

# 1. INTRODUCTION

The Integrated Chemical Effects Test (ICET) project was a joint effort by the United States Nuclear Regulatory Commission (NRC) and the Electric Power Research Institute (EPRI) to simulate the chemical environment present inside a containment water pool 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 was conducted by Los Alamos National Laboratory (LANL) at the University of New Mexico (UNM), with the assistance of professors and students in the Departments of Civil and Mechanical Engineering.

## 1.1. Objectives 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. Water collected in the sump from the reactor coolant system, the safety injection system, and the containment spray system is recirculated through the reactor core to remove residual heat. The recirculation sump contains a screen to protect components that are in the reactor coolant, containment spray, and ECCS flow paths from the effects of debris that could be transported to the sump. Concerns have been raised that fibrous material could form a mat on the screen, obstructing flow, and that chemical reaction products such as gelatinous or crystalline precipitates could migrate to the screen, causing further blockage and increased head losses across the debris bed. Another potential adverse chemical effect is increased bulk fluid viscosity that could also increase head losses through a debris bed.

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

The ICET test 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. It should be noted that the Test #5 initial boron concentration and buffering agent were different from the other four tests.\* All tests in the series included metal coupons with surface areas scaled to those in representative PWR containment and sump systems.

The ICET project was a joint effort by the U.S. NRC and the nuclear utility industry, undertaken through the Memorandum of Understanding on Cooperative Nuclear Safety between the NRC and EPRI, “Addendum on Integral Chemical Effects Testing for PWR ECCS Recirculation.” EPRI supplied many of the materials used in the ICET tests, and Appendix B provides

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\*An important distinction exists between the chemical reagents used to achieve bulk solution pH and those used to stabilize or “buffer” the pH within a desired range. However, there was no intent to control pH during the course of the tests. In this report series, discussions of additives that influenced pH refer exclusively to the sodium hydroxide, trisodium phosphate, and sodium tetraborate that, in addition to boric acid, were used to initiate the test conditions. The term “buffer” or “buffering agent” is sometimes used informally to refer to these chemicals.

descriptions of the materials used in the testing. EPRI and NRC also wrote a Project Test Plan (Appendix C, Ref. 1) that provides a complete rationale for the selection of the ICET test conditions.

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

Run	Temp (°C)	TSP <sup>a</sup>	NaOH	Sodium Tetraborate	pH <sup>b</sup>	Boron (mg/L)	Notes
1	60	N/A	Yes	N/A	10	2800	100% fiberglass insulation test. High pH, NaOH concentration as required by pH.
2	60	Yes	N/A	N/A	7	2800	100% fiberglass insulation test. Low pH, trisodium phosphate (TSP) concentration as required by pH.
3	60	Yes	N/A	N/A	7	2800	80% calcium silicate/20% fiberglass insulation test. Low pH, TSP concentration as required by pH.
4	60	N/A	Yes	N/A	10	2800	80% calcium silicate/20% fiberglass insulation test. High pH, NaOH concentration as required by pH.
5	60	N/A	N/A	Yes	8 to 8.5	2400	100% fiberglass insulation test. Intermediate pH, sodium tetraborate (Borax) buffer.

<sup>a</sup>TSP was added in hydrate form, i.e., Na<sub>3</sub>PO<sub>4</sub>·12H<sub>2</sub>O.

<sup>b</sup>Values shown were the target pH for Tests #1–#4. The value for Test #5 is the expected range.

The ICET test apparatus consisted 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 the relative surface areas found in containment and in proportion to a maximum test dilution volume of 250 gal. of circulating fluid. Representative chemical additives, temperature, flow rates, and material combinations were established in each test, and the system was then monitored while corrosion and fluid circulation occurred for a time comparable to the ECCS recirculation mission time.

## 1.2. Information Presented in This Report

Information on the ICET project is presented in six volumes. Volume 1 (this report) provides a compilation and comparison of key results from ICET Tests #1–#5. Volumes 2–6 (Refs. 2–6) are the test data reports for Tests #1–#5. The test data reports included detailed test information, and highlights from those data reports were assembled here to illustrate similarities and differences between the tests. Section 2 of this volume contains a brief description of the test apparatus and samples used in the tests. Test procedures, quality assurance, and test parameters are also presented. Section 3 summarizes for each individual test the results for daily water samples, insulation, sediment, and coupons. Section 4 contains comparisons of the test results for water samples, insulation, sediment, metal coupons, aluminum coupon passivation, and deposition products. Section 5 discusses conclusions, and Section 6 itemizes references. Appendix A

contains detailed background information on the test apparatus, test samples, analytical methods and measurements, and quality assurance (QA) program. Appendix B describes materials used in the ICET testing, and Appendix C is the Project Test Plan.

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## 2. TEST PROCEDURES

The functional description and physical attributes of the ICET apparatus are presented in detail in Appendix A. That appendix includes the Project Test Plan requirements, the test apparatus design, analytical methods and measurements, and the project QA program. The experimental apparatus is briefly described here, followed by a summary of the QA program, test parameters, and test operation.

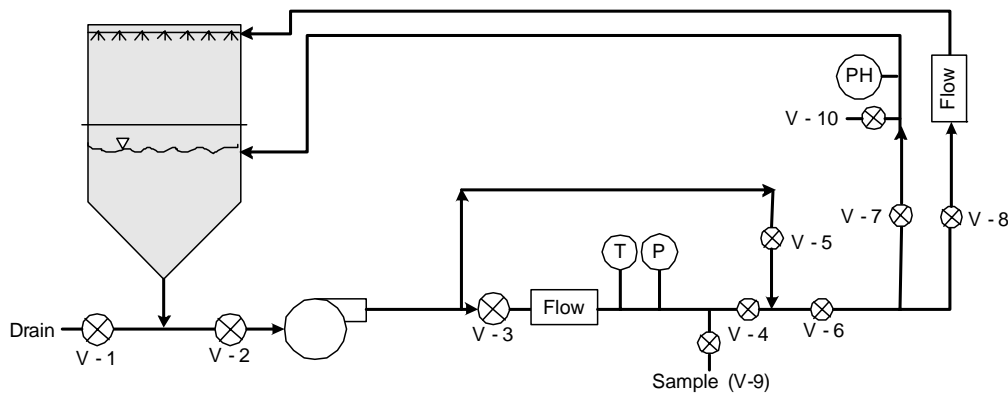
### 2.1. Chemical Test Apparatus Functional Description

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

1. The central component of the system was a test tank. The test apparatus was designed to prevent solids from settling in the test piping.
2. The test tank maintained both a liquid and vapor environment, as would be expected in a post-LOCA containment building.
3. The test loop controlled the liquid temperature at 60°C ( $\pm 3^\circ\text{C}$ ).
4. The system circulated water for spray that simulated volumetric containment spray rates per unit area of containment cross section.
5. The test tank contained submerged test coupons and provided a water flow that was representative of the containment pool fluid velocities expected at plants.
6. Piping and related isolation valves were provided such that a section of piping could be isolated without interrupting a test.
7. The pump discharge line was split along two branches, one branch directing flow to the spray header in the tank vapor space and the other branch returning flow to the liquid pool. Each branch was provided with an isolation valve and a flow meter.
8. The pump circulation flow rate was controlled at the pump discharge to be within  $\pm 5\%$  of the flow required to establish target fluid velocities in the tank. Flow was controlled manually by setting a throttle valve, but it was monitored automatically in a continuous data record.
9. The tank accommodated a rack of immersed sample coupons that included the potential reaction constituents identified in the test plan.
10. The tank also accommodated six racks of sample coupons that were exposed to a spray of liquid that simulated the chemistry of a containment spray system. Provisions were made for these racks to be visually inspected through polycarbonate windows.

11. The chlorinated polyvinyl chloride (CPVC) coupon racks provided sufficient space between the test coupons to minimize electrochemical interactions between the coupons. The different metallic test coupons were also electrically isolated from each other and from the test stand to prevent galvanic effects resulting from metal-to-metal contact either between specimens or between the test tank and the specimens.
12. The fluid dilution volumes and the sample corrosion surface areas were selected based on scaling considerations that relate the test conditions to representative plant conditions.
13. All components of the test loop were made of corrosion-resistant material (for example, SS for metallic components and CPVC for spray injection lines).

The as-built test loop consisted 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. The labels P, T, and pH represent pressure, temperature, and pH probes, respectively. Valves are numbered V-1 through V-10.



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

## 2.2. Pre-Test Preparation

### 2.2.1. Test Loop Cleaning

Before each test, the experimental loop was thoroughly cleaned to remove all deposits and residues remaining from the previous test. In addition to visual inspections, the apparatus was flushed and cleaned per written directions given in the pre-test operations project instruction (PI). The system was repeatedly flushed with dilute concentrations of ammonium hydroxide, followed by ethanol, then by nitric acid, and finally by reverse osmosis (RO) water, until the effluent water turbidity was less than 0.3 NTU and its conductivity was  $<50 \mu\text{S/cm}$ .

## 2.2.2. Test Coupons and Samples

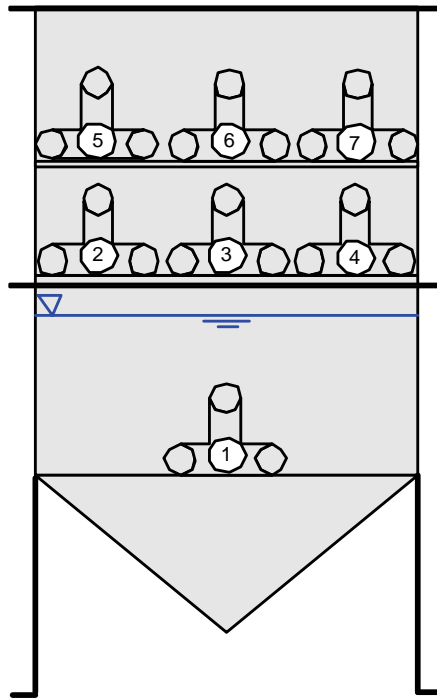
Each ICET experiment exposed an identical set of metallic and concrete coupons to simulated post-LOCA environments. Each coupon was ~12 in. square. The metallic coupons were ~1/16 in. thick, except for the inorganic zinc-coated steel coupons, which were ~3/32 in. thick. The concrete coupons (one per test) were ~1-1/2 in. thick. Insulation materials were also exposed. NUKON™ fiberglass insulation (hereafter referred to as fiberglass) samples were included in each test. Also, calcium silicate insulation (hereafter referred to as cal-sil) samples were included in Tests #3 and #4. Each test subjected seven racks of coupons to the specified environment, with one being submerged in the lower tank and the remaining six being suspended in the tank vapor space. The number of coupons of each type that were used in every test are listed in Table 2-1.

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

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

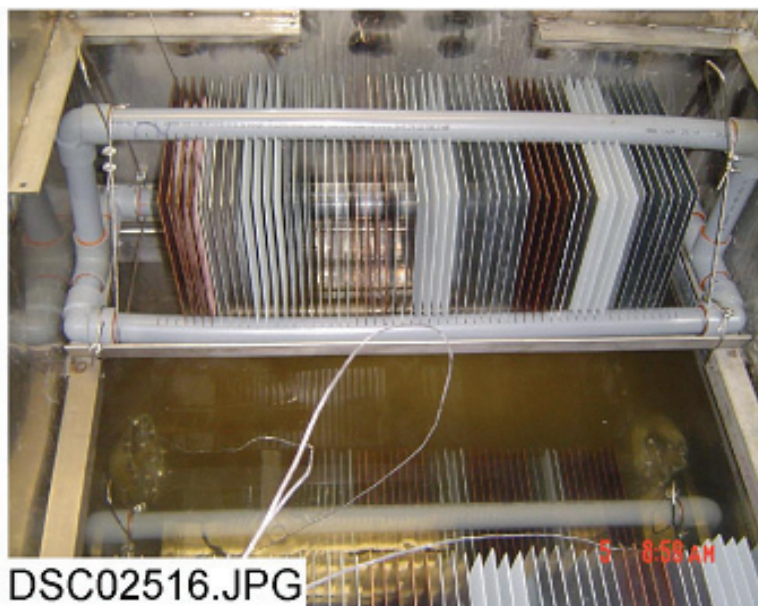
Note: IOZ refers to an inorganic zinc primer coating. IOZ and CS or coated steel (used elsewhere) refer to the same coupon type.

The arrangement of 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 labeled. The normal water level is indicated by the blue line in the figure. Rack 1 was the only submerged rack, and it sat on angle iron supports. It was centered in the tank so that flow from two opposing distribution headers reached it equally. Racks 2–4 were positioned above the water line, supported by angle iron brackets on the side of the tank. Racks 5–7 were positioned at a higher level and were also supported by angle iron brackets. Racks 2–7 were exposed to spray. In Figure 2-2, north is to the right and south is to the left. Compass directions were used subsequently to identify rack locations and sediment sample locations. Appendix A contains more details on the tank internals.



**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 a typical unsubmerged coupon rack loaded with metal coupons in the ICET tank. The loading pattern of all suspended 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: four Cu, four Al, four coated with inorganic zinc (IOZ), seven GS, four Cu, three Al, four IOZ, and seven GS.



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



The submerged rack had a slightly different configuration because of the ratios of submerged versus unsubmerged metals. The sole concrete coupon was also placed in the submerged rack. The Rack 1 coupon configuration was originally eight Cu, one Al, one IOZ, two GS, eight Cu, one Al, one IOZ, two GS, one US, nine Cu, one Al, one IOZ, three GS, eight empty slots, concrete, and then eight empty slots. Some copper deposition on the aluminum coupons was observed following Test #2. Subsequently, two of the three Al coupons were moved to empty slots on either side of the concrete coupon in Tests #3–#5.

Several fiberglass samples were placed in the ICET tank. These samples were either submerged or suspended above the water level. The unsubmerged fiberglass samples were positioned so that they would be exposed to sprays. All fiberglass samples were contained in SS wire mesh bags or “pouches” 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 contained ~5 g of fiberglass. Sacrificial samples were attached with SS wire; removed from the tank on Days 4, 15, and 30; and examined for possible time-dependent changes. As shown in the figure, bigger insulation bags were wrapped around the sacrificial specimens during the test. In addition, small sacrificial samples called fiber pucks were added to the solution beginning with Test #3. Fiber puck fabrication and post-test analyses are described in Ref. 5. Appendix A contains more detailed descriptions of the standard fiberglass samples. Appendix B contains additional details on the fiberglass insulation, including heat treating.



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

Several cal-sil samples were also placed in the ICET tank for Tests #3 and #4. Samples were either submerged or suspended above the water level. The unsubmerged cal-sil samples were positioned so they would be exposed to sprays. In addition to solid cal-sil pieces (Figure 2-5), 43.5 lb of cal-sil dust (Figure 2-6) were added to the tank solution before the test began. 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. Appendix B contains additional details on the cal-sil insulation, including heat treating.



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



**Figure 2-6. Pulverized cal-sil insulation before addition to the ICET tank.**

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

A project QA manual was developed to satisfy the contractual requirements that applied to the ICET project. Specifically, those requirements were to provide credible results by ensuring that the QA effort was consistent with the intent of the appropriate sections of 10CFR50, Appendix B, in the areas of test loop design, sampling, chemicals, operation, and analysis.

The 18 criteria of 10CFR50, Appendix B, were addressed separately in a Project QA Manual, and the extents to which they applied to the ICET project were delineated. A resultant set of QA procedures was developed. In addition, project-specific instructions (PIs) 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 all tests:

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

Project instructions specific to each test were written for the following:

- Pre-Test Operations
- Test Operations
- Post-Test Operations

### **2.2.4. Test Parameters**

ICET test parameters were selected based on literature surveys and on the results of surveys of U.S. 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 to the ICET tank simulated the post-LOCA sump environment. The Project Test Plan (Appendix C) is the basis for the following information in this section.

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

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

Material	Ratio Value (ratio units)	Percentage of Submerged Material (%)	Percentage of Unsubmerged Material (%)
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 that were critical for defining the tank environment and that had a significant effect on sump-flow blockage potential and gel formation are summarized in Tables 2-3 and 2-4, respectively. Note that of the chemical parameters listed, only boric acid, lithium hydroxide, and hydrochloric acid were present in all five tests.

**Table 2-3. Physical Parameters for the ICET Tests**

Physical Parameter	Test Value	
Tank water volume	949 L	250 gal.
Circulation flow	100 L/min	25 gpm
Spray flow	15 L/min	3.5 gpm
Sump temperature	60°C	140°F

**Table 2-4. Chemical Parameters for the ICET Tests**

Chemical	Concentration
H <sub>3</sub> BO <sub>3</sub> concentration	2800 mg/L as boron <sup>a</sup>
Na <sub>3</sub> PO <sub>4</sub> ·12H <sub>2</sub> 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
Sodium tetraborate (Borax)	As required to reach boron concentration of 2400 mg/L in Test #5
HCl concentration	100 mg/L*
LiOH concentration	0.7 mg/L*

<sup>a</sup>Concentrations applicable to Tests #1–#4. Concentrations for Test #5 were 2400 mg/L boron, 43 mg/L HCl, and 0.3 mg/L LiOH.

## 2.3. Test Operation

All of the ICET tests were conducted in environments that attempted to simulate expected containment pool conditions during recirculation. The tests were conducted for 30 days at a constant temperature of 60°C. The materials tested within each environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples. Representative amounts of concrete dust (21.2 g) and latent debris (63.7 g) were also added to the test solution. Each test also had representative amounts of insulation debris present, either as fiberglass or a mixture of fiberglass and cal-sil.

Preparation of the tests began with the heating of approximately 250 gal. of RO water to 65°C, which was 5°C above test temperature. This initial temperature was used to offset the 5°C reduction in water temperature caused by the addition of the metal coupons and insulation samples. The required chemicals for each test, pre-measured latent debris, and concrete dust were added to the heated RO water with the recirculation pump operating to ensure complete dissolution. Then the pump was stopped, and the metal coupons and fiberglass samples were put into the tank. Inserting the coupon racks and insulation samples required approximately 2 hours.

After all required items were added to the tank, base-line grab samples and measurements of the test solution were taken. Time zero of the test started with initiation of the tank sprays, which lasted for 4 hours. During the spray phase, additional chemicals were injected directly into the nozzle spray lines or into the recirculation line. The nozzle spray flow was set at 3.5 gpm. The recirculation flow was set at 25 gpm.

The initial 4-hour spray phase simulated containment spray interaction with the unsubmerged samples. The spray pH was controlled to be <12 for all tests. Water was circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens. Any water loss because of water sample removal and evaporation during the tests was replaced with RO water.

Test solution samples were taken during the first 24 hours of the test and daily thereafter. Water chemistry measurements included pH, turbidity, total suspended solids (TSS), kinematic viscosity, and strain-rate viscosity. Additionally, daily measurements of selected elemental concentrations (plus silica) with inductively coupled plasma-atomic emission spectroscopy (ICP-AES) were performed. Weekly size distributions of suspended particles were also determined. Sampling and analyses were conducted in accordance with approved project instructions.

Samples of fiberglass were taken from the tank at several day intervals for SEM/energy-dispersive spectroscopy (EDS) investigations. At test termination, water, insulation, metallic coupon, and sediment samples were obtained and various parameters investigated.

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### 3. SUMMARY OF TEST RESULTS

This section summarizes the key results obtained from each ICET test. These test summaries are organized for each test, with brief test descriptions that are supplemental to Section 2.3 and four results sections: (1) water samples, (2) insulation (fiberglass and cal-sil), (3) sediment, and (4) metal coupons.

Elemental chemical analyses with ICP-AES were performed similarly for each test. Daily samples were analyzed for aluminum, calcium, copper, iron, magnesium, nickel, silica, sodium, and zinc. Boron, lithium, potassium, lead, and chloride (analyzed using ion chromatography) were evaluated at the beginning of the tests, at the midpoint, and at the end of the tests. In the test summaries that follow, only those elements that were prevalent in solution (except for boron, which had a high concentration in all tests) are discussed. The water samples were totally digested before the ICP-AES measurements and represented the solution plus any precipitate present. Samples were kept at 4°C between the time they were extracted and when they were digested.

The measurements were within the accepted uncertainties of the laboratory quality control (QC) program (see Appendix A, Section A.4), or the analyses were rerun. Each batch of samples had its own specific uncertainty, depending on the results of the laboratory's QC checks. A typical set of measurement uncertainties are for aluminum,  $\pm 20\%$ ; calcium,  $-14\%$  and  $+19\%$ ; sodium,  $\pm 20\%$ ; magnesium,  $-13\%$  and  $+20\%$ ; and silica,  $-9\%$  and  $+12\%$ .

Deposits on the fiberglass samples were of three types: particulates, flocculence, and film or webbing. Particulate deposits were confined to the exteriors of the samples and were physically attached or retained. Flocculent deposits were found throughout the samples and were more prevalent on the fiberglass interior. It is likely that film or webbing deposits that were observed in Tests #1 and #4 were caused by chemical precipitation during the drying process.

#### 3.1. Test #1

ICET Test #1 was conducted using sodium hydroxide to obtain the target pH of 10. Fiberglass was the only type of insulation used in the test. Boric acid, lithium hydroxide, and hydrochloric acid were also added to the test solution.

Preparation of ICET Test #1 (Run 1 in Table 1-1) began with the heating of 200 gal. of RO water to 60°C. Boric acid, sodium hydroxide, lithium hydroxide, and hydrochloric acid were added to the heated RO water, with the recirculation pump operating to ensure complete dissolution. After the chemicals were observed to be well mixed, an additional 50 gal. of RO water were added to reach the required test volume. The solution again was brought to the desired test temperature. During the first 30 minutes of the spray period, additional sodium hydroxide was injected directly into the nozzle spray lines.

The fiberglass and metal coupons were added on the evening of November 20, 2004. Because of the quantity of the metal mass added to the tank, the solution temperature dropped below the desired test range. The test apparatus was held in recirculation mode for ~12 hours, until the next morning, when the temperature had again reached the desired value. Some settling of the added



particulates was observed overnight, with the turbidity decreasing from 12 to 8 NTU over that time.

The experiment commenced at 10:00 a.m. on Sunday, November 21, 2004, and it ended on Tuesday, December 21, 2004, at 10:00 a.m. Details of ICET Test #1 are reported in Ref. 2.

### **3.1.1. Water Samples**

Test #1 had a target pH of 10. The actual test solution pH at the end of the sodium hydroxide injection during the spray phase was 9.5. The pH decreased slightly during the test, and it was ~9.4 at the end of the test.

No chemical byproducts were visible at the test temperature of 60°C. However, microscopic evaluation revealed the presence of small amorphous particulates, which existed in the test solution before cooling. After the first 8 hours of testing, when the test solution was cooled to room temperature, precipitates became visible. The amount of visible room temperature precipitation increased, and the precipitates formed more quickly during cooling as the test progressed. Reheating of precipitated solution samples led to only partial dissolution of the solid back into solution. Additionally, analysis of the precipitates indicated that they were principally amorphous, and the fundamental particle size was ~10 nm. Much larger agglomerations of these basic particles were also visible.

Turbidity was measured at two temperatures: 60°C and 23°C. At 60°C, the turbidity remained relatively constant at around 1 NTU after 3 days of testing. At 23°C, the turbidity began rising at Day 3 to ~135 NTU at the end of the test.

The TSS increased from the base-line measurement of 27 mg/L to a peak concentration of 43 mg/L after the 30-minute injection of the additional sodium hydroxide. The baseline TSS sample was taken after the addition of the debris to the tank solution. By the end of the first day, the TSS measurement had declined to a value similar to that of the base-line measurement. For the first 13 days of testing, the TSS measurement oscillated within the range of 13–24 mg/L. On the 14th day, the TSS measurement increased notably above the previous range to a value of 29 mg/L. From that point, the TSS measurement fluctuated within the range of 20–29 mg/L.

The test solution was filtered through a 0.7- $\mu$ m filter, and the particle size distribution was determined. After Day 1, the particulates remained <1  $\mu$ m for the duration of the test.

Bulk kinematic viscosity values at 60°C remained steady throughout the test. However, at 23°C the kinematic viscosity began to increase after 5 days of testing and leveled out after ~20 days of testing. Strain-rate viscosity testing clearly demonstrated thinning or reduced viscosity with increasing strain rate for the 25°C samples (strain-rate viscosity was measured at 60°C and 25°C) after Day 23, which is indicative of non-Newtonian behavior. No strain-rate dependency was observed for the 60°C solution.

Results from ICP-AES analyses indicated that aluminum and sodium were present in the test solution in the greatest concentrations. The sodium concentration was ~2000 mg/L at the start of the test and rose to ~4500 mg/L after 3 days of testing. It stayed at that level for the duration of



the test. The aluminum concentration was near zero at the start of the test. It rose to 350 mg/L after ~20 days of testing and then evened out until the end of the test, similar to the behavior of the kinematic viscosity. The aluminum concentration in solution corresponds well with the mass lost from the submerged aluminum coupons.

### **3.1.2. Insulation**

Fiberglass insulation samples were removed from the test tank on Day 15 and Day 30 of the test and examined. The Day-15 samples exhibited evidence of particulate deposits on individual fibers and a web-like material spanning multiple fibers. The Day-30 samples exhibited similar substances that were more prevalent, and contiguous webbing appeared to span multiple fibers. The particulate deposits were observed on only the exterior surfaces of the insulation. The penetration depth spanned a few fiber diameters below the surface. Insulation within the remaining portion of the insulation holder bags was not subject to these deposits, and these fibers appeared relatively pristine after the test.

### **3.1.3. Sediment**

A total of 292 g (wet) of sediment were recovered from the tank bottom after the tank was drained. Photographs and SEM images showed that a large portion of the sediment was composed of fiberglass debris. Elemental composition analysis of the sediment indicated that silicon was the dominant element (27% by mass). This information is consistent with the fact that fiberglass was the only insulation material used in the test. Sodium was the next most prominent element (9% by mass). It should be noted that these compositions are somewhat qualitative because of the difficulty in obtaining an accurate spatial average using a 25- $\mu$ m beam spot.

The latent debris and concrete dust quantities added just before the onset of testing amounted to ~85 g. The additional amount of sediment appeared to be made up of fugitive insulation material that escaped from the insulation sample bags during testing. Generally, the sediment was found to contain no visible chemical byproducts that were similar to the web-like material present in the fibrous insulation. Although the sediment settled to the bottom of the tank during the test, subsequent settling tests revealed that the sediment was easily re-suspended and required a substantial amount of time (hours to days) before completely settling in a quiescent container.

### **3.1.4. Coupons**

The coupons were weighed after drying, and no cleaning was performed before weighing. Therefore, post-test weights included any corrosion products or surface deposits that were present on the test coupons. Weight loss/gain measurements indicated that only the aluminum specimens experienced a significant weight change. The aluminum specimens lost ~25% of their pre-test mass. The carbon steel sample lost ~2% of its pre-test mass, whereas the concrete sample gained ~3% more mass. The concrete weight gain may have resulted from retained water, since the weight measurement was performed after several days of air drying. Other samples either gained or lost much less than 1% of their pre-test mass.

## **3.2. Test #2**

ICET Test #2 was conducted using TSP as the buffering agent, with a target pH of 7. Fiberglass was the only type of insulation used in the test. Boric acid, lithium hydroxide, and hydrochloric acid were also added to the test solution.

The test began with 240 gal. of RO water and chemicals in the system. Beginning at 30 minutes into the spray phase, two batches of the TSP solution were metered into the recirculation line. Each batch consisted of boric acid and TSP in 5 gal. of RO water.

The experiment commenced at 9:45 a.m. on Saturday, February 5, 2005, and it ended on Monday, March 7, 2005. Details of ICET Test #2 are reported in Ref. 3.

### **3.2.1. Water Samples**

Test #2 had a target pH of 7. After the spray phase and during the first week of testing, the test solution pH averaged 7.2. There was a slight increase in the average pH to a value of 7.3 during the second week. The pH remained constant over the final 2 weeks of the test.

No chemical byproducts were visible in the daily water samples at the test temperature of 60°C or upon cooling to 23°C. Additionally, no precipitates or byproducts became visible as the samples were stored at room temperature after the end of the test.

Turbidity values at both test and room temperatures were very similar; thus, only the 60°C value is explained further. A sharp rise in water sample turbidity occurred after 4 hours, which coincided with the completed introduction of TSP. The maximum turbidity value at both test and room temperatures was reached within 1 day of testing. That value was 20 NTU when the test solution was at 60°C. The turbidity then gradually decreased to a level of 1 NTU by the sixth day of the test. Thereafter, the turbidity remained constant until the end of the test.

The TSS values exhibited similar trends. The TSS value was a maximum (36.2 mg/L) at the end of the 4-hour spray cycle, which coincided with the completed addition of TSP. After the first 24 hours of the test, the TSS sample dropped to 27.5 mg/L and continued to decrease to 10 mg/L by Day 5 of the test. The TSS concentration was constant at that point until the end of the test.

Measurements of water-sample particulate sizes indicated that 80%–90% of the particulates were between 50 and 100  $\mu\text{m}$  during the first 2 days of testing. Measurements of weekly samples thereafter indicated that the suspended particulates were between 1 and 25  $\mu\text{m}$ .

Kinematic viscosity measurements at 60°C and 23°C were constant during the entire test. Strain-rate viscosity measurements indicated that the water samples exhibited common Newtonian behavior, where the shear stress is directly proportional to the strain rate.

ICP-AES results indicated that the elements present in the highest concentrations were silica and sodium. The silica concentration at the beginning of the test was ~3 mg/L; it then increased nearly linearly to 85 mg/L by Day 19 of the test. The silica concentration then remained constant through the end of the test. The initial sodium concentration (after TSP injection) averaged

~900 mg/L during the entire test, but the sodium concentration measurements fluctuated between values of 700 and 1000 mg/L during the course of the test.

### **3.2.2. Insulation**

Fiberglass insulation samples were removed from the test tank on Days 4, 16, and 30 of the test and analyzed. Flocculent deposits were observed in the Day-4 fiberglass samples. The amount of the deposits increased with the test duration. At the end of the test, these deposits were dispersed throughout the insulation samples. However, the amount of deposits was greater on the exterior than on the interior of the samples. The fibers were not encrusted, and the deposits often appeared near the intersection of several fibers.

### **3.2.3. Sediment**

A total of 256 g (wet) of sediment was recovered from the tank bottom after the tank was drained. A large portion of the sediment consisted of fugitive fiberglass debris, and the sediment appeared to be heterogeneous throughout. Elemental composition analysis of the sediment indicated that silicon was the dominant element (18% by mass). This is consistent with the fact that fiberglass was the only insulation material used in the test. Aluminum was the next most prominent element (9% by mass), followed by phosphorus (4% by mass).

### **3.2.4. Coupons**

A copper layer was evident on the submerged aluminum samples. The layer can be attributed to electrochemical ion transfer. Large, white deposits also covered the submerged aluminum coupons. The samples were weighed after drying, and no cleaning was performed before weighing. No significant weight loss or gain occurred except for the submerged galvanized steel coupons and concrete coupon, which gained approximately 3% of their pre-test weights.

## **3.3. Test #3**

ICET Test #3 was conducted using TSP as the buffering agent, with a target pH of 7. A combination of 20% fiberglass and 80% cal-sil (by volume) was the insulation mixture used in the test. Boric acid, lithium hydroxide, and hydrochloric acid were also added to the test solution.

The test began with 240 gal. of RO water and chemicals in the system. Beginning at 30 minutes into the spray phase, two batches of the TSP solution were metered into the recirculation line. Each batch consisted of boric acid and TSP in 5 gal. of RO water. The second batch also included the pre-determined amount of HCl needed for the test.

The experiment commenced at 3:30 p.m. on Tuesday, April 5, 2005, and it ended on May 5, 2005. Details of ICET Test #3 are reported in Ref. 4.

### **3.3.1. Water Samples**

The target system pH was 7. The TSP injection was completed by the end of the spray phase, and the test solution pH was 7.3 at that time. The pH increased to a value of 7.9 by the third day of

testing. After Day 3, the average pH values increased slightly to a value of ~8.0 and remained there until the end of the test.

During the first few hours of the test, precipitates were visible through the tank's viewing window. Otherwise, no chemical byproducts were visible in the daily water samples at the test temperature of 60°C or upon cooling to room temperature. Additionally, no precipitates or byproducts became visible after the samples were stored at room temperature after the end of the test. The ICP-AES analyses of daily water samples did not provide sufficient time resolution to quantify the rapid transient at the beginning of the test.

Turbidity measurements at both test and room temperatures were very similar; thus, only the 60°C values are explained. At test initiation, the turbidity measured 54 NTU, which is attributable to the addition of cal-sil dust. Approximately 1 hour into the test, the turbidity had increased significantly (>200 NTU). By the end of the 4-hour spray phase, the turbidity decreased to 79 NTU. The turbidity continued to decrease, and by the end of the first day, the turbidity fell below 1 NTU and remained near that value for the remainder of the test.

The TSS values exhibited trends similar to those of the turbidity. During the TSP injection, white flocculent particles were visible within the solution. The TSS concentration rose from its baseline value of ~10 mg/L to 268 mg/L midway through the TSP injection. At the end of the 4-hour spray cycle, the concentration had decreased to 73 mg/L. After the first 24 hours of the test, the TSS concentration had dropped to 14 mg/L and remained in the 10–20 mg/L range for the remainder of the test.

Measurements of water-sample particulate sizes indicated that they were relatively unchanged during the test. Measurements of weekly samples indicated that the suspended particulate sizes were between 1 and 50 µm.

Kinematic viscosity measurements at 60°C and 23°C were constant during the entire test. Strain-rate viscosity measurements indicated that the water samples exhibited common Newtonian behavior.

ICP-AES results indicated that the elements present in the highest concentrations were silica, sodium, and calcium. The silica concentration increased quickly to a value of ~100 mg/L after the first day of testing and then remained relatively constant throughout the next 20 days. Starting at Day 20, the silica concentration appeared to decrease slowly, but it remained >82 mg/L by the end of the test. The sodium concentration (after TSP injection) averaged ~1090 mg/L during the entire test, but the sodium concentration measurements fluctuated between values of 900 mg/L and 1620 mg/L. The calcium concentration gradually increased asymptotically from a baseline value of 3 mg/L to a Day-30 value of 109 mg/L.

### **3.3.2. Insulation**

ICET Test #3 had the greatest amount of deposits on the fiberglass of all the ICET tests. In general, the amount of deposits increased as the test progressed, and the greatest amount of deposits was found on the Day-30 exterior samples. The deposits on these samples included particles that had likely been physically captured or attached. Based on EDS analysis, the

particulate deposits on the fiberglass exterior may be classified into two categories according to phosphorous (P) and silicon (Si) content. Particulate deposits of lower P and higher Si content were likely cal-sil particles. The particulate deposits with lower Si and high P, Ca, and O content were likely composed of calcium phosphate precipitates (specific phase was not identified), which explains the composition of the white gel-like cream deposit found on the tank bottom. Calcium phosphate precipitated when pre-dissolved calcium from the cal-sil reacted with phosphate from the TSP. After precipitation, the calcium phosphate was transported to the fiberglass exterior by sedimentation and flowing water. Both kinds of deposits (cal-sil and calcium phosphate precipitates) may be physically transported and/or deposited onto the fiberglass sample exterior in this manner.

In contrast to the exterior regions of the fiberglass, the interior fiberglass samples were relatively pristine, with few particulate deposits. However, the interior samples did contain significant flocculent deposits. EDS analysis indicated that the flocculent deposits contained insignificant amounts of P, meaning that the deposits were not related to the white gel-like cream deposit.

Cal-sil made up 80% by volume of the insulation in Test #3. A Day-30 baked cal-sil sample that had been submerged in the high-flow zone and a Day-30 unbaked cal-sil sample that had been submerged in the birdcage were examined. The exterior surface of the baked cal-sil sample from the high-flow region was examined by environmental scanning electron microscopy (ESEM) and EDS. These results showed a significant amount of phosphorus. Smaller amounts of phosphorus were found on the exterior of the unbaked cal-sil in the birdcage sample. However, almost no phosphorus was present in the interior of the unbaked cal-sil.

### **3.3.3. Sediment**

A large quantity (over 78 kg wet) of sediment was recovered from the bottom of the tank after it was drained. The sediment was ~9 in. deep in some places on the bottom of the test tank after the test. The main reason for the large amount recovered was that ~20 kg of the cal-sil added was in the form of dust. The sediment was similar in consistency to wet mud and was pink and yellow, consistent with the baked and unbaked cal-sil that was originally added to the tank. Calcium was the dominant element present (19% by mass), followed by silicon (17% by mass). Although dominated by cal-sil, the sediment was also likely composed of fiberglass debris and corrosion products. Though an accurate mass comparison is difficult because of the large amount of water retained in the recovered sediment, it is believed that a significant fraction of the sediment mass was composed of secondary chemical precipitates.

Test #3 was unique in that a pinkish-white, gel-like deposit was found after the tank was drained. That deposit had the consistency of face cream and covered portions of the sediment and submerged objects on the tank bottom. No effort was made to isolate all of the gel-like deposit. However, 500 mL were collected separately for analysis. EDS results showed that 92% of the deposit was composed of calcium, oxygen, and phosphorus. Implications of this gel-like material are discussed in Ref. 7.

### **3.3.4. Coupons**

All of the submerged coupons had white particulate deposits on them at the end of the test. As in Test #2, a copper layer was evident on the submerged aluminum samples. The galvanized steel coupons had an average weight gain of 15 g, and the others had weight changes of less than 2 g. The concrete coupon was covered with a white particulate and gained ~3% of its pre-test weight. Overall, the unsubmerged coupons had similar streaking deposits and weight changes of 2 g or less.

## **3.4. Test #4**

ICET Test #4 was conducted using sodium hydroxide to obtain the target pH of 10. A combination of 20% fiberglass and 80% cal-sil (by volume) was the insulation mixture used in the test. Boric acid, lithium hydroxide, and hydrochloric acid were also added to the test solution.

The test began with 249 gal. of RO water and chemicals in the system. Over the first 30 minutes of the test, 1 gal. of RO water containing 614 g of sodium hydroxide was metered into the spray. The test ran for 30 days, with one 2.25-hour interruption caused by a power outage, which occurred on Day 11.

The experiment commenced at 2:30 p.m. on Tuesday, May 24, 2005, and ended on June 23, 2005. Details of ICET Test #4 are reported in Ref. 5.

### **3.4.1. Water Samples**

The target pH for this test was 10, and the actual test solution pH was approximately 9.8 at the end of the sodium hydroxide injection into the spray. The pH of the test solution decreased slightly over the first 2 days of the test to a value of 9.6. After the second day, the pH rose steadily to a value of 9.9 by Day 8. Thereafter, the pH varied between 9.7 and 9.9 over the remainder of the test, and was ~9.8 at the end of the test.

No chemical byproducts were visible in the daily water samples at the test temperature of 60°C or upon cooling to room temperature. Additionally, no precipitates or byproducts became visible after the samples were stored at room temperature following the test.

Turbidity measurements at both test and room temperatures were very similar; thus, only the 60°C values are explained. The turbidity value at the start of the test was high (130 NTU), because of the addition of cal-sil before initiation of the test. Turbidity steadily decreased during the 4-hour spray phase and reached a value of 36 NTU by the end of this phase. The turbidity continued to decrease and, by the end of the first day, fell below 3 NTU and remained largely constant over the remainder of the test.

TSS values generally exhibited trends similar to the turbidity trends. At the start of the test, the TSS was 129 mg/L, which was the highest measured value. At the end of the 4-hour spray cycle, the concentration had decreased to 68 mg/L. After the first 24 hours of the test, the TSS concentration had dropped below 40 mg/L and remained roughly constant during the remainder

of the test. The TSS varied between 29 and 45 mg/L from Day 1 to Day 30, with an average value of ~35 mg/L.

Measurements of water-sample particulate sizes indicated that they were relatively unchanged during the test. Measurements of weekly samples indicated that the particulate sizes were between 1 and 50  $\mu\text{m}$ .

Kinematic viscosity measurements at 60°C and 23°C were constant during the entire test. Strain-rate viscosity measurements indicated that the water samples exhibited common Newtonian behavior.

ICP-AES results indicated that the elements present in the highest concentrations were silica, sodium, calcium, and potassium. The silica concentration increased quickly to a value of ~100 mg/L after the first day of testing and then continued to increase steadily until Day 15, when the concentration was ~170 mg/L. The silica concentration was approximately constant from Day 15 to the end of the test, but individual concentration measurements varied between ~160 and 190 mg/L. The sodium concentration at the start of the test was ~6,000 mg/L and increased fairly steadily over the course of the 30-day test. By Day 17, the sodium concentration variability had increased significantly from day to day. The calcium concentration rose from a 4-hour value of 37 mg/L to ~70 mg/L over the first 5 days of testing. After Day 5, the calcium concentration remained relatively constant for the duration of the test. The potassium concentration was measured five times throughout the duration of the test, and it rose from a baseline value of 7 mg/L to 35 mg/L at the end of the test.

### **3.4.2. Insulation**

The ICET Test #4 fiberglass contained a large amount of deposits. Fiberglass samples exhibited deposits throughout the fiber matrix after they had been exposed in the test solution for 30 days. Particulate deposits were generally found only on the exterior of the fiberglass and were likely physically retained or attached on the fiberglass exterior. EDS analyses indicated that these particulate deposits contained significant amounts of silica and calcium, suggesting they were from cal-sil debris. In contrast, the interior of the fiberglass samples was relatively clean, and only film deposits were observed. To investigate the formation of the film deposits, controlled experiments were performed by gently rinsing the interior fiberglass samples with several drops of RO water before ESEM analysis. After being rinsed with RO water, the film deposits disappeared from the fiberglass samples. These results suggest that the film was actually soluble, which is consistent with the explanation that the film was deposited during the drying process of the fiberglass.

As with Test #3, cal-sil made up 80% by volume of the insulation. However, the significant sodium concentration that was present in solution as a dissolved ion throughout the duration of the test (unlike phosphate in Test #3, which was quickly depleted) was able to diffuse to the interior of the cal-sil (the cal-sil was essentially soaked in a sodium solution for 30 days). Thus, it appears in relatively equal amounts in the interior and exterior cal-sil samples.

### **3.4.3. Sediment**

A large quantity (>86 kg wet) of sediment was recovered from the bottom of the tank after it was drained. The sediment was ~9 in. deep in some places on the bottom of the test tank. The main reason that the amount recovered was so large is that 80% of the fiberglass was replaced by cal-sil insulation, with ~20 kg being in the form of cal-sil dust. The sediment was similar in consistency to wet mud and was pink and yellow, consistent with the baked and unbaked cal-sil that was originally added to the tank. Calcium was the dominant element present in the sediment (20% by mass), followed by silicon (16% by mass). Although dominated by cal-sil, the sediment also contained fiberglass debris and corrosion products. No secondary chemical precipitates were observed during Test #4 that would increase the quantity of sediment. The larger amount of recovered mass from Test #4 compared to Test #3 is indicative of the uncertainty inherent to a bulk mass balance on the ICET tank. The difference in sediment masses may be because the Test #4 sediment was recovered sooner after the tank was drained, and it contained higher water content.

### **3.4.4. Coupons**

The unsubmerged zinc, aluminum, US, and copper samples exhibited evidence of corrosion upon removal from the test chamber. No significant corrosion products were observed on any of the metallic samples contained within the submerged coupon rack. Pre- and post-test weight measurements revealed insignificant changes (<1 g) in the bulk weight of the submerged and unsubmerged metallic coupons. A faint, white film was present on the submerged aluminum and zinc coupons. The film was much less than the corrosion products evident on the unsubmerged specimens. Test #4 was unique in the relatively inert response of the submerged coupons to the high-pH environment. Section 4.4.3 provides an hypothesis for the observed behavior.

## **3.5. Test #5**

ICET Test #5 was conducted using sodium tetraborate (borax) as the buffering agent, with an expected pH of 8–8.5. Fiberglass was the only type of insulation used in the test. Boric acid, lithium hydroxide, and hydrochloric acid were also added to the test solution.

The test began with 248 gal. of RO water and chemicals in the system. After 2 hours, 2 gal. of RO water containing 90.8 mL of hydrochloric acid were metered into the spray.

The experiment commenced at 11:00 a.m. on Tuesday, July 26, 2005, and ended on August 25, 2005. Details of ICET Test #5 are reported in Ref. 6.

### **3.5.1. Water Samples**

A target pH range of 8–8.5 was specified for the onset of this test. The test solution pH was 8.4 before initiation of the sprays, after the initial chemical environment had been established. The pH of the test solution decreased slightly during the last 2 hours of the spray phase to a value of ~8.3 because of the hydrochloric acid addition. The pH was 8.2–8.3 over the first 20 days of the test, and it was between 8.2 and 8.5 over the final 10 days of the test.



No chemical byproducts were visible at the test temperature of 60°C during this test. At room temperature, precipitates became visible at the bottom of the water samples, beginning with the Day-2 sample. The amount of visible room temperature precipitation appeared to increase only slightly in subsequent daily water samples. It should be noted that it required several days at room temperature before the precipitates were visible on the bottom of the sample bottles. The total amount of precipitates in the Day-30 water sample barely covered the bottom of the 250-mL sample bottles. When the precipitates were suspended upon shaking, they were no longer visible, and it required ~2–3 days before they settled back to the bottom of the sample bottles.

Test #5 solution (120 gal.) was archived at 60°C in constant-temperature ovens at the end of the test. After ~2 months of aging, similar small quantities of precipitates were visible at the bottom of the storage containers. Furthermore, it was found that when this test solution was decanted, its rapid cooling to ~40°C could quickly produce small amounts of visible precipitates. These findings are documented in Ref. 8.

Turbidity values at 60°C and 23°C were similar until Day 7. The turbidity values taken before the latent debris and concrete dust were added were less than 1 NTU. After addition of the latent debris and concrete dust samples, the turbidity increased to 14 NTU at the start of the test. During the 4-hour spray phase, the turbidity slowly decreased to 12 NTU at the end of the spray phase. As the test continued, the turbidity measurements at both temperatures continued to decrease and were <1 NTU by Day 8. The 60°C values generally remained below 1 NTU for the remainder of the test and had an average value of 0.7 NTU between Days 8 and 30. Beginning with the Day 8 measurement, the 23°C values began to deviate from, and were slightly higher than, the 60°C values. Between Days 12 and 14, the 23°C turbidity began to increase and reached a value of ~5 NTU by Day 18, where it remained for the remainder of the test.

The baseline TSS before the initiation of sprays was 16 mg/L. At the conclusion of the spray phase, the TSS value was ~27 mg/L. TSS then decreased until Day 5, when it was ~17 mg/L. From Days 5 through 9 of the test, TSS remained approximately constant, with an average value of 17 mg/L. On Day 10 it increased to a value of 25 mg/L. Between Days 10 and 21, the average TSS value was >20 mg/L, but the results were more variable. Individual measurements ranged from a low of 16 mg/L to a high of 32 mg/L during this time frame. After Day 22, the TSS measurements decreased to ~15 mg/L and remained approximately constant over the duration of the test.

Measurements of water-sample particulate sizes indicated that they were between 1 and 50 µm during the first 3 days of testing. Measurements of weekly samples indicated that the particulates were <1 µm until the last measurement, when 90% of the particulates were between 25 and 50 µm.

The bulk kinematic viscosity measurements at 60°C remained constant during the test. The 23°C measurements remained constant over the first 15–17 days of the test. Between Days 16 and 18, the 23°C viscosity increased slightly to ~0.96 mm<sup>2</sup>/second and remained constant until the end of the test. Strain-rate viscosity testing clearly demonstrated that the viscosity varied with the strain

rate for the 25°C samples, which is indicative of non-Newtonian behavior. No strain-rate dependency was observed for the 60°C solution.

ICP-AES results indicated that the elements present in the highest concentrations were sodium, aluminum, calcium, and silica. The sodium concentration was ~1200 mg/L between Days 1 and 17 of the test. On Day 18, the sodium concentration increased to 1400 mg/L and remained at this value through Day 20. Between Days 20 and 30, the sodium concentration gradually decreased to a value of ~1200 mg/L. The aluminum concentration was initially minimal and then rose to ~55 mg/L after ~12 days of testing. The Day 15 measurement indicated a sudden drop in the aluminum concentration to ~45 mg/L. The aluminum concentration remained below 50 mg/L between Days 15 and 20. The Day 21 measurement indicated that the aluminum concentration had dropped again to 37 mg/L. Beginning at the Day 23 measurement, the aluminum concentration rose from 35 to 45 mg/L by the end of the test.

The calcium concentration initially was minimal, but it rose to a value of >15 mg/L by Day 2 and reached 20 mg/L by Day 6. The calcium concentration peaked by Day 11 at ~22 mg/L and at Day 15 decreased slightly to 20 mg/L. Between Days 15 and 24, the concentration remained approximately constant at 20 mg/L. The Day-25 measurement indicated that calcium had increased to 30 mg/L, and it remained above this value for the remainder of the test. The initial silica concentration at test initiation was ~6 mg/L; it rose to 10 mg/L by the end of the spray phase and peaked at 12 mg/L by Day 3 of the test. The concentration slowly decreased after Day 3 until it reached a value of 7 mg/L by Day 22. The silica concentration remained <8 mg/L between Days 22 and 30.

### **3.5.2. Insulation**

ICET Test #5 had the smallest amount of deposits in the fiberglass of any of the ICET tests. Day-30 high-flow fiberglass exterior samples were relatively pristine, and no significant particulate deposits were found. The lack of deposits is likely because of the small amount of suspended particles in the test solution. The interior samples were also relatively pristine, and only flocculent deposits were observed. The flocculent deposits were primarily composed of oxygen, sodium, calcium, magnesium, aluminum, and possibly silicon.

### **3.5.3. Sediment**

A very small quantity of sediment (only 89 g wet) was found on the test tank floor after draining. For reference, that is only slightly higher than the 85 g of concrete dust and latent debris that were added prior to the test initiation. The sediment consisted of fiberglass debris and particulate deposits. Silicon made up 30% by mass of the sediment, and sodium was the next most prominent at 7%.

### **3.5.4. Coupons**

The samples were weighed after drying, and no cleaning was performed before weighing. Weight loss/gain measurements indicated that only the submerged aluminum specimens experienced a significant weight change. The submerged aluminum specimens lost ~5% of their pre-test mass. The concrete sample gained 226 g (3% of its pre-test mass). The concrete weight

gain may have resulted from retained water, since the weight measurement was performed after several days of air drying. Other samples either gained or lost <1% of their pre-test mass.

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