CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

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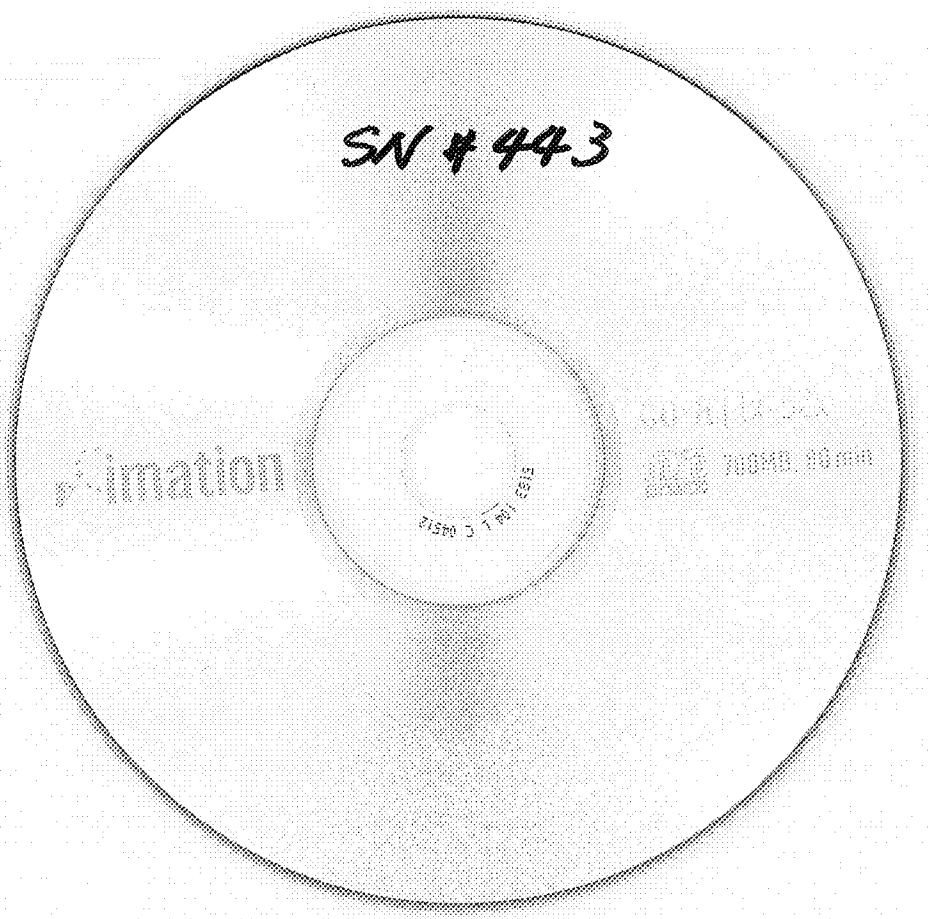
Yi-Ming Pan
ext. 6640

Brian K. Deaby - BKD

Yiming Pan YMP

SN # 443



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Initial Entry for In-Package Chemistry Study

Initial Scientific Notebook Entry for In-Package Chemistry Study

Title: In-package chemistry tests

Tests Performed by: Yi-Ming Pan, Darrell Dunn and Vijay Jain

Objectives: Determine changes to the chemistry of solutions dripping into waste package

Equipments: Internal chemistry test cell, Solartron SI 1287 potentiostat, CorrWare V2.2 data acquisition software, Net Force 586 computer, Waters capillary ion analyzer.

Materials: 316L Heat #P80746

Measurement Parameters: Current, potential, temperature, cation content.

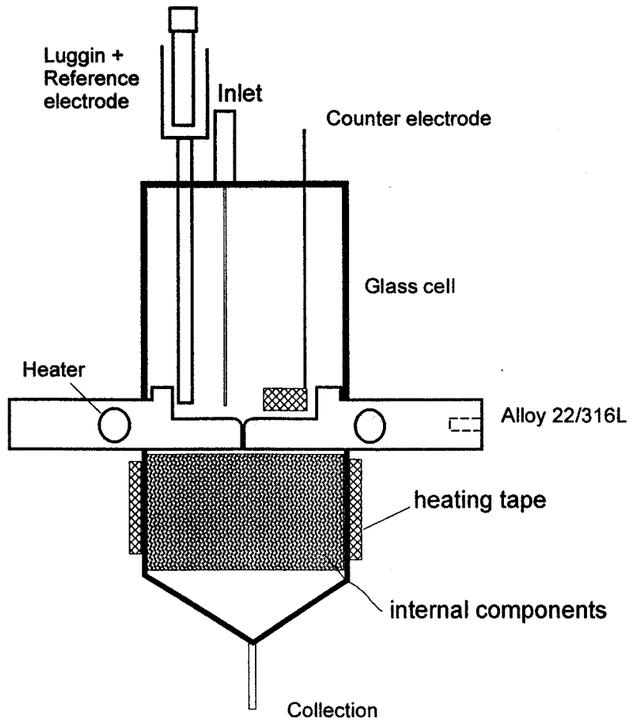
Required Level of Accuracy: Current: ± 100 pA, potential: ± 100 μV, temperature: ± 2.0°C, cation content: below 1 ppm.

Uncertainty and Sources of Error: The solution inside the pit may be diluted during solution extraction. Solution will be extracted using a Pt-syringe needle positioned at the top of the pit.

Yi Pan
2/21/2001

Internal Corrosion Cell Design

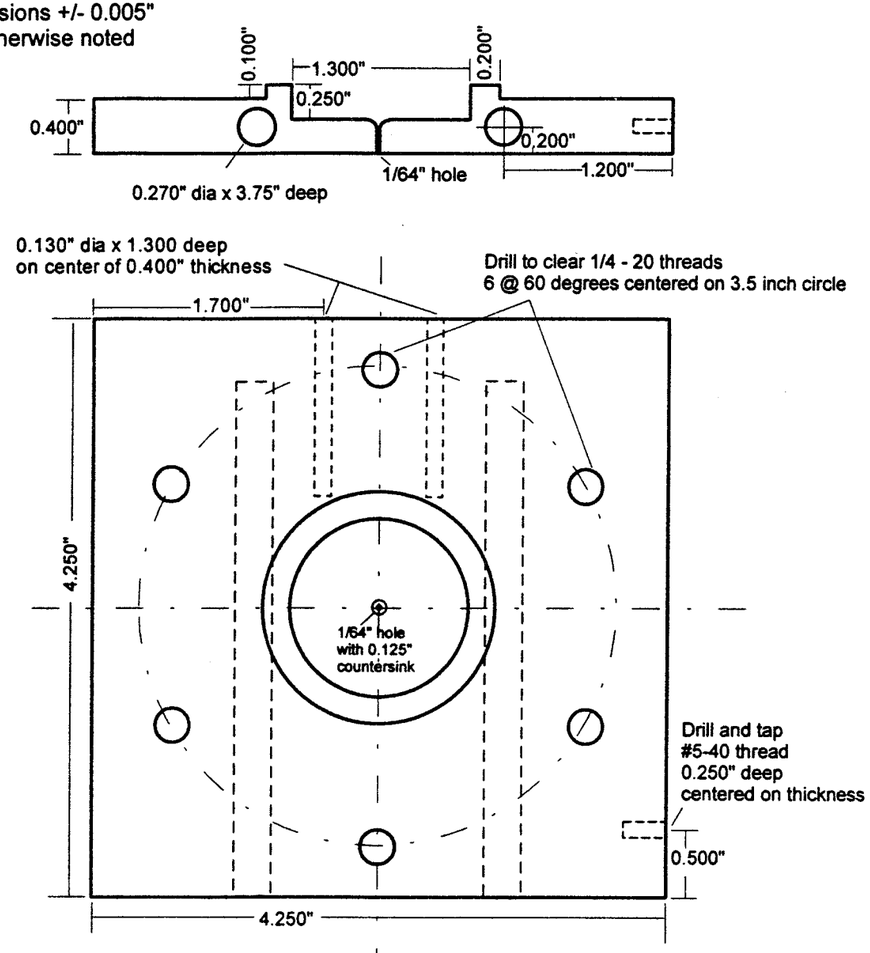
1. Conceptual Design



2. Test Specimen

Internal Chemistry Specimen
Drawing # 20-01402-571-009 rev.01

All dimensions +/- 0.005" unless otherwise noted



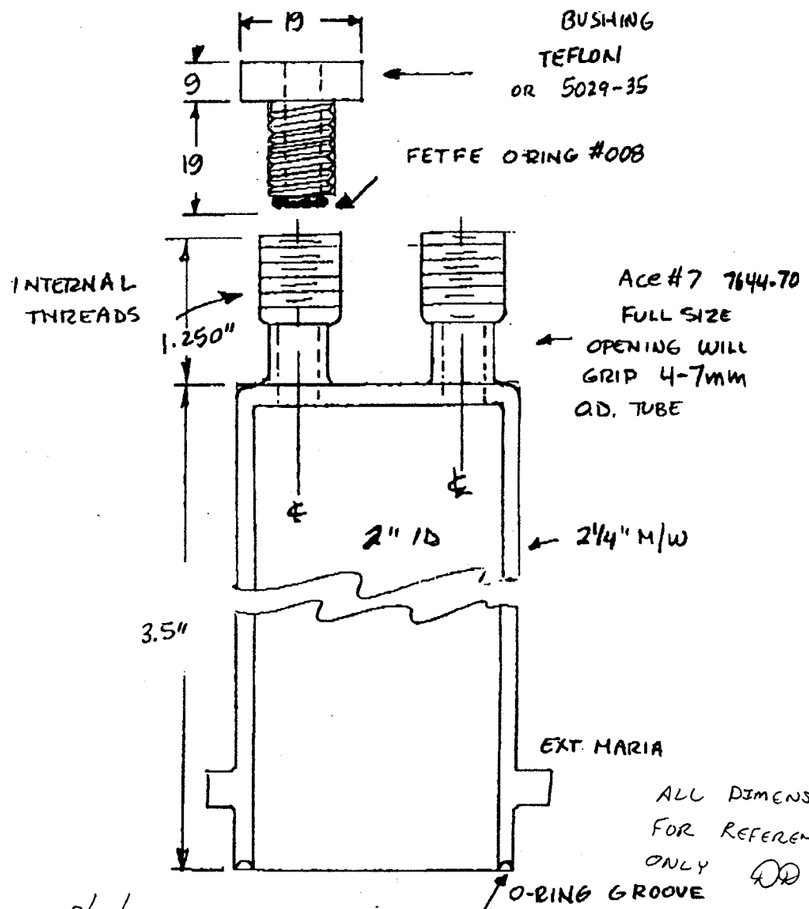
Initiated by D. Dunn 4/03/2001 date

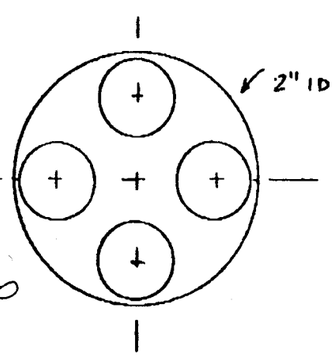
Reviewed by V. Jain 4/3/01 date

QA Approval: B. Mabrito 4/5/2001 date

3. Cell #1

Drawing # 20-01402-571-007
Internal corrosion cell #1





8/17/2000
Initiated by: D. Dunn Date

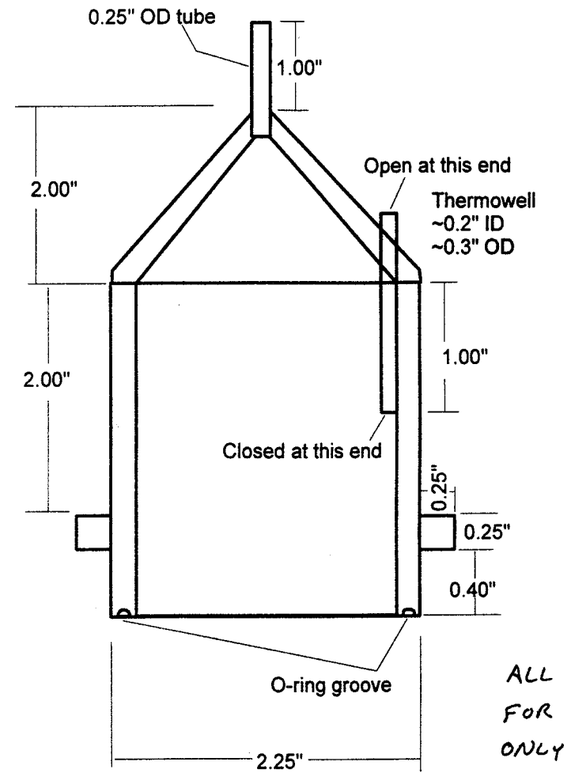
N. Sridhar 8/17/2000
Reviewed by: N. Sridhar Date

for Mark R. Ehrnstrom 8/17/2000
QA approval: B. Mabrito Date

4. Cell #2

Internal chemistry cell #2

SwRI Drawing Number 20.01402.571.008



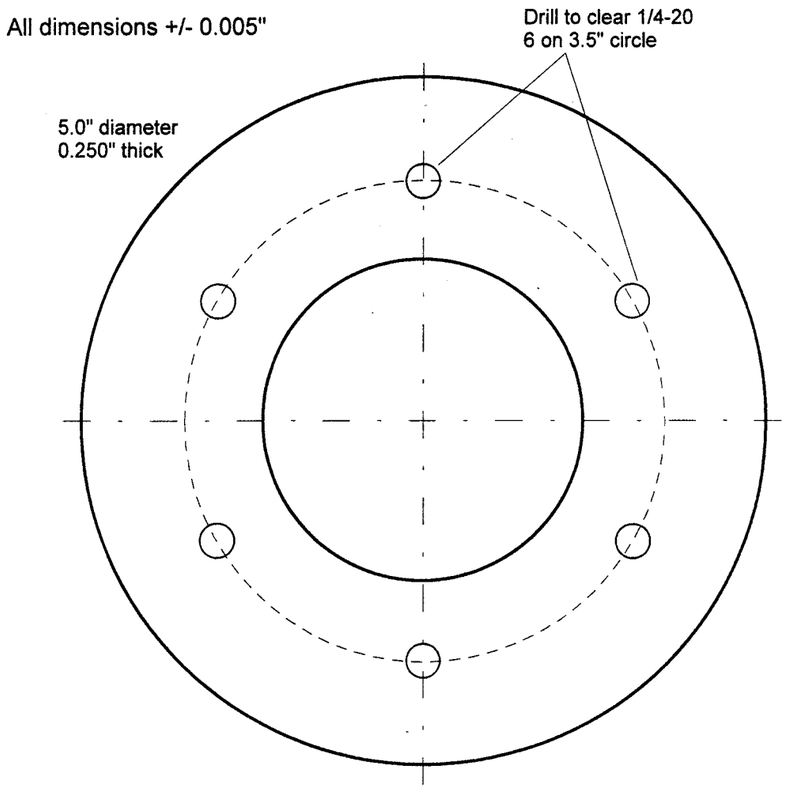
8/17/2000
Initiated by D. Dunn Date

N. Sridhar 8/17/2000
Reviewed by N. Sridhar Date

for Mark R. Ehrnstrom 8/17/2000
QA Approval: B. Mabrito Date

5. Cell Fixture

Drawing # 20-01402-571-010
Internal corrosion cell fixture



Initiated by D. Dunn date

QA Approval: B. Mabrito 8/25/2000 date

Reviewed by N. Sridhar for NS 8/25/00 date

12/1/01

Intchem 10-15 Internal Corrosion Tests

Objective: Measure Current Density at an applied potential of -150 mV$_{SCE}$, -100 mV$_{SCE}$, 0, 100 mV$_{SCE}$, 150 mV$_{SCE}$, and 200 mV$_{SCE}$.

Specimen: 316L SS Heat P80746

Test Environment:

Solution - 1000 ppm Cl^- (as KCl), 4mL in countersink and well

Temperature - R.T.

In Air

Duration: 30 min Rate: 2/sec

Potentiostat: Solartron SI 1287

Reference Electrode: FISHER SCE 13-620-52 SN00042119

Counter Electrode: Pt Flag

Data Files:

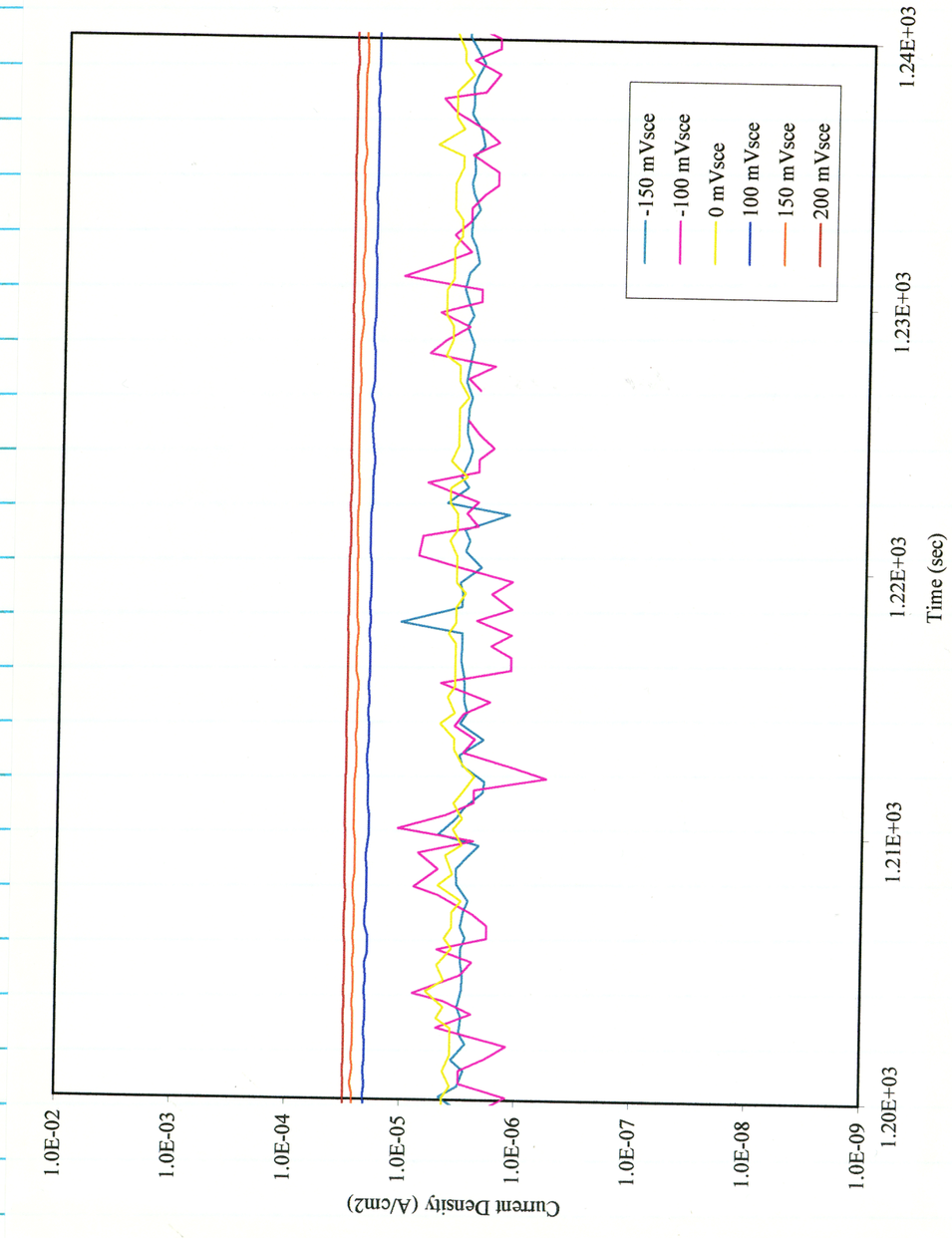
Applied Potential (mV$_{SCE}$)	File ID
-150	intchem 10
-100	intchem 11
0	intchem 12
100	intchem 13
150	intchem 14
200	intchem 15

12/4/01

Note: 30 µL solution extracted at the end of the tests with different applied potentials. This solution is designated as intchem 10.

12/4/01

Current Density For Internal Corrosion Tests Intchem 10-15



3/26/01

Intchem 20-24 Internal Corrosion Tests

Objective: Determine solution chemistry changes as a function of applied potential

Specimen: 316L SS Heat P 80746

Test Environment:

Solution - 1000 ppm Cl^- (as KCl), 4mL in countersink and well

Temperature - R.T.

In Air

Duration: 24 hrs Rate: 0.25/sec

Potentiostat: Solartron SI 1287

Reference Electrode: FISHER SCE 13-620-52 SN 00042119

Counter Electrode: Pt Flag

Solution Extractions:

First Extraction - 5μL from the countersink

Second Extraction - 25μL after first extraction

Data Files:

Applied Potential (mVSCE)	File ID	Date	
0	intchem 20	2/20/01	10:46am
50	intchem 21	2/21/01	11:22am
100	intchem 22	2/22/01	1:52pm
150	intchem 23	2/26/01	9:47am
200	intchem 24	2/27/01	10:40 am

3/1/01

Cation Ion Concentration Measurements by CIA

Objective: Measure cation ion concentration of solutions extracted from internal corrosion tests

Instrument: Waters Capillary Ion Analyzer

Method: Capillary Ion Analysis Method for Alkali and Alkaline Earth Cations

Data Files: data acquisition using Millennium System

Set #1 intchem 10_24 25μL 10_1 dil

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	intchem 10, 10:1 dilution	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 3:09:48 PM	Cation no anal_report
2	1	intchem 20, 10:1 dilution	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 3:22:30 PM	Cation no anal_report
3	1	intchem 21, 10:1 dilution	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 3:35:12 PM	Cation no anal_report
4	1	intchem 23, 10:1 dilution	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 3:47:54 PM	Cation no anal_report
5	1	intchem 24, 10:1 dilution	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 4:00:37 PM	Cation no anal_report
6	1	DI water	Unknown	intchem 10_24 25 uL 10_1 dil	3/8/01 4:48:39 PM	Cation no anal_report

3/8/01

Set #2 intchem 20_24 20_1_5μL_10_1 (20_1 dilution for the 25μL solutions)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	intchem20,20:1	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 3:08:56 PM	Cation no anal_report
2	1	intchem21,20:1	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 3:19:08 PM	Cation no anal_report
3	1	intchem23,20:1	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 3:29:22 PM	Cation no anal_report
4	1	intchem24,20:1	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 3:39:57 PM	Cation no anal_report
5	1	1000kcl,10:1	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 3:50:11 PM	Cation no anal_report
6	1	initchem20,10:1 5uL samples	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 4:00:25 PM	Cation no anal_report
7	1	initchem21,10:1 5uL samples	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 4:10:38 PM	Cation no anal_report
8	1	initchem22,10:1 5uL samples	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 4:20:50 PM	Cation no anal_report
9	1	initchem23,10:1 5uL samples	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 4:31:04 PM	Cation no anal_report
10	1	initchem24,10:1 5uL samples	Unknown	intchem20_24 20_1 _ 5uL_10_1	3/9/01 4:41:16 PM	Cation no anal_report

3/9/01

Set #3 intchem 20_24 5μL 100_1 dil

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	intchem20-2, 100:1 5ul sample	Unknown	intchem20_24_2 5uL 100_1 dil	3/12/01 5:14:46 PM	Cation no anal_report
2	1	intchem21-2, 100:1 5ul sample	Unknown	intchem20_24_2 5uL 100_1 dil	3/12/01 5:24:58 PM	Cation no anal_report
3	1	intchem22-2, 100:1 5uL sample	Unknown	intchem20_24_2 5uL 100_1 dil	3/12/01 5:35:12 PM	Cation no anal_report
4	1	intchem23-2, 100:1 5uL sample	Unknown	intchem20_24_2 5uL 100_1 dil	3/12/01 5:45:24 PM	Cation no anal_report
5	1	intchem24-2, 100:1 5uL sample	Unknown	intchem20_24_2 5uL 100_1 dil	3/12/01 5:55:38 PM	Cation no anal_report

3/12/01

Set #4 std 10 ppm samples

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	std_10ppm Fe2+	Standard	std 10ppm samples	3/27/01 4:11:07 PM	Cation no anal_report
2	1	std_10ppm Ni2+	Standard	std 10ppm samples	3/27/01 4:21:19 PM	Cation no anal_report
3	1	std_10ppm Cr3+	Standard	std 10ppm samples	3/27/01 4:31:33 PM	Cation no anal_report
4	1	std_10ppm Fe3+	Standard	std 10ppm samples	3/27/01 4:41:45 PM	Cation no anal_report
5	1	std_10ppm Fe2+/Ni2+/Cr3+/Fe3+	Standard	std 10ppm samples	3/27/01 4:51:59 PM	Cation no anal_report
6	1	std_10ppm Ni2+/Fe3+	Standard	std 10ppm samples	3/27/01 5:02:13 PM	Cation no anal_report

3/27/01

Set #5 intchem 24_5μL (including dilution of 10 ppm std solutions)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	std_10ppm K+	Standard	intchem 24_5uL	3/28/01 12:28:14 PM	Cation no anal_report
2	1	std_10ppm Na+	Standard	intchem 24_5uL	3/28/01 12:38:26 PM	Cation no anal_report
3	2	std_1ppm Ni2+	Standard	intchem 24_5uL	3/28/01 12:58:52 PM	Cation no anal_report
3	3	std_1ppm Ni2+	Standard	intchem 24_5uL	3/28/01 1:09:04 PM	Cation no anal_report
3	1	std_1ppm Ni2+	Standard	intchem 24_5uL	3/28/01 12:48:38 PM	Cation no anal_report
4	2	std_2ppm Ni2+	Standard	intchem 24_5uL	3/28/01 1:29:31 PM	Cation no anal_report
4	3	std_2ppm Ni2+	Standard	intchem 24_5uL	3/28/01 1:39:43 PM	Cation no anal_report
4	1	std_2ppm Ni2+	Standard	intchem 24_5uL	3/28/01 1:19:17 PM	Cation no anal_report
5	2	std_5ppm Ni2+	Standard	intchem 24_5uL	3/28/01 2:00:08 PM	Cation no anal_report
5	3	std_5ppm Ni2+	Standard	intchem 24_5uL	3/28/01 2:10:20 PM	Cation no anal_report
5	1	std_5ppm Ni2+	Standard	intchem 24_5uL	3/28/01 1:49:56 PM	Cation no anal_report
6	2	std_10ppm Ni2+	Standard	intchem 24_5uL	3/28/01 2:30:49 PM	Cation no anal_report
6	1	std_10ppm Ni2+	Standard	intchem 24_5uL	3/28/01 2:20:37 PM	Cation no anal_report
7	1	1:300 dil of #9 (intchem 24_5μL)	Standard	intchem 24_5uL	3/28/01 2:41:01 PM	Cation no anal_report
8	1	1:500 dil of #9	Standard	intchem 24_5uL	3/28/01 2:51:13 PM	Cation no anal_report
9	1	spike Ni2+ in #9(18uLF11+2uLA8)	Standard	intchem 24_5uL	3/28/01 3:01:27 PM	Cation no anal_report

3/28/01

Set #6 intchem 24_5uL dilution_b

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	1:1500 dilution of #9	Unknown	intchem 24_5uL diluton_b	3/28/01 5:19:47 PM	Cation no anal_report
2	1	1:1000 dilution of #9	Unknown	intchem 24_5uL diluton_b	3/28/01 5:29:59 PM	Cation no anal_report

YP 3/28/01

Set #7 intchem 24_spike

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	spike Fe/Ni in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 11:43:17 AM	Cation no anal_report
1	3	spike Fe/Ni in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 11:53:30 AM	Cation no anal_report
1	1	spike Fe/Ni in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 11:33:06 AM	Cation no anal_report
2	2	spike Cr/K/Na in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 12:13:56 PM	Cation no anal_report
2	3	spike Cr/K/Na in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 12:24:08 PM	Cation no anal_report
2	1	spike Cr/K/Na in 24_5uL_E10	Unknown	intchem 24_spike	3/29/01 12:03:44 PM	Cation no anal_report
3	2	spike Fe/Ni in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 12:44:33 PM	Cation no anal_report
3	3	spike Fe/Ni in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 12:54:47 PM	Cation no anal_report
3	1	spike Fe/Ni in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 12:34:20 PM	Cation no anal_report
4	2	spike Cr/K/Na in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 1:15:11 PM	Cation no anal_report
4	3	spike Cr/K/Na in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 1:25:25 PM	Cation no anal_report
4	1	spike Cr/K/Na in 24_25uL_G10	Unknown	intchem 24_spike	3/29/01 1:04:59 PM	Cation no anal_report

YP 3/29/01

Set #8 intchem 24_5uL dil_c

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	1:600 dilution #9	Unknown	intchem 24_5uL dil_c	4/4/01 3:22:21 PM	Cation no anal_report
2	1	1:800 dilution #9	Unknown	intchem 24_5uL dil_c	4/4/01 3:32:33 PM	Cation no anal_report
3	1	1:900 dilution #9	Unknown	intchem 24_5uL dil_c	4/4/01 3:42:47 PM	Cation no anal_report

YP 4/4/01

Cation Ion Concentration Calculations from CIA Analyses

Objective: Calculate cation ion concentration for solutions extracted from internal corrosion test of 316L SS at 200 mV$_{SCE}$

Method: Integrate the area of specific peak and compare with that of standard solution

Results:

Cation concentrations from anodic dissolution of Type 316L SS at 200 mV$_{SCE}$ in 1000 ppm chloride at room temperature (in unit of ppm)

Cation	5 uL Solution from the Countersink	25 uL Solution after 5 uL Extraction
Fe^{2+}	12,900 ①	550 ④
Ni^{2+}	270 ②	10 ⑤
Cr^{3+}	100 ③	–
K^+	1,650 ②	2,200 ⑤
Na^+	260 ②	30 ⑤

① Calculation using CIA file sample name "1:1500 dilution of #9"

② " " "intchem 24-2, 100:1 5uL sample"

③ " " "1:500 dil of #9"

④ " " "intchem 24, 10:1 dilution"

⑤ " " "intchem 24, 20:1"

Note that in the Cr^{3+} concentration calculation Cr^{3+} peak overlapped with Fe^{2+} peak. Area of the Cr^{3+} peak was estimated based on the peak height in comparison with that of the ~~Fe~~ Ni^{2+} peak.

YP 4/4/01

YP 4/5/01

Solution pH Calculations Based on Hydrolysis Reactions

Objective: Calculate solution pH on the basis of measured cation ion concentrations and associated hydrolysis reactions

I. Fe^{2+} = 12,900 ppm (see page 17)

$Fe^{2+} + H_2O \rightarrow FeOH^+ + H^+$

$pH = 4.75 - \frac{1}{2} \log [Fe^{2+}]$

$[Fe^{2+}] = 12{,}900 \times \frac{1}{10^3} \times \frac{1}{55.85} = 0.23\ M$

$pH = 4.75 - \frac{1}{2} \log 0.23 = 5.07$

II. Ni^{2+} = 270 ppm

$Ni^{2+} + 2H_2O \rightarrow Ni(OH)_2 + 2H^+$

$pH = 6.09 - \frac{1}{2} \log [Ni^{2+}]$

$[Ni^{2+}] = 270 \times \frac{1}{10^3} \times \frac{1}{58.71} = 4.6 \times 10^{-3}\ M$

$pH = 6.09 - \frac{1}{2} \log (4.6 \times 10^{-3}) = 7.26$

III. Cr^{3+} = 100 ppm

$Cr^{3+} + 3H_2O \rightarrow Cr(OH)_3 + 3H^+$

$pH = 1.60 - \frac{1}{3} \log [Cr^{3+}]$

$[Cr^{3+}] = 100 \times \frac{1}{10^3} \times \frac{1}{52.00} = 1.92 \times 10^{-3}\ M$

$pH = 1.60 - \frac{1}{3} \log (1.92 \times 10^{-3}) = 2.15$

Note: All hydrolysis reactions referred to

A. J. Sedriks. Corrosion of Stainless Steels. 2nd edition.

John Wiley & Sons, Inc. 1996. p. 179. Table 5.2.

4/5/01

Solution pH Measurements Using Micro-Reference Electrode

Method: MI-402 Micro-Reference Electrode with Flexible Barrel Microelectrodes, Inc.

Results:

1. Measurement Table

pH measurements of solutions extracted from internal chemistry tests using micro-electrode

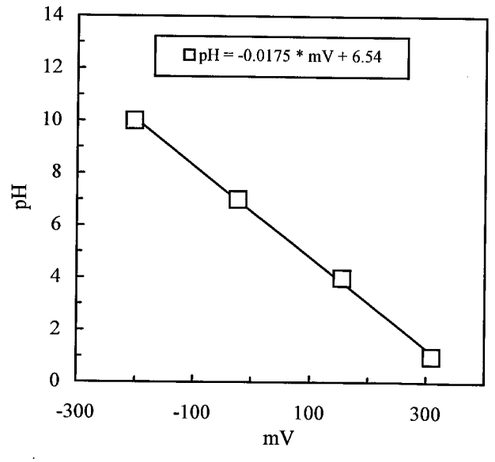
Calibration using buffer solutions

pH	Output(mV)
1	309.5
4	155.2
7	-22.9
10	-202.6

Sample measurements

Sample	Output(mV)	Calculated pH*	[H+]	[H+]x10	Solution pH+
intchem 20 (10:1 dil)	6.7	6.42	3.78E-07	3.78E-06	5.42
intchem 21 (10:1 dil)	-9.2	6.70	1.99E-07	1.99E-06	5.70
intchem 22 (10:1 dil)	2.6	6.49	3.20E-07	3.20E-06	5.49
intchem 23 (10:1 dil)	-13.1	6.77	1.70E-07	1.70E-06	5.77
intchem 24 (10:1 dil)	167.2	3.61	2.43E-04	2.43E-03	2.61

2. Calibration Curve



* Calculated pH was based on the linear regression of the calibration curve in Results 2.

For example,

intchem 20 (10:1 dil)

Calculated pH = $-0.0175 \times 6.7 + 6.54$

= 6.42

+ Solution pH refers to the pH of original solutions extracted from internal corrosion tests after adjustment of a dilution factor.

4/5/01

3. Solution pH summary

Solution pH measured by micro-reference electrode for Type 316L SS tested at various applied potentials in 1000 ppm chloride at room temperature

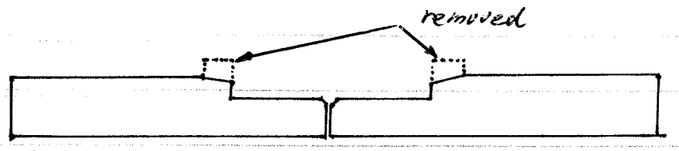
Test ID	Applied Potential (mV_{SCE})	pH 5 uL Solution from countersink
Intchem 20	0	5.42
Intchem 21	50	5.70
Intchem 22	100	5.49
Intchem 23	150	5.77
Intchem 24	200	2.61

4/5/01

Intchem 30-34 Internal Corrosion Tests

objective: Determine solution chemistry changes as a function of applied potential at 60°C

Specimen: 316L SS Heat P80746 with a modified geometry to allow the condensation on the test cell surface moving back to the well



* Reference to the original specimen drawing in page 7

Test Environment: Solution - 1000 ppm Cl^- (as KCl), 4 mL in countersink and well
Temperature - 60°C
In Air

Duration: 24 hrs Rate: 0.25/sec

Potentiostat: Solartron SI 1287

Reference Electrode: Fisher SCE 13-620-52 SN 00042119

Counter Electrode: Pt Flag

Solution Extraction:

First Extraction - 5 μL from the countersink

Second Extraction - 25 μL after the first extraction

Data Files:

Applied Potential (mV_{SCE})	File ID	Date
0	intchem 30*	6/11/01
50	intchem 31*	6/12/01

* Severe corrosion occurred and tests stopped. No solution collected. There may be a contact problem between the counter electrode and the test specimen.

6/12/01

Intchem 60C10-15 Internal Corrosion Tests

Objective: Measure current density variations as a function of applied potential at 60°C

Specimen: 316L SS Heat P80746

Test Environment: Solution - 1000 ppm Cl^- (as KCl), 4mL in countersink and well
Temperature - 60°C
In Air

Duration: 30 min Rate: 2/sec

Potentiostat: Solartron SI 1287

Reference Electrode: FISHER SCE 13-620-52 SN 00042119

Counter Electrode: Pt Flag

Data Files:

Applied Potential (mV$_{SCE}$)	File ID
-150	intchem 60C10
-100	intchem 60C11
0	intchem 60C12
100	intchem 60C13
150	intchem 60C14
200	intchem 60C15 * severe corrosion occurred

Jj Pan
6/18/01

Intchem 90C10-15 Internal Corrosion Tests

Objective: Measure current density variations as a function of applied potential at 90°C

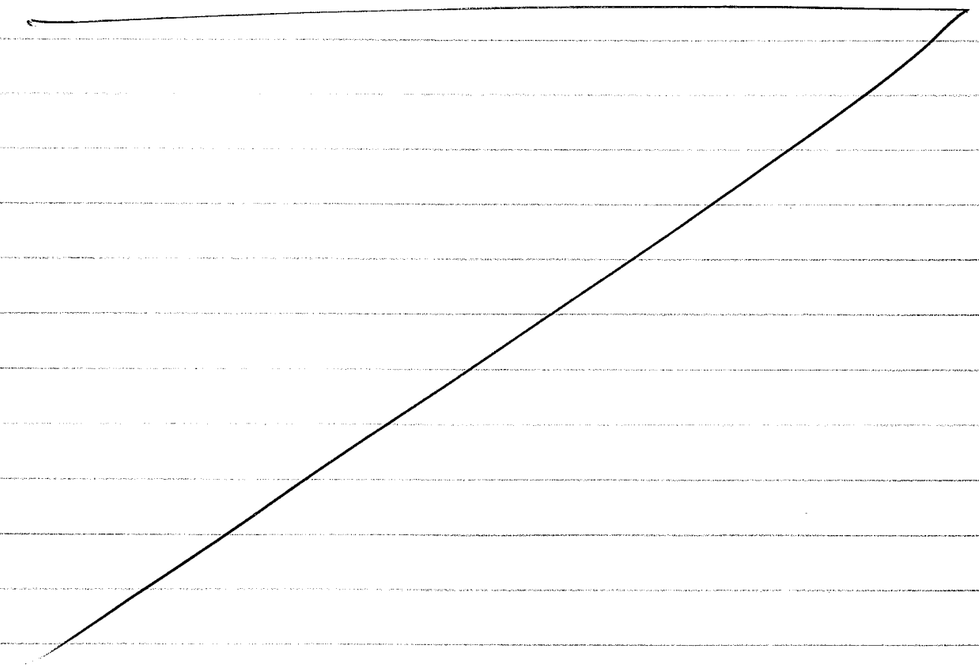
Test conditions: the same in page 22.

Data Files:

Applied Potential (mV$_{SCE}$)	File ID
-150	intchem 90C10
-100	intchem 90C11
0	intchem 90C12
100	intchem 90C13 *

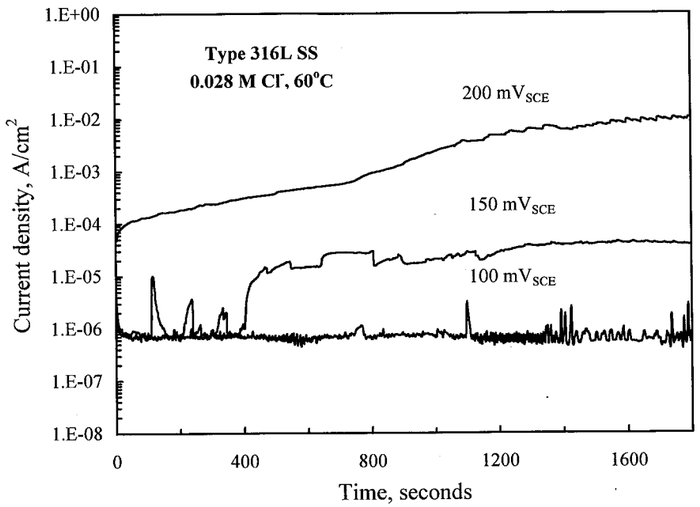
* Severe corrosion occurred and test series stopped.

Jj Pan
6/19/01

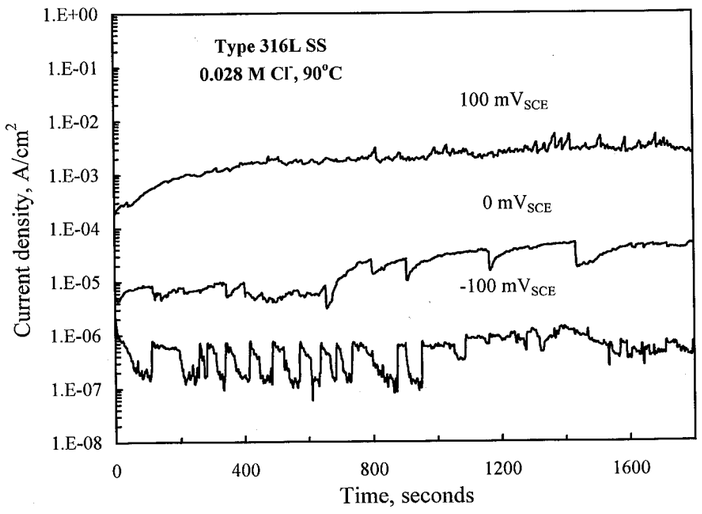


Current Density and Total Charge Results

1. Current density versus time for Intchem 60 C 10-15



2. Current density versus time for Intchem 90 C 10-15



7/12/01

3. List of total charge values for each in-package chemistry test

Total charge measured on type 316L SS in 0.028 M Cl⁻ at various applied potentials and temperatures for each in-package chemistry test

Test ID	Temperature (°C)	Applied Potential (mV$_{SCE}$)	Total Charge (Coulombs)
0.5 h Tests			
Intchem 10	20	-150	0.0056
Intchem 11	20	-100	0.0069
Intchem 12	20	0	0.0070
Intchem 13	20	100	0.0366
Intchem 14	20	150	0.0371
Intchem 15	20	200	0.0563
Intchem 60C10	60	-150	0.0119
Intchem 60C11	60	-100	0.0126
Intchem 60C12	60	0	0.0129
Intchem 60C13	60	100	0.0147
Intchem 60C14	60	150	0.439
Intchem 60C15	60	200	63.68
Intchem 90C10	90	-150	0.0080
Intchem 90C11	90	-100	0.0106
Intchem 90C12	90	0	0.440
Intchem 90C13	90	100	41.37
24 h Tests			
Intchem 20	20	0	0.057
Intchem 21	20	50	0.076
Intchem 22	20	100	0.17
Intchem 23	20	150	1.96
Intchem 24	20	200	3.38

7/12/01

Intchem 60C 20-21 Internal Corrosion Tests

Objective: Determine solution chemistry changes as a function of applied potential at 60°C

Specimen: 316L SS Heat P80746 with a modified geometry (see page 21)

Test Environment: Solution - 1000 ppm Cl^- (as KCl), 5 mL in countersink and well
Temperature - 60°C
In Air

Duration: 24 hrs Rate: 0.25/sec

Potentiostat: Solartron SI 1287

Reference Electrode: FISHER SCE 13-620-52 SN 00042119

Counter Electrode: Pt Flag

Solution Extraction: First extraction - 5 mL from the countersink
Second extraction - 25 mL after first extraction

Data Files:

Applied Potential (mV SCE)	File ID	Date
0	intchem60C20	7/9/01 2:35pm
0	intchem60C20a	7/11/01 2:15pm
50	intchem60C21*	7/10/01 4:26pm

* severe corrosion occurred and test stopped.

7/12/01

Copies sent to QA Records
9/20/2001

Cyclic Polarization of 316L

objective: See Pg #5

Specimen: 316L P80746 Cylinder 600 Grit Finish

Start wt 11.81745 g Sartorious Genius SN# 12809099 cal 5/22/01
End wt 11.81675 g

Solution: 0.028 M K_2SO_4 (~~1,000~~ 2,690 ppm SO_4) 7/18/01
4.880 g K_2SO_4 Lot# 706672
+ DI water To 1000 mls

PH Start: 6.706 Fisher Accumet 950 meter SN# 3440 Cal 7/20/01
PH End = 5.888 PH Probe 13-620-296 SN# 1100208

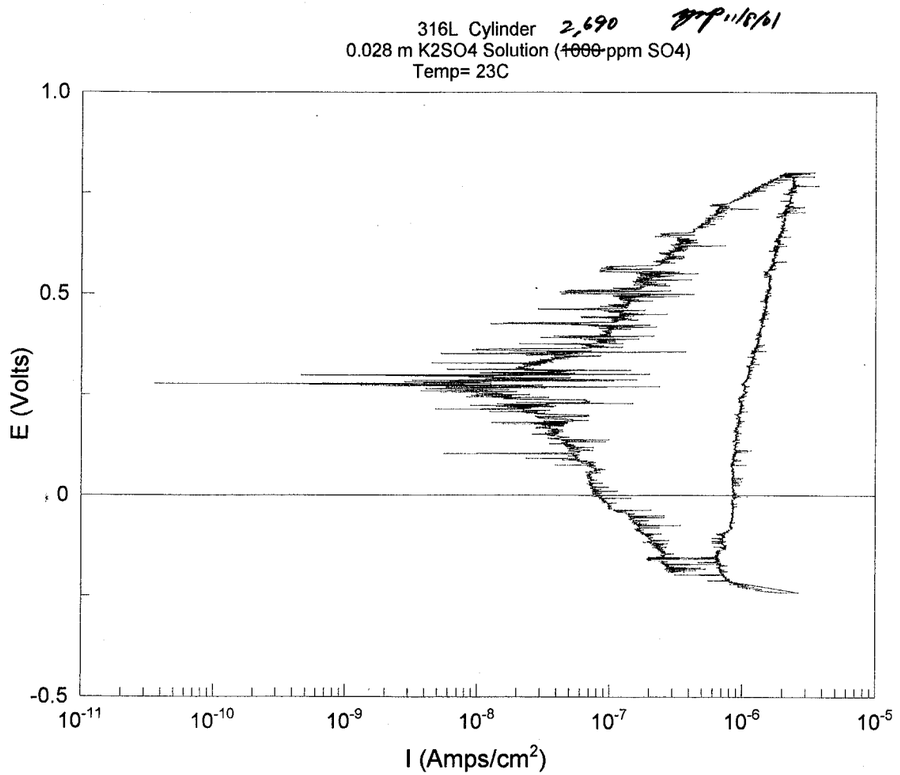
Potentiostat: Solotron 1287
Counter Electrode: PT Flag
Reference Electrode: Fisher 13-620-52 SN# 0042119
Temperature: 23°C Hg Thermometer SN# 183301 cal 6/26/01
Deaerated with 99.999% N_2
Econn: -415 mv Keithley SN# 537418 Cal 2/22/01
Ept: +257 mv

Specimen Examination: Specimen Looks Good No Signs of Pitting or Corrosion
Repolish Specimen for further Testing

Cocaph Pg #28 →

Data Saved As 316L cycpol Test1

10/4/01



10/8/01

Cyclic Polarization of 316L

objective: See Pg #5

Specimen: 316L P80746 Cylinder 600 Grit Finish

start wt: 11.81350g Santorious Genius SN#12809099 Cal 5/22/01

End wt: 11.81346g

Solution: 0.028 m K_2SO_4 (~~1000 ppm~~ 2,690 ppm SO_4) 11/8/01

4.880g K_2SO_4 Lot# 706672

+ DI water To 1000 mls

PH Start = 5.953 Fisher Accumet 950 meter SN#3440 cal 7/20/01

PH End = 4.707 PH Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: PT Flag

Reference Electrode: Fisher 13-620-52 SN# 0042119

Temperature: 23°C Hg Thermometer SN# 183301 Cal 6/26/01

Deaerated with 99.999% N_2

Econn = -248 mv Keithley SN# 537418 cal 2/22/01

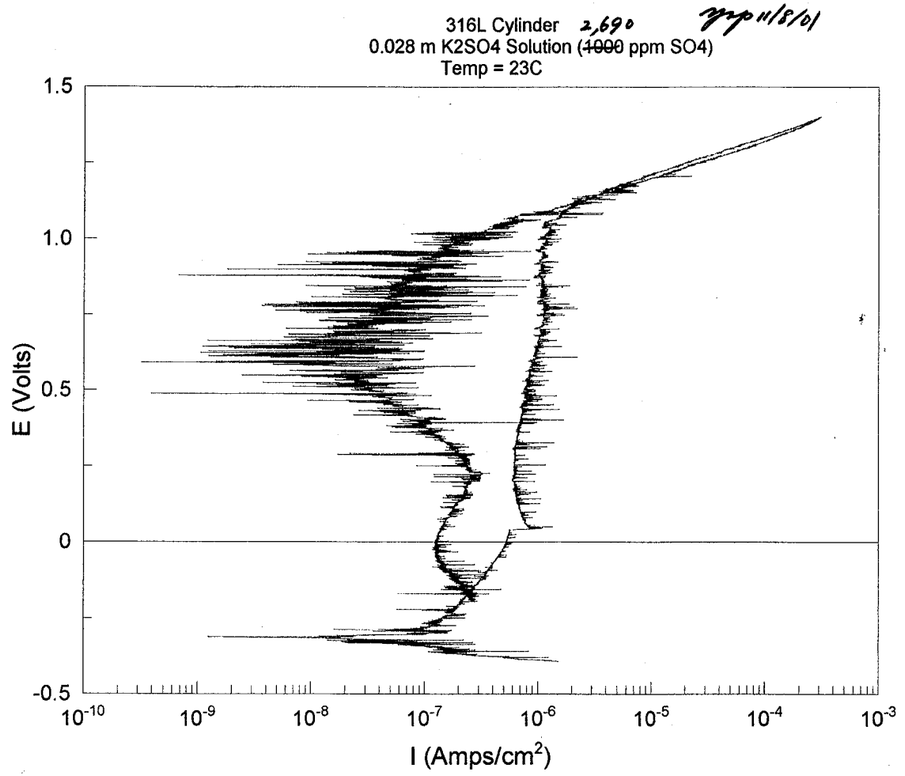
Ept = +409 mv

Specimen Examination: No signs of corrosion or Pitting

Graph on Pg #30 →

Data Saved As 316L CycPol Test2

10/8/01



10/5/01

Cyclic Polarization of 316L

objective: See Pg #5

Specimen: 316L P80746 Cylinder 600 Grit Finish

start wt: 11.81758g Santorious Genius SN# 12808099 Cal 5/22/01

End wt: 11.81781g

Solution: 0.028 m K_2SO_4 (2,690 ppm ypp 11/8/01 ~~4000 ppm~~ SO_4)

4.881 g K_2SO_4 Lot #706672

+ DI water To 1000 mls

pH start = 6.051 Fisher Accumet 950 meter SN# 3440 cal 7/20/01

pH End = 4.562 Fisher pH Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: PT Flag

Reference Electrode: Fisher 13-620-52 SN# 0042119

Temperature: 95°C Hg Thermometer SN# ~~00~~ 4211 (10-22-01) A2000-123 cal 8/23/01

Deaerates with 99.999% N_2

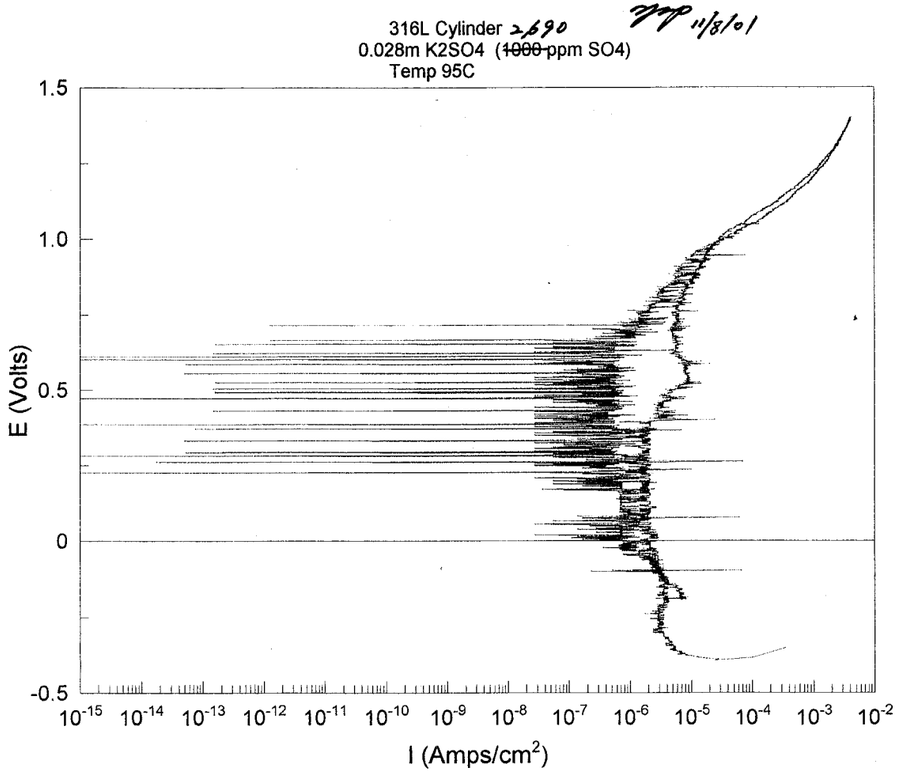
Econn -571mv Keithley 614 SN# 467374 cal 10/4/01

Ept +104mv

Specimen Examination - No sign of Corrosion - Mild staining on Specimen

Graph on Pg #32

10/23/01



11/8/01

10/24/01

Intchem 524C 10-16 Internal Corrosion Tests

objective: Measure Current Density At an Applied potential

Specimen: 316L Heat P80746 Specimen Dimensions on pg #7
with modified geometry on pg #21

Test Environment:

Solution - 1000 ppm ~~K_2SO_4~~ 3/10 11/13/01 SO_4 Lot# 706672

0.181 g K_2SO_4 + DI water To 100mls

4 mls of Test Solution In well

Temperature: 23°C

Duration: 30 min Rate: 5/sec.

Potentiostat: Solartron 1287

Reference: Fisher sc 13-620-52 SN# 8238341

Counter Electrode: PT Flag

Data Files:

Applied Potentials (mVSCE)	File ID
0	Interchem S24C10
200	Interchem S24C11
400	Interchem S24C12
600	Interchem S24C13
800	Interchem S24C14
1000	Interchem S24C15
1200	Interchem S24C16

11/13/01

Interchem S24C 10A-16A Internal Corrosion Tests

objective: Measure Current Density At AN Applied potential

Specimen: 316L Heat P80746 · Specimen Dimensions on pg #7
with modified geometry on pg #21

Test Environment:

Solution 2,690 ppm SO_4 - 0.028m K_2SO_4
.488g K_2SO_4 Lot # 706672
+ DI water To 100 mls
4 mls of Test Solution In well

Temperature: 24°C
Duration: 30 min Rate 5/sec
Potentiostat: Solartron 1287
Reference: Fisher SCE 13-620-52 SN# 8238341
Counter Electrode: PT Flag
Data Files:

Applied Potentials (mVSCE)	File ID
0	Interchem S24C10A
200	Interchem S24C11A
400	Interchem S24C12A
600	Interchem S24C13A
800	Interchem S24C14A
1,000	Interchem S24C15A
1,200	Interchem S24C16A

11/13/01

Interchem S60C 10-16 Internal Corrosion Tests

objective: Measure Current Density At AN Applied potential

Specimen: 316L Heat P80746 - Specimen Dimensions on pg #7
with modified geometry on pg #21

Test environment:

Solution 2,690 ppm SO_4 - 0.028m K_2SO_4
.488g K_2SO_4 Lot # 706672
+ DI water To 100 mls
4 mls of Test Solution In well

Temperature: 60°C
Duration: 30 min Rate: 5/sec
Potentiostat: Solartron 1287
Reference: Fisher SCE 13-620-52 SN# 8238341
Counter Electrode: PT Flag
Data Files:

Applied Potentials (mVSCE)	File ID
0	Interchem S60C10
0	Interchem S60C10A
200	Interchem S60C11
400	Interchem S60C12
500	Interchem S60C13
600	Interchem S60C14
800	Interchem S60C15
1,000	Interchem S60C16

11/14/01

Interchem S90C 10-16 Internal Corrosion Tests

objective: Measure Current Density At An Applied potential

Specimen: 316L Heat P80746 - Specimen Dimensions on pg #7
with modified geometry on pg #21

Test Environment:

Solution 2,690 ppm SO_4 - 0.028 m K_2SO_4
.488 g K_2SO_4 Lot #706672
+ DI water To 100 ml's

4 ml/s of Test Solution In well Brought Up To Temperature
then Added 1.5 ml/s Extra Test Solution To well - Allowed Temperature
To Stablize with Added Solution then Started Test.

Temperature: 90°C
Duration: 30 min Rate: 5/sec
Potentiostat: Solartron 1287
Reference: Fisher SCE 13-620-52 SN# 8238341
Counter Electrode: PT Flag
Data Files

Applied Potentials (mVSCE)	File ID
0	Interchem S90C10
200	Interchem S90C11
400	Interchem S90C12
500	Interchem S90C13
600	Interchem S90C14
700	Interchem S90C15
800	Interchem S90C16

11/14/01

Current Density and Total Charge Results for Tests in K_2SO_4 solutions

Current density and total charge measured on Type 316L stainless steel in K_2SO_4 solutions for 0.5 hr at various applied potentials and temperatures for each in-package chemistry test

Test ID	Temperature (°C)	Applied Potential (mV_{SCE})	Current Density (A/cm^2)	Total Charge (Coulombs)
0.028 M (2690 ppm) SO_4^{-2}				
Intchem S24C10a	20	0	$1.98x10^{-7}$	0.00074
Intchem S24C11a	20	200	$2.48x10^{-7}$	0.0090
Intchem S24C12a	20	400	$2.51x10^{-7}$	0.0010
Intchem S24C13a	20	600	$2.47x10^{-7}$	0.0008
Intchem S24C14a	20	800	$3.35x10^{-7}$	0.0012
Intchem S24C15a	20	1000	$4.94x10^{-7}$	0.0015
Intchem S24C16a	20	1200	$3.77x10^{-6}$	0.0082
Intchem S60C10a	60	0	$3.86x10^{-7}$	0.00096
Intchem S60C11	60	200	$5.62x10^{-7}$	0.0020
Intchem S60C12	60	400	$7.62x10^{-7}$	0.0022
Intchem S60C13	60	500	$1.90x10^{-6}$	0.0034
Intchem S60C14	60	600	$3.58x10^{-6}$	0.0065
Intchem S60C15	60	800	$9.91x10^{-6}$	0.018
Intchem S60C16	60	1000	$6.28x10^{-5}$	0.107
Intchem S90C10	90	0	$3.10x10^{-7}$	0.00089
Intchem S90C11	90	200	$5.65x10^{-6}$	0.0106
Intchem S90C12	90	400	$3.14x10^{-5}$	0.037
Intchem S90C13	90	500	$3.61x10^{-5}$	0.067
Intchem S90C14	90	600	$4.32x10^{-5}$	0.095
Intchem S90C15	90	700	$1.40x10^{-4}$	0.264
Intchem S90C16	90	800	$4.20x10^{-4}$	0.708
0.0104 M (1000 ppm) SO_4^{-2}				
Intchem S24C10	20	0	$1.06x10^{-6}$	0.0020
Intchem S24C11	20	200	$1.23x10^{-6}$	0.0023
Intchem S24C12	20	400	$5.68x10^{-7}$	0.00072
Intchem S24C13	20	600	$1.87x10^{-6}$	0.0024
Intchem S24C14	20	800	$6.21x10^{-7}$	0.0018
Intchem S24C15	20	1000	$2.87x10^{-6}$	0.0030
Intchem S24C16	20	1200	$5.60x10^{-6}$	0.012

11/22/01

Additional Internal Corrosion Tests in 0.028M K_2SO_4 solution

Objective: Measure current density variation at -100mV and -150mV for various temp.

Specimen: 316L SS Heat P80746

Test Environment: Solution - 0.028M K_2SO_4 solution
4mL of test solution in well
In Air

Duration: 30 min Rate: 5/sec

Potentiostat: Solartron SI 1287

Reference Electrode: FISHER SCE 13-620-52 SN 823 P341

Counter Electrode: Pt Flag

Data Files:

Applied Potential (mV$_{SCE}$)	Temperature (°C)	File ID
-100	RT	Interchem S24C9A
-150	RT	Interchem S24C 8A
-100	60	Interchem S60C 9
-150	60	Interchem S60C 8
-100	90	Interchem S90C9
-150	90	Interchem S90C8

Zj Pan
2/6/02

24-hour Internal Corrosion Tests in 0.028M K_2SO_4 solution

Objective: Determine solution chemistry changes under transpassive conditions

Test specimen, environment, and equipment set-up: the same in page 38

Note: the applied potential was determined based on the cyclic polarization curves in pages 30 and 32. For each test two solution extractions were made, both inside and outside the pit.

Data Files:

Applied Potential (V_{SCE})	Temperature (°C)	File ID	Date
0.5	RT	interchem S24C-20	2/7/02 1:42pm
1.2	RT	interchem S24C-21	2/11/02 9:06 am
0.5	90	interchem S90C-20	2/12/02 11:42am
Note: Test stopped after 5420 sec (~1.5hr) due to severe corrosion.			
0.5	90	interchem S90C-20a	2/12/02 3:07pm
Note: Test stopped after 1824 sec (30 min) due to severe corrosion.			

Zj Pan
2/13/02

Copies sent to QA Records
Zj Pan 4/4/2002

Initial Entry for Glass Wasteform Dissolution Study

Objectives

- To study interactions between simulated HLW glass and 316 L stainless steel
- To determine dissolution behavior of glass in presence of corrosion products

Staff: Vijay Jain, Yi-Ming Pan, Darrell Dunn, and Brian Derby

Samples should be weighed prior to testing.
Glass samples placed on Teflon cage.
SRL 202G glass samples supplied by Argonne National Laboratory.
316L SS samples Heat #P80746.
Use 250 ml glass vessel with Teflon liner.
Use 100 ml of KCl solution.
The vessel should be aerated.
Samples retrieved every day and analyzed for pH and chemical composition using ICP analysis.
Solution samples retrieved each day should not exceed 2 ml.

Test Matrix/Conditions

Test ID	Glass	316L SS	Temperature (°C)	Potential (mV_{SCE})	Duration (days)	pH	Cl Conc. (ppm)	ICP
BL-G	Yes	Yes	95	No	10	Yes	1000	Yes
BL-S	No	Yes	95	-200	10	Yes	1000	Yes
GS95-100	Yes	Yes	95	-100	10	Yes	1000	Yes
GS95-200	Yes	Yes	95	-200	10	Yes	1000	Yes
GS95-300	Yes	Yes	95	-300	10	Yes	1000	Yes
GS60-100	Yes	Yes	60	-100	10	Yes	1000	Yes
GS60-200	Yes	Yes	60	-200	10	Yes	1000	Yes
GS60-300	Yes	Yes	60	-300	10	Yes	1000	Yes
GSRT-100	Yes	Yes	RT	-100	10	Yes	1000	Yes
GSRT-200	Yes	Yes	RT	-200	10	Yes	1000	Yes
GSRT-300	Yes	Yes	RT	-300	10	Yes	1000	Yes
GS95-200(2)	Yes	Yes	95	-200	10	Yes	1000	Yes

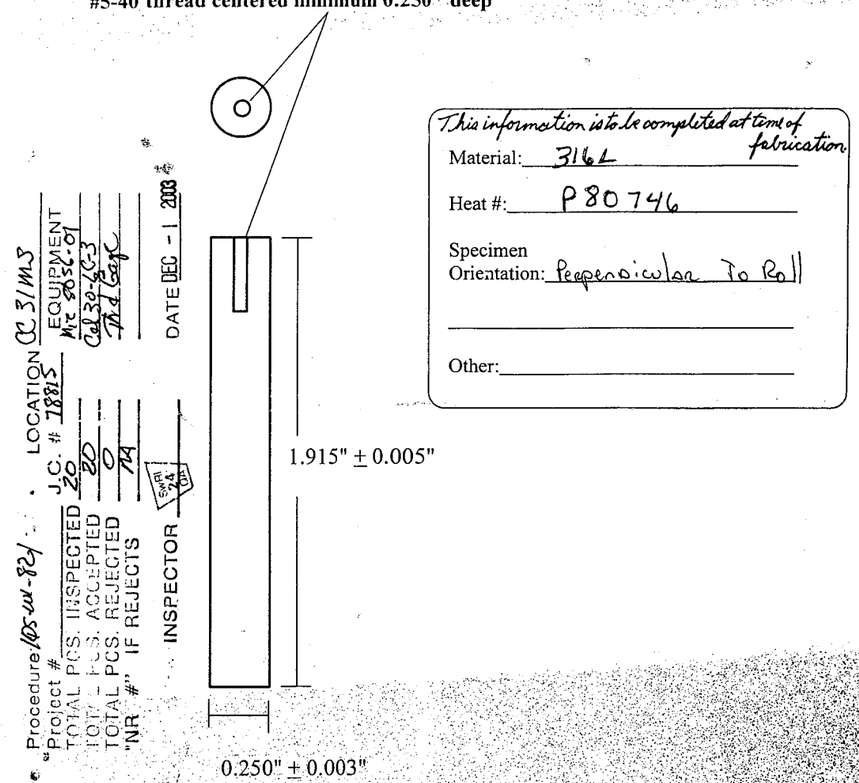
Vij Jain
8/1/03

Copies sent to QA records
Vij Jain
10/1/03

Darrell S. Dunn
SwRI-CNWRA
Phone: (210) 522-6090
Fax: (210) 522-5184

Cylindrical Test Specimen

CNWRA Drawing 20.01402.571.019



This information is to be completed at time of fabrication

Material: 316L

Heat #: P80746

Specimen Orientation: Perpendicular To Roll

Other:

Darrell S. Dunn 10/15/01
Initiated by. D. Dunn Date

Mark R. Shinstock for 10/15/01
QA Approval B. Mabrito Date

10/15/01
Reviewed by V. Jain Date

* New Test Specimens

1/27/04

In-Package Glass Dissolution Tests at 95°C

Test cell: three-electrode system

Potentiostat: Solartron 1480

Counter Electrode: Pt Flag

Reference Electrode SCE

Temperature: 95°C, Omega thermometer Model HH22

Initial pH: 4.82

pH Measurements

Test ID	1-Day	2-Day	3-Day	4-Day	5-Day	
GS95t-100	6.78 3:05pm 10/15/03	6.57 2:33pm 10/16/03	6.61 2:35pm 10/17/03	6.36 2:49pm 10/18/03	6.47 3:06pm 10/19/03	
GS95-200	6.76	6.56	6.57	7.74	—	Cl2 evolution severe corrosion test stopped
GS95-300	6.78	6.90	6.78	7.98	—	Cl2 evolution severe corrosion test stopped

Note: As a result of Cl2 evolution during the tests, modification of the test cells is proposed by separating the counter electrode from the test cell using a salt bridge.

Bill Pan
1/28/04

Revised Test Matrix using Modified Test Cells

Test ID	Glass	316L SS	Temperature (°C)	Potential (mV_{SCE})	Duration (days)	pH	Cl Conc. (ppm)	ICP
GS95-1 (Set #1)	Yes	Yes	95	-100	10	Yes	1000	Yes
GS60-1 (Set #1)	Yes	Yes	60	-100	10	Yes	1000	Yes
GSRT-1 (Set #1)	Yes	Yes	RT	-100	10	Yes	1000	Yes
GS95-2 (Set #3)	Yes	Yes	95	-50	10	Yes	1000	Yes
GS60-2 (Set #3)	Yes	Yes	60	-50	10	Yes	1000	Yes
GSRT-2 (Set #3)	Yes	Yes	RT	-50	10	Yes	1000	Yes
GS95-3 (Set #4)	Yes	Yes	95	0	10	Yes	1000	Yes
GS60-3 (Set #4)	Yes	Yes	60	0	10	Yes	1000	Yes
GSRT-3 (Set #4)	Yes	Yes	RT	0	10	Yes	1000	Yes
BL-G (Set #2)	Yes	Yes	95	No	10	Yes	1000	Yes
BL-S (Set #2)	No	Yes	95	-100	10	Yes	1000	Yes
GS95-1(2) (Set #2)	Yes	Yes	95	-100	10	Yes	1000	Yes

Bill Pan
1/28/04

In-Package Glass Dissolution Tests Set #1

Test Cell: three-electrode system with a counter electrode outside the test cell

Potentiostat: Solartron 1480

Counter Electrode: Pt Flag

Reference Electrode: SCE

Temperature: 60 and 95°C checked by Omega thermometer Model HH22

Initial pH: 5.347

pH measurements:

Date for pH	GS 95-1 +	GS60-1 ++	GSRT-1 ++
1/29/04 3:00pm	4.98	4.76	4.63
1/30/04 3:30 pm	4.32	5.11	5.59
1/31/04 5:20pm	2.87	7.05	8.12
2/2/04 3:15pm	2.68	6.23	5.82
2/3/04 3:00 pm	2.61	5.95	5.93

Note: "+" The applied potential of -100 mV varied with time.
"++" The applied potential of -100 mV remained constant.

Zi Pan
2/4/04

In-Package Glass Dissolution Tests Set # 2

Test set-up is the same as set #1.

pH measurements:

Date	BL-G	BL-S +	GS 95-1(2) +
2/10/04 3:10pm	5.52	5.84	5.06
2/11/04 3:30pm	5.76	6.11	5.19
2/12/04 3:30pm	5.87	6.02	5.32
2/13/04 3:15pm	5.69	6.18	5.32
2/16/04 3:15pm	6.03	6.64	5.89
2/17/04 3:15pm	5.16	6.73	6.70
2/18/04 3:25pm	6.64	7.83	7.90
2/20/04 6:38 am	7.13	7.92	8.24

Note: "+" The applied potential of -100 mV varied with time.

Zi Pan
2/23/04

In-Package Glass Dissolution Tests Set #3

Test Set-up is the same as Set #1.

pH measurements:

Date	GS 95-2+	GS 60-2+	GSRT-2+
2/23/04 3:00 pm	tests start		
2/24/04 3:00 pm	4.84	~~4.84~~ 5.83 (typ 3/1/04)	5.59
2/25/04 8:30 am	tests restart due to changes in applied potential	5.63	5.51
2/26/04 3:00 pm	3.39	~~3.39~~ 5.04 (typ 3/1/04)	5.68
2/27/04 8:30 am	tests restart due to changes in applied potential	5.46	5.28
2/28/04 3:45 pm	3.27	~~3.27~~ 5.90 (typ 3/5/04)	5.72
3/1/04 3:00 pm	3.22	5.92	5.95
3/2/04 3:00 pm	3.22	6.04	5.85
3/3/04 3:00 pm	3.23	6.11	5.83
3/4/04 3:00 pm	3.19	6.10	5.93

Note: "+" The applied potential of -50mV varied with time.

3/5/04

In-Package Glass Dissolution Tests Set #4

Test set-up is the same as set #1.

The applied potential changed to -100 mV to repeat test set #1.

Initial pH: 5.58

pH measurements:

Date	GS95-3	GS60-3 (typ 5/20/04)	BL-S3
5/14/04 10:45am	7.21	7.11	7.37
5/15/04 11:00am	7.13	7.04	7.27
5/16/04 11:00am	6.98	7.00	8.92
5/17/04 11:00am	7.02	6.65	3.89
5/18/04 11:00am	7.06	6.52	—*
5/19/04 11:00am	5.98	—* *(Lost of electric contact/no solution collected)	—*

Note: 1) Applied potential with time

GS95-3	start up to 75 hrs	-100 mV
GS60-3	start up to ~~119~~ 146 hrs (typ 5/20/04)	-100 mV
BL-S3	start up to 125 hrs	-100 mV

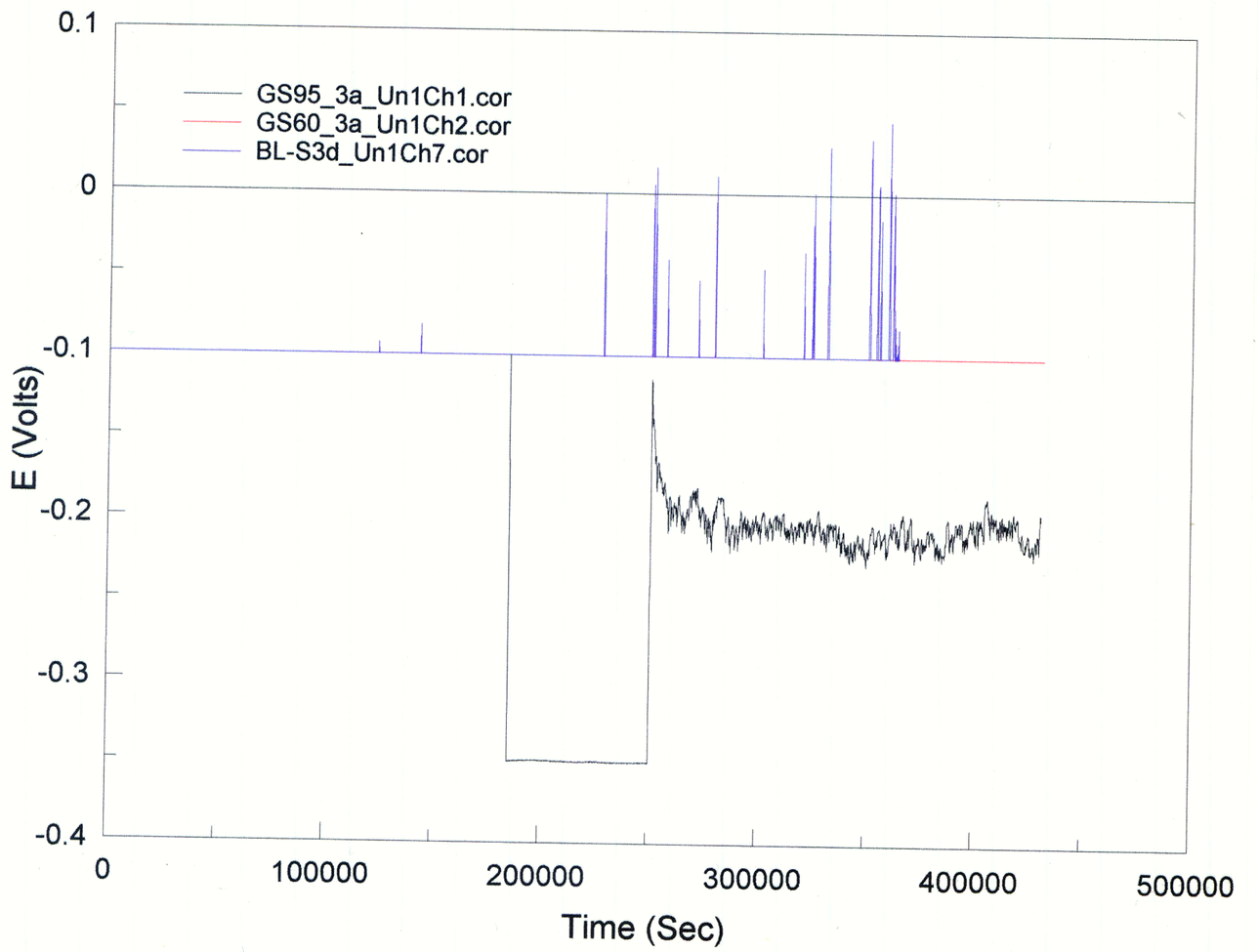
2) For GS60-3 and BL-S3 tests, high current density values recorded as a result of loss of electric contact.

5/20/04

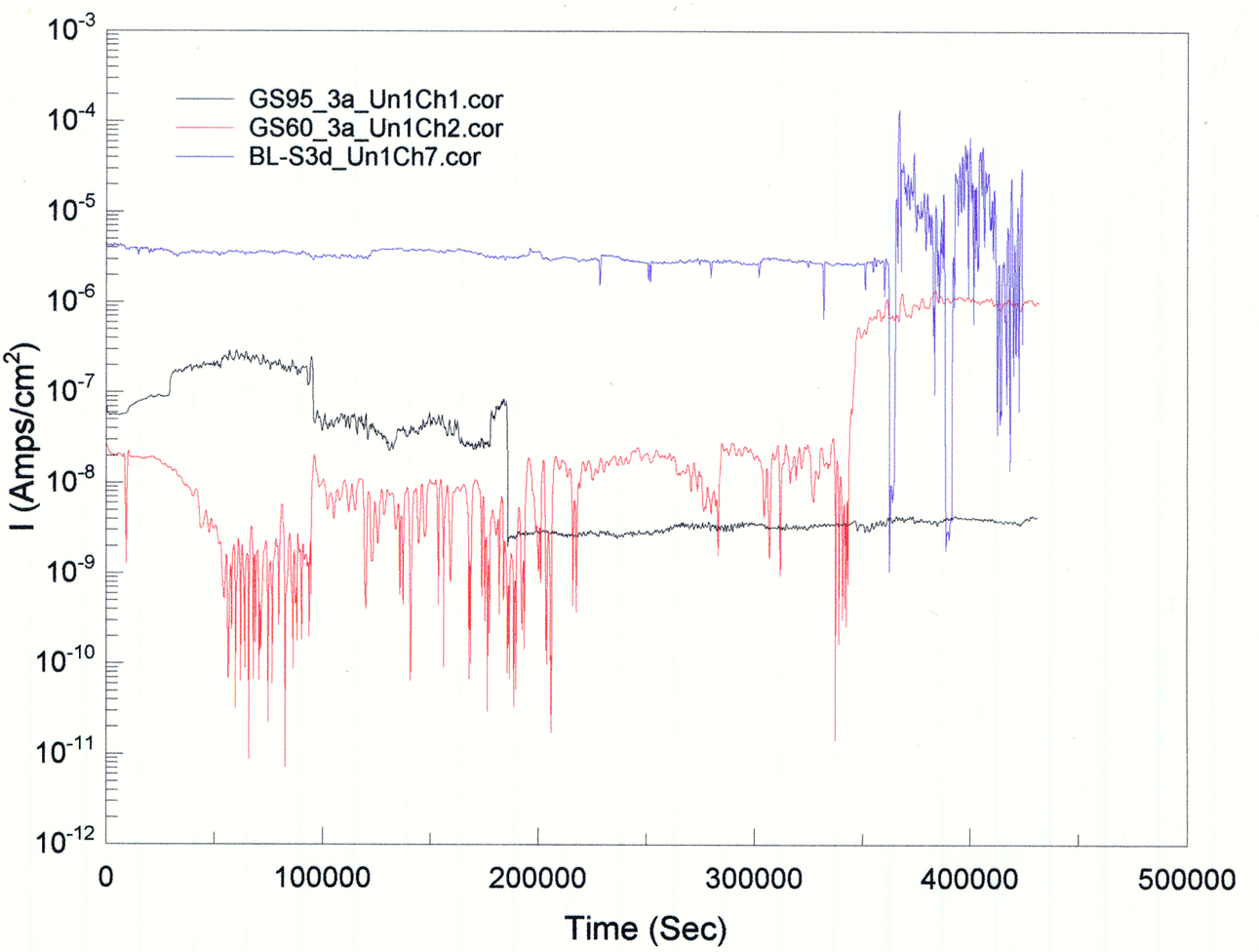
For the BL-S3 test, high applied potential values around -910 to 960mV after testing for 365,690 sec (or 101.6 hrs) are removed.

All the curves plotted represent the data collected starting second day of the test.

Bijay Rai 5/20/04



Bijay Rai 5/20/04



ICP Results for Solution Samples Collected from Tests Set #4

Element Concentrations for In-Package Tests at -100mV (mg/L)
* Numbers in parentheses are instrument detection limits.

Sample ID	Date	Test Duration (days)	Al (1.5)	B (1)	Ca (2.5)	Cr (0.25)	Fe (1.5)	Li 0.25	Mg (1.5)	Mn (0.25)	Ni (0.25)	P (1.5)	K (10)	Si (1.5)	Na (10)	Ti (0.25)	U (3.75)	Zr (0.25)	Cl (0.1)	Final pH
GS95-3A	5/14/2004	1	0	0	0	0	0	0	0	0	0	0	1451	4.29	1212	0	0	0	3151	7.21
GS95-3B	5/15/2004	2	0	0	0	0	0	0	0	0	0	0	1807	6.74	2250	0	0	0	5143	7.13
GS95-3C	5/16/2004	3	0	1.15	0	0	0	0	0	0	0	0	2217	9.7	3485	0	0	0	7602	6.98
GS95-3D	5/17/2004	4	0	1.54	0	0	0	0	0	0	0	0	2696	15.3	5557	0	0	0	11716	7.02
GS95-3E	5/18/2004	5	0	1.97	0	0	0	0.303	0	0	0	0	3150	20.6	7514	0	0	0	15287	7.06
GS95-3F	5/19/2004	6	0	1.88	0	0	0	0.309	0	0	0	0	3106	20.7	7476	0	0	0	15583	5.98

Sample ID	Date	Test Duration (days)	Al (1.5)	B (1)	Ca (2.5)	Cr (0.25)	Fe (1.5)	Li 0.25	Mg (1.5)	Mn (0.25)	Ni (0.25)	P (1.5)	K (10)	Si (1.5)	Na (10)	Ti (0.25)	U (3.75)	Zr (0.25)	Cl (0.1)	Final pH
GS60-3A	5/14/2004	1	0	0	0	0	0	0	0	0	0	0	1924	2.7	3737	0	0	0	7484	7.11
GS60-3B	5/15/2004	2	0	0	0	0	0	0	0	0	0	0	2011	3.09	3923	0	0	0	8053	7.04
GS60-3C	5/16/2004	3	0	0	0	0	0	0	0	0	0	0	2243	3.54	4226	0	0	0	8329	7.00
GS60-3D	5/17/2004	4	0	0	0	0	0	0	0	0	0	0	2509	3.8	4604	0	0	0	9077	6.65
GS60-3E	5/18/2004	5	0	0	0	0	0	0	0	0	0.41	0	2586	4.57	4655	0	0	0	9931	6.52

Sample ID	Date	Test Duration (days)	Al (1.5)	B (1)	Ca (2.5)	Cr (0.25)	Fe (1.5)	Li 0.25	Mg (1.5)	Mn (0.25)	Ni (0.25)	P (1.5)	K (10)	Si (1.5)	Na (10)	Ti (0.25)	U (3.75)	Zr (0.25)	Cl (0.1)	Final pH
BL-S3A	5/14/2004	1	0	0	0	0.808	15.4	0	0	0.66	3.63	0	1747	2.04	273	0	0	0	2048	7.37
BL-S3B	5/15/2004	2	0	0	0	0	11.4	0	0	0.674	3.62	0	2174	3.76	590	0	0	0	2907	7.27
BL-S3C	5/16/2004	3	0	1.17	0	0	8.6	0	0	0.692	3.74	0	2584	5.52	1049	0	0	0	4038	8.92
BL-S3D	5/17/2004	4	0	1.65	3.22	0	5.82	0	0	0.772	4.09	0	3355	7.66	1917	0	0	0	6280	3.89

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Following papers were published. VJ 12/2/05

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Effect of In-Package Chemistry on the Degradation of Vitrified High-Level Radioactive Waste and Spent Nuclear Fuel Cladding, CNWRA 2002-01, October 2001

Y.-M. Pan, C.S. Brossia, G.A. Cragnolino, D.S. Dunn, V. Jain, and N. Sridhar, Evolution of Solution Chemistry through Interactions with Waste Package Internal Structural Components, *Mat. Res. Soc. Symp. Proc.* Vol. **713**, 121-127, 2002

I have reviewed this scientific notebook and find it in compliance with QAP-001. There is sufficient information regarding procedures used for conducting tests, acquiring and analyzing data so that another qualified individual could repeat the activity.

12/2/05

ADDITIONAL INFORMATION FOR SCIENTIFIC NOTEBOOK NO. 443

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