ENCLOSURE 1

Non-Proprietary Technical Report, "BWR Material Dissolution and Corrosion Evaluation" (BWROG Report – GEH Class I)

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TECHNICAL DOCUMENT COVER PAGE

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Attachment A –BWR Chemical Dissolution Testing, Test Plan Development RFP: received e	e-mail
from Fred Emerson, BWROG, GE Hitachi Nuclear Energy (August, 2009)	3 Pages
Attachment B – DVD of All Test Data and PhotographsI Cover Page, I	DVD-R



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ACRONYMS AND DEFINITIONS

0	Degrees
AL	Aluminum (Test Series)
BWR	Boiling Water Reactor
BWROG	Boiling Water Reactor Owners' Group
С	Celsius
ECCS	Emergency Core Cooling System
F	Fahrenheit
ft	Feet
ft²	Square feet
ft³	Cubic feet
GC	Galvanic Cell (Test)
GS	Galvanized Steel (Test Series)
h, hr	Hour
ICP	Inductively Coupled Plasma (Spectrometry)
kg	Kilograms
lb/ft ³	Pounds per cubic foot
LCS	Low Carbon Steel (Test Series)
LOCA	Loss of Coolant Accident
mg/L	Milligrams per Liter
min	Minutes
mL	Milliliter
NaPB	Sodium Pentaborate
NaTB	Sodium Tetraborate
NRC	Nuclear Regulatory Commission
ррb	Parts per Billion
ppm	Parts per Million
RMI	Reflective Metal Insulation
RO	Reverse Osmosis
S	Seconds
SLCS	Standby Liquid Control System
ZN	Zinc (Test Series)

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I BACKGROUND

1.1 Chemical Effects Concerns

A major and relatively recent concern in evaluating the effects of the debris transported to the BWR ECCS strainer after a LOCA is the impact of chemical products that may form in a post-LOCA suppression pool environment. Materials present in the drywell and suppression pool may dissolve or corrode when exposed to the reactor coolant and spray solutions. This can lead to oxide particulate corrosion product formation as well as the potential for the formation of precipitates due to changes in temperature and reactions with other dissolved materials. These chemical products may be another source of debris loading to be considered in strainer performance.

The impact of chemical effects on head loss occurs due to the:

- corrosion/leaching of materials when exposed to coolant chemistry/pH and temperature;
- subsequent potential re-association of dissolved species in solution to form new compounds and precipitates dependent on the time, pH, temperature of the coolant; and
- impact of these precipitates on debris head loss over time.

The water chemistry and containment materials in BWRs differ from those of PWRs, and the corresponding corrosion and dissolution behavior is currently not well understood. Due to the severity of the chemical effects on strainer head loss in PWRs, the issue must be addressed for the BWR fleet, (per NRC Memo dated January 31st, 2008, titled "Revised Summary of Nov. 27, 2007 Public Meeting with the BWROG to Discuss GSI-191 Technical Issues as Applied to BWRs") [1].

1.2 BWRs vs. PWRs

Nearly all nuclear industry strainer chemical effect research and progress over the past five years has been focused on chemicals effects on PWR strainers. Although the general tenets of strainer chemical effects, as listed in Section 1.1, are applicable to both BWR and PWR strainers, the details vary significantly between the two reactor types and the dissimilarities necessitate BWR-specific evaluation.



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1.2.1 Chemistry of Reactor Coolant and Containment Sprays

Post-LOCA BWR suppression pools experience two distinctly different suppression pool initial chemistry scenarios, depending on whether the plant starts the Standby Liquid Control System (SLCS) during a LOCA [2]. The SLCS injects sodium pentaborate (NaPB) or a mixture of sodium tetraborate (NaTB, Borax) and boric acid solution (the mixture of which is equivalent to NaPB) into the RPV and ultimately, the suppression pool [2]. The two chemical scenarios will be referred to as "SLCS" and "non-SLCS" for the remainder of this document. The primary significance of the different SLCS injection scenarios is the effect on coolant pH and buffering capacity, i.e., pH stability.

The SLCS suppression pool chemistry scenario occurs when NaPB and/or boric acid dissolved in water is injected as a part of the SLCS. NaPB is an alkaline ionic compound, and when dissolved NaPB increases the suppression pool pH to a maximum of ~8.6, depending on the quantity of injected NaPB and the initial conditions of the suppression pool. The range of possible boron concentrations is 0 to ~250 ppm boron, which is approximately an order of magnitude below the average PWR containment sump boron concentration of approximately 3000 ppm boron. Dissolved NaPB/boric acid will buffer the suppression pool water, and the pool pH will be more predictable, constant and less affected by dissolving containment materials than in the non-SLCS scenario.

The non-SLCS suppression pool chemistry scenario involves "ultrapure" water, which is purified water with low ionic strength, and total dissolved solids, generally in the ppb range. The pH can range from 4.8 - 8.6 [3], but due to the low ionic strength and lack of any significant buffering capacity, the pH can change readily and significantly as containment materials dissolve and/or corrode. The non-SLCS suppression pool scenario is very different from that of a PWR, which contains significant concentrations of dissolved boric acid and alkaline buffer, whether it's sodium hydroxide, trisodium phosphate, or sodium tetraborate.

This test program evaluated the effects of both the SLCS and non-SLCS suppression pool chemistries on various reactive plant materials.

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1.2.2 Temperature Profile of Suppression Pool and Containment Sprays

Dissolved species tend to be more soluble at elevated temperatures, so the suppression pool temperature profile can affect the amount of chemical dissolution as well as the timing of the dissolution. The temperature profiles used for this test program were selected to be representative of typical BWR post-LOCA temperature profiles, as provided in Reference [3].

1.2.3 Released Elements and Debris Material Sources

A table of potential released elements and debris material sources is included as Table 1-1. This table is not comprehensive, but encompasses the post-LOCA containment materials that are most likely to dissolve in a BWR suppression pool environment based on existing industry test data.

Aluminum	Aluminum reflective metal insulation (RMI), containment components,
(AI)	aluminum silicate insulation, mineral wool, fiberglass, concrete
Calcium (Ca)	Fiberglass, calcium silicate, concrete, mineral wool
Iron (Fe)	Low carbon steel, sludge, galvanized steel, fuel cladding deposits
Sodium (Na)	Fiberglass, calcium silicate
Silicon (Si)	Fiberglass, calcium silicate, concrete, microporous insulation, mineral wool, aluminum silicate insulation, demineralizers
Zinc (Zn)	Galvanized steel, IOZ coatings, fuel cladding deposits

Table I-I - Released Elements and Sources

The released elements that produce the largest impact on debris bed head loss in PWRs are aluminum, calcium (when phosphate is present), and silicon. Although BWRs tend to have large exposed areas of aluminum due to the extensive use of aluminum RMI, the non-SLCS near-neutral chemistry is less corrosive to aluminum than the elevated pH in a buffered PWR sump but more corrosive to zinc than a basic pH environment.

2 TEST PROGRAM OBJECTIVE

The objective of this test program was to record test data and collect samples for material loss and dissolved/suspended concentrations of species released from aluminum, zinc, low carbon steel, galvanized steel, and Nukon insulation in unbuffered and buffered water at temperatures and chemistries that simulate post-LOCA conditions for a typical BWR nuclear power plant. The results, analysis, and observations are used to determine preliminary release rate correlations, predict possible problem areas for chemical effects for BWR strainers and to develop guidance for future testing.



3 TEST APPARATUS



Figure I – Test Apparatus

This BWR material dissolution testing program was comprised of both mixed materials and isolated (single-material) dissolution/material loss testing. A drawing of the test apparatus is included as Figure 1. Metallic coupons and samples of insulation were suspended in unbuffered water to simulate exposure to the post-LOCA sump fluid in a BWR containment and suppression pool environment, and the material-fluid systems were continuously agitated while subjected to various temperature profiles. Measurements of dissolved and suspended species were obtained over the test duration via inductively coupled plasma spectrometry (ICP) of fluid samples taken during the testing, and the ICP dataset was used as the primary measurement of material dissolution. Material samples were weighed and photographed before and after testing to evaluate possible formation of corrosion products on material surfaces. Additionally, the cooled test liquid was filtered after testing to collect and weigh any solids.



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4 TEST INPUTS

4.1 Test Materials

Commercially pure metals were used for zinc and aluminum, since alloys are generally more corrosion resistant than pure metals. Metallic coupons were not polished or sanded before use.

- Aluminum coupons (Alloy 1100 from McMaster Carr Part#88685K11)
- Zinc Coupons (Fisher Scientific S71226-4: Zinc)
- Low Carbon Steel (LCS) Coupons (Shim Stock Sheet, McMaster Carr Part# 9011K28)
- Galvanized Steel (GS) Coupons (Galvanized LCS Sheet, McMaster Carr Part# 8943K12)
- Nukon (supplied in blanket form by Performance Contracting, Inc.)

4.2 Test Scaling

This test program consisted of a parametric evaluation of material dissolution and temperature profiles. All tests except for the mixed-material and galvanic cell tests were performed in 0.40L test fluid volumes, and the mixed-material test and galvanic cell test were performed in a 0.80L fluid volume. The test materials and fluid volumes are listed in Table 4-1.

Test Type	Solution Mass	AI	Zn	Low Carbon Steel	Galvanized Steel	Nukon	Galvanic Cell - Zn	Galvanic Cell - LCS
	(g)	(cm ²)	(cm²)	(cm²)	(cm ²)	(g)	(cm²)	(cm²)
Ponchton	400.0	4.00	14.00	14.00	14.00	0.40	n/a	n/a
benchtop	800.0	8.00	28.00	28.00	n/a	0.80	14.00	28.00

Table 4-1 - Test Materials

Note: Listed nominal surface areas of coupons include the surface area of both major sides of the coupon. Areas of edges are accounted for in release rate determination calculations.

Approximate scaling ratios are listed in Table 4-2, based on an approximate average suppression pool volume of 100,000 ft³ [3].



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Table 4-2 – Test Scaling Ratios

Test	Scaling Ratio
l est volume	(Test Volume / Plant Volume)
400 mL	1.41×10 ⁻⁷
800 mL	2.82×10-7

Material surface areas and masses per unit of test vessel volume were selected to be within an order of magnitude of the average material quantities per unit of BWR sump volume based on a BWR survey provided by the BWROG (Attachment A). The area of aluminum was increased above that of the average plant area ratio to ensure that the coupon area could be accurately measured and yield a correspondingly accurate measurement of aluminum release rate.

4.3 Solution Chemistry

Dissolution tests were performed at BWR suppression pool chemistries representing the non-SLC injection scenario, which was simulated using unbuffered deionized water, and in a sodium pentaborate buffered chemistry, representing the SLC injection scenario. The pH was monitored but not controlled. Due to the lack of buffering capacity in the non-SLC cases, the pH of the fluid tended to drift due to material corrosion and dissolution, which would also be expected to occur in a plant suppression pool environment. The pH during the SLC tests was stable between 8.5 and 8.6 (See the results for the SLCS series tests in Section 6).

4.4 Solution Temperature and Timing

Dissolution tests were performed over the temperature profiles included in Table 4-3 through Table 4-9. Temperature profiles were selected to represent the range of temperature profiles included in the BWR survey provided by the BWROG (Attachment A). The temperature and sampling schedule in Table 2 (and similarly for all tables) is interpreted as "...after 23 hours, capture an ICP sample, and keep temperature at 200°F. After 24 hours have passed, capture an ICP sample, reduce temperature to 180°F...." etc. Although each listed time and sample number may not involve a temperature change, each listed time does include removal of a sample.

Additionally, some tests were conducted at a constant temperature, and are denoted by the "C" suffix in the test label.



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Table 4-3 - Temperature Profile B-200M200°F Initial Temperature, Mixed Materials

Initial Temperature: 200°F			
ICP	Elapsed	т/°б\	
Sample #	Time (h)		
I	0	200	
2	I	200	
3	4	200	
4	7	200	
5	23	200	
6	24	180	
7	25	180	
8	28	180	
9	32	180	
10	47	180	
11	48	160	
12	52	160	
13	56	160	
14	71	160	
15	72	I 40	
16	76	I 40	
17	80	140	
18	104	140	
19	119	140	
20	120	110	
21	144	110	
22	168	110	



Elapsed Time (h)





Table 4-4 - Temperature Profile B-200 200°F Initial Temperature, Single Material







Elapsed Time (h)



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80

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Table 4-6 - Temperature Profile B-180 180°F Initial Temperature, Single Material





Table 4-7 - Temperature Profile B-160 160°F Initial Temperature, Single Material





Table 4-8 - Temperature Profile B-140 140°F Initial Temperature, Single Material



_				
Initial Temperature: 140°F				
ICP	Elapsed	T (%E)		
Sample #	Time (h)	I(F)		
Ι	0	140		
2	I	140		
3	6	140		
4	24	140		
5	47	110		
6	48	110		



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Table 4-9 - Temperature Profile B-200X200°F Initial Temperature, Single Material







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Temperature was controlled with a circulating regulated hot water bath and agitation was provided by a magnetic stirrer (See Figure I). Thermal transients were as conducted as quickly as possible, and typically occurred within 10 minutes. 1000-ml Pyrex flasks were used for mixed materials tests and 500-mL pyrex and polycarbonate flasks were used for single material tests. Stoppers were used to minimize solution evaporation throughout the test. Temperature and pH were measured and recorded with each sample removal, conductivity was measured at the end of the test, and temperature was recorded continuously using LabView software and in-vessel thermocouples. The fluid within the test vessels was typically not replenished over the duration of each test, and the final mass and volume of the fluid was weighed at the end of the test to calculate evaporation loss. Samples were measured volumes, so sample volume loss could be calculated.

Elemental release was measured by ICP, which provided the dissolved elemental concentrations but not details about the molecular structures. For all samples, concentrated nitric acid was added to the sample being analyzed to redissolve any suspended solids prior to ICP analysis. The exceptions were some of the LCS tests where not all iron corrosion products could be redissolved, even when stirred with pure concentrated nitric acid.

5 TEST PROCEDURE

Each test used a similar procedure based on the test matrix shown in Table 5-1 and the temperature profiles included as Table 4-3 through Table 4-9.

5.1 Materials

5.2 Pre-Test Preparation

Metals: Metallic coupons were precision cut by a machine shop from metallic sheets and drilled to allow for secure suspension with stainless steel wire in the test flasks, as shown in Figure 2. Coupons were used as-is (i.e., no polishing), and rinsed with RO water followed by methanol or acetone, and dried in a 105°C oven for at least two hours prior to pre-test weighing, measurement, and photographing.





Figure 2 – Sample Pre-Test Metallic Coupon

Nukon:

Nukon was used as provided by the manufacturer in a single shredded state, and dried for at least 2 hours at 105°F prior to weighing. To ensure that the Nukon was representative of the Nukon/fiberglass installed in the BWRs, it was not boiled (as is common in head loss testing) prior to dissolution testing. Nukon was weighed and photographed prior to testing, and was not rinsed prior to testing. Nukon was caged in a section of Teflon tubing with stainless steel wire as shown in Figure 3.



Figure 3 - Sample Pre-Test Nukon

Galvanic-Effect Metals: A separate galvanic effect test (GC-B-200) evaluated the effect of a galvanic cell on corrosion rates. For the galvanic effect test(s), the zinc coupon was attached to the LCS coupon using a small stainless steel hardware. A tight electrical connection was used to allow for a galvanic cell to develop and accelerate material release.



Figure 4 – Galvanic Cell Construction



Figure 5 – Galvanic Cell

5.3 Apparatus

Tests were conducted in Erlenmeyer flasks that were partially submerged in hot water baths (See Figure 1) with temperature control capability ranging from 110° to 205°F. The single material tests used 500mL flasks, and the control and mixed material tests, including the galvanic cell test, used 1000mL flasks. Initially Pyrex flasks were used, but polycarbonate flasks were used for the majority of tests. The temperature of the fluid in the flasks were monitored for the test duration and recorded.

During testing, flasks were loosely sealed using rubber stoppers to avoid evaporation. A typical flask layout is shown in Figure 6, and galvanic cell construction is shown in Figure 4.

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Figure 6 – Single Material (Metallic Coupon) Flask Layout (LabView Thermocouples were used in most tests instead of standalone thermometers)

5.4 Post-Test Processing

All Metals:

Coupons were photographed, then rinsed with ASTM Type I DI water at the conclusion of each test to remove any residual impurities and the waste solution captured and labeled, followed by methanol or acetone, and the coupons dried in a 105°C oven for at least two hours prior to post-test weighing and measurement. Galvanic cell metals were disassembled and weighed separately. Observations were noted and the coupons individually bagged, labeled, and stored for post-test analysis.

Nukon:

After test completion, Nukon was photographed. Nukon was not rinsed to avoid loss of loose material. Nukon samples were dried in an oven at 105°C for at least 8 hrs prior to final weighing, and individually bagged, labeled and stored for posttest analysis.

5.5 Sample collection and Measurement

Solution samples were collected using a pipette and stored in polypropylene centrifuge tubes according to the schedules included with each test procedure. Each sample was observed before



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and after cooling to detect visible solids. ICP samples were 6 mL and their volumes measured precisely and recorded, and the pH of ICP samples was measured after cooling to 76°F, before storage for ICP analysis. ICP analytes included Na, Al, Si, Ca, Fe, and Zn, depending on the dissolution sample being measured. The samples for any given test were all analyzed after a single ICP calibration to minimize measurement variability.

After each test was completed, a filtered sample was taken at the test temperature to remove any possible suspended solids. The difference in measured concentrations between the final unfiltered sample and filtered sample provides insight into the amount of suspended solids in the solution.

Sample conductivity was measured using a diluted portion of the sample due to sample volume requirements. Final conductivity was measured directly.

5.6 Test Fluid

After each test, the remaining volume of fluid was weighed and the volume measured. Any visible solids were photographed while still in the flask, filtered and stored.

5.7 Test Matrix

Table 5-1 contains the test matrix for dissolution testing performed in this test program.

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ļ	au	ne	J -	1 -		est	1	Iauix	

Test Label	Material	Amo	unt	Temperature Profile	Test Duration	
	AI	8.00	cm ²			
	Zn	28.00	cm²			
MIXED-B-200M	Low Carbon Steel (LCS)	28.00	cm²	B-200M	7 days	
	NUKON	0.80	g			
AI-B-200	Al	4.00	cm²	B-200	48 hrs	
AI-B-160/200	Al	4.00	cm²	B-160/200	72 hrs	
AI-B-180	Al	4.00	cm ²	B-180	48 hrs	
AI-B-160	Al	4.00	cm²	B-160	72 hrs	
Al-B-140	AI	4.00	cm ²	B-140	48 hrs	
Zn-B-200	Zn	14.00	cm²	B-200	48 hrs	
Zn-B-160/200	Zn	14.00	cm ²	B-160/200	72 hrs	
Zn-B-180	Zn	14.00	cm ²	B-180	48 hrs	
Zn-B-160	Zn	14.00	cm ²	B-160	72 hrs	
Zn-B-140	Zn	14.00	cm ²	B-140	48 hrs	
LCS-B-200	LCS	14.00	cm ²	B-200	48 hrs	
LCS-B-160/200	LCS	14.00	cm ²	B-160/200	72 hrs	
LCS-B-180	LCS	14.00	cm ²	B-180	48 hrs	
LCS-B-160	LCS	14.00	cm ²	B-160	72 hrs	
LCS-B-140	LCS	14.00	cm ²	B-140	48 hrs	
NUKON-B-200	NUKON	0.40	g	B-200	48 hrs	
NUKON-B-160/200	NUKON	0.40	g	B-160/200	72 hrs	
NUKON-B-180	NUKON	0.40	g	B-180	48 hrs	
NUKON-B-160	NUKON	0.40	g	B-160	72 hrs	
NUKON-B-140	NUKON	0.40	g	B-140	48 hrs	
GS-B-200	GS	14.00	cm ²	B-200	48 hrs	
GS-B-160/200	GS	14.00	cm ²	B-160/200	72 hrs	
GS-B-180	GS	14.00	cm²	B-180	48 hrs	

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Test Label	Material	Amount		Temperature Profile	Test Duration	
GS-B-160	GS	14.00	cm²	B-160	72 hrs	
GS-B-140	GS	14.00	cm ²	B-140	48 hrs	
CONTROL-B-200M	NONE	NONE	n/a	B-200M	7 days	
	Zn	14.00	cm ²			
GC-B-200	Low Carbon Steel (LCS)	28.00	cm²	B-200	48 hrs	
AI-SLCS-140	AI	4.00	cm²	B-160/200	48 hrs	
AI-SLCS-160	AI	4.00	cm ²	B-160	72 hrs	
AI-SLCS-160/200	AI	4.00	cm²	B-160/200	72 hrs	
AL-SLCS-180	AI	4.00	cm ²	B-180	48 hrs	
AL-SLCS-200	AI	4.00	cm²	B-200	48 hrs	
LCS-SLCS-200	LCS	14.00	cm ²	B-200	48 hrs	
ZN-SLCS-160	ZN	14.00	cm²	B-160	72 hrs	
CONTROL-PC-200	NONE	NONE	cm ²	B-200	48 hrs	
NUKON-SLCS-200	NUKON	0.40	g	B-200	48 hrs	
	AI	8.00	cm ²			
and the second second	Zn	28.00	cm ²	The Alter		
MIXED-PC-200MA	Low Carbon Steel (LCS)	28.00	cm²	B-200M	7 days	
	NUKON	0.80	g			
	AI	8.00	cm²			
	Zn	28.00	cm²		and and and and a second and as second and a	
MIXED-SLCS-200MA	Low Carbon Steel (LCS)	28.00	cm²	B-200M	7 days	
	NUKON	0.80	g			
AI-SLCS-200X	AI	4.00	cm ²	B-200X	15 days	
GS-B-200X	GS	14.00	cm²	B-200X	15 days	
ZN-B-160C	Zn	14	cm ²	160° Constant		
AL-SLCS-200A	AI	4.00	cm²	B-200	48 hrs	
LCS-SLCS-200A	LCS	14.00	cm ²	B-200 48 hrs		

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Test Label	Material A		unt	Temperature Profile	Test Duration
LCS-B-160A	LCS	14.00	cm ²	B-160	72 hrs
NUKON-SLCS-200A	NUKON	0.40	g	B-200	48 hrs
	AI	8.00	cm²	in the second seco	
	Zn	28.00	cm²		
MIXED-PC-200MA	Low Carbon Steel (LCS)	28.00	cm²	B-200M	15 days
	NUKON	0.80	g		
AL-SLCS-160-2011	AI	4.00	cm ²	B-160	72 hrs
ZN-B-160-2011	Zn	14.00	cm²	B-160	72 hrs
AL-SLCS-160C	AI	4.00	cm ²	160° Constant	72 hrs
ZN-B-160-C-2011	Zn	14.00	cm²	160° Constant	72 hrs
	AI	8.00	cm ²		
	Zn	28.00	cm ²		
MIXED-PC-200X	Low Carbon Steel (LCS)	28.00	cm²	B-200X	15 days
	NUKON	0.80	g		
	Al	8.00	cm²		
	Zn	28.00	cm²		
MIXED-SLCS-200X	Low Carbon Steel (LCS)	28.00	cm²	B-200X	15 days
	NUKON	0.80	g		

6 TEST RESULTS AND ANALYSIS

Note: All test data, photographs and logs are included as Attachment B.

6.1 Experimental Control (CONTROL)

6.1.1 Experimental Control - ICP Data

The CONTROL-B-200M test was conducted in parallel with MIXED-B-200M, but included no coupons or insulation for dissolution. The test was conducted as a control in a Pyrex flask to

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evaluate changes in the test fluid chemistry due to the test vessel. ICP data for the control test is included as Figure 7.

Note: Where temperature differentials are indicated on ICP plots, the accuracy of the temperature change timing indication is approximately ± 1 hr.





6.1.2 Experimental Control - Mass and Dimensional Changes

	Table 0-1	Summary of reserveasure	intents control [/.cc. b]	
		Maximum Measured	Filtered Sample	Mass of
	ICP Constituents	Concentrations	Concentrations	Precipitate
		mg/L	mg/L	g
Al	Al	0.046	0.036	
LCS	Fe	0.003	0.062	
Zinc	Zn	0.106	0.002	1.002-03
NUKON	Ca / Na / Si	2.032 / 1.089 / 4.026	1.9 / 0.812 / 3.976	

Tat	ole 6	- -	Summary	y of	Test	Measur	ements	- 1	Control	[Att.	B	
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Note: Balance is ± 0.0005 g.



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6.1.3 Experimental Control - Photographs



Figure 8 CONTROL-B-200M – After I hr



Figure 9 CONTROL-B-200M – After 168 hr

No changes to the test fluid were observed, and there were no samples to observe during the experimental control test.

6.1.4 Experimental Control - Discussion

The Pyrex test vessel released ~4 ppm of silicon into the test fluid, as well as 1 ppm Na, and 2 ppm Ca. The measured levels of aluminum, iron and zinc were all below 0.2 ppm, which is the "Practical Limit of Quantification" for the ICP analyzer, and values below this limit are of questionable accuracy. Measured analyte concentrations increased essentially monotonically over time, indicating that no measurable precipitation or redeposition occurred. Filtered sample concentrations were essentially equal to measured solution concentrations, indicating that no precipitation or deposit of dissolved materials occurred. The measured mass of solids was 1.0 mg, which is likely due to measurement variability rather than actual precipitation.



The experimental control ICP results show that Pyrex releases measurable amounts of Na, Ca, and Si. In this test program, Pyrex test vessels were used for the mixed materials test as well as several of the earlier tests in the test program before the change to polycarbonate test vessels. A table listing tests performed in Pyrex glassware is included as Table 6-2.

Table 6-2 – Dissolution	Tests	using	Pyrex	Glassware
		•	٦	

CONTROL-B-200M
GC-B-200
GS-B-160/200
GS-B-180
LCS-B-140
LCS-B-160/200
MIXED-B-200M
NUKON-B-140
NUKON-B-160/200
NUKON-B-200
ZN-B-160/200
ZN-B-180

The amount of solids collected at the end of the experimental control test was 1.0 mg. Since no materials were added to the experimental control and no dissolved materials show any reduction in concentration from the maximum concentration to the filtered sample, or the room-temperature end-of-test sample, the 1.0 mg of measured solids is likely due to measurement uncertainty and experimental variability.

6.2 Aluminum

6.2.1 Aluminum - ICP Data - Set I

Aluminum ICP and pH data for the first set of aluminum tests is included below in Figure 10 and Figure 11. Figure 11 provides a detailed view of the AL-B-140, -180, -160 and -200 tests.



Figure 10 - Aluminum Concentrations and pH vs. Time - AL-B Series Tests










Figure 13 – Long Term Aluminum SLCS Test Results – AL-SLCS-200X

6.2.2 Aluminum - Mass and Dimensional Changes

Table 6-3 – Summary of Single Material Test Measurements - All	Aluminum
--	----------

	Maximum Dissolved Aluminum	Filtered Sample Dissolved Aluminum	Mass of Solids Collected	Sample Mass Loss	Final Fluid Mass
Test Label	(mg/L)	(mg/L)	(g)	(g)	(g)
AL-B-140	0.016	n/a	0.0002	-0.0022	348.08
AL-B-160A	0.034	0.006	0.005	-0.0008	309.44
AL-B-160-200	0.051	0.013	0.0003	-0.0017	291.96
AL-B-180	0.026	0.012	0.0004	-0.0006	291.32
AL-B-200	0.029	0.009	0.001	-0.0005	295.23
AL-SLCS-160	7.576	9.403	0.0004	0.0017	313.58
AL-SLCS-160C	6.437	6.543	0.0049	0.0017	305.49
AL-SLCS-160-200	8.962	9.531	0.0003	0.0014	265.05
AL-SLCS-200A	7.258	6.977	0.0002	0.0014	292.7
AL-SLCS-200X	4.487	1.235	0.0064	0.0007	359.7

Note: Balance is \pm 0.0005 g. No measurable dimensional changes were observed.



6.2.3 Aluminum - Photographs



Figure 14 Aluminum coupon before test – AL-B-160



Figure 16 Aluminum coupon during test – AL-B-160



Figure 18 - Coupon at 23 hr point of test AL-



Figure 15 Aluminum coupon after test – AL-B-160



Figure 17 Aluminum coupon after test – AL-B-200



Figure 19 - Coupon at end of test AL-SLCS-



SLCS-160 (note, the white spots are bubbles on

the beaker)



Figure 20 – Solution of AL-SLCS-160C after 71 hours

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24,200	2018-0-1						
10000	108.0						
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125.0	1224						
100.50	38.5						
20275	1992						
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1200	20.02.2						
20000	122.22						
100000	10.21						
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Figure 21 – Coupon at end of test AL-SLCS-160C

6.2.4 Aluminum - Discussion

Aluminum coupons did not show any significant changes during single-material dissolution testing with unbuffered water, and no precipitation or color changes in the test fluid were observed during those tests. Coupons did not appear to corrode or react with the unbuffered water.

During the buffered aluminum dissolution tests, some changes to the coupons were observed, including a change to a golden color and darkening of the coupon, as shown in Figure 18 and Figure 19 from test AL-SLCS-160. The concentration of dissolved aluminum measurably increased in all SLCS-series tests, and the final concentration of aluminum ranged from 4.5 mg/L to 9.0 mg/L.

The solution of test AL-SLCS-160C became cloudy after sitting at room temperature for 71 hours, although only 0.2 mg of precipitate was collected, so the precipitate may have either redissolved or passed through the filter paper.

The mass of the coupon did not measurably decrease during any of the unbuffered tests, although it did decrease by as much as 1.7 mg for the buffered tests. This mass decrease corresponds with a dissolved aluminum concentration of 4.25 mg/L, which is the same order of magnitude as the concentrations detected via ICP measurement.

No significant dimensional changes were observed in any of the aluminum tests.



Maximum dissolved aluminum concentrations were, as expected, lower in unbuffered water than in a buffered PVVR environment. All aluminum dissolution tests in unbuffered water rapidly approached a low maximum concentration of aluminum (less than 0.05 mg/L), as shown in Figure 10 and Figure 11, which is well below the practical limit of quantification for the ICP analyzer used to determine elemental concentrations in this test program, which is generally 0.2 mg/L.

Based on the results of the aluminum dissolution tests, it is clear that aluminum dissolution and precipitation do not occur to the extent in an un-buffered BWR suppression pool environment as they do in buffered, basic PWR sump. However, dissolution of aluminum in a basic, buffered environment does occur, and should be considered in BWR chemical effects evaluations.

6.3 Low Carbon Steel (LCS)

6.3.1 LCS - ICP Data - Fe

Iron ICP and pH data is included below in Figure 22 and Figure 23. Figure 23 provides a detailed view of the LCS-B-140, LCS-B-180, and LCS-SLCS-200A tests, which resulted in significantly less observed and measured LCS corrosion than in the rest of the LCS tests. The pH of the LCS tests was generally under pH 7, except for LCS-B-160/200 which climbed to 8.5 and the stable pH of 8.5 of test LCS-SLCS-200A. The reason for the elevated pH of LCS-B-160/200 was unclear, but the elevated pH likely led to the reduced Fe release for that test as compared to LCS-B-160.









6.3.2 LCS - Mass and Dimensional Changes

Table 6-4 –	Summary	of 1	Fest I	Measure	ements -	LCS
	the second s					

	Maximum Dissolved Iron	Filtered Sample	Mass of Precipitate	Sample Mass Loss	Final Fluid Mass
Test Label	(mg/L)	(mg/L)	(g)	(g)	(g)
LCS-B-140	0.2000	0.187	0.0004	0.0013	337.1
LCS-B-160	79.70	2.20	0.0842	0.0644	309.1
LCS-B-160-200	20.40	10.2	0.0236	0.0301	267.5
LCS-B-180	0.036	0.0000	0.0012	0.0006	292.8
LCS-B-200	7.83	0.0002	0.0202	0.0151	277.3
LCS-SLCS-200A	0.011	0.003	0.0003	0.0000	294.9

Note: Balance is \pm 0.0005 g. No measurable dimensional changes were observed.



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6.3.3 LCS - Photographs

LCS corrosion occurred rapidly in LCS-B-160, LCS-B-160/200, and LCS-B-200, but LCS-B-140 and -180 resulted in comparatively low levels of LCS corrosion.





Figure 24 LCS coupon @ 1h – LCS-B-140

Figure 25 LCS coupon @ 47h – LCS-B-140



Figure 26 LCS coupon before test- LCS-B-160



Figure 27 LCS coupon @ 7h– LCS-B-160





Figure 28 LCS Fluid @ 72h – LCS-B-160



Figure 29 EOT Rinse Sample for ICP – LCS-B-160



Figure 30 LCS Coupon @ EOT – LCS-B-160



Figure 31 Solids @ EOT – LCS-B-160





Figure 32 LCS Fluid @ 7h – LCS-B-180



Figure 33 LCS Fluid @ 48h – LCS-B-180



Figure 34 LCS Coupon @ EOT – LCS-B-180



Figure 35 Solids @ EOT – LCS-B-160



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6.3.4 LCS - Discussion

Some LCS coupons lost significant mass during the dissolution tests, with the LCS-B-160 coupon losing 64.4 mg of material over the test duration, corresponding with a dissolved concentration of 161 ppm. The maximum concentration of iron measured during test LCS-B-160 was only ~80 ppm, however, the sample of coupon rinse effluent taken at the end of the test contained over 600 ppm Fe (See Figure 29). This can be explained via the iron corrosion mechanism: the iron on the surface of the LCS coupon reacts with dissolved oxygen to form iron hydroxides (Fe(OH)₂ and Fe(OH)₃) and finally, iron oxide (Fe₂O₃), i.e., rust. These substances tend to be relatively insoluble in water and rust tends to form on and adhere to the metallic surface. Rinsing the coupon physically removes the iron hydroxides and oxides and allows them to be measured via ICP, resulting in the elevated final dissolved iron ICP measurement.

Tests LCS-B-140 and LCS-B-180 lost far less mass than the rest of the tests, losing only 1.3 and 0.6 mg, respectively. The rate of corrosion observed in LCS-B-140 is consistent with the low temperature, but it is not yet clear why LCS-B-180 resulted in so little corrosion and dissolved iron. Adherence of corrosion products to the coupons may also have contributed to the low observed mass loss.

All tests resulted in some collected solids, with the LCS-B-160 resulting in approximately 460 kg of plant equivalent solid mass collected in the filter after testing and cooling to room temperature (\sim 70°F). The easily visible corrosion product (See Figure 31) was rust-colored and appears to be iron oxide.

Iron coupons tended to corrode, lose mass, and dissolve rapidly in most cases, with LCS-B-140 and LCS-B-180 being the exceptions. A comparison between LCS-B-160 and LCS-B-160/200 suggests that corrosion of steel and dissolution of iron is accelerated by reduced pH; this assertion is also supported by existing literature on steel corrosion in aqueous solutions. As expected with steel corrosion, no limit to the quantity of steel that will corrode was observed. Corrosion products continued to form throughout the test, and although solubility limits were most likely reached, the process of steel corrosion continued without apparent limit. Significant variation existed in the measured iron concentration profiles, and more investigation is necessary to quantify the effects of pH and temperature on steel corrosion and dissolution, as well as the effects of steel corrosion and iron dissolution on pH.

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Essentially zero corrosion of the LCS sample was observed during the buffered test, which is consistent with the expected corrosion behavior of iron in a high-pH buffered solution.

6.4 Nukon

6.4.1 Nukon - ICP Data

ICP and pH data for the NUKON-B test series is included in Figure 36 through Figure 39.







Figure 35 – NUKON-B Test Series ICP Data – AI – Higher Range for SLCS





The pH of all unbuffered tests climbed rapidly after the introduction of Nukon, and the rate and maximum pH increased with increasing temperature. The maximum pH measured in the first 24 hours was of the fluid in test NUKON-B-200 of pH 9.5, and the lowest was for tests NUKON-B-140, -160, and -160/200 of approximately pH 8.6. The increase in measured pH is most likely due to dissolving urea in the binder used in Nukon manufacture, and this would explain the rapid shift in pH, as opposed the slower process of dissolving the Nukon itself. Aluminum concentrations increased to measurable levels for all tests, with test NUKON-SLCS-200A showing the fastest aluminum dissolution rate and highest concentration.

Reductions of measured constituent concentrations were observed with changes in pH or temperature, although the reductions were relatively small, \sim 0.5 ppm. No visible precipitation was observed in any of the Nukon tests. There was a slight yellowing of the water observed in the longer-term tests, likely due to Nukon binder dissolution and/or small particles of suspended Nukon fiber, as described in Section 6.4.2.



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The dissolved calcium, silicon and sodium dissolved from Nukon at parallel, although not equal rates, and the dissolution rates increased with increasing temperature. In all but the B-160/200 and B-140 cases, after the 24 hr temperature reduction the concentrations of Ca, Si and Na plateaued. These plateaus correspond with the small drops in measured aluminum concentration measured in the B-160 and B-200 cases, which may indicate a redeposition of species on the Nukon fiber surface as a result of cooling.

Calcium, sodium and silicon concentrations increased without apparent bound, and concentrations were still increasing essentially linearly at the conclusions of all five tests. Longer test durations would be required to determine solubility limits and develop a more representative model of release than linear extrapolation. The sodium concentration for test NUKON-SLCS-200A is above the visible range on the plot below due to the addition of the buffer, which increased the sodium concentration into the 200+ ppm range. Release rates of Ca, Na and Si appeared to increase with increasing temperature.



Figure 38 – NUKON-B Test Series ICP Data - Na

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6.4.2 Nukon - Mass and Dimensional Changes

Table 6-5 - Summary of Test Measurements - Nukon

	Maximum/Filtered Dissolved Aluminum	Maximum/Filtered Dissolved Silicon	Maximum/Filtered Dissolved Sodium	Maximum/Filtered Dissolved Calcium	Mass of Solids Collected	Sample Mass Loss	Final Fluid Mass
Test Label	(mg/L)	(mg/L)	(mg/L)	(mg/L)	(g)	(g)	(g)
NUKON-B-140	0.200/0.213	9.7/9.6	5.0/4.8	2.1/2.0	0.0029	0.0248	334.6
NUKON-B-160	0.275/0.154	23.21/23.19	11.13/11.24	4.47/4.798	0.0047	0.0447	304.4
NUKON-B-160-200	0.535/0.150	80.83/80.29	36.73/36.29	14.15/13.89	0.0001	0.0708	189.2
NUKON-B-180	0.657/0.470	20.57/20.25	10.39/9.866	3.864/3.788	0.0019	0.0437	287.8
NUKON-B-200	0.97/0.55	39.49/41.34	18.04 / 18.24	5.557/ n/a	n/a	0.0416	254.9
NUKON-SLCS-200A	2.25/1.47	24.6/24.85	241.5 / 234.7	4.71 / 4.82	0.0037	0.0438	314.3

Note: Balance is \pm 0.0005 g.

All Nukon samples lost considerable mass, corresponding with a total dissolved concentration range of ~60-180 ppm, not including the sodium in the SLCS test. The mass losses correspond with the measured dissolved concentration range for the Nukon samples, the total of which ranged from ~20-130 ppm. The "missing material" is likely due to physical loss of fiber pieces and binder dissolution, as the amount of material associated with the measured concentrations was



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too small (< 5 ppm) to effect this variance of measurement. All tests resulted in some collected solids, with the NUKON-B-160 resulting in approximately 33.3 kg of plant equivalent solid mass collected in the filter after testing and cooling to room temperature (~70°F). Based on the yellow appearance of the collected solids, the collected mass is most likely a result of fiber erosion. Figure 42 shows collected Nukon fibers on the filter at the end of the test, further supporting the assertion of mechanical fiber loss.

Nukon sample dimensions were not measured.

6.4.3 Nukon - Photographs



Figure 40 Nukon Sample Preparation



Figure 41 Nukon @ 71h – NUKON-B-160





Figure 42 Filter Paper @ EOT, showing Nukon Fibers – NUKON-B-160



Figure 43 Nukon @ Ih – NUKON –B-160

6.4.4 Nukon - Discussion

Nukon elemental release rates appeared to be directly correlatable with temperature, though on the timescales and concentration ranges tested, elemental concentrations (except for aluminum) increased linearly without approaching any apparent limits. Additional testing may require longer timescales to determine solubility limits and reductions of release rate due to dissolved constituents. No precipitation was observed or measured during any of the Nukon tests, although Nukon fibers did erode and were collected during end-of-test filtration.

pH @ 76°F

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6.5 Zinc

6.5.1 Zinc - ICP Data

Zinc and GS ICP data is included below in Figure 44.



Figure 44 - Zinc Concentrations and pH vs. Time

Up to ~2 ppm of zinc (corresponding with 6 kg of dissolved zinc in a typical suppression pool) dissolved in the zinc test series. As with the galvanized steel coupon test results, zinc release was the most rapid over the first 24 hours in the B-160 temperature profile. The B-160/200 case was conducted in Pyrex and had otherwise the same conditions for the first 24 hours as the B-160 case, which was conducted in polycarbonate. The B-160/200 case suggests a zinc-release/solubility inhibitive effect as compared to the B-160 case. The B-160/200 case reached a maximum of only 1.4 ppm in the first 24 hours while the B-160 case reached a maximum of 2.1 ppm in the first 24 hours. The results for the galvanized steel series were similar, as discussed in Section 6.6.1, and support the observed inhibitive effect. Based on the releases from Pyrex (Primarily Si, some Na and Ca), and the known reactivity of zinc with silicon, the observed inhibitive effect may be due to silicon inhibition.

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The zinc concentration profiles measured in tests ZN-B-200 and ZN-B-160/200 indicate some reduction of zinc concentration, as the measured zinc concentration in both tests increased to an upper limit, then decreased over time. Zinc redeposition on the metallic surface and/or oxide layer is a possible mechanism for the reduction of zinc concentration these tests, since no measurable precipitate was collected or observed and the zinc coupons did not lose appreciable mass. The zinc concentration in test ZN-B-160/200 sharply decreased after the 160°-200°F temperature change, which suggests some retrograde solubility behavior. The highest zinc concentrations measured were for the two lowest temperature tests, ZN-B-160 and ZN-B-140.

The galvanic couple resulted in a less total zinc concentration than the ZN-B-200 case for most of the test duration, although there was some scatter of measured concentrations around the 200°-180°F temperature reduction, and the final sample at 48 hrs contained 3 ppm zinc, compared to the previous sample at 47 hrs which contained only 0.75 ppm zinc. Notably, the GC-B-200 test was performed in 800g of water, so twice as much zinc was actually released to achieve the same concentrations as in the 400g tests. GC-B-200 was performed in a Pyrex flask, so silicon inhibition may have reduced the zinc dissolution rate at the higher test temperatures, since an increased rate due to the galvanic effect would be expected. However, tests ZN-B-160/200 and ZN-B-180 were both performed in Pyrex flasks, and GC-B-200 still resulted in a less zinc dissolution for most of the test. It appears that the galvanic couple did increase the zinc release rate, although more testing is required in order to quantify the effect. The pH profile of the galvanic test was similar to that of the ZN tests.

Table 0-0 - Summary of Test Tleasurements - Zinc						
	Maximum Dissolved Zn	Filtered Sample Dissolved Zn	Mass of Solids Collected	Sample Mass Loss	Final Fluid Mass	
Test Label	mg/L	mg/L	g	g	(g)	
ZN-B-140	1.7520	1.649	0.0003	-0.0051	344.2	
ZN-B-160	2.25	2.16	0.0009	-0.0102	312.4	
ZN-B-160/200	1.41	0.4	0.0006	-0.0001	223.8	
ZN-B-180	1.035	0.4710	0.0002	0.0000	279.9	
ZN-B-200	1.45	1.0930	0.0004	-0.0027	278	
GC-B-200	3.017	0.142	0.0321	0.0011	648.8	
ZN-SLCS-160	0.165	0.195	0.0005	0.0000	311.8	

6.5.2 Zinc - Mass and Dimensional Changes

Table 6-6 - Summary of Test Measurements - Zinc

Note: Balance is \pm 0.0005 g. No measurable dimensional changes were observed.



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Zinc coupons did not lose any appreciable mass or undergo any appreciable dimensional changes in any of the ZN series tests, and all coupons except ZN-B-180 gained some mass. During testing, white crystalline growths on coupons were observed, which likely accounts for the measured weight gain. Based on the white appearance of the crystalline growths and the known stability and insolubility in water of zinc oxide, it is likely the nature of these growths. Figure 46 shows some surface discoloration and white depositions on the zinc coupon. The collected solids for all ZN tests were less than what was collected in the experimental control (See Section 6.1). The collected solids for the GC test appear to be primarily iron oxide, as shown in Figure 58.

Mass loss for test GC-B-200 (which was conducted in 800g of water as opposed the 400g used for the ZN series), was 1.1 mg, which correlates with a dissolved zinc concentration of approximately 1.4 ppm. The difference in mass loss between the ZN series and GC test may be due to the larger volume of fluid used, so in the GC test it takes twice as much zinc to dissolve to reach the measured concentrations as it does in the ZN tests. It is not clear whether the zinc release rate was increased by the galvanic couple or if the increased release rate was a result of the increased volume of fluid.

The ZN-SLCS-160 test showed significantly less material dissolution than the B-series tests, with a maximum dissolved concentration of only 165 ppb. This result is consistent with the expected stable behavior of zinc in an alkaline buffered solution.

6.5.3 Zinc - Photographs

Zinc coupons developed some discoloration during testing, and white deposits were observed on the surfaces of some coupons. SEM micrographs of coupons revealed nucleation and growth of material that appeared crystalline in tests ZN-B-160 and ZN-B-200 (See Figure 50 and Figure 52).





Figure 45 Zinc coupon before test – ZN-B-140



Figure 47 Zinc coupon after test – ZN-B-160



Figure 46 Zinc coupon after test – ZN-B-140





Figure 48

Figure 50 SEM of Zinc coupon after test – ZN-B-160



Figure 49 SEM of Zinc coupon after test – ZN-B-140







Figure 51 SEM of Zinc coupon after test – ZN-B-180

Figure 52 SEM of Zinc coupon after test – ZN-B-200



Figure 53 GC-B-200 – Galvanic Couple Coupons and Hardware – Pre-Test



Figure 54 GC-B-200 @ 7 hrs





Figure 55 GC-B-200 @ 23 hrs







Figure 57 GC-B-200 – Galvanic Couple Coupons and Hardware – 168 hrs (Zinc in front)



Figure 58 GC-B-200 – End of Test Filter





Figure 59 Zinc coupon before test – ZN-SLCS-160



Figure 61 Zinc coupon after test – ZN-SLCS-160



Figure 60 Zinc coupon after test – ZN-SLCS-160



Figure 62 Aluminum coupon after test – ZN-SLCS-160

6.5.4 Zinc - Discussion

Measured zinc corrosion and dissolution were measurable in the unbuffered test cases, and although the release rate was highest at 200°F, the highest solubility limit was observed at lower temperatures, 140° and 160°F. Small amounts of collected solids were measured at the conclusion of all tests, but the quantities were significantly lower than those for galvanized steel. Measured pH during all zinc tests was in the 5.9 – 7.2 range, with most tests reaching around pH

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6.6, and ZN-B-200 stabilizing around pH 6.0. The galvanic couple may have elevated the release rate of zinc, but the results are not clear due to the size of the flask used and the use of Pyrex.

Essentially zero zinc corrosion was observed in the buffered ZN-SLCS-160 test case, no dissolved solids were collected via filtration, and no mass loss of the test article was measured.

6.6 Galvanized Steel (GS)

6.6.1 GS - ICP Data

Galvanized steel zinc ICP and pH data is included below in Figure 63. Measured Fe concentrations were generally undetectable, except for less than 2 ppm Fe during test ZN-B-160/200, so Fe concentration plots are not included.



Figure 63 – GS Zinc Concentrations and pH vs. Time

Measured zinc concentrations in galvanized steel dissolution tests followed similar trends as the series of pure zinc tests. Pyrex test flasks were used for the B-160/200 and B-180 temperature profiles for both materials. Most zinc was released in the first ten hours of each test, and the



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asymptotic release profiles indicated that solubility limits were approached, reached, and in the cases of the B-160/200 and B-200 tests, reductions were observed in zinc concentration over time and over temperature changes, notably the 160°-200°F temperature increase. This concentration reduction associated with temperature increases suggests either retrograde solubility behavior or precipitation. Releases were similar for zinc and galvanized steel coupons, and a clear trend differentiating the two was not apparent. Zinc release was maximized at the minimum temperature for both galvanized steel and zinc.

As with the zinc coupon test results, galvanized steel released zinc the most rapidly over the first 24 hours in the B-160 temperature profile. The B-160/200 case was conducted in Pyrex and had otherwise the same conditions for the first 24 hours as the B-160 case, which was conducted in polycarbonate. The B-160/200 case strongly indicates a zinc-release/solubility inhibitive effect as compared to the B-160 case, as the B-160/200 case reached a maximum of only 1.05 ppm in the first 24 hours while the B-160 case reached a maximum of 3.03 ppm in the first 24 hours. The results for the zinc series were similar, as discussed in Section 6.5.1.

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6.6.2 GS - Mass and Dimensional Changes

	Maximum Dissolved Zinc	Filtered Sample Dissolved Zn	Mass of Solids Collected	Sample Mass Loss	Final Fluid Mass
Test Label	mg/L	mg/L	g	g	(g)
GS-B-140	3.152	2.983	0.0010	0.0017	346.2
GS-B-160	3.35	2.86	0.0030	-0.0070	317.2
GS-B-160/200*	1.77	0.2	0.0019	0.0040	230.8
GS-B-180*	1.172	1.1340	0.0003	0.0017	291.65
GS-B-200	1.59	0.6800	0.0021	-0.0043	287.0
GS-B-200X	18.69	2.9790	0.0255	-0.0119	339.5

Table 6-7 – Summary of Test Measurements – Galvanized Steel

* Test conducted in Pyrex flask instead of polycarbonate

Note: Balance is \pm 0.0005 g. No measurable dimensional changes were observed.

Appreciable solids were captured in all galvanized steel tests, although the amount of captured solids were in the range of the experimental control (See Section 6.1) so the extension of these measurements to the plant condition is questionable. Significantly more solids were collected than in the ZN series of tests. GS-B-180 yielded the lowest amount of collected solids, likely due to inhibition of zinc dissolution as discussed in Section 6.6.1. Coupon mass loss was inconsistent, and galvanized steel likely gains some mass due to corrosion product nucleation and growth on the material surface, as observed with zinc coupons in Sections 6.5.2 and 6.5.3.

6.6.3 GS - Photographs

Galvanized steel coupons developed deposits similar to those observed on zinc coupons. A close up of the zinc coupon deposits is shown in Figure 46. The surface appearance changes observed during the GS-B-160 test indicate surface chemical reactions with the test fluid and corrosion of the sample (See Figure 64, Figure 65 and Figure 47). Small amounts of rust were observed along the drilled hole and cuts, due to exposure of the uncoated steel from the drilling and cutting procedures.





Figure 64 GS coupon before test – GS-B-160







Figure 66 GS coupon during test – ZN-B-160



Figure 67 GS coupon after test – GS-B-140

6.6.4 GS - Discussion

As with the ZN series, measured galvanized steel zinc corrosion and dissolution were significant, and although the release rate was highest at 200°F, the highest solubility limit was observed at lower temperatures, 140° and 160°F. Larger amounts of collected solids were measured at the conclusion of all tests than in the ZN test series, and were generally an order of magnitude higher than in the ZN tests, but are still near the experimental control collected solids mass (1.0 mg, see Section 6.1). Measured pH during all GS tests was in the 6.1 – 7 range, with GS-B-160/200 reaching a high of 7.5.



6.7 Mixed Materials

6.7.1 MIXED-B-200M - ICP Data



Figure 68 - MIXED-B-200M - Concentrations and pH vs. Time

6.7.2 MIXED-B-200M - Mass and Dimensional Changes

Table 6-8 – Summar	y of Tes	t Measureme	nts – Mixed	Materia	Test

	ICP	Maximum Measured Concentations	Filtered Sample Concentrations	Mass of Solids Collected	Sample Mass Loss	
Sample	Constituents	mg/L	mg/L mg/L	g	g	
Al	Al	2.040	0.418		0.0025	
LCS	Fe	13.370	3.980	0.0600	-0.0192	
Zinc	Zn	6.020	3.278		0.0015	
Nukon	Ca/Na/Si	5.096 / 12.34 / 17.74	4.902 / 12.29 / 9.217		0.0486	

Note: Balance is \pm 0.0005 g. Nukon also releases aluminum, but that quantity is contained in the elemental aluminum measurement. The metallic coupons did not undergo significant dimensional changes, and the dimensions of the Nukon were not measured. The final mass of the test fluid was 586.1g.



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6.7.3 MIXED-B-200M - Photographs



Figure 69 MIXED-B-200M – 1 hr



Figure 70 MIXED-B-200M – 7 hr



Figure 71 MIXED-B-200M – 144 hr



Figure 72 MIXED-B-200M – 168 hr





Figure 73 MIXED-B-200M – Zinc Coupon – 168 hr



Figure 74 MIXED-B-200M – LCS Coupon – 168 hr



Figure 75 MIXED-B-200M – Aluminum Coupon – 168 hr



Figure 76 MIXED-B-200M – End of Test Filter

6.7.4 MIXED-B-200M - Discussion

The aluminum coupon lost 2.5 mg of mass, corresponding with ~3 ppm of dissolved aluminum, which is I ppm less than the measured maximum aluminum concentration. This differential is likely due to Nukon dissolution which can contribute dissolved aluminum to the solution, as shown in the NUKON test series. The aluminum coupon turned completely black (See Figure 75), which is consistent with behavior observed during PWR testing of aluminum corrosion in a high-phosphate environment. Phosphorus concentration was not measured since none of the



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materials added to the test were expected to release phosphorus. The pH of the solution was above 8.5 for most of the test and reached as high as 9.3, so far more than 3.0 ppm of aluminum would be expected to dissolve.

The LCS coupon gained mass unlike in previous single material LCS tests, and although the cause is not completely clear, the coupon does appear to have undergone significantly less corrosion than in the single material tests (See Figure 74 vs. Figure 30). The oxide layer may have remained on the coupon rather than sloughing off as in other tests The reduced LCS corrosion is likely due to the increased pH of the mixed material test fluid (pH 9) as compared to that of the single material LCS tests (pH ~6-7), since steel corrosion is known to be reduced with an increase in pH.

The zinc coupon lost 1.5 mg of mass, which corresponds to a zinc concentration of ~2 ppm. The maximum zinc concentration was 6.0 mg/L, which is approximately double that of the ZN or GS series tests. The zinc coupon appeared to collect rust spots and white spotting of zinc corrosion products were observed on the coupon; these two factors are the probable causes of the differential between coupon mass lost and measured zinc in solution. Most of the zinc release occurred during the 48-hr 140°F hold, supporting the retrograde solubility behavior seen in the GS and ZN series. The measured zinc concentration dropped sharply during and after the 140°-110°F temperature reduction. Over the first 24 hours, the measured zinc concentration reached a maximum of only 0.5 ppm, contrasting with the 1.5 ppm reached over the same time period and temperature (200°F) in test ZN-B-200. The reduction of zinc release rate may be due to inhibition as a result of the elevated silicon concentrations from dissolving Nukon.



6.7.5 MIXED-PC-200MA ICP Data



Figure 77 - Plot of ICP Data from Test MIXED-PC-200MA

6.7.6 MIXED-PC-200MA - Mass and Dimensional Changes

Table 6-9 -	Summary	of Test	Measurements -	- MIXED-PC-2	00MA

Sample	ICP Constituents	Maximum Measured Concentations	Filtered Sample Concentrations mg/L	Mass of Solids Collected	Sample Mass Loss
Al	Al	1.324	0.346	0.0908	0.0044
LCS	Fe	8.432	7.982		0.0241
Zinc	Zn	2.594	2.502		-0.0172
Nukon	Ca/Na/Si	6.379/21.310/30.76	6.302/21.080/25.24		0.1000

Note: Balance is \pm 0.0005 g. Nukon also releases aluminum, but that quantity is contained in the elemental aluminum measurement. The metallic coupons did not undergo significant dimensional changes, and the dimensions of the Nukon were not measured. The final mass of the test fluid was 588.7g.



6.7.7 MIXED-PC-200MA- Photographs



Figure 78 MIXED-PC-200MA – 7 hr



Figure 79 MIXED-PC-200MA – 28 hr



Figure 80 MIXED-PC-200MA – 52 hr



Figure 81 MIXED-PC-200MA – 96 hr





Figure 82 MIXED-PC-200MA Zn at 120 hr



Figure 83 MIXED-PC-200MA LCS at 120 hr



Figure 84 MIXED-PC-200MA at 168 hr



Figure 85 MIXED-PC-200MA – Al Coupon – End of Test

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Figure 86 MIXED-PC-200MA – End of Test Filter



Figure 87 MIXED-PC-200MA – LCS – End of Test



Figure 88 MIXED-PC-200MA – Zn – End of Test



Figure 89 MIXED-PC-200MA - LCS End of Test

6.7.8 MIXED-PC-200MA - Discussion

The aluminum coupon lost 4.4 mg of mass, corresponding with ~7.5 ppm of dissolved aluminum, which is 6 ppm less than the measured maximum aluminum concentration. This differential is likely due to Nukon dissolution, which can contribute dissolved aluminum to the solution. The aluminum coupon turned black/brown (See Figure 78) as in previous tests. Phosphorus concentration was measured to be essentially zero for the entire test. The pH quickly rose to 9.3 in first 24 hours of the test, and remained very steady at ~9.3 for the remainder of the test.


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The LCS lost 24.1 mg of mass, unlike test MIXED-B-200MA where it gained mass. The variation observed in mass-loss of corroded coupons may be due to the lack of surface rinsing, so only loss due to corrosion products that physically detach from coupon are measured by the end of test weighing. The reduced LCS corrosion as compared to the single material tests is likely due to the increased pH of the MIXED-PC-200MA test fluid (pH 9.3) as compared to that of the single material LCS tests (pH ~6-7), since steel corrosion is known to be reduced with an increase in pH. The maximum iron concentration was 8.4 mg/L, which corresponds with a total dissolved iron quantity of 5.0 mg. The difference between the dissolved iron quantity and mass loss of the coupon is likely due to both precipitation of iron oxide and the lack of coupon rinsing which can leave corrosion products adhered to the coupon.

The zinc coupon gained 1.7 mg of mass, probably due to the formation of the white material on the surface which appears to be zinc oxide. The maximum zinc concentration was 2.5 mg/L, which is similar to that of the ZN and GS series tests. The zinc coupon appeared to collect rust spots and white spotting of zinc corrosion products were observed on the coupon. Most of the zinc release occurred during the 48-hr 140°F hold, supporting the retrograde solubility behavior seen in the GS and ZN series. The measured zinc concentration did not drop after the 140°-110°F temperature reduction as it did in test MIXED-B-200M. Over the first 24 hours, the measured zinc concentration reached a maximum of 1.3 ppm, similar to the 1.5 ppm reached over the same time period and temperature (200°F) in test ZN-B-200.

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6.7.9 MIXED-SLCS-200MA ICP Data



Figure 90 - Plot of ICP Data from Test MIXED-SLCS-200MA

6.7.10 MIXED-SLCS-200MA - Mass and Dimensional Changes

	Table 6-10 – Summar	y of Test Measurements -	– MIXED-SLCS-200MA
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2007 	ICP	Maximum Measured Concentations	Filtered Sample Concentrations	Mass of Solids Collected	Sample Mass Loss
Sample	constituents	mg/L	mg/L	g	g
Al	Al	10.81	10.890	- 0.0551	0.0044
LCS	Fe	0.026	0.013		0.0005
Zinc	Zn	0.688	0.277		-0.0049
Nukon	Ca/Na/Si	1,758 / 152 6 / 5 41	1.75 / 156.1 / 5.01		n/a

Note: Balance is \pm 0.0005 g. Nukon also releases aluminum, but that quantity is contained in the elemental aluminum measurement. The metallic coupons did not undergo significant dimensional changes, and the dimensions of the Nukon were not measured. The final mass of the fluid was 613.3g.



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6.7.11 MIXED-SLCS-200MA - Photographs



Figure 91 MIXED-SLCS-200MA – 5 hr



Figure 92 MIXED-SLCS-200MA – 28 hr



Figure 93 MIXED-SLCS-200MA – 52 hr



Figure 94 MIXED-SLCS-200MA – 144 hr









Figure 96 MIXED-SLCS-200MA – AI Coupon – End of Test



Figure 97 MIXED-SLCS-200MA – LCS Coupon – End of Test



Figure 98 MIXED-SLCS-200MA – Zn Coupon – End of Test





Figure 99 MIXED-SLCS-200MA – NUKON Sample – End of Test



Figure 100 MIXED-SLCS-200MA – End of Test Filter

6.7.12 MIXED-SLCS-200MA - Discussion

The aluminum coupon lost 4.4 mg of mass, corresponding with ~2.7 ppm of dissolved aluminum, which is 8.1 ppm less than the measured maximum aluminum concentration. This differential is likely due to Nukon dissolution. The aluminum coupon turned completely black (See Figure 96), as in MIXED-B-200M. Phosphorus concentration was undetectable. The pH of the solution was stable at 8.5 for the entire test, due to the use of the sodium tetraborate buffer.

The LCS coupon did not corrode measurably (via ICP or weight loss) or visibly, which is typical of steel corrosion in high-pH aqueous solutions. The maximum Fe concentration was 0.026 ppm.

The zinc coupon gained 4.9 mg of mass, which may just be attributable to measurement error, since the maximum zinc concentration was only 0.7 mg/L. The zinc coupon did not show any signs of corrosion after the test, zinc, rust or otherwise.



6.7.13 MIXED-PC-200X ICP Data



Figure 101 – Plot of ICP Data from Test MIXED-PC-200X

6.7.14 MIXED-PC-200X - Mass and Dimensional Changes

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Sample	ICP Constituents	Maximum Measured Concentations mg/L	Mass of Solids Collected	Sample Mass Loss
Al	Al	4.43		0.0097
LCS	Fe	11.800	0.0500	0.0709
Zinc	Zn	6.977	0.2533	0.0134
Nukon	Ca/Na/Si	4 566 / 17 720 / 16 300	· · · · · · · · · · · · · · · · · · ·	n/a

Note: Balance is \pm 0.0005 g. Nukon also releases aluminum, but that quantity is contained in the elemental aluminum measurement. The metallic coupons did not undergo significant dimensional changes, and the dimensions of the Nukon were not measured. A filtered sample was not taken at the end of the test. The final mass of the fluid was 888.7g.



6.7.15 MIXED-PC-200X - Photographs



Figure 102 MIXED-PC-200X – 0.5 hr





Figure 104 MIXED-PC-200X – 47 hr



Figure 105 MIXED-PC-200X – 71 hr



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Figure 106 MIXED-PC-200X – 96 hr



Figure 107 MIXED-PC-200X - 119 hr



Figure 108 MIXED-PC-200X – 144 hr



Figure 109 MIXED-PC-200X – 168 hr



Figure 110 MIXED-PC-200X – 192 hr



Figure 111 MIXED-PC-200X – 216 hr



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Figure 112 MIXED-PC-200X – 240 hr



Figure 113 MIXED-PC-200X - 264 hr



Figure 114 MIXED-PC-200X – 336 hr



Figure 115 MIXED-PC-200X – Final Cooled Solution





Figure 116 MIXED-PC-200X - End of Test Filter



Figure 117 MIXED-PC-200X - Aluminum Coupon at End of Test



Figure 118



Figure 119 MIXED-PC-200X – Zinc Coupon at End of Test MIXED-PC-200X – LCS Coupon at End of Test



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Figure 120 MIXED-PC-200X – Nukon at End of Test

6.7.16 MIXED-PC-200X - Discussion

The aluminum coupon lost 9.7 mg of mass, corresponding with ~8.6 ppm of dissolved aluminum, which is ~4 ppm more than the measured maximum aluminum concentration. This differential may be due to redeposition of dissolved aluminum on the Nukon. The aluminum coupon became completely black (See Figure 110), but gradually returned to a gray metallic color during the final hours of the test (See Figure 114). Phosphorus concentration was measured to be essentially zero for the entire test. The pH quickly rose to 9.0 in first 48 hours of the test, and slowly dropped to 7.8 - 8 for the remainder of the test.

The LCS coupon lost 70.9 mg of mass, and based on the plot of the Fe concentration vs. time, most of the corrosion of the LCS occurred during the 140°F and 110°F temperature holds. Since the formation of iron oxide requires oxygen from the air, and oxygen is more soluble at lower temperatures, this result is consistent with expectations. The LCS coupon was not rinsed prior to the final weighing, so more corrosion products may have been formed than what were collected. The maximum iron concentration of 11.8 ppm corresponds to a total dissolved iron mass of 10.5 mg. The variance between the dissolved iron and the mass loss of the coupon can be accounted for by the collected solids at the end of the test, which had a mass of 363.3 mg.

The zinc coupon lost 13.4 mg of mass, although more might have been lost if the sample was rinsed prior to the final weighing. The maximum zinc concentration was 7.0 mg/L, which is higher



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than that of the ZN and GS series tests, and equates to 4.1 mg of dissolved zinc. Examination of the plot of Zn concentration vs. time in Figure 101 indicates that approximately half of the zinc dissolution occurred during the extended 110°F temperature hold, and the concentration at the beginning of the 110°F hold was 3.7 mg/L, which is consistent with the single material tests. The zinc coupon appeared to collect rust spots and white spotting of zinc corrosion products were observed on the coupon. Most of the zinc release occurred during the 48-hr 140°F hold, supporting the retrograde solubility behavior seen in the GS and ZN series. The measured zinc concentration did not drop after the 140°-110°F temperature reduction as it did in test MIXED-B-200M. Over the first 24 hours, the measured zinc concentration reached a maximum of 1.1 ppm, similar to the 1.5 ppm reached over the same time period and temperature (200°F) in test ZN-B-200.

The Nukon released calcium, silicon and sodium at rates that appeared dependent on temperature for the first 120 hours of the test. After the temperature reduction to 110°F at 120 hours, the calcium and sodium concentrations only varied by approximately 15% for the remaining 240 hours. The silicon concentration decreased sharply and steadily during the 110°F temperature hold from 16.3 mg/L to 3.6 mg/L, suggesting precipitation or redeposition on the samples.



6.7.17 MIXED-SLCS-200X ICP Data



6.7.18 MIXED-SLCS-200X - Mass and Dimensional Changes

Table 6-12	 Summary 	of Test	Measurements	– MIXED-SLCS-200X
	our minut y	0	r reason erriertes	

Camala	ICP Constituents	Maximum Measured Concentations	Mass of Solids Collected	Sample Mass Loss
Sample	No. I Later and the second	ing/L	5	5
Al	Al	11.73	0.0376	0.0048
LCS	Fe	0.089		0.0006
Zinc	Zn	0.203	0.0270	-0.0007
Nukon	Ca/Na/Si	2.082 / 143.4 / 6.065		n/a

Note: Balance is \pm 0.0005 g. Nukon also releases aluminum, but that quantity is contained in the elemental aluminum measurement. The metallic coupons did not undergo significant dimensional changes, and the dimensions of the Nukon were not measured. The final mass of the fluid was 599.8g.



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6.7.19 MIXED-SLCS-200X - Photographs



Figure 122 MIXED-SLCS-200X – 0.5 hr



Figure 123 MIXED-SLCS-200X – 23 hr



Figure 124 MIXED-SLCS-200X – 47 hr



Figure 125 MIXED-SLCS-200X – 71 hr





Figure 126 MIXED-SLCS-200X – 96 hr



Figure 127 MIXED-SLCS-200X – 119 hr



Figure 128 MIXED-SLCS-200X – 144 hr



Figure 129 MIXED-SLCS-200X – 168 hr



Figure 130 MIXED-SLCS-200X - 192 hr



Figure 131 MIXED-SLCS-200X – 216 hr









Figure 133 MIXED-SLCS-200X – 264 hr



Figure 134 MIXED-SLCS-200X – 336 hr



Figure 135 MIXED-SLCS-200X – Final Solution







Figure 136 MIXED-SLCS-200X – Aluminum Coupon at End of Test

Figure 137 MIXED-SLCS-200X – LCS Coupon at End of Test





Figure 138 MIXED-SLCS-200X – Zinc Coupon at End of Test

Figure 139 MIXED-SLCS-200X – 168 hr



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Figure 140 MIXED-SLCS-200X – Filters at End of Test

6.7.20 MIXED-SLCS-200X - Discussion

The aluminum coupon lost 4.8 mg of mass, corresponding with 6ppm of dissolved aluminum, which is 8.8 ppm less than the measured maximum aluminum concentration. This differential is likely a result of Nukon dissolution. The aluminum concentration reached 11.7 mg/L, and remained essentially constant for the entire test. The aluminum coupon turned completely black within 23 hours (See Figure 123), which is consistent with previous mixed material tests in this test program. Phosphorus concentration was zero throughout the test. The pH of the solution was 8.5 for the entire test due to the sodium tetraborate buffer.

The LCS coupon did not visibly corrode during this test, as shown in Figure 119, which is consistent with other SLCS LCS tests and the expected behavior of LCS in a high pH, buffered environment. The maximum measured iron concentration was 0.089 mg/L, and the iron coupon lost only 0.6 mg of mass.

The zinc coupon did not gain significant mass, and did not visibly corrode or develop white spots that may be zinc oxide. The maximum zinc concentration was 0.2 mg/L, which is consistent with the single material zinc test results from ZN-SLCS-160.

The Nukon sample released calcium, silicon at rates that appeared dependent on temperature for the first 48 hours of the test. Sodium concentration was not plotted due the high concentration of sodium as a result of the added buffer. Notably, the silicon concentration reached a maximum of only 6 mg/l, far less than the 16.3 mg/L that was reached in test MIXED-PC-200X, which used identical temperature and timing to test MIXED-SLCS-200X, but without the sodium tetraborate buffer. This phenomenon may be due to the increased dissolved aluminum redeposition on the



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Nukon, which could inhibit the release of silicon. Calcium release behaved similarly and reached a maximum concentration of approximately half of what was measured in test MIXED-PC-200X, further supporting this hypothesis. After the temperature reduction to 110°F at 120 hours, the calcium and sodium concentrations only varied by approximately 15% for the remaining 240 hours. The silicon concentration decreased sharply and steadily during the 110°F temperature hold from 16.3 mg/L to 3.6 mg/L, suggesting precipitation or redeposition on the samples.

7 CONCLUSIONS

Since the goal of this test program was to successfully measure and record test data, this test program was a success. The conclusions that can be drawn from each test are indicated in the discussion section of the relevant test or test series.

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