ENCLOSURE 1

Report BWROG-ECCS-WP-4-1 R4, "BWR Material Dissolution Test Plan," – Non-Proprietary Information (GEH Class I)

BWROG-ECCS-TP-4-1 Revision 4 Project No. 1300470 November 2013

BWR Material Dissolution Test Plan

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		REVIS	SION CONTR	OL SHEET
Documer	nt Number:	BWROG-ECC	S-TP-4-1	
Title:	BWR Materi	al Dissolution T	est Plan	
Client:	General Elec	tric-Hitachi Nuc	clear Energy An	nericas LLC
SI Projec	t Number: _1	300470	Qualit	y Program: 🛛 Nuclear 🔲 Commercial
Section	Pages	Revision	Date	Comments
All	All	0	11/5/2012	Original Issue.
n/a	n/a	1	11/16/2012	Removed copyright, corrected page count and footers, and corrected the original description. Finally, updated the Test Acceptance section.
n/a	n/a	2	4/12/2013	Updated Revision History Log to be current with QA. Updated and clarified objectives to ensure that test plan is achievable.
All	All	3	7/15/2013	Complete rewrite of document.
n/a	n/a	4	11/14/2013	Incorporated BWROG comments, added calcium silicate tests, added baking procedure to insulation processing, added earlier sampling times to all tests, increased sample storage time to 2 years. Inserted updated equipment diagram. Small technical revisions throughout.

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1.0 TEST OBJECTIVE

The objective of this test program is to quantify the amount and characteristics of species released to the coolant when BWR containment materials are exposed to simulated suppression pool/torus water and a sodium pentaborate solution during a bounding post-LOCA (post-loss of coolant accident) temperature excursion at a BWR. The ratio of the coolant mass to the exposed area or mass of each material during testing will simulate the ratio at a typical BWR. Elemental release rates from materials such as aluminum, carbon steel, galvanized carbon steel and NUKON® insulation during exposure of mixtures of the materials to simulated suppression pool/torus water and sodium pentaborate solutions will be quantified. Tendencies for precipitate formation also will be assessed.

2.0 TEST PLAN

2.1 Overview

During post-LOCA simulation tests, chemical release from selected materials during exposure of multiple materials to simulated suppression pool/torus water and sodium pentaborate solutions over a period of 18 days will be quantified. Tendencies for precipitate formation will also be assessed. Metallic coupons and samples of insulation will be exposed at conditions simulating exposure to the post-LOCA sump fluid in a BWR containment environment. In the test loop configuration, the solution will flow continuously over the test materials at low velocity. The temperature of the fluid will be varied to simulate a bounding post-LOCA BWR suppression pool temperature profile.

Coolant samples will be obtained intermittently during each period of exposure during the post-LOCA simulation tests to quantify concentrations of dissolved and suspended solids. Release rates from the exposed solid materials will be calculated per unit surface area or per unit mass of each material. The extent of precipitate formation during the exposure period will be quantified.

During a series of tests, tendencies for precipitate formation during cool down to 110 to 140°F after an initial 24 hour exposure of selected material combinations at 200°F will be assessed. Identification of the chemical form of the precipitate will be attempted if sufficient amounts of

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precipitates are formed. Releases during the initial 24 hour exposure at 200°F will be quantified to supplement the post-LOCA simulation test database.

It currently is planned to use graphite furnace or flame atomic adsorption for elemental analyses of the liquid solutions. However, alternate analysis approaches such as ICP may be used. Reactive silica concentrations will be determined colorimetrically. Concentrations of dissolved species will be determined after filtration of the solution through a 0.45 micron membrane. Concentrations of particulate species will be determined after dissolution of particulates from the filter membranes in an appropriate media.

Samples of the test materials will be weighed before and after exposure to quantify corrosion and release rates. In some cases, weight losses or gains may not be sufficient to estimate releases, and reliance will be placed on estimates developed from solution mass and concentration increases. Deposition or compound formation on the surfaces will be assessed. Materials will be photographed when appropriate.

Releases to the solution will be calculated from solution concentration increases and volumes. The calculated releases will be compared to the weight loss of each material. The release rate results will be used to estimate release rates following a LOCA.

Multiple materials will be exposed during most tests to account for possible interactions between the dissolved species that could lead to precipitate formation or affect the release rate of an individual material.



2.2 Test Materials

It currently is planned to quantify release rates from the following materials during exposures of mixtures of materials to simulated torus/suppression pool and borated water environments. The list of materials will be finalized following completion of the BWROG site survey of materials.

- Aluminum
- Carbon Steel (CS)
- Galvanized Steel (GS)
- Inorganic Zinc Coating (IOZ)
- NUKON[®]

2.3 Exposed Material Area/Mass to Coolant Mass Ratios

A critical requirement for simulating the conditions present in the BWR containment during a LOCA event is to approximate to the extent reasonable the ratios of the area or mass of each major material that is exposed to the coolant to the total coolant mass. These ratios will be determined from the detailed site surveys of materials being prepared by BWROG members and will be employed during the proposed dissolution and precipitation tests.

2.4 Solution Chemistry

Release rate tests will be performed at BWR suppression pool chemistries representing the non-SLC (Standby Liquid Control) injection scenario (unbuffered), which will be simulated using representative suppression pool/torus water, and the SLC injection scenario (buffered), which will be simulated using a sodium pentaborate solution chemistry.

2.5 Temperature Control

2.5.1 Assessment of Tendency for Precipitate Formation

An initial series of tests will be performed with the temperature maintained for ~24 hours at ~200°F, the expected maximum post-LOCA containment solution temperature. Selected mixes of the test materials will be suspended in the test solutions. Solutions will then be cooled to 140° F, maintained at this temperature for ~24 hours and visually inspected for precipitate



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formation. The turbidity will then be measured, and an aliquot of the solution will be filtered. Duplicate solutions will be cooled to 110° F, maintained at this temperature for ~24 hours, visually inspected for precipitate formation, and then filtered. The filtered and unfiltered solutions will be analyzed. The filters will be weighed to quantify the amount of precipitate. If a sufficient quantity of material is present. precipitate identification will be pursued.

2.5.2 LOCA Temperature Profile Simulation

Since the rate of dissolution or film formation for most materials is expected to be a function of solution chemistry, the solution temperature and the prior exposure of the material, the test solution temperature during the post-LOCA simulation tests, which will be performed in a low flow rate test loop, will be controlled to simulate that during the post-LOCA event. Maximum BWR suppression pool temperature profiles from Reference 1 are shown in Figure 2-1.



Figure 2-1 – BWR Suppression Pool Post-LOCA Maximum Temperature Profiles [1]

Since there are only three BWR containment designs, and the BWR suppression pool temperature variations during a LOCA are generally similar, the materials test program will be



conducted using a bounding BWR suppression pool temperature profile. This will allow quantification of material release rates during a solution temperature and chemistry transient simulating that during the LOCA. Release rate estimates as a function of temperature from ~140 to 210° F, which will reflect the exposure of the material during the early stages of the LOCA, can be developed from such data.

As shown in Figure 2-1, Plant M exhibits the highest suppression pool coolant temperature during the LOCA. However, since the initial portion of the Plant M temperature profile is below the Plant K temperature profile, the Plant M temperature profile was adjusted to bound the Plant K values as shown in Figure 2-2. The bounding plant temperature profile displayed on a linear time axis (Figure A-1 of Appendix A) will be used during the material release testing program.



Figure 2-2 – Adjusted Plant M post-LOCA Suppression Pool Temperature Profile and Fleet post-LOCA Suppression Pool Temperature Profiles

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3.0 TEST PROGRAM

3.1 Test Apparatus

3.1.1 Precipitate Formation Assessment

Mixtures of selected materials will be suspended in polycarbonate containers. Containers will be placed in a constant temperature bath or oven and will be sealed (or a vapor phase condenser will be employed) to minimize solution loss by evaporation. The solution temperature will be maintained for ~24 hours at ~ 200°F. Solutions will then be cooled to 140°F, maintained at this temperature for ~24 hours, visually inspected for precipitate formation, and then filtered. Duplicate solutions will be cooled to 110°F, maintained at this temperature for ~24 hours, visually inspected for precipitate formation. The filters will be weighed to quantify the amount of precipitate and then analyzed to identify the precipitant if a sufficient quantity of material is present.

A preliminary test matrix is given in Section 4. Previous testing has suggested that precipitate formation will occur during cooling in some cases when the temperature is reduced to \sim 140°F [2]. The test matrix will be finalized following completion of the detailed BWROG containment materials survey.

3.1.2 Post-LOCA Simulation Testing

A preliminary schematic of the test apparatus for post-LOCA simulation testing is provided in Figure 3-1.





Figure 3-1 – Post-LOCA Simulation Apparatus

The facility will be fabricated primarily from stainless steel. Polycarbonate will be used in selected regions to allow process fluid visualization. The feed tank volume will be ~10 liters. The specimen exposure vessel volume will be ~5 liters. Samples of test materials will be suspended from the top of the exposure vessel and will be submersed in the solutions during each test. The NUKON[®] cake will be loaded onto a stainless steel screen prior to initiating testing. The solution will be recirculated through the system using a stainless steel pump at a flow rate of 1 to 5 gallons/minute. This will yield a velocity through the column of ~ 0.1 ft/second. Provision will be made for measuring temperature at the exposure vessel inlet. Temperature will be controlled to simulate the BWR post-LOCA profile. The feed tank solution will be preheated prior to test initiation to allow the exposure vessel temperature to be increased rapidly. After exiting the exposure vessel, the solution will be passed through the bed of NUKON[®]. Sample points are shown in Figure 3-1. Each sample point will be provided with an in-stream filter for prefiltering solution samples. A nitrogen or helium overpressure will be maintained in the feed tank vapor space. Degassing of the fluid will be performed if necessary.



The specimen surface area to volume or mass to volume ratio will be adjusted to simulate post-LOCA BWR conditions based on the detailed BWROG survey results [1].

3.2 Solution Chemistry

Testing will be performed in simulated suppression pool/torus water and in a sodium pentaborate solution simulating that expected to be present during a BWR LOCA, based on inputs received from the BWROG [1].

3.3 Test Temperature and Duration

Post-LOCA simulation testing will be performed with selected combinations of materials over an 18 day period following the temperature profile shown in Figures 2 and A1. In the tests to assess precipitate formation, the temperature will be maintained at 200°F for approximately 24 hours followed by cooling to 140 or 110°F.

3.4 Test Materials

3.4.1 Metal Coupons

Aluminum, carbon steel and galvanized carbon steel coupons shall be purchased from a commercial supplier. Surfaces will be finished to 120 grit. This is expected to maximize release rates during exposure to the test solutions and yield conservative results relative to release rates and precipitate formation. Material composition certificates will be obtained. After visual inspection, coupons will be measured, rinsed with acetone, rinsed with demineralized water, dried for 2 to 4 hours and then weighed.

3.4.2 Nukon[®] Insulation

The NUKON® specimen will be provided by the manufacturer in a single shredded state and baked for 10 hours on a hot plate at 260°C to simulate aging. Prior to weighing, it shall be dried for at least 2 hours at 105°C. The NUKON® material shall not be boiled prior to testing (as is common in head loss testing). The NUKON[®] specimen shall be weighed prior to testing. The material will not be rinsed prior to testing.



3.4.3 Cal-Sil

Calcium silicate will be provided by the manufacturer, baked for 10 hours on a hot plate at 260°C to simulate aging, and shredded. Prior to weighing, it shall be dried for at least 2 hours at 105°C. The calcium silicate material shall not be boiled prior to testing (as is common in head loss testing). The calcium silicate specimen shall be weighed prior to testing. The material will not be rinsed prior to testing.

3.4.4 IOZ

Inorganic zinc coating shall be applied to carbon steel such that it is representative of the IOZ coating in a BWR and allowed to cure for an appropriate amount of time prior to testing.

3.4.5 Concrete

Preliminary results of the BWROG site materials surveys indicate that significant areas of uncoated concrete are not generally present in the BWR containment. As such, it is not currently planned to consider this material during the dissolution or precipitation tests.

3.5 Sampling and Analysis

3.5.1 Precipitate Formation Assessment

Solution samples will be taken every 2 hours for the first 8 hours, and then every 8 hours during the initial 24 hour exposure at 200°F. Twenty four hours after cooling to 140°F or 110°F, the solutions will be visually assessed for precipitate formation, and the solution sampled through a 0.45 micron membrane. The solution samples will be analyzed by atomic absorption or inductively coupled plasma (ICP). The filter membrane will be weighed and then analyzed by x-ray diffraction (XRD) or an alternate technique for identification of crystalline forms (if a sufficient amount of precipitate can be collected.). The membrane will also be analyzed for individual elements if a significant amount of precipitate is collected.

3.5.2 Post-LOCA Simulation Test Solution

Solution samples will be taken every 2 hours for the first 8 hours, then every 8 hours for the first day of the exposure. After that, samples will be obtained approximately every 12 to 18 hours over the 18 day test period. The liquid samples will be passed through a .45 micron filter



membrane installed in the filter housing attached to the test loop. Analyses for major constituents of the test samples will be performed as outlined in Section 2.1. Analytical requirements will be detailed in individual test procedures. Filtered samples generally will be acidified following collection. Filter membranes will be removed, weighed, dissolved in an appropriate media and then analyzed. (Alternate filter analysis techniques are also being considered.) Conductivity and pH@25°C of the solution will be measured. Measurements will be done in-line or in the laboratory.

The solution volume in the loop before and after sampling will be recorded. At the end of each test, an aliquot of the residual solution will be collected and archived.

XRD may be used for solid phase identification if a significant particulate concentration is observed and a sufficient amount of precipitate is present on the filter membranes. Alternate filter analysis techniques may be considered.

3.5.3 Test Materials

3.5.3.1 Metal Coupons

Coupons shall be dried and photographed at the end of each test. They will then be weighed, brushed to remove residual deposits or oxides, and reweighed. The material removed by brushing shall be collected and reserved for possible analysis. Each specimen will be archived for 2 years following the test.

3.5.3.2 NUKON® and Calcium Silicate

After test completion, the NUKON® and calcium silicate samples shall be photographed, dried in an oven at 105°C for ~8 hours prior to weighing, and then stored for possible post-test analysis.

3.5.3.3 IOZ

After test completion, IOZ samples shall be photographed and dried. They will then be weighed, brushed to remove residual deposits or oxides, and reweighed. The material removed by brushing shall be collected and reserved for possible analysis. Each specimen will be stored for possible post-test analysis.



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4.0 TEST MATRICES

Tests will be performed at BWR suppression pool chemistries representing the non-SLC injection scenario (non-alternate source term), which will be simulated using representative suppression pool/torus water, and the SLC injection scenario, which will be simulated using a sodium pentaborate solution chemistry following SLC injection into the suppression pool. The specimen surface area to volume or mass to volume ratio will be adjusted to simulate post-LOCA BWR conditions based on the detailed BWROG survey results. A list of initial post-LOCA simulation tests is given in Table 4-1. A responsive test matrix will be developed following completion of the BWROG containment materials survey.

Table 4-1 – Pre	liminary Test	Matrix f	for Post-L	OCA Simu	lation Testing

Test	Materials	Solution Chemistry
1-S-B	Al, NUKON®, Galvanized Steel	Borated
2-S-NB	Al, NUKON®, Galvanized Steel	Suppression Pool/Torus
3-S-CS-B	Al, NUKON®, Galvanized Steel, CS	Borated
4-S-CS-NB	Al, NUKON®, Galvanized Steel, CS	Suppression Pool/Torus

A preliminary test matrix for the evaluations of precipitate formation tendencies is given in Table 4-2. The final test matrix will be developed following completion of the detailed BWROG containment materials survey.



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Test	Temperature, °F	Materials	Solution
1-PF-B	200 to 140 °F	Al, NUKON®	Borated
2-PF-NB	200 to 140 °F	Al, NUKON®	Suppression Pool/Torus
3-PF-GS-B	200 to 140 °F	Al, NUKON®, Galvanized Steel	Borated
4-PF-GS-NB	200 to 140 °F	Al, NUKON®, Galvanized Steel	Suppression Pool/Torus
5-PF-CS-B	200 to 140 °F	Al, NUKON®, Carbon Steel	Borated
6-PF-CS-NB	200 to 140 °F	Al, NUKON®, Carbon Steel	Suppression Pool/Torus
7-PFC-B	200 to 110 °F	Al, NUKON®	Borated
8-PFC -NB	200 to 110 °F	Al, NUKON®	Suppression Pool/Torus
9-PFC-GS-B	200 to 110 °F	Al, NUKON®, Galvanized Steel	Borated
10-PFC-GS-NB	200 to 110 °F	Al, NUKON®, Galvanized Steel	Suppression Pool/Torus
11-PFC-CS-B	200 to 110 °F	Al, NUKON®, Carbon Steel	Borated
12-PFC-CS-NB	200 to 110 °F	Al, NUKON®, Carbon Steel	Suppression Pool/Torus
13-PF-CAL-B	200 to 110 °F	Al, NUKON®, Calcium Silicate	Borated
14-PF-CAL-NB	200 to 110 °F	Al, NUKON®, Calcium Silicate	Suppression Pool/Torus

	Table 4-2 – Preliminary	/ Test Matrix	for Precipitate	Formation Assessment
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5.0 INSTRUMENT CALIBRATION

Calibration of thermocouples used for temperature measurement during the tests will be performed by an approved calibration facility using National Institute of Standards and Technology (NIST) traceable standards. The pH and conductivity meters shall be calibrated before, during and after each measurement campaign using traceable pH buffers and conductivity standards, respectively. The flow meter will be calibrated on a mass basis (Accuracy \sim 3%).

6.0 DATA ACCEPTANCE CRITERIA

The test objectives are to determine the corrosion and/or release rates of selected materials and assess the tendencies for precipitate formation during a simulated BWR LOCA event. Data to be collected will include sample mass before and after testing as well as dissolved and filterable species concentrations, solution conductivity and pH@25°C during testing. Visual observations and selected photographs will also be obtained. If the data are insufficient to quantify release rates at the required level or with the required accuracy, the test procedure will be modified to allow development of the necessary data.

7.0 TEST PROCEDURES

Separate procedures will be developed for each test of the finalized test matrix. Sample preparation, sampling and analysis requirements, and temperature, flow rate, solution volume, conductivity, pH monitoring and data recording requirements will be detailed. The procedures will document each required test step and will include test specific logs for manually recording data required to meet test objectives.

8.0 REFERENCES

- 1. BWR Chemical Effects DIR Responses
- 2. Furman, J., *BWR Material Dissolution and Corrosion Evaluation*, ALION-RPT-LAB-5810-001, Revision 0, Alion Science and Technology Corporation, August, 2012.
- Sawochka, S. G., and R. Eaker, *Review of ALION BWR GS-191 Database*, NWT 863, NWT Corporation, San Jose, CA. July 2013.

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Appendix A Test Temperature Profile







Figure A1-1 – Test Temperature Profile

Interval (hrs)	Temperature (°F)	
0 - 8.4*	207	
8.4 - 11.8	207 - 204	
11.8 - 14.3	204 - 202	
14.3 - 17.0	202 - 199	
17.0 - 28.4	199 – 187	
28.4 - 42.0	187 – 179	
42.0 - 48.0	179 – 176	
48.0 - 306.0	176 - 132	
306.0 - 432.0	132 - 110	

Table A1-1 – Test Temperature Profile

*Following initial rapid heat-up

