ICET Test #4: Test Conditions and Observations
(NaOH and 80% Cal-Sil and 20% Fibrous Insulation)

Test Conditions

The general conditions, approach, and requirements for the ICET program are contained within the test plan that governs this test series (Adams Accession # ML051100357). All tests are being conducted in an environment that represents expected containment pool conditions during recirculation. The initial chemical environment contains 2800 mg/L of boron, 100 mg/L of hydrochloric acid (HCl), and 0.7 mg/L of lithium hydroxide (LiOH). 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 un-submerged 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.

The primary differences among the ICET tests are the buffering agent and the insulation materials. Test #4 incorporated a sodium hydroxide (NaOH) buffering agent with 80% calcium silicate (Cal-Sil) insulation and 20% fibrous (NUKON™) insulation. An initial amount of NaOH was added to the aqueous chemical environment. The remaining NaOH was injected during the first 30 minutes of the 4-hour spray phase of the test. The amount of injected NaOH was determined so that the spray fluid pH did not exceed a value of 12 during the injection phase. The actual test solution pH was approximately 9.8 at the end of the NaOH injection into the spray. The pH of the test solution declined slightly over the first two 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 approximately 9.8 at the end of the test. This 30-day test was initiated on May 24, 2005, and was completed on June 23, 2005.

Important Test Observations

The following general visual observations were made during the test. Four hours after the test began, the solution was murky as the Cal-Sil had not completely settled. However, after the first day of testing, the test solution was clear with no observed by-products. Most of the Cal-Sil had settled to the bottom of the tank by this point. After the first day, the tank clarity and color appeared constant throughout the test, even during the addition of the make-up water to the tank. A total of 31 gallons of water was added in five gallon increments over the course of the test. On Day 11 of the test, a power outage occurred for a 2 hour, 15 minute interval. Pump circulation and temperature control was lost during this interval. During this outage, the test chamber temperature peaked at 62 °C (144 °F), compared to the 60 °C (140 °F) target test temperature, before the process control hardware was restarted. It is not expected that this outage significantly affected chemical product formation in this test.

Several products are being evaluated for the presence of chemical effects in these tests: the test solution, fibrous insulation samples, test chamber sediment, and sample coupons. There is currently evidence of chemical by-products in the fibrous insulation samples and possibly in the sediment that should be considered when addressing possible chemical effects implications as part of the generic letter (GL) 2004-02 evaluation. The test observations relevant to each product form are described as follows:

Water Samples

No chemical by-products were visible at the test temperature of 140°F or upon cooling to room temperature in the daily water samples. Additionally, no precipitates or by-products have become visible during storage of samples at room temperature since the end of the test. The turbidity at 140 °F was high (130 NTU) at the start of testing due to the addition of Cal-Sil debris prior to initiating the test. Turbidity continued to steadily decrease 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, the turbidity fell below 4 NTU and remained largely constant over the remainder of the test. Over the last three weeks of testing, the turbidity at 140 °F averaged 0.6 NTU. Small turbidity increases were measured after the make-up water was periodically added. These increases peaked at a high of 9.8 NTU during the day 3 water addition and decreased with each subsequent water addition. The turbidity increased to less than 0.7 NTU after the final day 27 water addition. These turbidity trends are consistent with the earlier stated visual observations of activity within the test chamber.

The total suspended solids (TSS) values generally exhibit similar 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 30 and 45 mg/L from day 1 to day 30, with an average value of approximately 35 mg/L. Kinematic viscosity measurements at 140°F and room temperature were constant during the entire test and consistent with measured values obtained in ICET tests 2 and 3.

The most prevalent elements in solution, in addition to the species introduced to achieve the initial chamber chemical composition (i.e, boron, NaOH, LiOH, and HCl), included silica, calcium, and potassium. The silica concentration increased quickly to a value of approximately 100 mg/L after the first day of testing, and then continued to steadily increase until day 15 when the concentration was approximately 170 mg/L. The silica concentration was approximately constant from day 15 to the end of the test, but individual concentration measurements varied between approximately 160 and 190 mg/L. The sodium concentration at the start of the test was approximately 6000 mg/L and increased fairly steadily over the course of the 30-day test. By day 17, the sodium concentration was approximately 8500 mg/L. After this point, sodium mean concentration continued to increase but the variability between measurements taken at different times increased substantially. Variations of 2000 mg/L between measurements taken 1 day apart were not uncommon after day 17. These variations are attributed to data analysis variability for the most part. By the end of the test, the sodium mean concentration appeared to be greater than 10000 mg/L.

The calcium concentration started below 4 mg/L. It then rose sharply over the first day of testing to approximately 40 mg/L. The calcium continued to increase until approximately day 5 of the test. Between days 5 and 21, the calcium concentration was approximately a constant value of 50 mg/L. Variability was less than ± 5 mg/L. After day 21, the calcium appeared to slowly decrease over the remainder of the test. Aluminum concentrations were detectable only during the initial 24 hours of testing and decreased steadily to non-detectable levels over this time. The highest concentration measured was less than 6 mg/L within a few hours of initiating the test. The potassium concentration was only measured five times during the testing. At the onset of testing, the potassium concentration was 7 mg/L. The concentration increased to 46 mg/L by the end of the first day. The day 17 potassium concentration was approximately 70

mg/L. By day 30, potassium appeared to decrease somewhat. The final measured value was approximately 35 mg/L.

Fibrous Insulation Materials

Fibrous insulation materials are being evaluated that were extracted from the tank during the beginning, middle, and end of the test. Initial evaluations revealed that some web-like structures formed between the fibers intersection points. These structures were evident in the day 15 and day 30 insulation samples and appear to be similar to structures formed during ICET test #1. However, the web-like structures were only sporadically located throughout the insulation and in significantly less quantities than evident in ICET test #1. Unlike ICET test #1, these structures did not appear to be preferentially located on insulation at the interface with the stainless steel sample holder.

Sediment

There was a large quantity of sediment found on the test chamber floor after the test. As expected, much of this sediment appeared to be from the small Cal-Sil particles introduced during the beginning of the test. The heat treated and unheat treated Cal-Sil materials appeared to be well-mixed in the sediment. There was no visibly apparent corrosion by-product on the top of the sediment layer as in ICET test #3. A more detailed analysis of the sediment for evidence of corrosion by-product formation is underway.

Sample Coupons

The unsubmerged zinc, aluminum, uncoated steel, and copper samples exhibited evidence of corrosion upon removal from the test chamber. Conversely, there were no obvious corrosion product on any of the metallic samples contained within the submerged specimen rack. Pre- and post-test weight measurements revealed insignificant changes (less than 1 g) in the bulk weight of the submerged and unsubmerged zinc, aluminum, uncoated steel, and copper samples. Lack of submerged coupon corrosion directly contrasts with ICET test #1 results, which exhibit significant wastage of the submerged aluminum specimens (approximately 25% less weight in post-test measurements) in a similarly buffered NaOH solution. There was a faint white film on the aluminum and zinc specimens that was much lighter than the corrosion products evident on the unsubmerged specimens. This film has been speculated to be calcium carbonate which acts to inhibit corrosion on these specimens. These specimens will be analyzed to determine the film's composition.

A more complete data report for ICET test #4, containing additional observations, is currently being prepared and will be made publicly available once it has been completed.