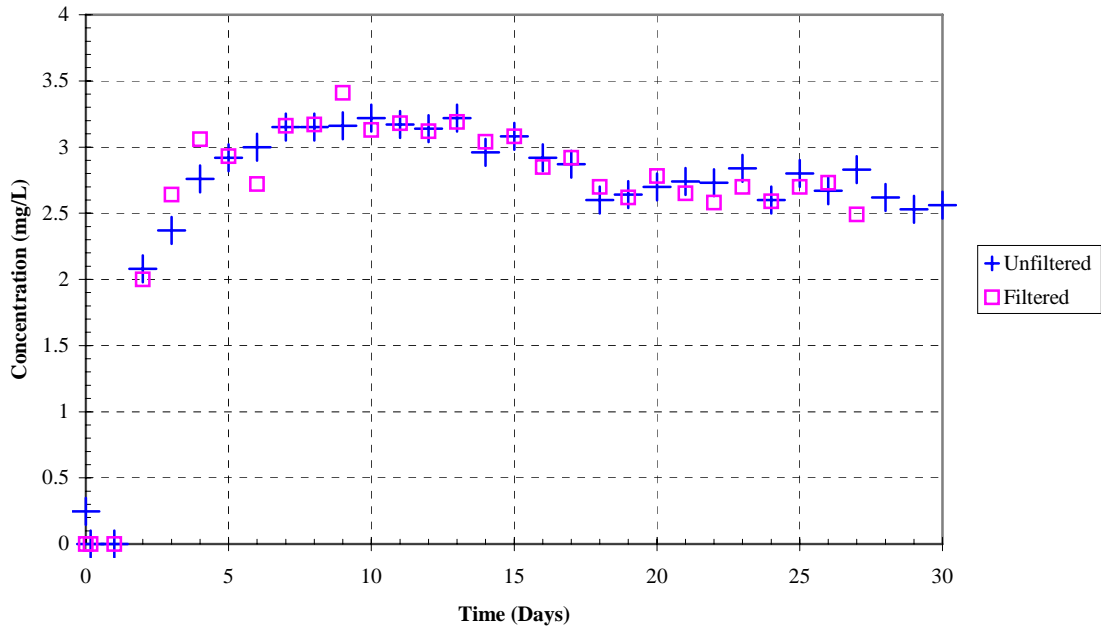


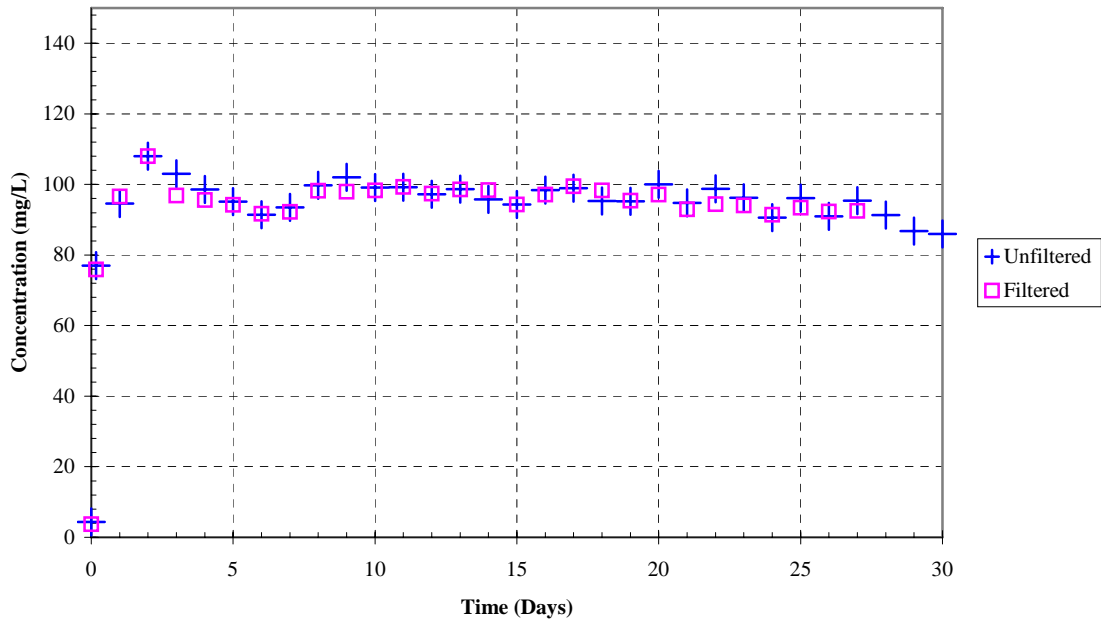
Figure 3-11. Iron concentration.

**Magnesium Concentration**

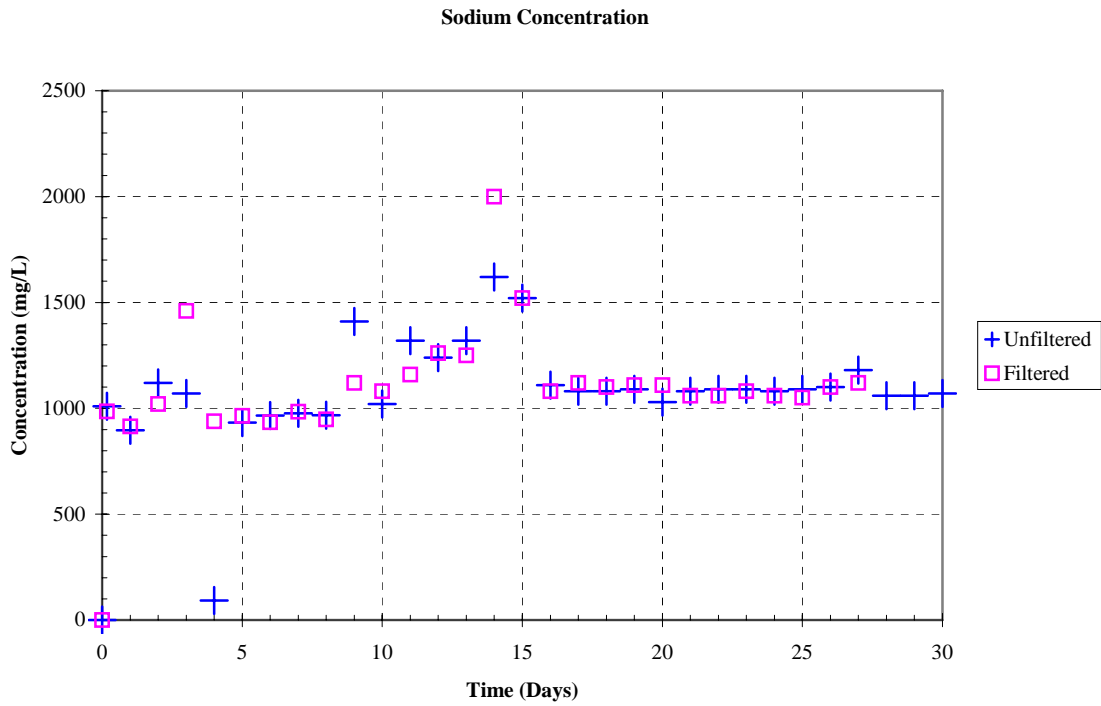


**Figure 3-12. Magnesium concentration.**

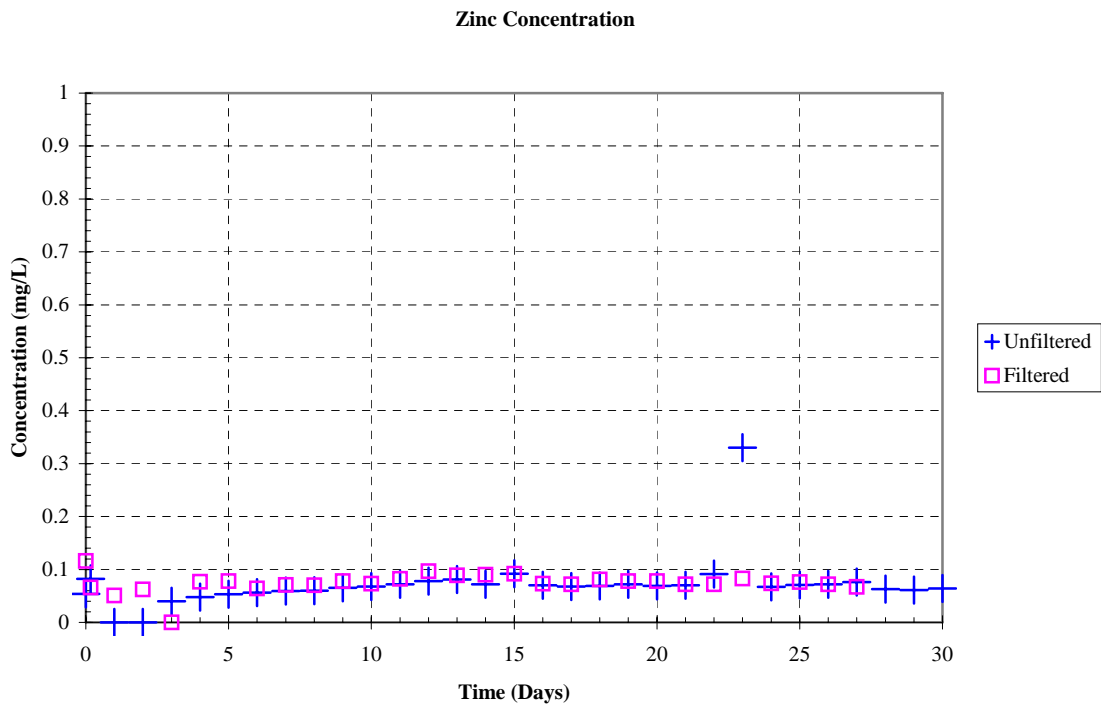
**Silica Concentration**



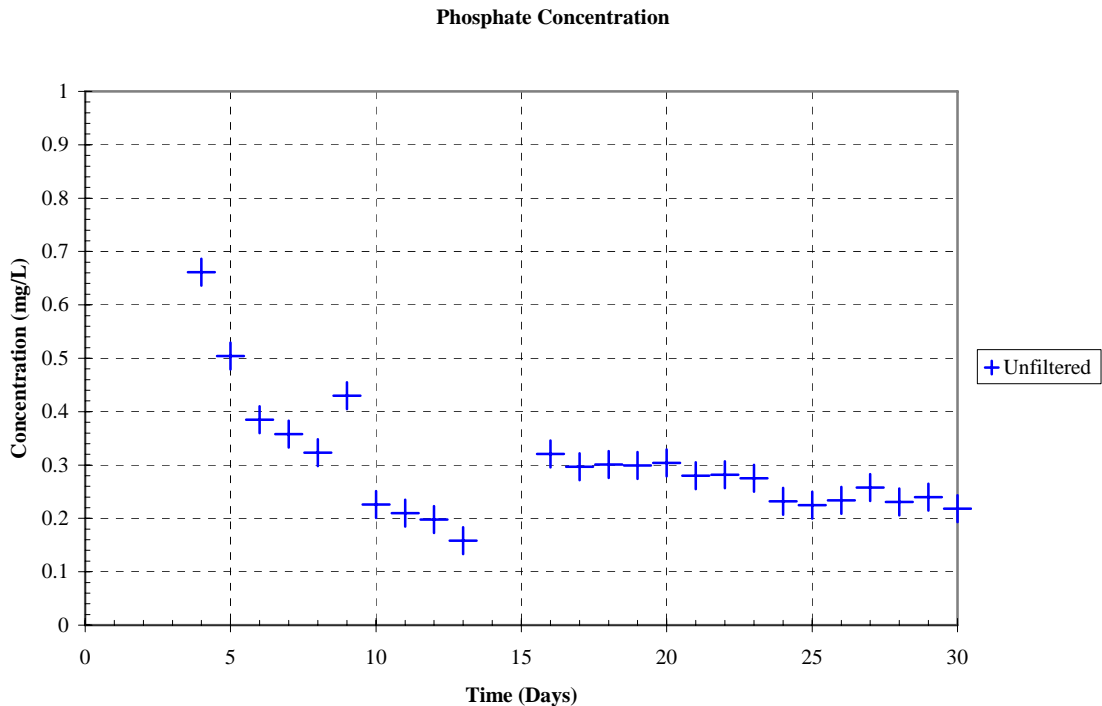
**Figure 3-13. Silica concentration.**



**Figure 3-14. Sodium concentration.**



**Figure 3-15. Zinc concentration.**



**Figure 3-16. Phosphate concentration.**

### 3.2.3. Optical/TEM Images from Filtered and Unfiltered Samples

TEM/EDS and diffraction patterns analysis were performed for Test #3, Day-4, Day-15, and Day-30 filtered and unfiltered solution samples. The filtered solution samples were passed through a 0.7- $\mu\text{m}$  fiberglass filter at 60°C. The unfiltered solution samples were extracted from the tank directly. The results showed no significant diffraction pattern, due to the amorphous nature of the samples. In addition, no significant presence of colloidal particles was observed. Appendix J contains the TEM data.

## 3.3. Insulation

Test #3 was the first ICET test that included cal-sil insulation in addition to NUKON™ fiberglass samples. The fiberglass samples received more thorough investigations, with samples removed from the tank on Day 4, Day 15, and Day 30. The cal-sil was analyzed based on its Day-30 character. In addition, analyses were performed on the raw cal-sil, both baked and not baked.

### 3.3.1. Deposits in Fiberglass Samples

The fiberglass debris was contained in SS mesh bags to minimize migration of the fiberglass throughout the tank and piping. Small mesh envelopes, approximately 4 in. square, containing approximately 5 g of fiber, were pulled out of the tank periodically for SEM examination. These sample envelopes were placed in a range of water flow