Appendix H

TEM Data for Test #4 Solution Samples

Figures

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This appendix presents TEM images and EDS results for Test #4, Day-4, Day-15, and Day-30 unfiltered solution samples. The unfiltered solution samples were extracted from the tank directly. A tiny drop of the testing solutions was transferred to a copper mesh, followed by drying in air for TEM analysis. The TEM and EDS results were obtained on June 27, 2005.

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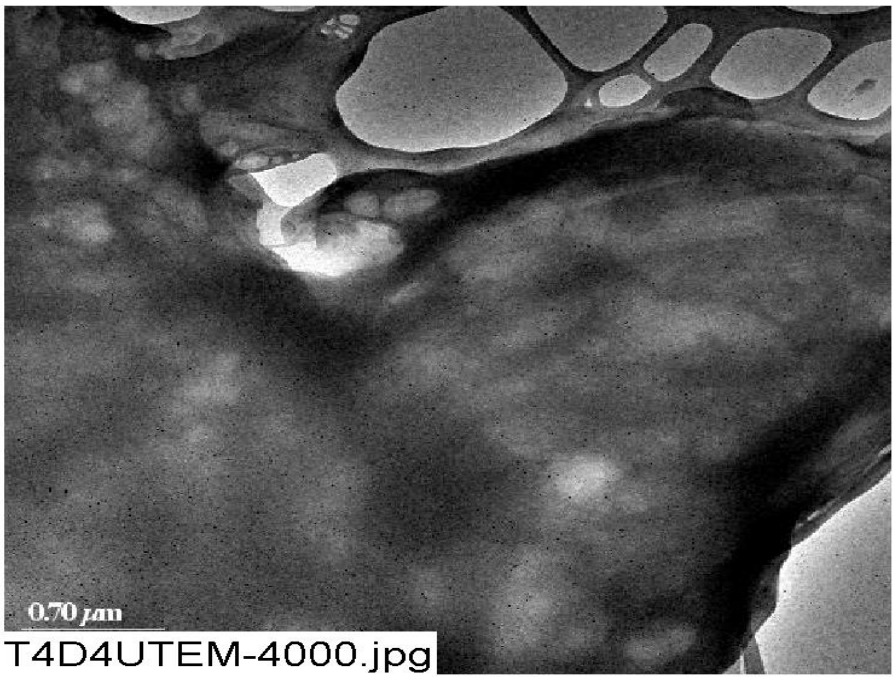

Figure H-1. TEM magnified 4000 times for one Test #4, Day-4 unfiltered sample location. (T4D4UTEM-4000.jpg)

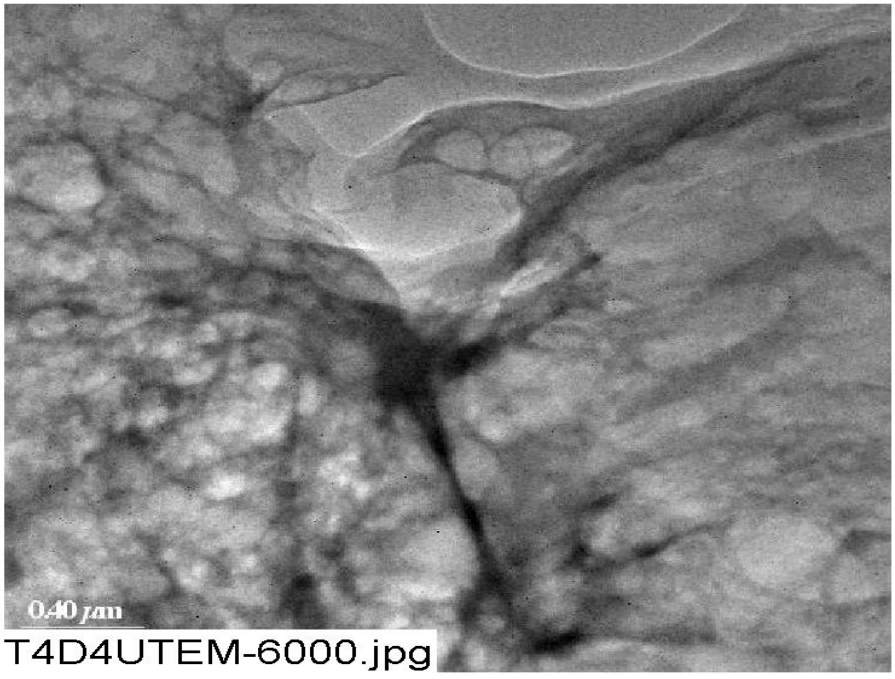

Figure H-2. TEM magnified 6000 times for one Test #4, Day-4 unfiltered sample location. (T4D4UTEM-6000.jpg)

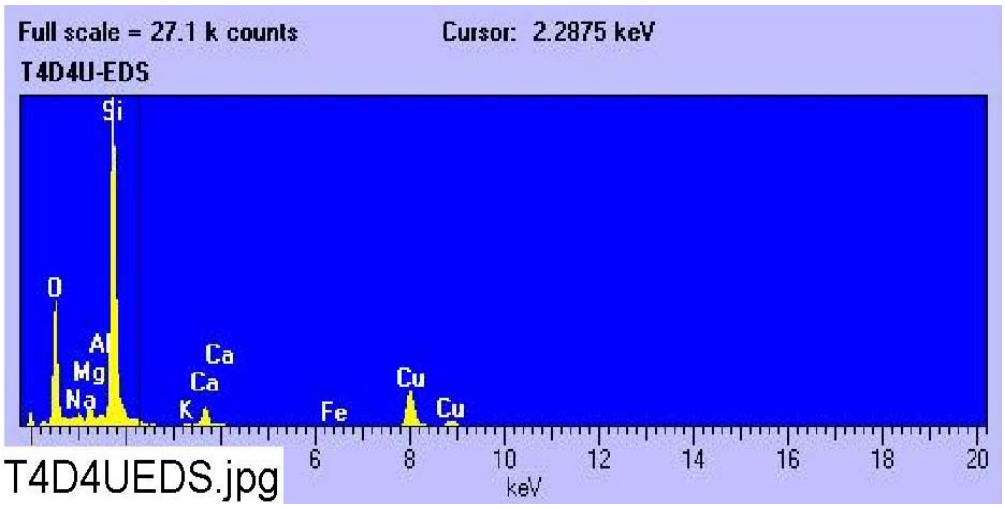

Figure H-3. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-4 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D4UEDS.jpg)**

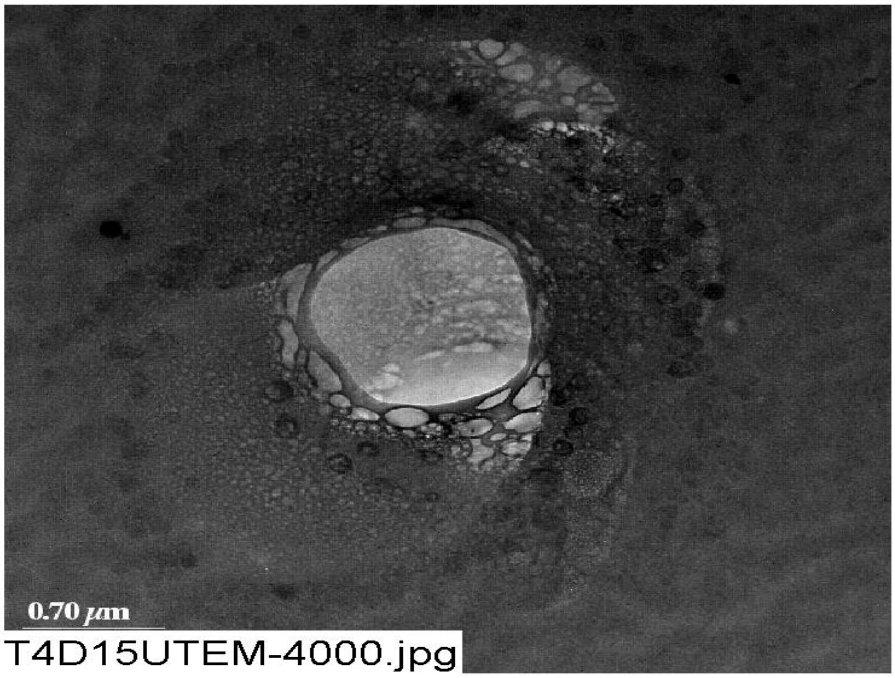

Figure H-4. **TEM magnified 4000 times for one Test #4, Day-15 unfiltered sample location. (T4D15UTEM-4000.jpg)**

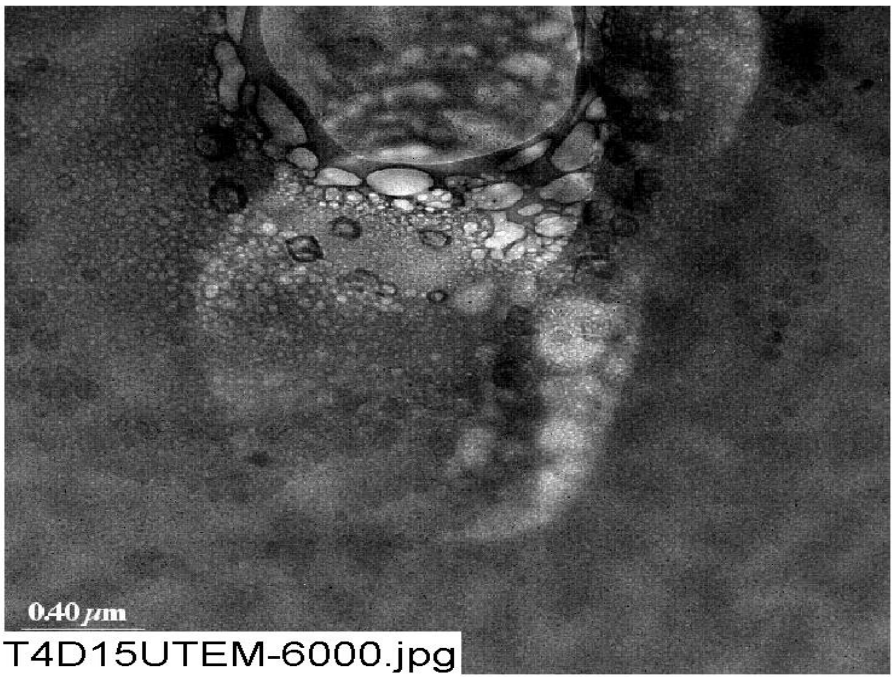

Figure H-5. **TEM magnified 6000 times for one Test #4, Day-15 unfiltered sample location. (T4D15UTEM-6000.jpg)**

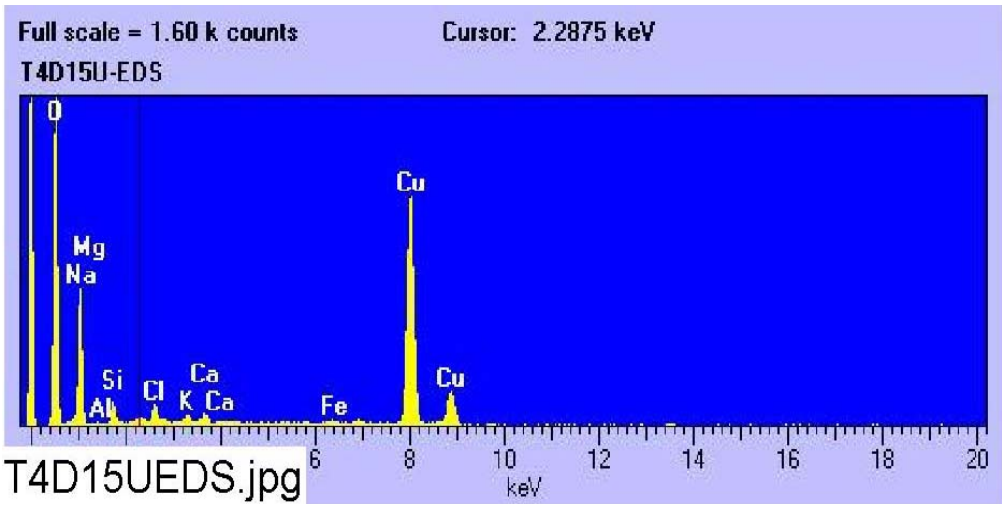

Figure H-6. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-15 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D15UEDS.jpg)**

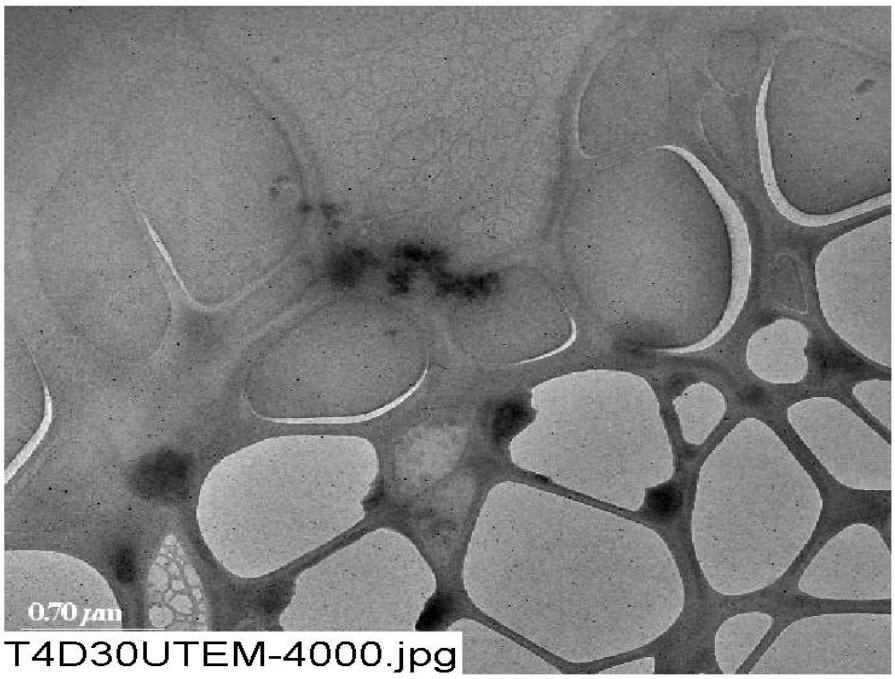

Figure H-7. TEM magnified 4000 times for one Test #4, Day-30 unfiltered sample location. (T4D30UTEM-4000.jpg)

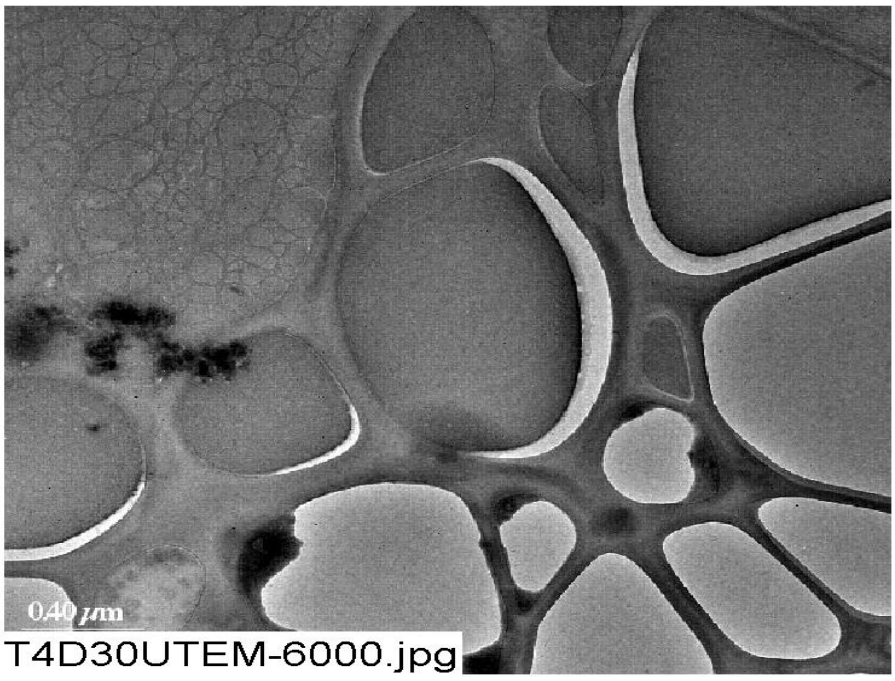

Figure H-8. TEM magnified 6000 times for one Test #4, Day-30 unfiltered sample location. (T4D30UTEM-6000.jpg)

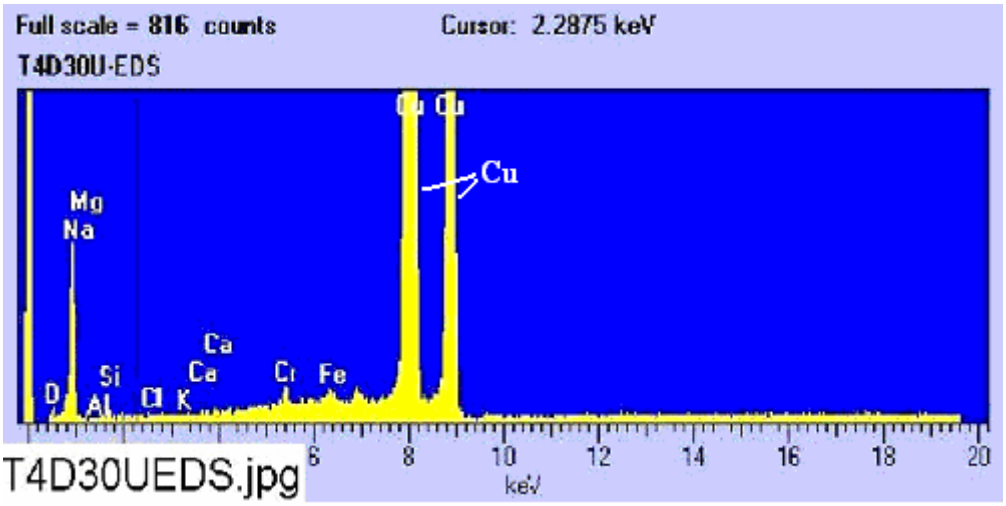

Figure H-9. **TEM energy-dispersive x-ray spectrum for a Test #4 Day-30 unfiltered sample. The copper peak is likely from the copper sample holder for TEM analysis. (T4D30UEDS.jpg)**

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Appendix I

UV Absorbance Spectrum—Day-30 Solution Samples

Figures

Tables

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This appendix presents the UV absorbance result of the Test #4, Day-30 solution sample. The purpose of this analysis was to find any distinguishing absorbance peaks to identify the organics present in the solution. The solution sample was collected through a 0.7-μm fiberglass filter at 60^{o}C to remove particulate impurities, followed by being scanned over the wavelength ranging from 200 to 800 nm by a UV-visible spectrophotometer. The spectrum of DI water was used as background subtraction. From the result, no distinguishing absorbance peaks were found because of the well-mixed nature of the test solution.

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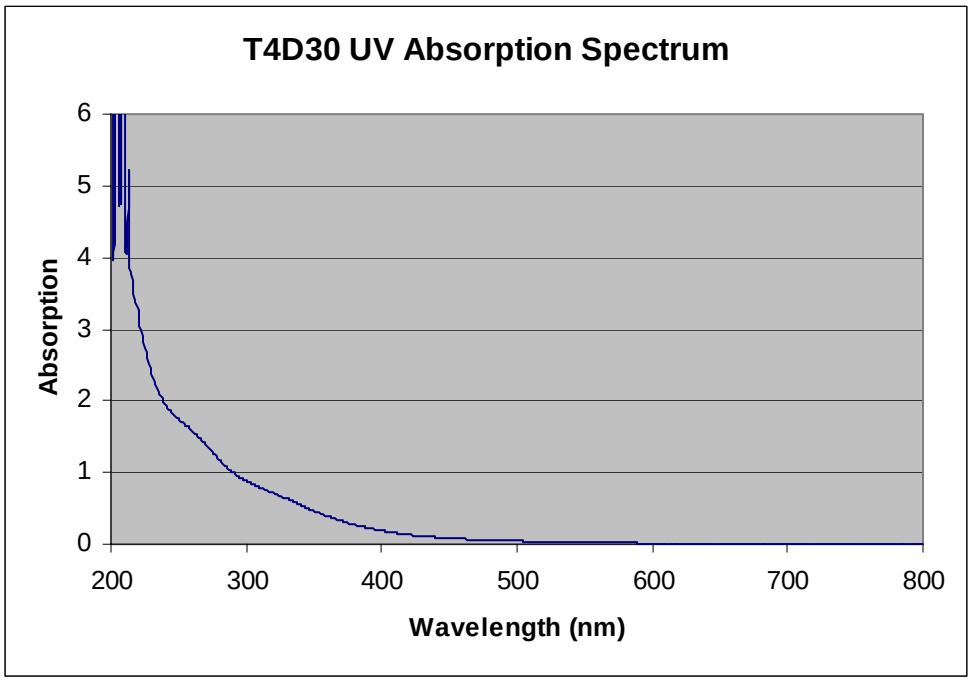

Figure I-1. UV absorbance spectrum for Test #4, Day-30 solution samples.

Table I-1. Test #4, Day-30 Solution Sample Laboratory Settings

	Test #4, Day 30
Collection Time:	7/21/2005 5:15:30 PM
Operator Name:	
Scan Software Version:	3.00(182)
Parameter List:	
Instrument:	Cary 50
Instrument Version:	3.00
Start (nm):	800.0
Stop (nm):	200.0
X Mode:	Nanometers
Y Mode:	Abs
UV-Vis Scan Rate (nm/min):	600.00
UV-Vis Data Interval (nm):	1.00
UV-Vis Ave. Time (sec):	0.1000
Beam Mode:	Dual Beam
Baseline Correction:	On
Baseline Type:	Baseline Correction
Baseline File Name:	
Baseline Std Ref File Name:	
Cycle Mode:	Off
Comments:	
Method Log:	
Method Name:	Default
Date/Time stamp:	7/21/2005 5:09:23 PM
Method Modifications:	
Cell Changer 6x6 Changed:	7/21/2005 5:09:27 PM/Old:1/New:0
UVVIS SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
NIR SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
Common SAT Changed:	7/21/2005 5:10:05 PM/Old:0.0125/New:0.1000
Baseline Correction Changed:	7/21/2005 5:10:10 PM/Old:0 / New:1
Temp Controller Changed:	7/21/2005 5:10:10 PM/Old:0 / New:2
Sipper Type Changed:	7/21/2005 5:10:10 PM/Old:Internal RSA/New:External Sipper
End Method Modifications	
<Current Wavelength>	200

Appendix J

ICET Test #4: Pre-Test, Test, and Post-Test Project Instructions

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The ICET series is conducted under the guidance of project instructions (PIs), which identify the steps to follow for certain activities. These PIs are revised or rewritten as needed for each test. For Test #4, new PIs were written to address pre-test operations and test operations. The post-test operations PI was not changed from Test #3. These three PIs are included in this appendix to more completely describe the test apparatus and chemical solution preparations, the test startup and daily sampling, and the steps followed after test shutdown.

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1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this instruction is to ensure that all data acquisition, test samples, testing supplies, chemicals, and related materials are ready and accounted for prior to testing. In addition, this instruction provides instructions on preparing the chemical test apparatus for testing.

1.2 SCOPE

The pre-test operations preparation will ensure that successful initiation of the testing activity is achieved.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #4 Addendum, May 10, 2005
- Laboratory Safety Guidelines
- ASTM A 380 – 99, Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems
- Material Safety Data Sheets (MSDS) for all chemicals involved

2.0 PREREQUISITES

The data acquisition setup and inspection; instrument calibration; and the coupon receipt, preparation, inspection, and storage tasks must be completed in full prior to the completion of this activity. Fiberglass and calcium silicate (cal-sil) samples must be weighed and their planned locations in the tank identified. That data must be recorded.

2.1 Training Requirements

The following personnel training is required for this task:

1) LabVIEW and computer data acquisition training
2) Chemical handling training, specifically for ethyl alcohol, ammonium hydroxide, and lithium hydroxide.
3) Safe lift execution training

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, eye wash station.

3.0 DOCUMENTATION REQUIRED

MSDSs must be available for all chemicals used.

A lab notebook must be maintained throughout the pre-test operations instruction. Contained within the lab notebook will be the date, times, description of activities, and quantities of chemicals added, number of cleanings, and physical observations of the tank cleaning and preparation procedures.

4.0 HAZARDS

The hazards associated with this activity include potential injuries associated with chemical handling.

5.0 INSTRUCTIONS

1. Ensure that all testing materials and supplies are ready and on-site. See checklist at the end of this document. Verify that eye wash station is operational. Note: The following solutions are not used in this instruction, but are to be prepared in advance of entering ICET-PI-016, "Test Operations, Test #4 (cal-sil, fiberglass, and NaOH at pH 10)." After preparation, clearly label the containers with the solutions and place in an area restricted for ICET Project test use.
2. Prepare 21. 2 g of concrete dust and 63.7 g of latent debris.
3. Prepare LiOH solution: dissolve 0.663 g of lithium hydroxide (LiOH) into about 100 mL water in a 250-mL sample container.
4. Prepare NaOH-LiOH-HCl solution.
   a. Add about 10 gallons of RO water to a 10-L polypropylene container.
   b. Add 212 mL of 12.24 N HCl to the water in the container.
   c. Dissolve 0.663 g of LiOH into the water in the container.
   d. Dissolve 8.47 kg of NaOH into the water in the container. Add the NaOH slowly and mix until dissolved so the solution does not over-heat.
   e. Properly label and store the container.
5. Prepare NaOH solution for spray nozzle feed.
   a. Add about 0.5 gallons of RO water to a 1-gallon polypropylene container.

b. Dissolve 614 g of NaOH into the water in the container. Add the NaOH slowly and mix until dissolved so the solution does not over-heat.
c. Dilute with additional RO water until the volume is 1 gallon.

6. Prepare laboratory control sample (LCS). See ICET-PI-005, "Chemical Sampling and Analysis," for details on the laboratory control sample.
7. Start the data acquisition system. Verify that the data acquisition system is monitoring flow rate, pump speed, temperature, and pH.
8. Clean the tank and piping.
   a. Cleaning should commence as soon after a test is completed as possible, to prevent material from hardening in the tank or piping and to maximize the time available for cleaning.
   b. Cleaning chemicals may consist of weak acids (e.g., acetic acid, citric acid, or dilute mineral acids), weak bases (e.g., ammonium hydroxide), week organic solvents (e.g., ethanol), or detergents/surfactants (e.g., trisodium phosphate, sodium dodecyl sulfate), as necessary. Cleaning solutions can be heated if necessary. Note that the discharge limit to the sanitary sewer is a maximum temperature of 140 °F and pH between 5.0 and 11.5. Cleaning solutions that are not within this range should be neutralized before discharge.
   c. During cleaning, the pump should be run and water directed through both recirculation lines (through the spray nozzles and lower headers)
   d. The sample line should be removed from the piping, physically cleaned, and carefully inspected. If the sample line cannot be adequately cleaned, it should be replaced.
   e. After each cleaning step, the tank and piping should be thoroughly rinsed with tap water or demineralized water.
   f. After each cleaning step, a segment of pipe should be removed, and the interior of the pipe visually inspected.
   g. Cleanliness criteria: When the tank visually appears to be satisfactorily cleaned, the tank and piping should be thoroughly rinsed with demineralized water. The interior surfaces of the tank and piping shall be free of any deposits that can be removed by vigorous scrubbing. Demineralized water drained from the tank should have turbidity less than 0.3 NTU and conductivity less than 50 uS/cm.
9. Tank is now ready for testing. Proceed immediately to Instruction No. ICET-PI-016, "Test Operations, Test #4 (cal-sil, fiberglass, and NaOH at pH 10).

6.0 ATTACHMENTS

No forms are attached to this document.

7.0 Materials Checklist

_____ lithium hydroxide, 0.663 g

_____ NaOH pellets, 9.085 kg

_____ 212 mL of 12.24 N HCl

_____ tap water supply

_____ demineralized water production system

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ analytical balance

_____ top loading balance

_____ chemical spatula

_____ chemical scoop

_____ weigh boats

_____ two 5-gallon plastic containers

_____ 250 mL graduated cylinder

_____ 250-mL HDPE or PP bottle

_____ 2.5 gallons ethanol

_____ 2.5 gallons ammonium hydroxide

_____ turbidimeter and associated equipment

_____ conductivity meter and associated equipment

1.0 INTRODUCTION

1.1 PURPOSE

The intent of the instruction is to outline the steps that are to be followed during testing.

1.2 SCOPE

This activity forms the core of the entire Chemical Effects Testing project. All activities involved in this project affect and are affected by this activity.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard D 3370-95a
- ASTM Standard G 31-72
- Material Safety Data Sheets (MSDS) for all chemicals involved
- LabVIEW operation manual
- Laboratory Safety Guidelines
- Chemical Additive Analysis Revisions – ICET-CALC-007, November 11, 2004; Test #4 Addendum, May 10, 2005. Note: This addendum used a target pH range of 9.7-10.3.
- John Gisclon email to Bhagwat Jain, Cal-sil Information Used in Test 3, March 31, 2005

2.0 PREREQUISITES

All sample coupons must be placed in their corresponding racks. Also, the pre-operation test preparation activity must be completed in full.

2.1 Training Requirements

The following personnel training is required for this task:
1) LabVIEW and computer data acquisition training.
2) Chemical handling training for all chemicals involved.

2.2 Equipment Requirements

The following equipment is required to perform this activity: computer with installed LabVIEW software, data acquisition system, and fully assembled and calibrated ICET test apparatus.

Safety equipment must be available: goggles, gloves, lab coats, hard hats, steel-toed shoes, eye wash station, hydrogen detector and hydrogen removal system.

3.0 DOCUMENTATION REQUIRED

A lab notebook must be maintained throughout the testing procedure. In addition, a binder will be maintained that includes pertinent test instructions and the completed daily log sheets (see Attachment A). The daily log sheet contains the date, times, physical description, and quantity of fiberglass and water samples obtained each day. In addition, the daily log sheet contains information from the data acquisition system (DAS), the water samples taken, and other test information.

The electronic data that are acquired are backed up daily and stored in a separate location each testing day. Refer to ICET-PI-001, Data Acquisition Setup and Inspection.

4.0 HAZARDS

The hazards associated with this activity include tipping of the chemical tank assembly, ingestion and/or respiration of any chemicals involved, and scalding and/or burning hazards involved in daily tank venting, and possible hydrogen gas generation from corrosion reactions. Appropriate measures to control hydrogen gas must be in place before operations commence.

Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1. Because of the time required for heating the tank contents and dissolving chemicals, this sequence should be started at least 48 hours before the scheduled time t = 0. Pre-test operations preparation should be complete before proceeding with this sequence.
2. Ensure that all testing materials and supplies are ready and on-site (see checklist at end of this instruction).
3. Add 239 gallons of RO water to the tank by pumping water from the RO skid through the totalizing flow meter. Record flow to the nearest 0.5 gallon.
4. Verify valves are positioned as follows:

Valve	Description	Position
V-1	tank drain	closed
V-2	pump isolation	open
V-3	instrument loop supply	open
V-4	instrument loop discharge	open
V-5	instrument loop bypass	closed
V-6	in-line filter isolation	open
V-7	recirculation line supply	open
V-8	tank spray nozzle supply	closed
V-9	sample line	closed
V-10	recirculation line injection	closed

5. Start pump and adjust to flow rate of approximately 25 gpm.
6. Start computer, start LabVIEW, verify that flow rate, pump speed, temperature, and pH are being recorded properly.
7. Turn on heater and allow water in tank to heat to 60 °C ± 2 °C. (This may take up to 20 hours.)
8. Add the pre-mixed NaOH-LiOH-HCl solution.
9. Add 15.14 kg of boric acid (H_3BO_3), weighing in approx. 2 kg increments, recording the weight of each increment to the nearest 10 g.
10. Allow the water to circulate until the solution is visibly clear, indicating that the boric acid is completely dissolved.
11. Allow water in tank to heat to 65 °C ± 2 °C.
12. Take grab water sample for analysis for the parameters identified in steps a – h below. Also record physical appearance of the sample (clarity, presence of gelatinous material, etc). All Day 1 and subsequent samples will be analyzed by Assaigai Analytical Laboratory. In addition, periodic test samples and laboratory control samples (LCSs) will also be analyzed by the UNM laboratory.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity
    e. total suspended solids (TSS)
    f. dissolved oxygen (DO)
    g. chloride
    h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved
13. Add 21.2 g of concrete dust and 63.7 g of latent debris samples (prepared earlier), wait 10 minutes, take 100 mL water sample for particulate size distribution, density, and TSS.
14. Stop pump.
15. Add the pre-determined amount of cal-sil dust. This will be approximately 43.5 lb.
16. Place coupon racks, fiberglass holders, and cal-sil holders into tank. This is done in accordance with previously determined quantities, size distributions, and locations.

(Details of the cal-sil preparation and size distributions are given in the referenced email.)

17. Verify locations of coupon racks, fiberglass holders, and cal-sil holders.
18. Verify the tank temperature is 62 °C. (Because the tank lid may have to be removed to unplug nozzles, the test will be started with the water temperature at its upper limit.)
19. Start pump and adjust pump speed to 25 gpm.
20. Open valve V-8 (tank spray nozzle supply) to direct water to nozzles and adjust valves V-7 (recirculation line supply) and V-8 (tank spray nozzle supply) until nozzle flowmeter is reading 3.5 gpm. Verify total flow is still 25 gpm and adjust variable frequency drive (VFD) if necessary.
21. Record date and time at which nozzle flow started. This is time t = 0 for the test.
22. Note that the spray phase will begin with the crane attached to the tank lid. The objective is to be able to carefully monitor possible nozzle blockage and take immediate action to prevent it. At the first sign that a nozzle may be starting to plug, the spray flow rate should be increased rapidly to 5-10 gpm for approximately 5 s. (As long as the nozzle spray pattern is not affected however, the spray flow should remain at 3.5 gpm.) If a nozzle should block in spite of the increased flow rate, the tank lid should be removed and a stainless steel wire used to clear the nozzle exit. The tank lid must be replaced as soon as possible to limit the expected temperature decrease.
23. Flow through the nozzles should be monitored every 5 minutes by looking through the tank view windows. After 15 minutes, if there have been no spray nozzle blockages, the monitoring frequency can be increased to every 15 minutes. (Note that the spray flow pattern from each nozzle can be observed through the view windows and restrictions to the spray pattern are readily observable.)
24. At t = 0, start chemical metering pump and inject pre-measured NaOH solution into the spray line. The objective here is to add the 1-gallon NaOH solution in 30 minutes.
25. At 30 minutes, shut off chemical metering pump and isolate this line.
26. Take a measurement of hydrogen concentration. At 2-hour increments, repeat the hydrogen concentration measurement. If the concentration reaches 10% of the flammability limit, purge the tank atmosphere. This needs to be repeated until the hydrogen concentration has been determined to be below 10% of the flammability limit, and then the frequency of hydrogen concentration measurements is to be re-evaluated.
27. At t = 4 hours, stop the spray flow by closing valve V-8.
28. At any time following the spray phase, the crane may be removed from the tank lid.
29. After closing valve V-8 (at t = 4 hours), take water grab sample for analysis for the parameters listed below. Record the time of sample collection.
    a. pH
    b. temperature
    c. turbidity
    d. viscosity

e. chloride
f. total suspended solids (TSS)
g. dissolved oxygen (DO)
h. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved.

30. At t = 24 hours, and daily thereafter, take water grab sample for analysis for the parameters listed below. (The LANL PI will propose a different sampling frequency to the project sponsors if test data support it.) Record the time of sample collection.
    a. pH
    b. turbidity
    c. viscosity
    d. temperature
    e. total suspended solids (TSS)
    f. metals (Al, B, Ca, Cu, Fe, Pb, Li, Mg, Ni, K, Si, Na, and Zn), total and dissolved. An exception is that B, Li, K, Pb, and chloride analyses will be performed only at t = days 15 and 30. Also, dissolved oxygen will be measured at day 30.
31. During each daily water sample collection, look inside tank (through windows) and record observations. If the tank water level indicates that the water volume is 245 gallons or less, add RO water to bring the volume up to 250 gallons and record the amount added.
32. At t = 24 hours, weekly thereafter, and at the end of the test, collect 100 mL water sample for particulate size distribution and density analysis, to be performed at AALI. The particulate size ranges to be used will be as close as possible to those called out in the test plan: (in microns), 1-10, 11-25, 26-50, 51-75, 76-100, and > 100 microns.
33. At t = 24 hours, weekly thereafter, and at the end of the test, collect water samples for strain rate viscosity measurements (see PI-010 for sample details.)
34. After 2 to 5 days of testing, it is anticipated that the solution will be stable and no suspended particles will be visible. If that is the case, insert the three types of fiberglass samples described in Attachment B. Note that one of the samples (long, narrow stainless steel holder) is to be placed in front of the water distribution headers, and the others are to be placed behind the headers. (The date and time of the addition of these samples will be recorded in the lab notebook.)
35. At 3 days $\leq$ t $\leq$ 5 days, 14 days $\leq$ t $\leq$ 16 days and at the end of the test, collect a sacrificial fiberglass sample to be inspected and examined with SEM.
36. At 24 hours, at 14 days $\leq$ t $\leq$ 16 days and at the end of the test, run 1L of water through a nucleopore filter. The filter will be taken for SEM analysis as specified in ICET-PI-007. (Note that depending on the solution, some filter material will not work well for this operation. If possible, use a nucleopore filter for SEM analysis, and then collect a second sample on nitrocellulose filter for later digestion and ICP analysis.)
37. Shut down pump
38. Indicate end of test on the data acquisition system and shut down the data acquisition software.
39. Proceed directly to PI-008 Post-Test Operations.

6.0 ATTACHMENTS

Attachment A. Daily Log Sheet

Attachment B. Test #4 Fiberglass Sample Addition after Test Start

7.0 MATERIAL CHECKLIST

_____ boric acid, 15.14 kg

_____ pre-mixed NaOH-LiOH-HCl solution

_____ pre-mixed NaOH solution

_____ concrete dust, 21.2 g

_____ latent debris, 63.7 g

_____ Nucleopore filter

_____ chemical handling safety equipment (lab coat, goggles, rubber gloves)

_____ top-loading balance

_____ weigh pan for 2 kg aliquots of boric acid

_____ stainless steel filter paper holder

_____ 500 mL graduated cylinder (for TSS)

_____ totalizing flow meter

_____ sample containers (see Chemical Sampling Instruction)

_____ analytical equipment (see Chemical Sampling Instruction)

_____ pre-assembled coupon racks

_____ pre-assembled fiberglass baskets, total of 2.2 lb of fiberglass

_____ pre-assembled cal-sil baskets, total of 26.7 lb of cal-sil

_____ pre-measured cal-sil dust, 43.5 lb

_____ coupon handling safety equipment (hard hat, leather gloves, boots)

_____ computer disks for backup of Labview data

_____ Masterflex peristaltic pump and tubing

_____ demineralized water production system

Attachment A. Daily Log Sheet

Daily Log Sheet
Integrated Chemical Effects Test (Test # 4)

Date: _____ Time of sample collection: _____
Sample taking and data reduction by _____ and_____

Sample bottle identification:
Assaigai (total): _____
Assaigai (filtered): _____
UNM (total): _____
UNM (filtered): _____

Control system readings:
Temperature: _____ Flow: _____ pH: _____

Analyses:
Volume filtered for TSS: _____ pH: _____
Temperature: _____ Dissolved oxygen: _____
Turbidity (at 60 °C): _____ (at 23 °C; and 10 min.)_____
Viscosity, unfiltered (60 °C): _____ (at 23 °C) _____
Viscosity, filtered (60 °C): _____ (at 23 °C) _____
Water Level: _____ Water Added: _____
Hydrogen: _____ Other: _____
Fiberglass or other samples taken: _____
TSS filter #: _____ TSS (mg/L): _____

Comments:

Observations written in lab notebook by _____
☐ Continued on back

Attachment B. Test #4 Fiberglass Sample Addition after Test Start

Recent experience gained in ICET Test #3 with adding large quantities of Calcium Silicate (Cal-Sil) debris to the tank suggests that contamination of fiber samples with suspended particulate may complicate the post-test identification and analysis of chemical products that may be contained within the samples. Past experience also suggests that the circulating tank solution will clarify after 1 to 2 days, providing an opportunity to introduce fiber samples for immersion in the chemical environment without substantially shortening the exposure time and while avoiding the complications of Cal-Sil contamination. (Several other fiber samples are immersed at the initiation of the test so that they are directly exposed to large quantities of the Cal-Sil particulate).

This attachment to ICET-PI-16, Rev 0 addresses the addition of three types of containers for fiberglass samples that are to be inserted in the test solution between Days 2 and 5 after substantial water clarity has been achieved and as needed to match operations schedules. These containers include: (A) a long (5-6 in.), thin (1/4 to ½ in.), narrow (1-2 in.) stainless steel mesh envelope that will be placed in front of a discharge hole on one of the water distribution headers (a "high-flow" area of the tank); (B) a nylon mesh envelope of approximately 4 to 6 inches square containing 5 to 10 g of fiberglass, and (C) 3 to 5, two-inch diameter pucks of fiberglass that are prepared in rings of CPVC and encased in typical envelopes of stainless steel mesh. Each container will hold less than 10 g of fiberglass and their fiberglass contents are considered negligible with respect to the total test amount of fiberglass. The nylon mesh envelope and pucks will be placed in "low-flow" areas of the tank behind the water distribution headers. Technical descriptions and justifications for each item follow.

Test Item (A): thin sample in high flow

This sample may provide evidence of whether chemical deposits are enhanced or inhibited by the direct impingement of water flow. The large aspect ratio of this envelope (length/width) is designed to avoid large perturbations in the inlet flow patterns that would occur by placing a large flat object near the distribution headers. Post-test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (B): nylon mesh envelope

ICET Test #1 results indicated a possible preference for chemical deposits to form near the interface between fiberglass and the stainless steel mesh that was used to form the sample envelopes. The introduction of a nonmetallic casing material may permit a comparison of this effect under exposure to a similar chemical environment. Nylon mesh was selected as a suitably inert material for constructing an envelope for this fiber. Based on qualitative assessments and recommendations of material performance made by chemical supply vendors (see for instance, chemical resistance information given at

www.eldonjames,com/html), nylon is expected to exhibit “Excellent” resistance to NaOH solutions of up to 50% concentration and “Good” resistance to industrial concentrations of boric acid. This container material has not been submitted for independent bench-scale leaching tests in the ICET solution. The small quantity of foreign material is not expected to perturb interpretation of results from ICET Test #4 regardless of its performance characteristics. Post test examination of this sample will be made using typical ESEM and SEM/EDS survey techniques.

Test Item (C): Fiber pucks

The MOU established between NRC and EPRI for conduct of the ICET series specifically excludes modification of the apparatus for the purpose of obtaining in-line flow head loss data. However, the presence of chemical deposits observed on and within fiberglass samples obtained from ICET Test #1 to Test #2 raises questions regarding the potential of these products to impede water flow and about their behavior under flowing conditions, for example, whether they will be adherent or wash out of the fiber matrix. Unanswered questions also remain regarding the possible formation of deposits in the presence of flow, but without a direct mechanism of studying formation under flow, it may be useful to examine whether the deposits can form in fiberglass that represents a prototypical debris bed. Two of the attributes that may distinguish fiberglass on a debris bed from fiberglass in a debris flock are (a) degree of mechanical separation between fibers, and (b) degree of compaction in the bed.

Between 3 and 5 fiberglass pucks will be prepared as shown in Figure 1 for jacketing in a stainless steel envelope and immersion in the Test #4 test solution after the water clarity has improved. Approximately 5-10 grams of dry fiberglass are required to fill the ½-inch thick, 2-in. diameter sample ring. The sample ring is cut from a 2-inch diameter CPVC pipe to provide a standard dimension for any flow testing that may be desired after the conclusion of Test #4 and to avoid the introduction of unapproved foreign materials in the test tank. Before introducing the fiberglass to the mold, it will first be agitated in a kitchen blender for at least 2 minutes in two batches, each batch containing approximately half the debris and approximately 2 quarts of water. The purpose of agitation is to separate fibers from the raw flocks of manufactured insulation. The batches will be sequential poured into the mold placed across a mesh screen. Gentle manual tamping may be required to ensure uniformity of the bed.

Figure 1. Example fiber pucks prepared for immersion in ICET test solution.

The introduction of fiberglass pucks will provide the following technical opportunities for post-test examination:

1) Any observed chemical deposits will be relatively free from contamination of Cal-Sil.
2) ESEM and SEM/EDS examines can be made for the presence of deposits inside of a relatively compact debris bed.
3) If exams 1 and 2 are positive, the pucks will provide a concentrated quantity of the deposit for possible extraction, isolation and identification.
4) The pre-measured dry mass of the fiber may permit a determination of dry mass for any deposited chemical products. This may provide a first step towards quantifying rates and quantities of formation.
5) The convenient form of the debris in the mold will facilitate any head loss testing that is deemed interesting or necessary as a post-test analysis activity. Samples of this type could be placed either within a continuously circulating closed loop or within a static head drain column for measurement of flow loss. The primary objective of any such examinations would be the direct comparison of fresh, identically prepared samples with cultured samples that have been exposed to the test environment. Expected variability between the samples suggests that several replicates should be prepared for comparison. Any work of this type will be conducted under a separate approved procedure.

<u>Note:</u> The purpose of this attachment is to describe the samples to be added to Test #4 after test initiation. Any post-test evaluations of these samples other than ESEM and SEM/EDS will be done under separate procedure/project instructions. In

addition, any post-test head loss testing will require appropriate documentation and sponsor approvals.

1.0 INTRODUCTION

1.1 PURPOSE

The intent of this instruction is to ensure that the experimental samples are removed from the test apparatus, the test apparatus is rinsed and inspected, and the test apparatus is made ready for subsequent pre-test operations.

1.2 SCOPE

This activity marks the end of one chemical effects test run. Experimental sample removals and inspections, test apparatus rinsing, and preparations for cleaning and subsequent tests are addressed here.

1.3 REFERENCES

- Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 12.c, March 30, 2005
- ASTM Standard G 4-01
- ASTM Standard G 31-72
- ICET-PI-002, Coupon Receipt, Preparation, Inspection, and Storage, November 19, 2004
- ICET-PI-014, Rev. 0, Test Operations, Test #3 (cal-sil and fiberglass, with TSP, April 5, 2005
- ICET-PI-005, Rev. 1, Chemical Sampling and Analysis, February 3, 2005
- Laboratory safety guidelines
- ICET Project Safety Plan

2.0 PREREQUISITES

All test operation PI criteria must be completed prior to conducting this task.

2.1 Training Requirements

- Laboratory Safety Guidelines
- ICET Project Safety Plan

2.2 Equipment Requirements

A city tap water supply outlet is required for this activity and chemical handling and lifting safety equipment. A reverse osmosis unit is required for the final flush.

3.0 DOCUMENTATION REQUIRED

Documentation related to test parameters, chemical water analyses, coupon and fiberglass examinations, and daily test operations are outlined elsewhere. In this instruction, the steps required to remove samples from the test apparatus and to make it ready for the next test are outlined. In addition, observations as to the test apparatus' condition are obtained and recorded here.

4.0 HAZARDS

The hazards associated with this activity include ingestion/respiration and/or dermal and eye contact with residual chemicals. Lifting hazards associated with the tank lid and coupon racks are also associated with this activity.

5.0 INSTRUCTIONS

1) On the last day of testing, collect water samples and perform analyses as outlined in ICET-PI-014 and ICET-PI-005.
2) Remove 10L of water from the test apparatus and store at test temperature, for future analyses
3) Shut off the recirculation pump.
4) Remove the small fiberglass samples for SEM examination.
5) Leave one heater on and continue to monitor tank water temperature.
6) Isolate and drain the test apparatus piping.
7) Remove the tank lid.
8) Before removing coupon racks or insulation samples, examine and take photographs and notes of the inside of the tank, the coupons and racks, and the insulation samples.
9) Remove the six non-submerged coupon racks to a staging area for drying and post-test examinations (refer to ICET-PI-002).
10) Take additional photographs of the inside of the tank.
11) Drain the tank slowly, down to the level that uncovers the submerged rack, but keeping the water level above the heater.
12) Remove the submerged coupon rack to the staging area.
13) Repeat step # 10.
14) Turn off the heater.
15) Completely drain the tank, taking precautions so that the sediment on the bottom of the tank is not disturbed any more than necessary.
16) Store water that was drained from the test apparatus until it is cleared for disposal or shipment. (This step was just moved from later in the PI – the old step #26.)
17) When the tank is drained, repeat step # 10. Note especially the locations and orientations of the remaining samples.
18) Remove the remaining insulation samples to the staging area to dry.

19) Ensure that all samples removed from the tank are clearly marked as to their location and orientation within the tank.
20) After all samples have been removed, repeat step # 10.
21) Inspect the interior of the tank, noting any observations.
22) Note the presence of any sediment. Carefully remove as much sediment as possible, noting any unique aspects of it, such as location. Place the sediment in plastic containers with lids, marking the location of the sediment in the tank.
23) Remove the tank drain screen and remove the insulation sample for future analysis.
24) Remove the flow meter from the loop and take pictures of the flow meter interior.
25) Remove any deposits within the flow meter and place the deposits in plastic containers with lids. This is to keep the samples hydrated.
26) Remove a section of pipe, take pictures of the pipe interior, and remove and store any deposits there.
27) Replace the flow meter and piping section.
28) Rinse the tank with tap water and drain the water.
29) Fill the system with 250 gallons of tap water and circulate water through the spray nozzles and recirculation headers for at least 60 minutes. Repeat with de-mineralized water.
30) If any signs of deterioration are observed on the inside of the test apparatus tank, remove selected insulation on the tank. Inspect the stainless steel tank for any abnormalities.

6.0 ATTACHMENTS

No forms are attached to this document.

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