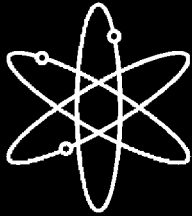




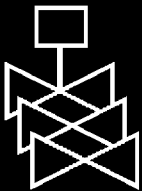
Integrated Chemical Effects Test Project: Test #4 Data Report



Los Alamos National Laboratory



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INTEGRATED CHEMICAL EFFECTS TEST PROJECT: TEST #4 DATA REPORT

ABSTRACT

A 30-day test was conducted in the Integrated Chemical Effects Test (ICET) project test apparatus. The test simulated the chemical environment present inside a pressurized water reactor containment water pool after a loss-of-coolant accident. The initial chemical environment contained 15.41 kg of boric acid, 8.47 kg of sodium hydroxide, 0.663 g of lithium hydroxide, and 212 mL of hydrochloric acid. An additional amount of sodium hydroxide (614 g) was added with the spray beginning at time zero and lasting until 30 minutes into the test. The test was conducted for 30 days at a constant temperature of 60°C (140°F). The materials tested within this environment included representative amounts of submerged and unsubmerged aluminum, copper, concrete, zinc, carbon steel, and insulation samples (80% calcium silicate and 20% fiberglass). Representative amounts of concrete dust and latent debris were also added to the test solution. Water was circulated through the bottom portion of the test chamber during the entire test to achieve representative flow rates over the submerged specimens. The test solution pH varied from 9.5 to 9.8 over the first two days, rose to 9.9 on Day 8, and then stayed between 9.7 and 9.9 for the remainder of the test. The test solution turbidity decreased to less than 3 nephelometric turbidity units (NTU) after 24 hours. The turbidity continued to decrease and averaged 0.5 NTU over the last three weeks of the test. Observations of the test solution indicated that no chemical byproducts were visible in the water and no precipitation occurred as samples cooled from test temperature to room temperature. The appearance of the submerged metallic coupons was largely unchanged throughout the test. Post-test examinations showed very little weight changes and only minimal visible deposits on some coupons. The unsubmerged coupons exhibited some streaking, but little or no weight changes. The bottom of the tank was filled with reddish-brown sediment, but no gel-like deposits were present. The test solution remained clearly Newtonian for the entire test. Aluminum was detectable in the solution for only the first 24 hours.

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INTEGRATED CHEMICAL EFFECTS TEST PROJECT: TEST #4 DATA REPORT

EXECUTIVE SUMMARY

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

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

This report describes the ICET experimental apparatus and surveys the principal findings of Test #4. This interim data report summarizes both primary and representative findings that were available at the time the report was prepared. The NRC and the nuclear power industry may conduct additional analyses to enhance the understandings obtained from this test.

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

ICET Test #4 was conducted using sodium hydroxide to control pH, with a target pH of 10. Insulation samples consisted of scaled amounts of NUKON™ fiberglass and calcium silicate (cal-sil) material. In addition, the test apparatus contained 373 metal coupon samples and 1 concrete sample. Process control consisted of monitoring online measurements of recirculation flow rate, test solution temperature, and pH. Flow rate and temperature were controlled to maintain the desired values of 25 gpm and 140°F. Daily water samples were obtained for measurements of pH, turbidity, total suspended solids, kinematic viscosity, and shear-dependent

viscosity and for analytical laboratory evaluations of the chemical elements present. In addition, microscopic evaluations were conducted on water sample filtrates, fiberglass, cal-sil, coupons, and sediment.

Before time zero, 15.14 kg of boric acid, 0.663 g of lithium hydroxide, 8.47 kg of sodium hydroxide, and 212 mL of 12.29 N hydrochloric acid were dissolved into the ICET tank. The measured pH was 9.5, which was the expected value obtained from analytical predictions. An additional 614 g of sodium hydroxide were added through the spray line for the first 30 minutes of the test. The pH rose to a value of 9.8 at the end of the spray cycle.

The test ran for 30 days, and all conditions were maintained within the accepted flow and temperature ranges, with two exceptions. On Day 11, a power outage occurred which caused a recirculation pump trip. Recirculation flow was re-established 2 hours and 15 minutes later. During that time, the maximum solution temperature rose to 62.4°C, which is 0.4°C above the target maximum. The maximum temperature was above 62.0°C for approximately one hour.

Observations of the test solution indicated similar behavior of the solution at both room temperature and test temperature. After the initial cloudiness caused by the cal-sil dust had cleared up, no chemical byproducts were visible in the water and no precipitation occurred as samples cooled from test temperature to room temperature.

Analyses of the test solution revealed aluminum in the solution, but only for the first 24 hours and at trace amounts. Calcium, silica, and sodium were prevalent in the solution.

Examinations of fiberglass taken from the test apparatus revealed chemical products and a film that spanned individual fibers. Some coating was also observed on individual fibers. The amounts of these deposits did not increase significantly from Day 5 until Day 30. The coating was likely formed by chemical precipitation. In addition to films and coatings, samples in high-flow regions had significant amounts of particulate deposits that were likely physically attached.

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

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

ACKNOWLEDGMENTS

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ACRONYMS AND ABBREVIATIONS

| | |
|---------|---|
| Al | Aluminum |
| ASTM | American Society for Testing and Materials |
| Au | Gold |
| cal-sil | Calcium Silicate |
| CPVC | Chlorinated Polyvinyl Chloride |
| CS | Coated Steel |
| Cu | Copper |
| DAS | Data Acquisition System |
| DHR | Decay Heat Removal |
| ECCS | Emergency Core-Cooling System |
| EDS | Energy-Dispersive Spectroscopy |
| EPA | Environmental Protection Agency |
| ESEM | Environmental Scanning Electron Microscopy |
| F | Filtered |
| GS | Galvanized Steel |
| GSI | Generic Safety Issue |
| HCl | Hydrochloric Acid |
| HI | High-Flow Interior Fiberglass Sample |
| HX | High-Flow Exterior Fiberglass Sample |
| ICET | Integrated Chemical Effects Tests |
| ICP | Inductively Coupled Plasma |
| ICP-AES | Inductively Coupled Plasma—Atomic Emission Spectroscopy |
| IOZ | Inorganic Zinc |
| LANL | Los Alamos National Laboratory |
| LCS | Laboratory Control Sample |
| LI | Low-Flow Interior Fiberglass Sample |
| LiOH | Lithium Hydroxide |
| LOCA | Loss-of-Coolant Accident |
| LX | Low-Flow Exterior Fiberglass Sample |
| MOU | Memorandum of Understanding |
| MSDS | Material Safety Data Sheet |
| NIST | National Institute of Standards and Technology |
| NRC | Nuclear Regulatory Commission |

| | |
|-----|---|
| NTU | Nephelometric Turbidity Unit |
| P | Phosphorous |
| Pd | Palladium |
| PI | Project Instruction or Principal Investigator |
| PVC | Polyvinyl Chloride |
| PWR | Pressurized Water Reactor |
| QA | Quality Assurance |
| RO | Reverse Osmosis |
| SEM | Scanning Electron Microscopy |
| SS | Stainless Steel |
| TEM | Transmissive Electron Microscopy |
| TOC | Total Organic Carbon |
| TSP | Trisodium Phosphate |
| TSS | Total Suspended Solids |
| U | Unfiltered |
| UNM | University of New Mexico |
| US | Uncoated Steel |
| VFD | Variable Frequency Drive |
| WD | Working Distance |
| XRD | X-Ray Diffraction |
| XRF | X-Ray Fluorescence |

1. INTRODUCTION

The Integrated Chemical Effects Test (ICET) project represents a joint effort by the United States Nuclear Regulatory Commission (NRC) and the nuclear utility industry to simulate the chemical environment present inside a containment 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 is being conducted by Los Alamos National Laboratory (LANL) at the University of New Mexico (UNM), with the assistance of professors and students in the civil engineering department.

1.1. Objective and Test Conditions

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

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

The ICET series was conceived as a limited-scope suite of five different 30-day tests with different constituents. The conditions selected for each test are shown in Table 1-1. As shown in this table, Test #4 of the ICET series was to be operated at a high pH (~10) and included substantial quantities of calcium silicate insulation material as well as fiberglass. All tests in the series included metal coupons whose surface areas were scaled to those in representative PWR containment and sump systems. A complete rationale for the selection of these test conditions is provided in Ref. 1.

Table 1-1. Test Series Parameters

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

^aTSP = trisodium phosphate.

The ICET apparatus consists of a large stainless-steel (SS) tank with heating elements, spray nozzles, and associated recirculation pump and piping to simulate the post-LOCA chemical environment. Samples of structural metals, concrete, and insulation debris are scaled in proportion to their relative surface areas found in containment and in proportion to a maximum test dilution volume of 250 gal. of circulating fluid. Representative chemical additives, temperature, and material combinations are established in each test; the system is then monitored while corrosion and fluid circulation occur for a duration comparable to the ECCS recirculation mission time.

1.2. Information Presented in This Report

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

2. TEST PROCEDURES

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

2.1. Chemical Test Apparatus Functional Description

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

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

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

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

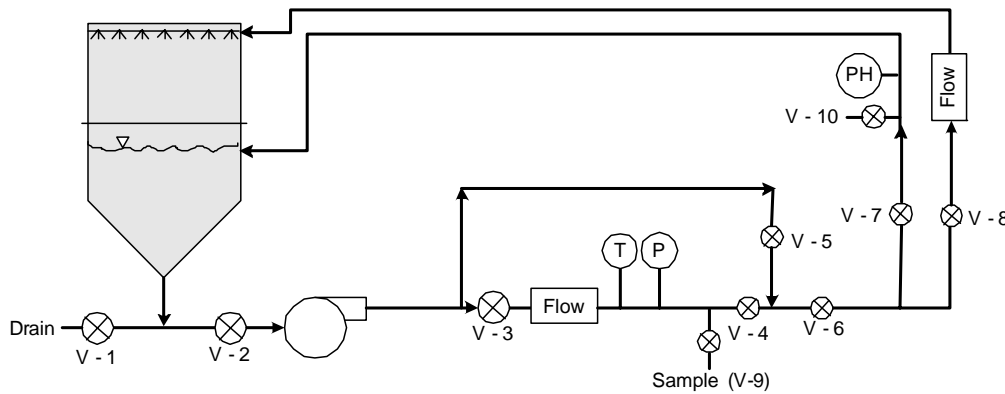


Figure 2-1. Test loop process flow diagram.

2.2. Pre-Test Preparation

2.2.1. Test Loop Cleaning

In preparation for Test #4, the experiment test loop was thoroughly cleaned to remove all Test #3 deposits and residues. In addition to visual inspections, the test apparatus was flushed and cleaned per the written direction given in the pre-test operations project instruction (PI) (Ref. 3). The system was flushed with ammonium hydroxide, followed by ethanol, and then nitric acid until it was visually clean and the water conductivity was $<50 \mu\text{S}/\text{cm}$.

2.2.2. Test Coupons and Samples

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

approximately 3/32 in. thick. The concrete coupons (one per test) are approximately 1-1/2 in. thick. Insulation materials are also exposed. For Test #4, fiberglass insulation samples and calcium silicate samples were included in the test. As with previous tests, Test #4 subjected seven racks of coupons to the specified environment, with one being submerged in the test tank and the remaining six being held in the tank's gas/vapor space. The Test #4 coupons of each type were as shown in Table 2-1.

Table 2-1. Quantity of Each Coupon Type in Test #4

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

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

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

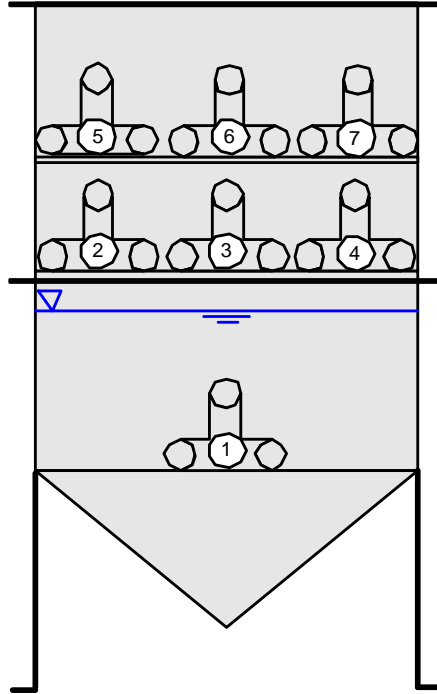


Figure 2-2. Coupon rack configuration in the ICET tank. The blue line represents the surface of the test solution.

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



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

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



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

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



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



Figure 2-6. Cal-sil dust.

2.2.3. Quality Assurance Program

A project QA manual was developed to satisfy the contractual requirements that apply to the ICET project. Specifically, those requirements were to provide credible results by maintaining an appropriate level of QA in the areas of test loop design, sampling,

chemicals, operation, and analysis. These requirements were summarized in the contract requirement that QA was to be consistent with the intent of the appropriate sections of 10CFR50, Appendix B.

The 18 criteria of 10CFR50, Appendix B, were addressed separately in the QA manual, and the extents to which they apply to the ICET project were delineated. A resultant set of QA procedures was developed. In addition, project-specific instructions were written to address specific operational topics that required detailed step-by-step guidance. PIs generally applicable to all tests were written for the following topics and were followed for Test #4:

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

Project instructions specific to Test #4 were written for the following:

- Pre-Test Operations
- Test Operations, Test #4 (cal-sil, fiberglass, and NaOH at pH 10)
- Post-Test Operations

The pre-test, test, and post-test operations PIs that were used in Test #4 are included in Appendix J.

2.2.4. Test Parameters

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

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

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

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

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

Physical Parameters

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

Chemistry Parameters

| | |
|--|--|
| • H_3BO_3 concentration | 2800 mg/L as boron |
| • $Na_3PO_4 \cdot 12H_2O$ concentration | As required to reach pH 7 in the simulated sump fluid |
| • NaOH concentration | As required to reach pH 10 in the simulated sump fluid |
| • HCl concentration | 100 mg/L |
| • Lithium hydroxide (LiOH) concentration | 0.7 mg/L as Li |

The parameters planned for each ICET test run are described in Table 1-1.

2.3. Test Operation

2.3.1. Description

Preparation of ICET Test #4 (Run #4 in Table 1-1) began with 239 gal. of reverse osmosis (RO) water heated to 65°C. (Adding the metal coupons and insulation samples reduces the water temperature by approximately 5°C, so the water was heated initially to 65°C.) With 25 gpm circulating through the loop, the predetermined quantities of boric acid (15.41 kg), sodium hydroxide (8.47 kg), lithium hydroxide (0.66 g), and hydrochloric acid (212 mL) were added and dissolved in the ICET tank solution. The chemicals were added with 10 gallons of RO water. After the chemicals were added and observed to be well mixed, a baseline grab sample and measurements of the test solution were taken. Then the premeasured latent debris and concrete dust were added to the tank solution. After the solution circulated for 10 minutes, the pump was stopped and the coupon racks and insulation samples were put into the tank (see Section 2.2.2).

The test commenced with initiation of the tank sprays (3.5 gpm). For the first 30 minutes, one gallon of RO water containing 614 g of sodium hydroxide was metered into the spray. The sprays were terminated after 4 hours. The test ran for 30 days, with one 2-hour, 15-minute interruption due to a power outage, which occurred on Day 11.

The experiment commenced at 2:30 p.m. on Tuesday, May 24, 2005, and it ended on June 23, 2005. During the test, grab samples were taken daily for wet chemistry and inductively coupled plasma – atomic emission spectroscopy (ICP-AES) analyses. Water loss due to water sample removals and evaporation was made up with RO water. Water samples, insulation, and metal coupons were analyzed after the test. Sampling and analyses were conducted in accordance with approved project instructions (Refs. 3, 4, and 5).

2.3.2. Process Control

During the test, critical process control parameters were monitored to ensure that the test conditions met the functional test requirements. Recirculation flow rate and temperature were controlled throughout the test. The solution pH was initially targeted to reach the prescribed value of 10 after the spray phase ended. The predetermined amounts of chemicals were added, and pH was not controlled.

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

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

2.4. Analytical Methods

Data collected during Test #4 included the in-line measurements of temperature, pH, and loop flow rate. During the daily water grab sample analysis, bench-top measurements were obtained for temperature, pH, turbidity, total suspended solids (TSS), and kinematic viscosity. The concentration of hydrogen in the tank atmosphere was also measured and could be used as an indicator of chemical reactions taking place. Water, fiberglass, and metal coupon samples were taken to other laboratory locations for additional analyses. These analyses included strain-rate viscosity, ESEM, SEM, EDS, TEM, ICP-AES, x-ray fluorescence (XRF), and x-ray diffraction (XRD). EDS provided a semi-quantitative elemental analysis after calibration of the instrument's x-ray signal using an internal element standard. Descriptions of the principles of operation and limitations of these analytical methods were provided in the Test #1 report (Ref. 2).

2.4.1. Data Compilation and Nomenclature

This section provides a brief guide to assist the reader in interpreting the ICET Test #4 information and data presented in the following sections and in the appendices. Standardized nomenclature is defined first to clarify the origin of samples that are described in the data sets. The appendices are listed, and a description is provided of how they were compiled.

2.4.1.1. Nomenclature

Many spatially unique but physically similar sample types were collected in ICET Test #4. To ensure that consistent interpretations and comparisons of data sets are made, it is imperative that a standardized nomenclature be adopted when referring to each sample type. Many different qualitative descriptions of these samples might be equally suitable, but different adjectives convey different connotations to each observer. Therefore, the following definitions establish the convention used in this report when

making generic references to sample type. Every effort should be made to adhere to this standard when interpreting the data so that all future audiences will have a common understanding of sample origins from the ICET series.

- White Precipitate** The behavior of test solution at test temperature and upon cooling is observed during testing. Precipitates and their prominence indicate chemical interactions occurring in the solution. White precipitate formed in Test #1 water solution samples drawn from the test loop. Upon cooling below the test temperature, Test #1 daily water samples extracted from the tank formed a visible white material that is referred to as a precipitate. This precipitate was absent in Test #4 water samples. Test #1 had a high pH of about 9.5, and the pH for Test #4 was about 9.8. It is unclear what caused precipitates to form in Test #1 but not in Test #4.
- Latent Debris** Commercial power plant containments gradually accumulate dust, dirt, and fibrous lint that are generically referred to as latent debris. This classification distinguishes resident material from debris generated during an accident scenario. At the beginning of Test #4, measured quantities of crushed concrete and soil were added to simulate the latent debris present in containment. These materials were examined via SEM/EDS to establish a baseline composition for comparison with sediment samples (see “Sediment” below).
- Sediment** Surrogate latent debris particulates and fugitive fiberglass fragments that were initially suspended in water at the beginning of Test #4 gradually settled to the bottom of the tank. In addition, a large amount of cal-sil dust was added to the tank solution. Most of that dust ended up on the tank bottom. A deep layer of sediment formed, which was 8-in. deep in some places at the end of the test. At the conclusion of the test, the sediment was recovered as completely as possible.
- Powder** At the conclusion of Test #4, fine particulate deposits were found on the submerged CPVC coupon rack. They were referred to as powder and examined by SEM/EDS. These deposits are also referred to as deposition products (see Appendix E).
- Fiberglass** One of the principal debris types introduced to Test #4 was shredded fiberglass insulation. This debris was bundled in 3-in.-thick bags (or blankets) of fiberglass confined in SS mesh to prevent ingestion through the pump and to better control the placement of debris in various flow regimes. Fiberglass samples are designated by their placement in high-flow and low-flow areas of the tank. Fiberglass in the “big envelope” sat on the tank bottom in a low-flow area of the tank. Additional 4-in.-square envelopes of

fiberglass were also prepared for extraction during the course of the test. These samples are referred to as “sacrificial” samples. One sample, called the “birdcage,” was constructed so that the fiberglass within was loose and not compacted. The birdcage fiberglass sat on the tank bottom and was removed on Day 30. Some amount of fiber, especially short-fiber fragments, escaped the mesh bags and was deposited in other locations within the tank. This material is referred to as “fugitive” fiberglass. Two additional fiberglass samples were added after the test began and the water clarity improved. A sacrificial sample was placed directly in front of one of the flow headers (high-flow area) on Day 2. Another sacrificial sample enclosed in nylon mesh was placed in a low-flow area on Day 3.

- Cal-sil The insulation samples used in Test #4 were 80% cal-sil by volume. That amount was divided into 4 size categories, in pieces that were roughly cubes. Of the total cal-sil, 14% was over 3 in., 19% was 1–3 in., 5% was less than 1 in., and 62% was “dust.” The dust consisted of cal-sil pieces that were ground into a fine powder. With the exception of the dust, the cal-sil pieces were contained within SS mesh and apportioned into submerged and unsubmerged samples. The dust was put into the tank solution before the test started, and it became the primary constituent of the sediment.
- Drain Screen A 12-in.-tall screen made of coarse SS mesh (1/8-in. holes) wrapped into a 2-in.-diam cylinder was inserted into the outlet drain at the bottom of the tank to protect the pump from ingesting large debris items. Two inches of the screen were inserted into the tank outlet to provide a solid base and stability. A 6-in.-tall drain collar was installed around the drain screen. This drain collar was a cylinder of fiberglass held in SS mesh. The drain collar was exposed to higher-velocity water flow than other samples in the tank. However, when the sediment built up around the drain collar, it was only exposed to water flow down through the drain screen. The drain collar fiberglass was examined as a separate debris location to identify any apparent differences with other sample locations.
- Gelatinous Material This term generically refers to any observed sample constituent with amorphous, hydrated, or noncrystalline physical characteristics. When Test #4 was shut down, there was no evidence of gel-like precipitates in the tank or piping.
- Water Sample Daily water samples were extracted from the ICET tank for elemental concentration analyses. After the sample line was properly flushed, some of this water was extracted directly from

the tap. An equal amount of water was also generally collected through a micropore filter. Thus, daily water samples were designated as filtered (F) and unfiltered (U), and a corresponding filter paper exists in the sample archive for each daily sample that was collected.

- High-Volume Filter** If white precipitates are observed in the tests, larger quantities of test solution are periodically extracted for filtration to determine whether suspended chemical products are present in the test liquid under in situ conditions. The intent of this exercise is to maintain the liquid temperature while forcing the liquid through a micropore filter under vacuum. Because the precipitates were not present in Test #4, these high-volume filter samples were not obtained.
- Filter Paper** Many different samples of tank solution were fractionated by micropore filtration into a liquid supernate and a solid filtrate that existed at the time and temperature conditions of the filtering process. These samples included (1) daily water samples filtered during extraction, (2) daily water samples filtered after cooling to room temperature, and (3) high-volume water samples.
- Chemical Deposits** Sacrificial fiberglass samples that were extracted at Day 5, Day 15, and Day 30 showed evidence of chemical products forming on and between fiber strands. These products are referred to as “deposits,” although the exact physical mechanism of formation is not well understood. The physical appearance suggests growth, agglomeration, or crystallization on and around the fiber strands over time rather than capture or impaction of particles from the bulk solution. This observation is supported by the fact that the small sacrificial fiberglass samples were located in a region of lower-velocity water flow (i.e., in the interior of larger blankets).
- Concrete Sample** Several chips of concrete (1/4–3/4 in. diam) were broken from the primary slab of submerged concrete and introduced to the tank in a small SS envelope at the start of the test. Examinations of these chips were conducted to determine if concrete surfaces provide a preferential site for gel formation.

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

2.4.1.2. Usage

The 10 appendices listed below are provided to present data collected for the sample types and analysis methods listed below. In addition, an appendix is provided with pertinent Test #4 project instructions.

- Appendix A ESEM/EDS Data for Test #4, Day-5 Fiberglass in Low-Flow Zone
- Appendix B ESEM Day-15 Fiberglass
- Appendix C ESEM Day-30 Fiberglass
- Appendix D ESEM/EDS Data for Test #4, Day-30 Low-Flow Cal-Sil Samples
- Appendix E ESEM and SEM/EDS Data for Test #4, Day-30 Deposition Products
- Appendix F SEM Day-30 Coupons
- Appendix G SEM/EDS Data for Test #4, Day-30 Sediment
- Appendix H TEM Data for Test #4 Solution Samples
- Appendix I UV Absorbance Spectrum – Day-30 Solution Samples
- Appendix J ICET Test #4: Pre-Test, Test, and Post-Test Project Instructions

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

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

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

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

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

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

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

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

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

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

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

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

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