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Q200409290001

Scientific Notebook No. 571: Alloy 22  
Repassivation Tests - Continuation of S/Ns  
366, 485, 520, 531, and 541 (02/08/2002  
through 03/03/2004)

# LABORATORY NOTEBOOK

CNWRA/SwRI

CNWRA  
CONTROLLED  
COPY 571

NOTEBOOK NO. 571  
ISSUED TO DARRELL DUNN *Darrell Dunn*  
ON FEB 14 2003  
DEPARTMENT \_\_\_\_\_  
RETURNED \_\_\_\_\_ 20  
*Brian K. Deeby - B. K. Deeby BKO*

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Continued Testing From NB # 366, # 485, # 520, # 528, # 531  
And # 541

Copies from Previous Notebooks

**Initial Scientific notebook entry for repassivation potential measurements**

**Title:** Alloy 22 Repassivation Tests

**Tests Performed by:** Darrell S. Dunn, Letai Yang, Div 20; Brian Derby, Div. 18

**Objectives:** Determine the effect of thermal aging time and temperature on the localized corrosion susceptibility of Alloy 22.

**Equipment:** Laboratory oven for exposure of test specimens at 600 to 900 °C, Thermocouple and thermocouple meter. Keithley 614/617. Solartron 1287 Potentiostat and CorrView Software or equivalent, Electrochemical test cell.

**Materials:** Alloy C-22, heat 2277-8-3175. Other materials and heats to be added and identified prior to testing.

**Specimen specifications:** Specimens will be equivalent to 20.01402.571.006 unless otherwise specified.

**Measurement Parameters:** Temperature and time of exposure, Potential and Current of specimen during test.

**Required level of accuracy:** Temperature  $\pm 2$  °C, Time of exposure  $\pm 1$  minute, Potentials  $\pm 1$  mV, Current  $\pm 0.01$  microamp.

**Uncertainty and Sources of Error:** Current measurement error can occur for localized corrosion processes because the actively corroding area is not the same as the surface area of the test specimen.

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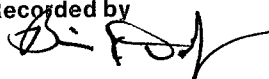
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Date \_\_\_\_\_

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Date

Recorded by



2/18/03

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**Initial Scientific notebook entry for electrochemical impedance measurements**

**Title:** Electrochemical Impedance Tests

**Tests Performed by:** Darrell S. Dunn, Brian Derby, Div. 18

**Objectives:** Determine uniform corrosion rate of passive alloys such as Alloy 22 using electrochemical impedance

**Equipment:** Keithley 614/617. Solartron 1287 Potentiostat, Solartron 1260 Impedance/Gain-Phase Analyzer, and ZPlot and ZView Software or equivalent, Electrochemical test cell.

**Materials:** Alloy C-22, heat 2277-8-3175. Other materials and heats to be added and identified prior to testing.

**Specimen specifications:** Specimens will be equivalent to 20.01402.571.006 unless otherwise specified.

**Measurement Parameters:** Temperature, Potential, and Current of specimen during test.

**Required level of accuracy:** Temperature  $\pm 2^\circ\text{C}$ , Potentials  $\pm 1\text{mV}$ , Current  $\pm 0.01$  microamp.

**Uncertainty and Sources of Error:** Models for oxide solution interface. Multiple models exist and may be applicable. Models used will be identified.

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Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>Darrell Dunn</i>	3/5/2003

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*FROM NB 157 P 208*

**Initial Scientific notebook entry for corrosion resistant material potentiostatic and potentiodynamic polarization tests.**

**Title:** Potentiostatic tests, cyclic polarization tests, crevice repassivation tests, passive current density tests, critical pitting temperature tests critical repassivation temperature tests.

**Tests Performed by:** Darrell S. Dunn

**Objectives:** Measure passive current densities, crevice repassivation potentials, critical pitting temperature and critical repassivation temperature for corrosion resistant candidate materials.

**Equipment:** EG&G Versastat Serial Number 20104. EG&G model 352 corrosion software. NEC 586 computer. Keithley Electrometer model 614 SN 555368 or equivalent. ASTM G-5 polarization cell, Large 2 L glass cells with Teflon tops, Electrochemical Impedance Spectroscopy system including Solartron 1260 FRA and Solartron 1287 Potentiostat. ESC 440 multichannel potentiostats with National instruments Labview data acquisition software or Strawberry Tree data acquisition software.

**Materials:** Alloy C-22, Alloy 825, Alloy 625 and Type 316 L stainless steel

**Specimen specifications:** Cylindrical CPP specimens 1.915"  $\times$  0.250" and Crevice repassivation specimens with Teflon crevice washers attached to surface.

**Measurement Parameters:** Current and Potential as described in TOP-008. Temperature of solution  $\pm 2^\circ\text{C}$

**Required level of accuracy:** Potentials  $\pm 5\text{mV}$ . Current less than 0.1 microamp.

**Uncertainty and Sources of Error:** Current density calculated as current divided by sample area. Actual current density of corroding areas is not determined. Resolution limit of data acquisition systems may limit accuracy of passive current density measurements.

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**Electrochemical Impedance Spectroscopy ALLOY 22**

OBJECTIVE SAME AS P 2

SPECIMEN ALLOY 22 HEAT 2277-8-3175

600 CRIT FINISH

START WT ~~12.29366~~ Sartorius Genius SN 12889099 CAL 11/5/02  
START ENO ~~12.29441~~ DD 3/5/2003 12.28178, DUE 5/15/03

START WT ENO WT 12.26212

SOLUTION 35% MgCl<sub>2</sub>  
2000.49 g MgCl<sub>2</sub> · 6H<sub>2</sub>O LOT 989283 +  
667 mL DI H<sub>2</sub>O

pH START 3.842 Fisher Account 950 SN 3340 CAL 8/7/02 DUE 8/7/03

pH ENO 5.172 pH probe 13-620-296 SN 1100202

SOLUTION DEAERATED W/ N<sub>2</sub>

CELL INFO AREA = 8 cm<sup>2</sup>  
DENSITY 8.69 g/cm<sup>3</sup>  
EQ WT 26.04

Impedance Analyser Solartron 1260

Counter electrode Pt Felag

Reference Fisher 13-620-52 SN 0052132

Temp 25-120°C Hg THERMOMETER H98-162 CAL 4/22/02 DUE 4/23/03

Ecorr -248 Keithley 614 0704936 CAL 5/26/02 DUE 5/26/03  
Ept +291

DATA FILES C226IS 92 - C22EIS 98

POST TEST SPECIMEN EXAMINATION: Shallow pitting on all surfaces

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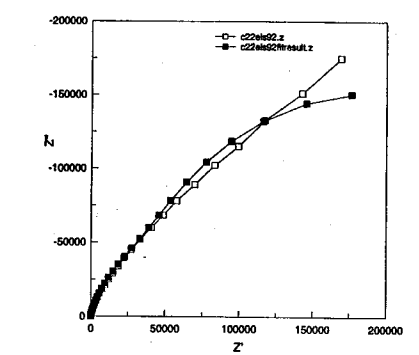
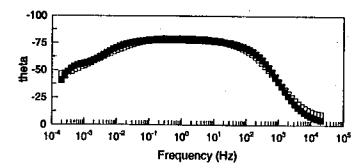
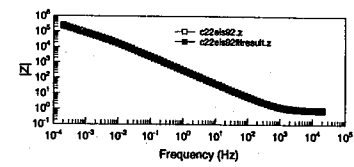
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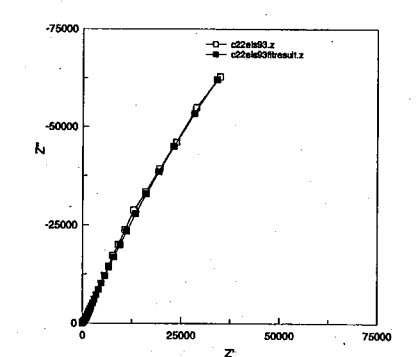
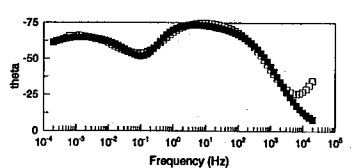
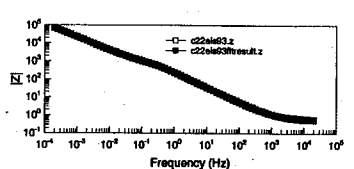
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25°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.78238	0.0088738	0.97859
Rox porous	Free(+)	49462	4778.9	9.6618
CPEporous-T	Free(+)	0.0008215	4.735E-5	5.7638
CPEporous-P	Free(+)	0.94291	0.0022724	0.28959
Rox barrier	Free(+)	2.9828E5	20273	6.7744
CPE barrier-T	Free(+)	0.0027197	0.0005577	20.508
CPE barrier-P	Free(+)	0.98228	0.028278	2.8788

Chi-Squared: 0.0028848  
Weighted Sum of Squares: 0.44401  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eis92.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.51273	0.014446	2.8173
Rox porous	Free(+)	421.4	35.713	8.4748
CPEporous-T	Free(+)	0.0015397	9.0487E-5	5.8758
CPEporous-P	Free(+)	0.97005	0.0000898	2.9825
Rox barrier	Free(+)	8.3118E5	3.8487E5	46.047
CPE barrier-T	Free(+)	0.0020214	4.2082E-5	2.0818
CPE barrier-P	Free(+)	0.75008	0.0040396	0.53857

Chi-Squared: 0.012343  
Weighted Sum of Squares: 1.9132  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eis93.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

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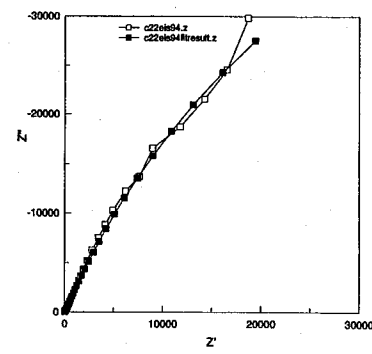
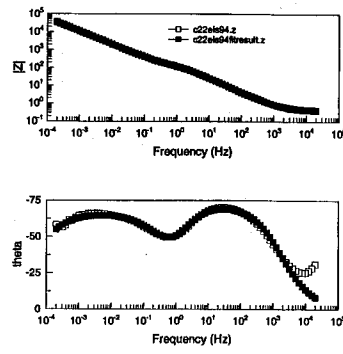
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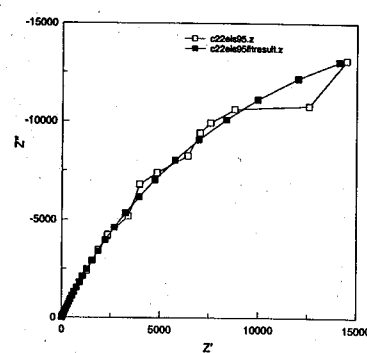
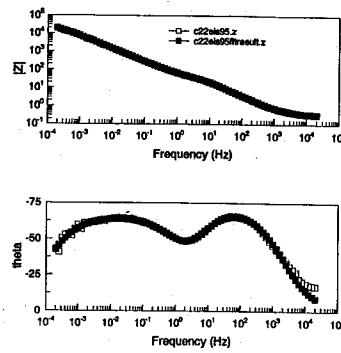


60°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.37968	0.0086301	2.273
Rox porous	Free(+)	77.71	7.4885	9.6483
CPEporous-T	Free(+)	0.0018938	0.00013089	6.9009
CPEporous-P	Free(+)	0.87341	0.020717	2.372
Rox barrier	Free(+)	1.48855	47940	31.356
CPE barrier-T	Free(+)	0.0037877	9.2184E-5	2.4338
CPE barrier-P	Free(+)	0.74352	0.0064519	0.86775

Chi-Squared: 0.006283  
 Weighted Sum of Squares: 1.2538  
 Data File: D:\corrosion tests\alloy c-22\impedance\c22le64.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



80°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.31409	0.0036992	1.1778
Rox porous	Free(+)	24.17	1.2062	4.9988
CPEporous-T	Free(+)	0.0025072	0.00010351	4.0954
CPEporous-P	Free(+)	0.84784	0.0084985	1.0012
Rox barrier	Free(+)	44274	1830	4.1537
CPE barrier-T	Free(+)	0.0052481	5.1893E-5	0.98861
CPE barrier-P	Free(+)	0.73484	0.0028986	0.40537

Chi-Squared: 0.0022065  
 Weighted Sum of Squares: 0.34247  
 Data File: D:\corrosion tests\alloy c-22\impedance\c22le85.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

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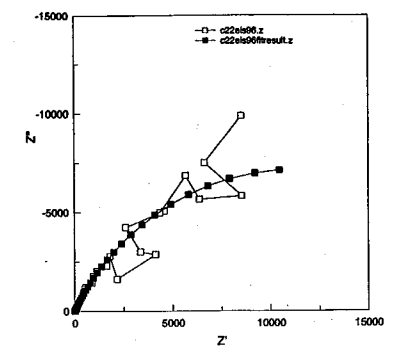
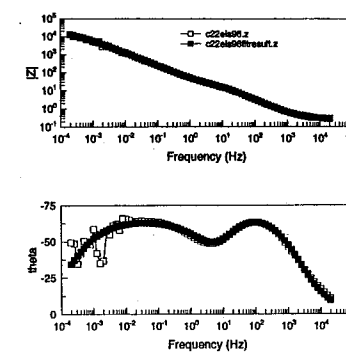
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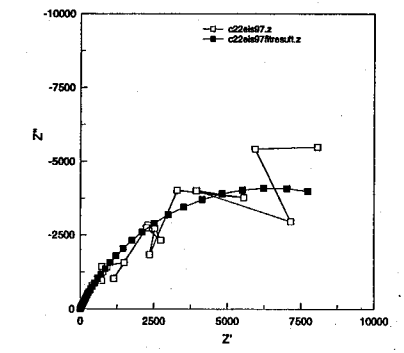
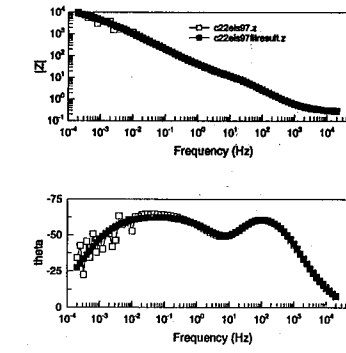


95°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.25289	0.0055969	2.2047
Rox porous	Free(+)	9.537	1.1328	11.854
CPEporous-T	Free(+)	0.0024775	0.0003428	13.841
CPEporous-P	Free(+)	0.87867	0.025521	2.9111
Rox barrier	Free(+)	22588	1435.8	6.3612
CPE barrier-T	Free(+)	0.008018	0.00010194	1.2698
CPE barrier-P	Free(+)	0.71958	0.0057778	0.78693

Chi-Squared: 0.010111  
 Weighted Sum of Squares: 1.5672  
 Data File: D:\corrosion tests\alloy c-22\impedance\c22le95.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



110°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.28681	0.0074968	2.6097
Rox porous	Free(+)	8.387	0.82299	14.027
CPEporous-T	Free(+)	0.0029572	0.00054582	18.461
CPEporous-P	Free(+)	0.86884	0.031876	3.6458
Rox barrier	Free(+)	12852	712.77	5.5032
CPE barrier-T	Free(+)	0.0070283	0.00012175	1.7323
CPE barrier-P	Free(+)	0.71727	0.0062779	0.87525

Chi-Squared: 0.012871  
 Weighted Sum of Squares: 1.995  
 Data File: D:\corrosion tests\alloy c-22\impedance\c22le97.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

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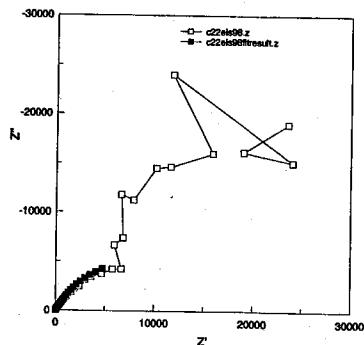
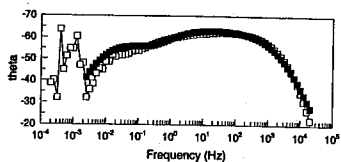
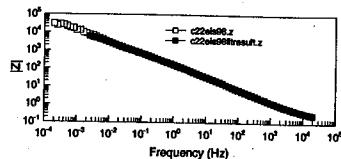
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Element	Freedom	Value	Err	Err%
R <sub>s</sub>	Free(*)	0.19124	0.0071502	3.7389
R <sub>porous</sub>	Free(*)	95.57	12.31	12.881
CPE <sub>porous-T</sub>	Free(*)	0.0053673	0.00077184	14.58
CPE <sub>porous-P</sub>	Fixed(0)	0.77853	N/A	N/A
R <sub>barrier</sub>	Free(*)	15005	863.77	5.4308
CPE <sub>barrier-T</sub>	Free(*)	0.0021482	3.2985E-5	1.5392
CPE <sub>barrier-P</sub>	Free(*)	0.86535	0.0027979	0.40237

Chi-Squared: 0.005257  
 Weighted Sum of Squares: 0.70038  
 Data File: D:\comsol\test\alloy c-22\impedance\c22al98.z  
 Circuit Model File: D:\comsol\test\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / Freq. Range (0.00252 - 20000)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

120°C

Fit range limited to 20000 to 0.00252 Hz  
 Data below 0.00252 Hz too noisy to  
 be used.

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### THERMAL AGING / SOLUTION ANNEALING

SPECIMEN ALLOY 22 HEAT 2277-8-3175  
 CYLINDER 0.250" DIA X 1.91" LONG  
 THERMAL AGED AT 870°C FOR 5 MIN

OVEN LIND BERG MODEL 51333 SN 909172  
 SET POINT 873°C

TEMP RECORDING / CHECKED WITH OMEGA HH22 SN T94140  
 CAL 10/29/02 DUE 4/29/02 WITH THERMOCOUPLE  
 #332 CAL 1/14/03 DUE 7/14/03

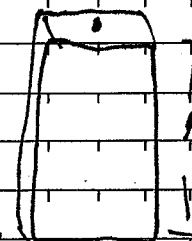
AFTER 5 min specimen removed and water quenched.

### SOLUTION ANNEALING

OVEN LIND BERG MODEL 51333 SN 909172  
 SET POINT 1125°C

TEMP CHECKED W OMEGA HH22 SN T94140 CAL 10/29/02  
 DUE 4/29/02 WITH THERMOCOUPLE #332  
 CAL 1/14/03 DUE 7/14/03 = 1129°C

TEST SPECIMEN ALLOY C22 HEAT 2277-8-3175



NOLE IN TOP FOR THERMOCOUPLE

SPECIMEN PLACED IN OVEN AT 1125°C

TIME (S)	TEMP °C	TIME (S)	TEMP °C
10	295	180	1116
30	580	210	1124
60	803	240	1128
90	944	270	1129
150	1098		

Witnessed & Understood by me, \_\_\_\_\_

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SOLUTION ANNEALING CONT FROM PAGE 9

TEST SPECIMEN W THERMO COUPLE HEATED TO 1125°C AND ALLOWED TO REM 20 3/5/2003 REMAIN IN OVEN FOR 10 min SPECIMEN WAS REMOVED AND PLACED IN CERAMIC CRUCIBLE AT ROOM TEMP TEMPERATURE OF SPECIMEN MONITORED WITH TIME

TIME (s)	TEMP °C
30	1015
40	931
60	837
75	778
90	729
105	687
120	654
135	620
150	589
180	538
210	495
240	450
270	425

Approximately 30 seconds was needed to remove specimen from the oven and place in ceramic crucible

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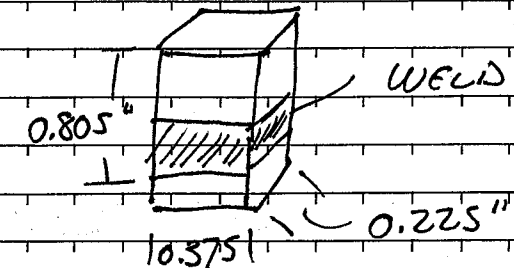
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SOLUTION ANNEALING CONTINUED FROM PAGE 9

SPECIMENS ALLOY 22 WELDED BY FRAMATOME HEAT 059902LLZ FILLER 622 HEAT XX2048BG PLATE P62X DETAILS OF PLATE WITH CHEMICAL ANALYSES IN NOTEBOOK

SPECIMEN DIMENSIONS TYPICAL SIZE



SPECIMENS PLACED IN OVEN AT 1125°C

LINDBERG S1333 SN 909172

TEMP CHECK W OMEGA HN 22 SN T 94140 CAL 10/29/02

DUE 4/29/03 WITH THERMOCOUPLE # 332 CAL 1/14/03

DUE 7/14/03 TEMP = 1129°C

FOUR (4) SPECIMENS PLACED IN OVEN

SPECIMENS REMOVED AT

15 min - WATER QUENCH

30 min - WATER QUENCH

45 min - WATER QUENCH

60 min - WATER QUENCH

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Electrochemical Impedance Spectroscopy Alloy 22

Objective: Same As pg #2

specimens: Alloy C-22 heat 2277-8-3175 polished To A 600 Coat Finish  
 Also In Cell  
 Alloy C-22 heat 2277-8-3175 Thermally Aged 820°C for 4 hrs then polished To A 600 Coat Finish

(MA)  
 start wt: 12.44534g Santorius Genius SN# 12589099 cal 11/15/02 due 5/15/03  
 End wt: 12.44315g

(TA)  
 start wt: 12.22954g Santorius Genius SN# 12589099 cal 11/15/02 due 5/15/03  
 End wt: 12.22802g

Solution  
 0.028 M NaCl  
 3.278g NaCl lot# 02787Y  
 + DI H<sub>2</sub>O To 200ml

pH Start: 6.112 Fisher Accumat 950 meter SN# 3340 cal 8/7/02 due 8/7/03  
 pH End: 7.514 pH probe # 13-670-296 SN# 2291257P6

Cell Info: Area = 8 cm<sup>2</sup> Density = 8.69 g/cm<sup>3</sup> Eq wt: 26.04

Impedance Analyzer: Solartron 1260  
 Counter Electrode: Pt Fly  
 Reference: Fisher 13-620-52 SN# 052137  
 Temperature: 25°C - 95°C Hg thermometer SN# 98-162 cal 9/22/02 due 4/22/03

(MA) E<sub>corr</sub> = -300mV  
 E<sub>pt</sub> = +309mV  
 (TA) E<sub>corr</sub> = -320mV  
 E<sub>pt</sub> = +309mV  
 Keithley 617 SN# 0579622 cal 9/22/02 due 9/22/03

Solution Bubbles with 99.999% N<sub>2</sub>

Data Files C22EISMA1 → C22EISMA9 / C22EISTA1 → C22EISTA9 To Page No. \_\_\_\_\_

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Continued from pg #12

Specimen Examination (MA):  
 No Sign of ~~measured~~ corrosion - will Repolish for Next Test

Specimen Examination (TA): 820°C for 4hrs  
 No Sign of Corrosion - will Repolish for future Testing

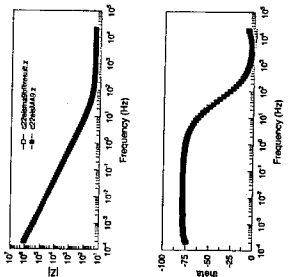
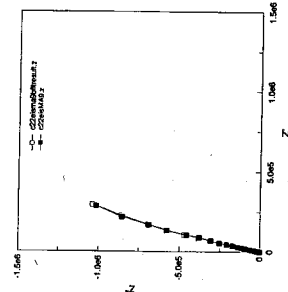
*(A large diagonal line is drawn across the page from the top left to the bottom right.)*

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	4/1/03





From Page No. \_\_\_\_\_



25°C



Element	Type	Value	Units	Estimate	Size
R1	Resistor	1.178	Ohms	1.178	1.0
C1	Capacitor	0.000149	F	0.000149	1.0
CPE1	Constant Phase Element	0.000149	F	0.000149	1.0

MILL ANNEALED  
AND PAGES  
14 & 15

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

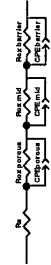
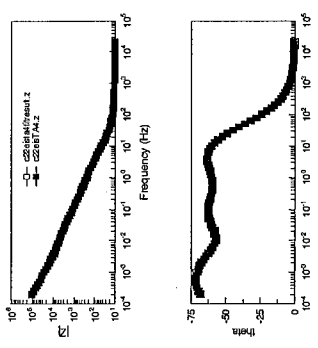
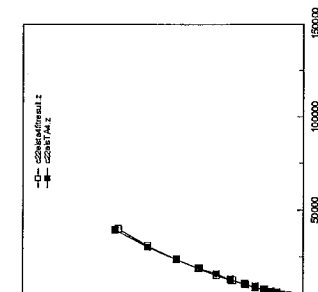
Recorded by \_\_\_\_\_

Date \_\_\_\_\_

3/4/2003

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From Page No. \_\_\_\_\_



Element	Type	Value	Units	Estimate	Size
R1	Resistor	1.178	Ohms	1.178	1.0
C1	Capacitor	0.000149	F	0.000149	1.0
CPE1	Constant Phase Element	0.000149	F	0.000149	1.0

THERMALLY AGED  
AND PAGES  
17 & 18

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

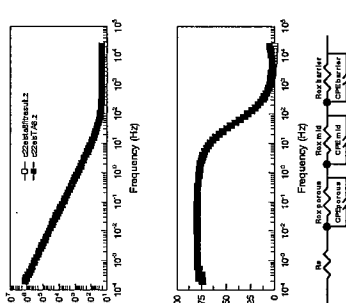
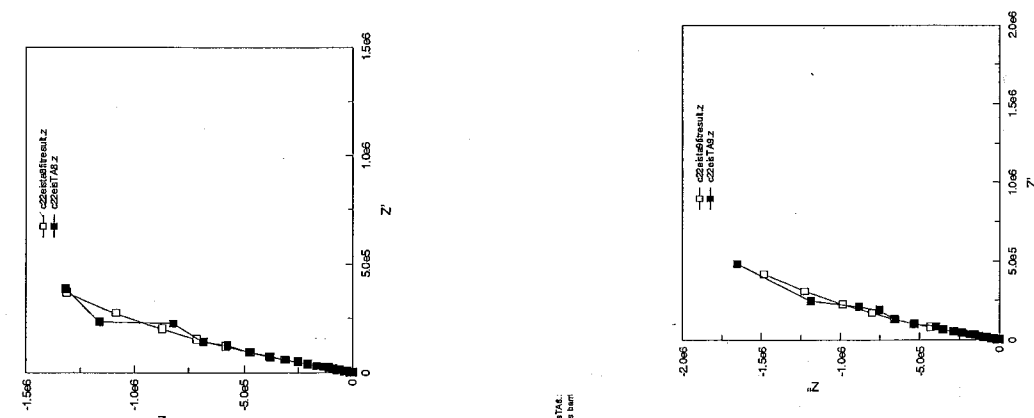
Recorded by \_\_\_\_\_

Date \_\_\_\_\_

3/7/2003

To Page No. \_\_\_\_\_

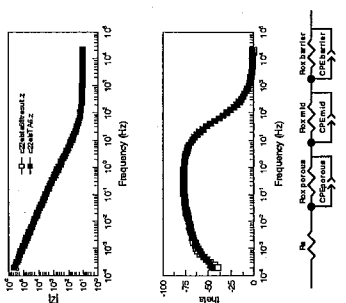
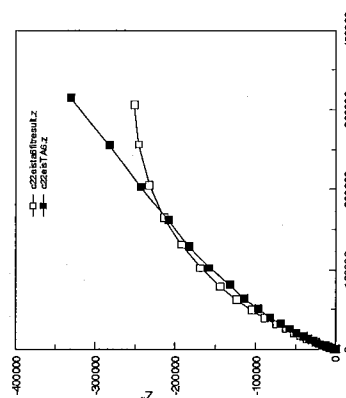
From Page No. \_\_\_\_\_



Element	Value	Error
R1	25.42	0.0786
CPE1	0.011178	0.0003754
CPE2	0.7378	0.01184
R2	0	N/A
CPE3	0	N/A
R3	0.16185	0.0002546
CPE4	0.0002546	2.011E-5
R4	0.0002546	0.00027
CPE5	0.0002546	0.01084
R5	0.0002546	1.171

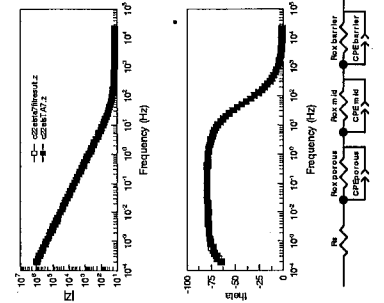
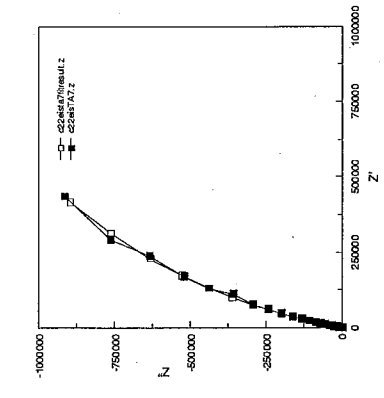
Chi-Square: 0.021925  
Weighted Sum of Squares: 0.2965  
Data File: C:\Program Files\Paragon Software\ZView\ZView746...  
Circuit Model File: C:\Program Files\Paragon Software\ZView\ZView746...  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

Element	Value	Error
R1	27.26	0.13477
CPE1	0.0003075	0.0000075
CPE2	0.73718	0.015309
R2	0	N/A
CPE3	0	N/A
R3	0.16085	1.591E-6
CPE4	0.0003418	3.559E-5
R4	0.0003418	10.034
CPE5	0.0003418	0.01842



Element	Value	Error
R1	11.42	0.3792
CPE1	0.002595	0.000045
CPE2	0.6281	0.00964
R2	0	N/A
CPE3	0	N/A
R3	0.0002595	0.000002595
CPE4	0.0002595	0.000002595
R4	0.0002595	0.000002595
CPE5	0.0002595	0.000002595

Chi-Square: 0.002595  
Weighted Sum of Squares: 0.000002595  
Data File: C:\Program Files\Paragon Software\ZView\ZView746...  
Circuit Model File: C:\Program Files\Paragon Software\ZView\ZView746...  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Value	Error
R1	14.3	0.4942
CPE1	0.00042	0.0000042
CPE2	0.6281	0.00964
R2	0	N/A
CPE3	0	N/A
R3	0.00042	0.0000042
CPE4	0.00042	0.0000042
R4	0.00042	0.0000042
CPE5	0.00042	0.0000042

Chi-Square: 0.00042  
Weighted Sum of Squares: 0.0000042  
Data File: C:\Program Files\Paragon Software\ZView\ZView746...  
Circuit Model File: C:\Program Files\Paragon Software\ZView\ZView746...  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by *[Signature]* 3/7/2003

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Electrochemical Impedance Spectroscopy Alloy 22

Objective: Sam As pg #2

Specimen: Alloy C-22 Heat 2277-8-3175 polished To A 600 Grit Finish  
Plus Alloy C-22 Heat 2277-8-3175 polished To A 600 Grit Finish  
Thermally Aged @ 870°C for 5 min

(MA)  
Start wt = 12.40887g Santaricus Genius SN# 12889099 cal 11/15/02 due 5/15/03  
End wt = 12.40888g

(JA)  
Start wt = 12.75820g Santaricus Genius SN# 12889099 cal 11/15/02 due 5/15/03  
End wt = 12.75819g

Solution: 0.028 M NaCl  
3.280g NaCl Lot# 027678  
+ DI water To 2000 ml

pH Start = 6.047 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/7/03  
pH End = 8.036 pH probe # 13-620-296 SN# 2291257 PL

Solution Degassed with 99.999% N<sub>2</sub>  
Cell Info: Area = 8 cm<sup>2</sup> Density = 1.69 g/cm<sup>3</sup> Eq wt = 26.04

Impedance Analyzer = Solartron 1260

Counter Electrode: Pt Flay

Reference: Fisher 13-620-52 SN# 0052132

Temperature: 95°C - 25°C H<sub>2</sub> Thermometer SN# 198-162 cal 4/22/02 due 4/22/03

(MA) E<sub>corr</sub> = -598 mV E<sub>pot</sub> = -115 mV  
(JA) E<sub>corr</sub> = -623 mV E<sub>pot</sub> = -115 mV  
Keithley 617 SN# 0579628 cal 9/22/02 due 9/22/03

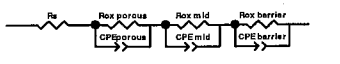
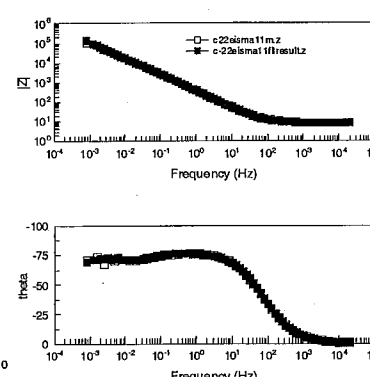
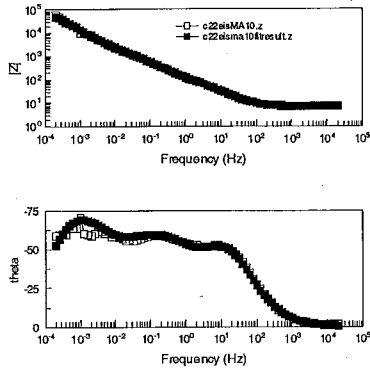
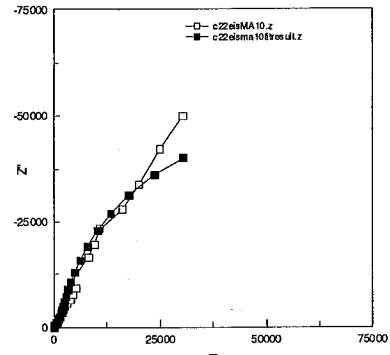
Specimen Examination  
(MA) = No Sign of Corrosion

(JA) = 870°C for 5 min = NO Sign of Corrosion  
Data Files C22EISJA10 - C22EISJA14  
Data Files C22EISMA10 - C22EISMA14

To Page No. \_\_\_\_\_

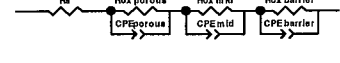
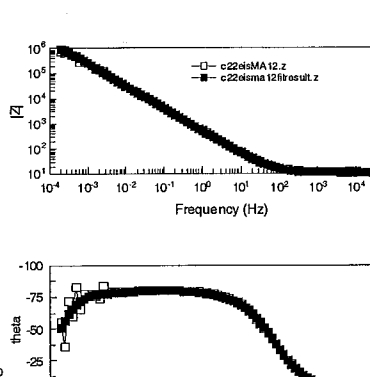
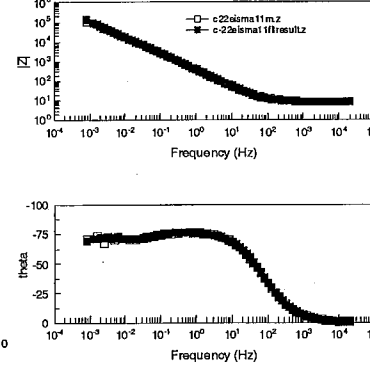
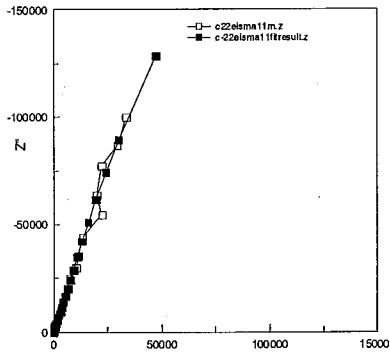
Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by *[Signature]* 3/20/03

From Page No. \_\_\_\_\_



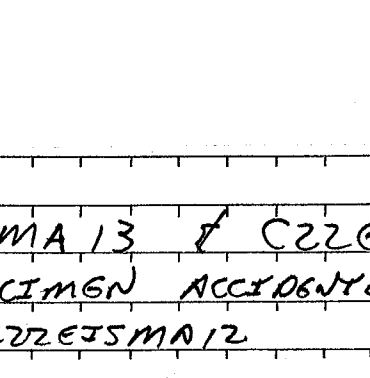
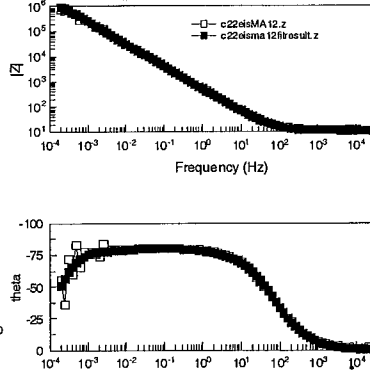
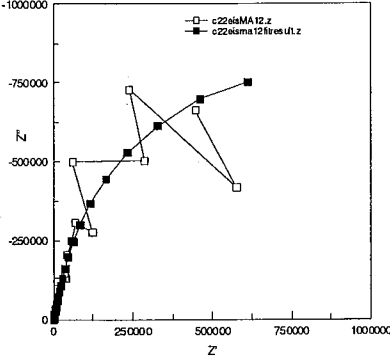
Element	Freedom	Value	Error	Error %
R0	Free(+)	7.451	0.098867	1.3289
Rox porous	Free(+)	1795	345.17	19.23
CPEporous-T	Free(+)	0.0032417	0.00017931	5.5114
CPEporous-P	Free(+)	0.70267	0.023267	3.3255
Rox mid	Free(+)	25.96	9.0391	34.819
CPE mid-T	Free(+)	0.0018358	0.00039909	21.739
CPE mid-P	Free(+)	0.92899	0.094286	10.147
Rox barrier	Free(+)	1.019855	16979	10.472
CPE barrier-T	Free(+)	0.0078458	0.001058	13.459
CPE barrier-P	Free(+)	0.92155	0.021247	2.3058

Chi-Squared: 0.009067  
 Weighted Sum of Squares: 1.3693  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisMA10.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous bar  
 Mode: Run Batch Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
R0	Free(+)	8.982	0.053938	0.60051
Rox porous	Free(+)	1596	249.91	15.659
CPEporous-T	Free(+)	0.0034337	0.00039322	11.452
CPEporous-P	Free(+)	0.97975	0.048623	4.9628
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	1.3202E8	1.4125E5	10.699
CPE barrier-T	Free(+)	0.00062671	9.781E-6	1.5575
CPE barrier-P	Free(+)	0.84601	0.0028814	0.34059

Chi-Squared: 0.003731  
 Weighted Sum of Squares: 0.52607  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisMA11.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous bar  
 Mode: Run Batch Fitting / All Data Points (1 - 74)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
R0	Free(+)	11.72	0.12945	1.1045
Rox porous	Free(+)	32108	18972	59.088
CPEporous-T	Free(+)	0.00081109	0.00019549	24.102
CPEporous-P	Free(+)	0.79555	0.018642	2.3433
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	1.5593E6	1.3064E5	8.3943
CPE barrier-T	Free(+)	0.00064892	0.00015263	23.521
CPE barrier-P	Free(+)	0.93689	0.033952	3.4065

Chi-Squared: 0.014598  
 Weighted Sum of Squares: 2.2627  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisMA12.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous bar  
 Mode: Run Batch Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

TESTS C22EISMA13 & C22EISMA14 NOT ANALYZED SPECIMEN ACCIDENTLY DAMAGED DURING TESTS AFTER C22EISMA12

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

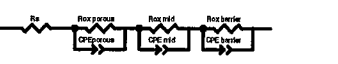
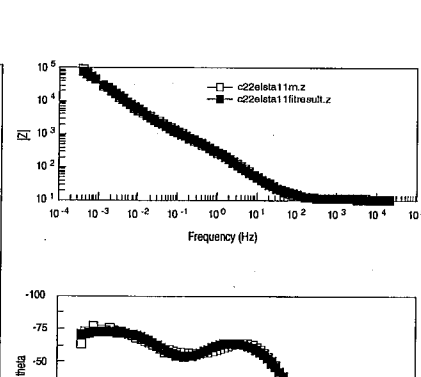
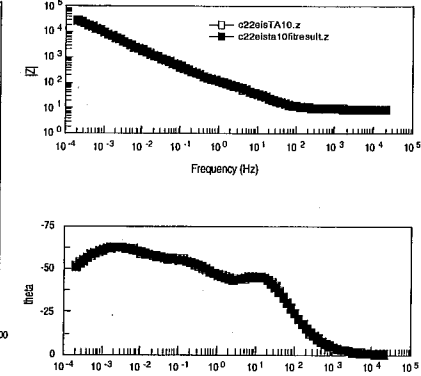
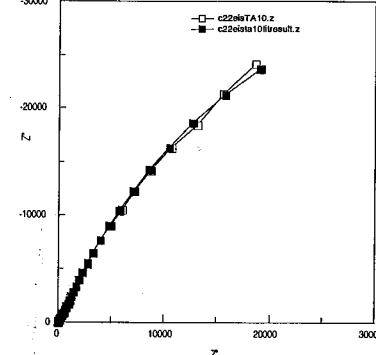
Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

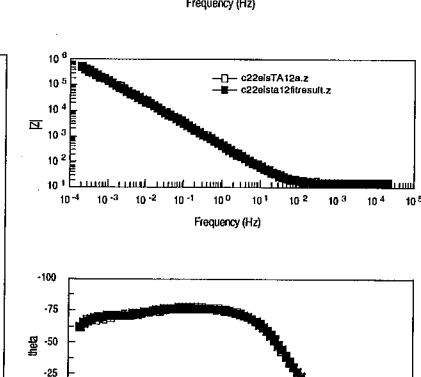
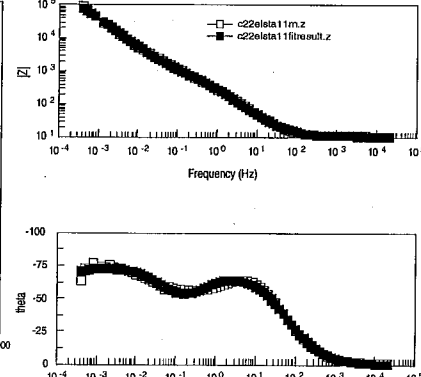
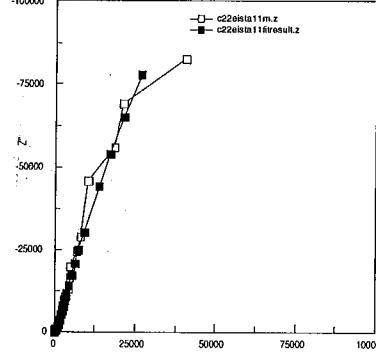
3/26/2003

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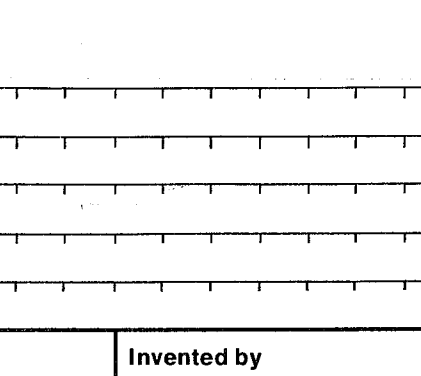
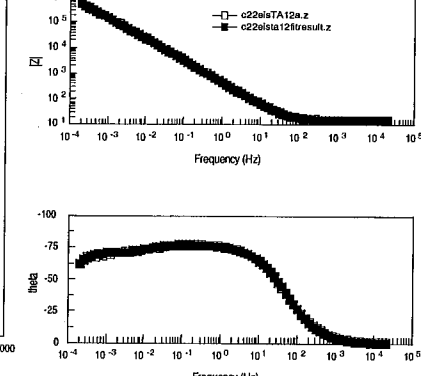
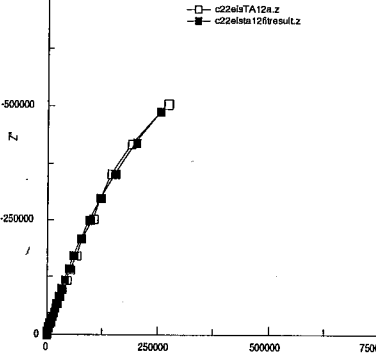
Element	Freedom	Value	Error	Error %
R0	Free(+)	9.656	0.022079	0.22798
Rox porous	Free(+)	37.54	2.8143	7.4968
CPEporous-T	Free(+)	0.0014365	3.369E-5	2.7864
CPEporous-P	Free(+)	0.85265	0.01465	1.7359
Rox mid	Free(+)	319.8	33.922	10.607
CPE mid-T	Free(+)	0.007655	0.0003828	4.786
CPE mid-P	Free(+)	0.70871	0.020073	3.2556
Rox barrier	Free(+)	299.3	255.5	2.7821
CPE barrier-T	Free(+)	0.0047734	9.472E-5	1.9845
CPE barrier-P	Free(+)	0.77058	0.003721	0.48288

Chi-Squared: 0.0033055  
 Weighted Sum of Squares: 0.046154  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisTA10.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous barrier oxide 3 layer.mtl  
 Mode: Run Batch Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
R0	Fixed(X)	11.3	N/A	N/A
Rox porous	Free(+)	613.7	34.736	5.6601
CPEporous-T	Free(+)	0.0011505	2.658E-5	2.487
CPEporous-P	Free(+)	0.75887	0.005927	0.75015
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	9.8493E5	2.478E5	25.081
CPE barrier-T	Free(+)	0.001896	4.402E-5	2.3222
CPE barrier-P	Free(+)	0.84688	0.0057613	0.6803

Chi-Squared: 0.004287  
 Weighted Sum of Squares: 0.63151  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisTA11.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous barrier oxide 3 layer.mtl  
 Mode: Run Batch Fitting / All Data Points (1 - 77)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
R0	Free(+)	14.93	0.008156	0.2557
Rox porous	Free(+)	599	528.16	10.358
CPEporous-T	Free(+)	0.0046699	0.00038059	8.1493
CPEporous-P	Free(+)	1.011	0.022843	2.2594
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	2.2668E6	69917	3.0644
CPE barrier-T	Free(+)	0.000491	3.388E-5	0.6588
CPE barrier-P	Free(+)	0.85995	0.00090941	0.10687

Chi-Squared: 0.0008272  
 Weighted Sum of Squares: 0.12442  
 Data File: D:\corrosion test\alloy c-22\impedance\c22eisTA12a.z  
 Circuit Model File: D:\corrosion test\alloy c-22\impedance\porous barrier oxide 3 layer.mtl  
 Mode: Run Batch Fitting / All Data Points (1 - 81)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

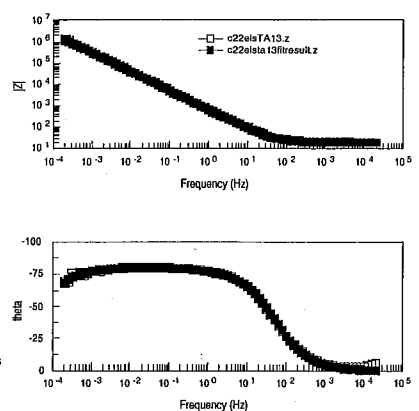
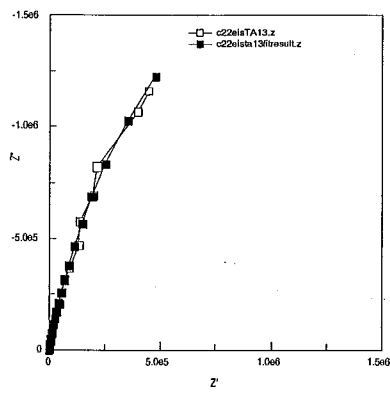
Date \_\_\_\_\_

Recorded by \_\_\_\_\_

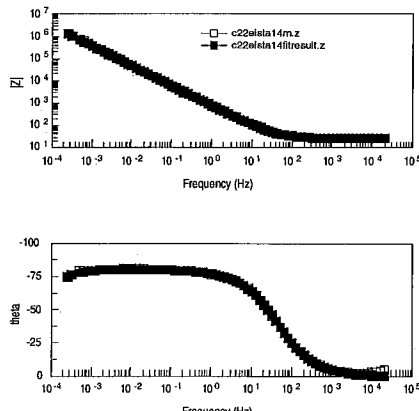
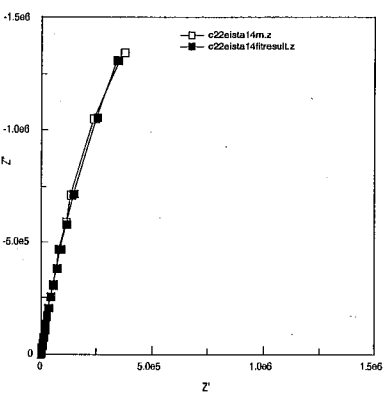
3/26/2003

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Element	Function	Value	Error	Error %
Rs	Free(+)	20.46	0.073041	0.35599
Rox porous	Free(+)	92561	24585	26.447
CPEporous-T	Free(+)	0.00074389	0.00016021	21.537
CPEporous-P	Free(+)	0.78416	0.011699	1.4919
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	4.811955	3.258955	6.8507
CPE barrier-T	Free(+)	0.00047277	4.2465E-5	8.922
CPE barrier-P	Free(+)	0.96452	0.013405	1.3892
Chi Squared		0.001698		
Weighted Sum of Squares		0.26316		



Element	Function	Value	Error	Error %
Rs	Free(+)	26.77	0.10715	0.40026
Rox porous	Free(+)	1516	1897	124.47
CPEporous-T	Free(+)	0.005312	0.002229	41.725
CPEporous-P	Free(+)	0.56775	0.035421	6.2388
Rox mid	Fixed(X)	0	N/A	N/A
CPE mid-T	Fixed(X)	0	N/A	N/A
CPE mid-P	Fixed(X)	0	N/A	N/A
Rox barrier	Free(+)	1.1901E7	1.3596E8	11.424
CPE barrier-T	Free(+)	0.00025512	5.5962E-6	2.1937
CPE barrier-P	Free(+)	0.90879	0.0041254	0.45394
Chi Squared		0.00075571		
Weighted Sum of Squares		0.11411		

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

3/26/2003

From Page No. \_\_\_\_\_

Electrochemical Impedance Spectroscopy of Alloy 22

Objective: Same As pg #2

Specimens: C-22 Base Alloy, 059902LL2 DOE material Plate D62X

(See NB #505 pg #2-25)

C-22 weld Alloy 059902LL2 DOE material Plate D62X - filler XX204866

(See NB #505 pg #2-25)

(BA)

Start wt = Not Taken

Sartorius Genius SN# 1288999 cal 11/15/02 due 5/15/03

End wt = 12.83304g

(WA)

Start wt = Not Taken

Sartorius Genius SN# 1288999 cal 11/15/02 due 5/15/03

End wt = 12.76518g

Solution

0.028M NaCl  
3.280g NaCl lot # 027828  
+ DI water To 200ml

pH start = Not Taken

Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/3/03

pH End =

pH probe # 15-620-296 SN# 2291257 P6

Cell Info: Area = 8cm<sup>2</sup> Density = 8.69g/cm<sup>3</sup> Eq. wt = 26.04

Impedance Analyzer = Solartron 1260

Counter Electrode = Pt Flag

Reference: Fisher 13-620-52 SN# 0052152

Temperature: 95°C - 25°C H<sub>2</sub> thermometer SN# 498162 cal 4/26/02

(BA) apu/lls (JA) due 4/22/03

E<sub>corr</sub> = -434mV (-65)mV E<sub>corr</sub> = -434mV Keithley 614 SN# 0704934

E<sub>pot</sub> = -118mV E<sub>pot</sub> = -118mV cal 5/20/02 due 5/20/03

Solution Bubbles - Deaerated with 99.999% N<sub>2</sub>

Data File see pg #28

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Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

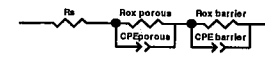
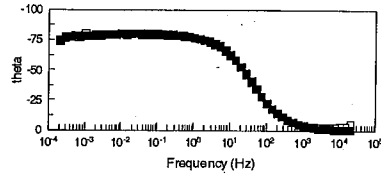
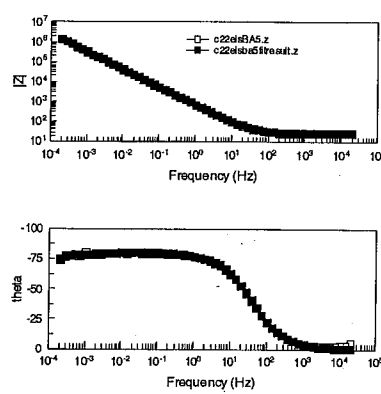
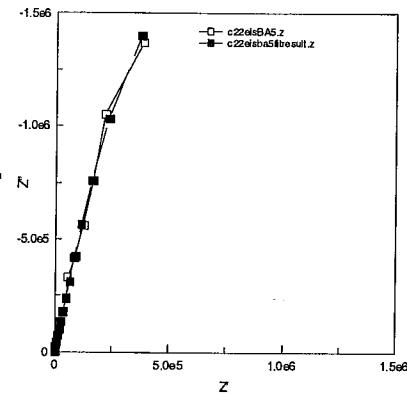
Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

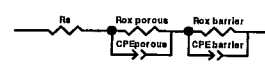
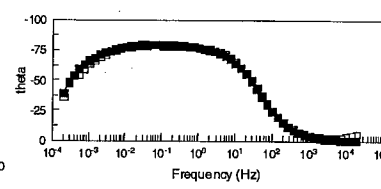
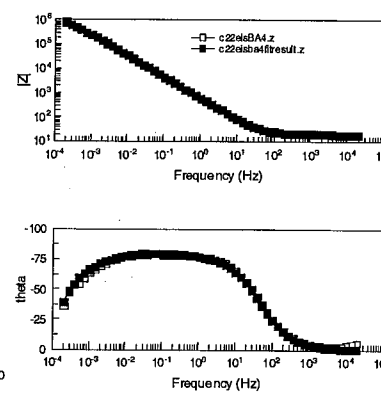
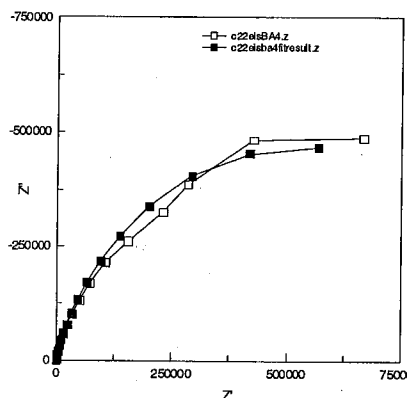
4/1/03

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Element	Freedom	Value	Error	Error %
Rs	Free(+)	28.68	0.10824	0.4057
Rox porous	Free(+)	1.0495E5	11899	11.338
CPEporous-T	Free(+)	0.00053568	6.8837E-6	1.2865
CPEporous-P	Free(+)	0.80042	0.0022557	0.28181
Rox barrier	Free(+)	6.9438E6	5.022E5	7.2322
CPE barrier-T	Free(+)	0.00055419	7.5895E-6	1.3859
CPE barrier-P	Fixed(X)	1	N/A	N/A

Chi-Squared: 0.001849  
Weighted Sum of Squares: 0.18899  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA5.  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous bart  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	20.09	0.1149	0.57193
Rox porous	Free(+)	1.2954E5	27585	21.295
CPEporous-T	Free(+)	0.00052567	1.8892E-5	3.5925
CPEporous-P	Free(+)	0.82791	0.0044789	0.54099
Rox barrier	Free(+)	8.7853E5	23794	2.7084
CPE barrier-T	Free(+)	0.0008771	5.3985E-5	6.1527
CPE barrier-P	Fixed(X)	1	N/A	N/A

Chi-Squared: 0.0031987  
Weighted Sum of Squares: 0.34416  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA4.  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous bart  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

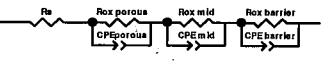
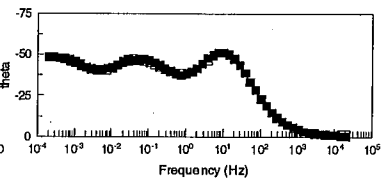
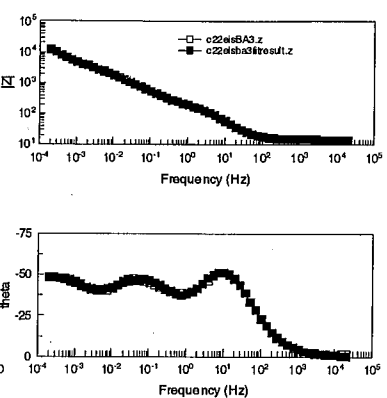
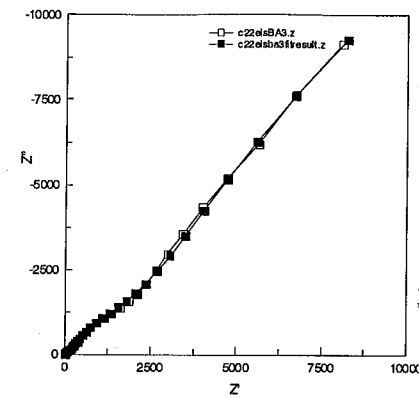
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Date \_\_\_\_\_

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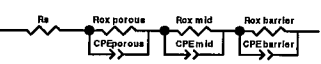
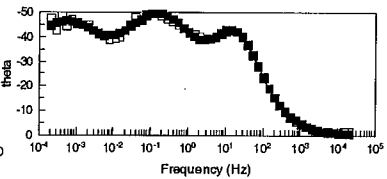
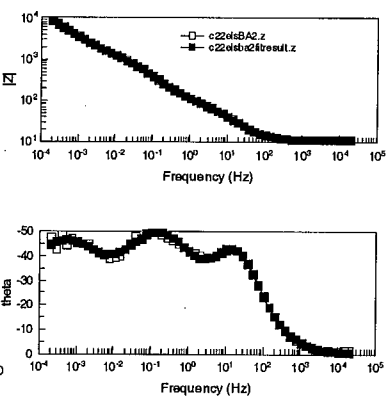
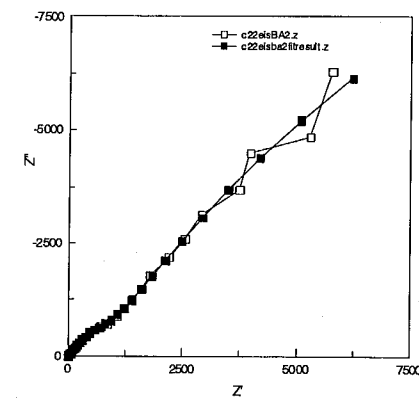
4/22/03

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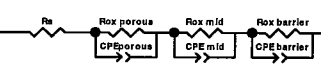
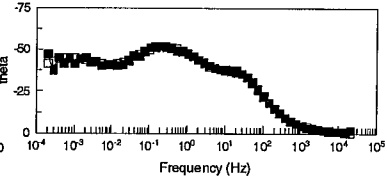
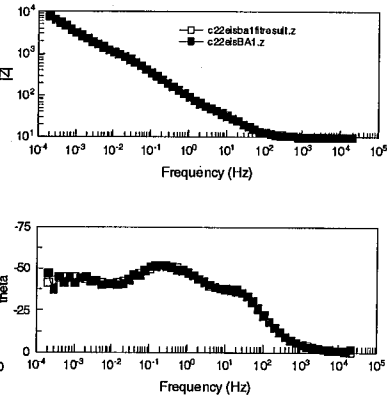
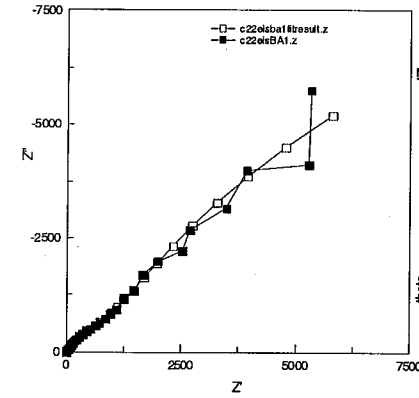
Element	Freedom	Value	Error	Error %
Rs	Free(+)	14.6	0.041843	0.2866
Rox porous	Free(+)	118	4.2544	3.6054
CPEporous-T	Free(+)	0.00069841	1.43E-5	2.0833
CPEporous-P	Free(+)	0.89766	0.0006388	0.71237
Rox mid	Free(+)	1823	189.95	10.965
CPE mid-T	Free(+)	0.0041154	0.00031188	7.5784
CPE mid-P	Free(+)	0.70985	0.01454	2.0488
Rox barrier	Free(+)	65246	12060	18.484
CPE barrier-T	Free(+)	0.0008485	0.0014817	15.355
CPE barrier-P	Free(+)	0.71393	0.023733	3.3243

Chi-Squared: 0.00042204  
Weighted Sum of Squares: 0.043882  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA3.  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous bart  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	11.13	0.054515	0.4888
Rox porous	Free(+)	39	2.3689	6.0759
CPEporous-T	Free(+)	0.0010024	5.788E-5	5.7548
CPEporous-P	Free(+)	0.88018	0.018119	1.8313
Rox mid	Free(+)	782.3	105.01	13.551
CPE mid-T	Free(+)	0.0053374	0.00050408	9.1028
CPE mid-P	Free(+)	0.74266	0.022157	2.9835
Rox barrier	Free(+)	38124	6085.1	16.79
CPE barrier-T	Free(+)	0.00065	0.0010209	15.292
CPE barrier-P	Free(+)	0.6652	0.022313	3.3543

Chi-Squared: 0.0010509  
Weighted Sum of Squares: 0.1093  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA2.  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous bart  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	9.66	0.060955	0.6182
Rox porous	Free(+)	15.4	1.6424	10.685
CPEporous-T	Free(+)	0.0013287	0.00016931	12.743
CPEporous-P	Free(+)	0.89909	0.032852	3.6361
Rox mid	Free(+)	604.1	97.326	16.111
CPE mid-T	Free(+)	0.0059883	0.00063728	10.642
CPE mid-P	Free(+)	0.7421	0.029281	3.9887
Rox barrier	Free(+)	27446	4116.2	14.997
CPE barrier-T	Free(+)	0.00851	0.0011696	13.744
CPE barrier-P	Free(+)	0.64611	0.023346	3.6133

Chi-Squared: 0.0014476  
Weighted Sum of Squares: 0.16055  
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA1.  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous bart  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

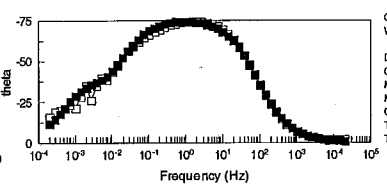
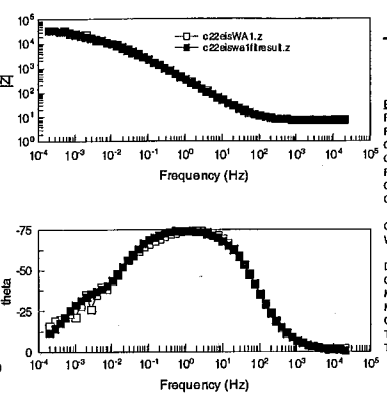
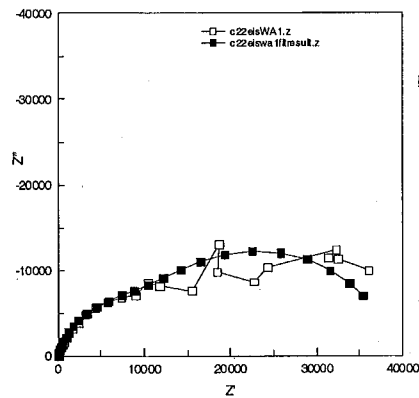
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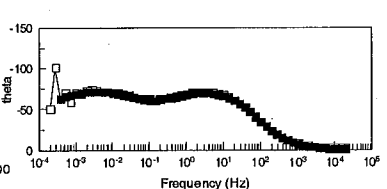
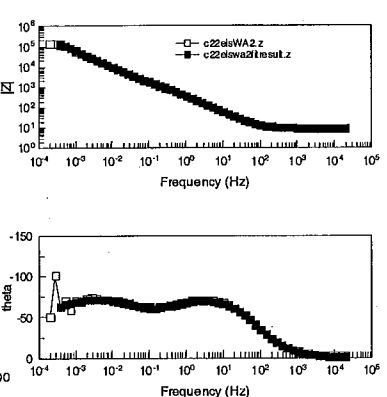
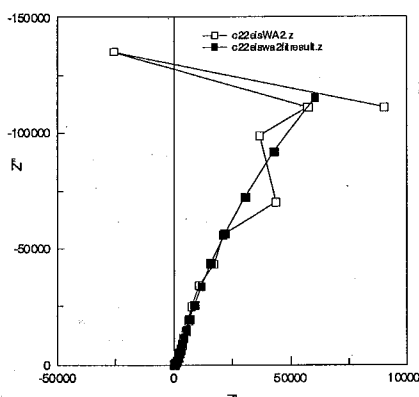
From Page No. \_\_\_\_\_



Element	Freedom	Value	Error	Error %
Rs	Free(+)	7.544	0.078254	1.0108
Rox porous	Free(a)	9828	2068.3	21.059
CPEporous-T	Free(+)	0.00078094	0.00010609	13.585
CPEporous-P	Free(+)	0.85522	0.022575	2.6397
Rox barrier	Free(+)	29419	3322.2	11.293
CPE barrier-T	Free(+)	0.0026771	0.0011828	44.182
CPE barrier-P	Free(+)	0.83831	0.059939	7.15

Chi-Squared: 0.006322  
Weighted Sum of Squares: 0.67846

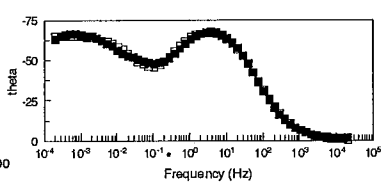
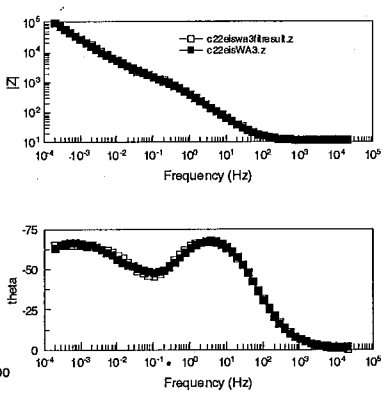
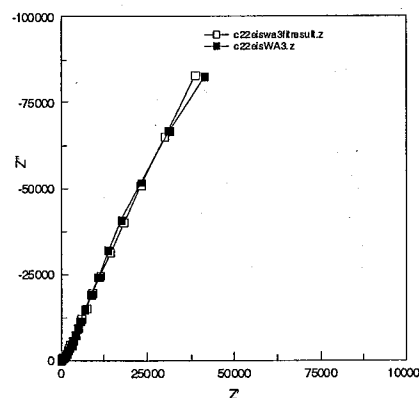
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Circuit Model File: D:\corrosion tests\valley c-22\impedance\porous barrier  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	9.001	0.058178	0.64635
Rox porous	Free(a)	758.6	67.094	8.8446
CPEporous-T	Free(+)	0.0011676	4.0034E-5	3.4287
CPEporous-P	Free(+)	0.83672	0.0088163	1.0548
Rox barrier	Free(+)	5.5584E5	51468	9.0959
CPE barrier-T	Free(+)	0.0010983	2.6892E-5	2.453
CPE barrier-P	Free(+)	0.83872	0.0059502	0.70944

Chi-Squared: 0.0025358  
Weighted Sum of Squares: 0.28119

Data File: D:\corrosion tests\valley c-22\impedance\c22slwa2  
Circuit Model File: D:\corrosion tests\valley c-22\impedance\porous barrier  
Mode: Run Fitting / Freq. Range (0.0039 - 20000)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	11.83	0.060354	0.51895
Rox porous	Free(a)	891.5	35.356	3.9659
CPEporous-T	Free(+)	0.00075441	1.2434E-5	1.5482
CPEporous-P	Free(+)	0.88495	0.0081824	0.92236
Rox barrier	Free(+)	2.4061E6	1.3073E6	54.333
CPE barrier-T	Free(+)	0.0016144	3.2489E-5	2.0125
CPE barrier-P	Free(+)	0.74959	0.0044256	0.5904

Chi-Squared: 0.0014747  
Weighted Sum of Squares: 0.1578

Data File: D:\corrosion tests\valley c-22\impedance\c22slwa3  
Circuit Model File: D:\corrosion tests\valley c-22\impedance\porous barrier  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

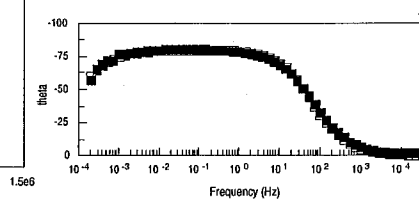
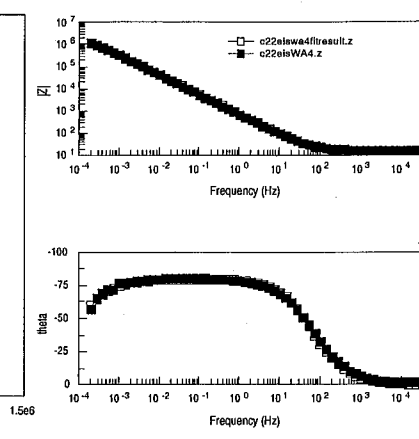
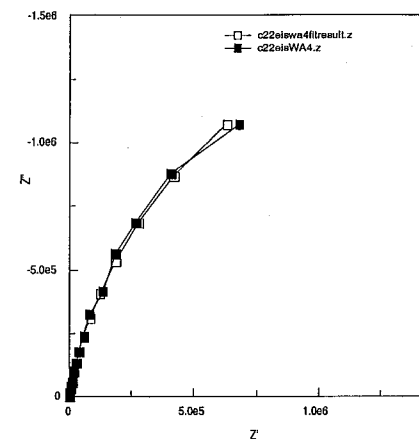
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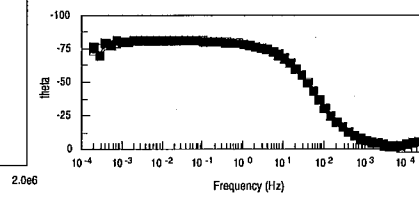
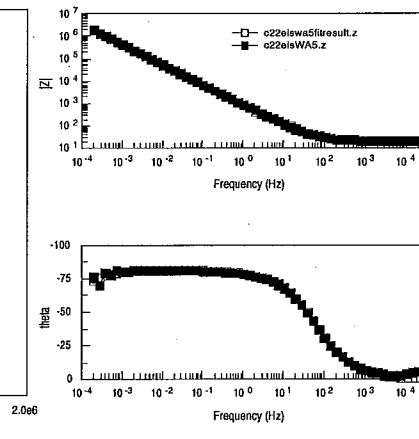
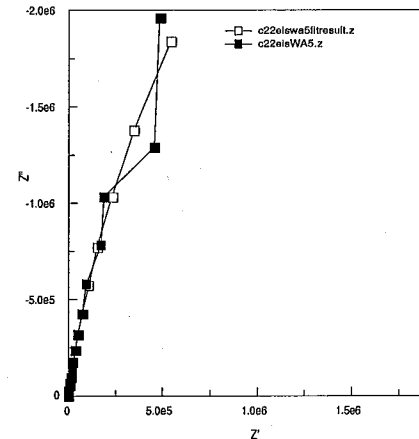
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Element	Freedom	Value	Error	Error %
Rs	Free(+)	18.24	0.082467	0.5078
Rox porous	Free(a)	1.55	0.24424	15.757
CPEporous-T	Free(+)	1.6896E-14	3.8806E-15	22.846
CPEporous-P	Free(+)	4.372	0.34738	7.9456
Rox barrier	Free(+)	3.4838E6	1.2508E5	3.5905
CPE barrier-T	Free(+)	0.000268	8.0445E-7	0.30017
CPE barrier-P	Free(+)	0.88552	0.00078218	0.87933

Chi-Squared: 0.0020028  
Weighted Sum of Squares: 0.2143

Data File: D:\corrosion tests\valley c-22\impedance\c22slwa4.z  
Circuit Model File: D:\corrosion tests\valley c-22\impedance\porous barrier oxide.mtl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	21.21	0.14856	0.70514
Rox porous	Free(a)	894.2	2354.1	264.38
CPEporous-T	Free(+)	0.0048737	0.0024757	50.787
CPEporous-P	Free(+)	0.58281	0.05131	8.8069
Rox barrier	Free(+)	1.258E7	1.6305E6	12.961
CPE barrier-T	Free(+)	0.00022658	7.3978E-6	3.285
CPE barrier-P	Free(+)	0.9141	0.0060308	0.65522

Chi-Squared: 0.0021028  
Weighted Sum of Squares: 0.225

Data File: D:\corrosion tests\valley c-22\impedance\c22slwa5.z  
Circuit Model File: D:\corrosion tests\valley c-22\impedance\porous barrier oxide.mtl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

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4/22/03



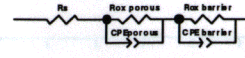
From Page No.



Element	Freedom	Value	Error	Error%
Rs	Free(+)	20.87	0.14482	0.69296
Rox porous	Free(+)	169.8	227.86	134.08
CPEporous-T	Free(+)	0.0037286	0.0051135	13.71
CPEporous-P	Free(+)	0.5799	0.01851	3.2022
Rox barrier	Free(+)	1.5218E7	1.738E9	11.656
CPEbarrier-T	Free(+)	0.0002222	2.8285E-6	1.273
CPEbarrier-P	Free(+)	0.88832	0.028429	0.32003



Element	Freedom	Value	Error	Error%
Rs	Free(+)	16.03	0.09178	0.57127
Rox porous	Free(+)	5401	81.436	1.5078
CPEporous-T	Free(+)	0.00043099	4.1682E-6	0.9712
CPEporous-P	Free(+)	0.81891	0.025277	0.30867
Rox barrier	Free(+)	2.7496E9	7.7734E5	28.281
CPEbarrier-T	Fixed(0)	0.0011575	N/A	N/A
CPEbarrier-P	Free(+)	0.82385	0.014343	0.17284



Element	Freedom	Value	Error	Error%
Rs	Free(+)	11.58	0.056239	0.48568
Rox porous	Free(+)	879.8	24.88	2.8258
CPEporous-T	Free(+)	0.00061051	8.4704E-6	1.3874
CPEporous-P	Free(+)	0.84912	0.051689	0.60674
Rox barrier	Free(+)	1.1333E9	3.2535E5	28.548
CPEbarrier-T	Free(+)	0.0018995	3.4617E-6	1.8253
CPEbarrier-P	Free(+)	0.74733	0.040704	0.54488

Chi-Squared: 0.0018055  
 Weighted Sum of Squares: 0.1719

Data File: D:\corrosion tests\alloy c-22\impedance\elstest30.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Batch Fitting / All Data Points (1 - 57)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

Chi-Squared: 0.0018352  
 Weighted Sum of Squares: 0.1982

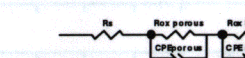
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 Mode: Run Fitting / All Data Points (1 - 57)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

Chi-Squared: 0.0011618  
 Weighted Sum of Squares: 0.12429

Data File: D:\corrosion tests\alloy c-22\impedance\elstest18.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 57)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error%
Rs	Free(+)	7.515	0.045505	0.60552
Rox porous	Free(+)	57.45	2.7429	4.7744
CPEporous-T	Free(+)	0.00086363	4.958E-5	5.7525
CPEporous-P	Free(+)	0.98176	0.017025	1.7341
Rox barrier	Free(+)	1.3522E9	13714	10.142
CPEbarrier-T	Free(+)	0.0029815	2.8491E-5	0.98205
CPEbarrier-P	Free(+)	0.64467	0.027404	0.42509



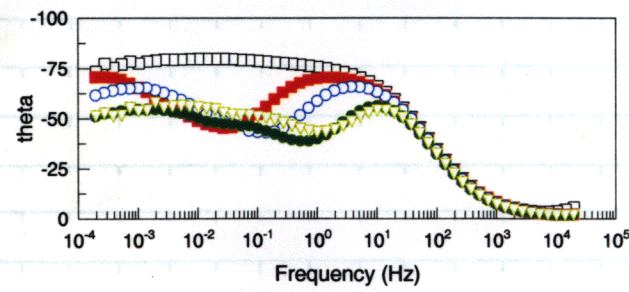
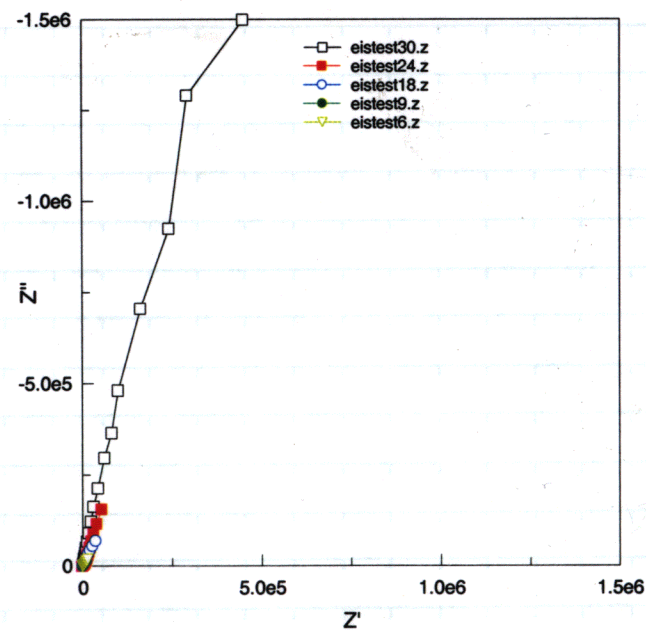
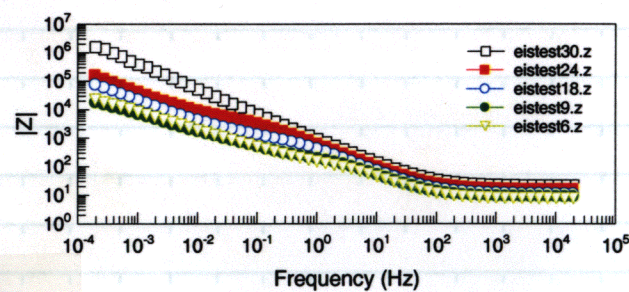
Element	Freedom	Value	Error	Error%
Rs	Free(+)	8.599	0.054417	0.74912
Rox porous	Free(+)	110.4	4.8913	4.4215
CPEporous-T	Free(+)	0.0007224	3.3242E-5	4.6785
CPEporous-P	Free(+)	0.98285	0.016182	1.6416
Rox barrier	Free(+)	2.1829E9	57940	26.543
CPEbarrier-T	Free(+)	0.0031842	4.3828E-5	1.3796
CPEbarrier-P	Free(+)	0.61685	0.036782	0.59586

Chi-Squared: 0.0013856  
 Weighted Sum of Squares: 0.14826

Data File: D:\corrosion tests\alloy c-22\impedance\elstest15.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 57)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus

Chi-Squared: 0.0018607  
 Weighted Sum of Squares: 0.19909

Data File: D:\corrosion tests\alloy c-22\impedance\elstest19.z  
 Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
 Mode: Run Fitting / All Data Points (1 - 57)  
 Maximum Iterations: 500  
 Optimization Iterations: 200  
 Type of Fitting: Complex  
 Type of Weighting: Calc-Modulus



WELD METAL FROM PAGE 23

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

4/29/03

To Page No.

From Page No.

# Electrochemical Impedance Spectroscopy Alloy 22

Objective: Same As pg #2

Specimen: C-22 Base Alloy 05990242 DOE material Plate 0.62X  
 (See NB # 505 pg # 2-25)  
 C-22 Weld Alloy 05990242 DOE material Plate 0.62X  
 filler X# 204886 (See NB # 505 pg # 2-25)

(BA)  
 Start wt = 12.81995, Santorius Genius SN# 12809099 cal 11/15/02 due 5/15/03  
 End wt = 12.82037

(WA)  
 Start wt = 12.74897, Santorius Genius SN# 12809099 cal 11/15/02 due 5/15/03  
 End wt = 12.74881

Solution: 0.028M NaCl \* same solution as previous test \*  
 3.250g NaCl lot # 027828  
 + DI water to 2000ml

pH start: Not taken, Fisher Accuret 950 meter SN# 3340 cal 8/7/02 due 8/7/03  
 pH End: 8.067, pH probe # 13-620-296 SN# 2251257 PL

Cell Info: Area = 8 cm<sup>2</sup>, Density = 8.69 g/cm<sup>3</sup>, E<sub>g</sub> WT = 26.04

Impedance Analyzer = Schlumberger 1260  
 Counter Electrode = Pt Plug  
 Reference = Fisher 13-620-52 SN# 0052132  
 Temperature = 95°C - 25°C, H<sub>2</sub> thermometer SN# 98-393 cal 8/11/03 due 8/11/03  
 (WA) (BA)  
 E<sub>corr</sub> = -571 mV, E<sub>corr</sub> = -649 mV, Keithley 617 SN# 537418  
 E<sub>p</sub> = -49 mV, E<sub>p</sub> = -49 mV, cal 4/2/03 due 10/2/03

Solution Deaerated with 99.999% N<sub>2</sub>

Witnessed & Understood by me,

Date

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Date

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*[Signature]*

4/30/03

To Page No.



From Page No. \_\_\_\_\_



Element	Freedom	Value	Error	Error %
Rs	Free(+)	20.24	0.22647	1.1189
Rox porous	Free(+)	548.1	374.47	68.321
CPEporous-T	Free(+)	0.0017769	0.00031918	17.963
CPEporous-P	Free(+)	0.63785	0.023098	3.6212
Rox barrier	Free(+)	1.4382E7	2.0041E6	13.935
CPE barrier-T	Free(+)	0.0001704	3.4498E-6	2.0245
CPE barrier-P	Free(+)	0.90652	0.0046713	0.5153

Chi-Squared: 0.0054928  
Weighted Sum of Squares: 0.58773

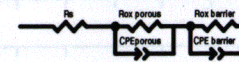
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisWA14.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	15.3	0.13917	0.90861
Rox porous	Free(+)	64.34	35.027	54.44
CPEporous-T	Free(+)	0.0034469	0.00064823	18.748
CPEporous-P	Free(+)	0.61232	0.029446	4.8089
Rox barrier	Free(+)	9.6148E6	1.0244E6	10.655
CPE barrier-T	Free(+)	0.00021965	1.731E-6	0.79995
CPE barrier-P	Free(+)	0.89134	0.0021183	0.23785

Chi-Squared: 0.0022058  
Weighted Sum of Squares: 0.28912

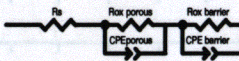
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	11.36	0.050849	0.44761
Rox porous	Free(+)	17.129	1789.6	10.331
CPEporous-T	Free(+)	0.00073389	4.5293E-5	6.1716
CPEporous-P	Free(+)	0.78159	0.0020549	0.26291
Rox barrier	Free(+)	3.3573E5	3.5641E5	10.616
CPE barrier-T	Free(+)	0.00064241	3.3343E-5	5.1903
CPE barrier-P	Free(+)	0.89708	0.0084111	0.98554

Chi-Squared: 0.0010684  
Weighted Sum of Squares: 0.11432

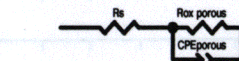
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisWA11.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	8.623	0.042887	0.49736
Rox porous	Free(+)	215.9	5.3358	2.4714
CPEporous-T	Free(+)	0.00078641	1.6815E-5	2.1382
CPEporous-P	Free(+)	0.83409	0.0054335	0.65143
Rox barrier	Free(+)	2.8169E5	27614	9.803
CPE barrier-T	Free(+)	0.0028491	3.182E-5	1.1168
CPE barrier-P	Free(+)	0.73296	0.0028137	0.38388

Chi-Squared: 0.0010769  
Weighted Sum of Squares: 0.11523

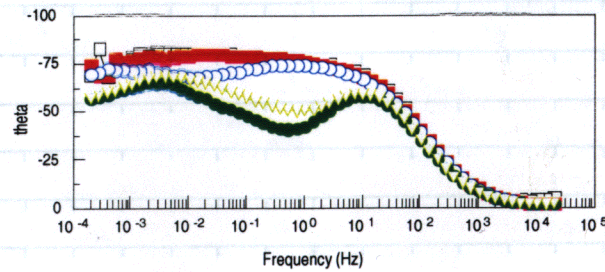
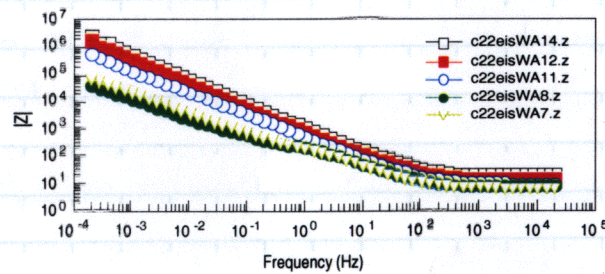
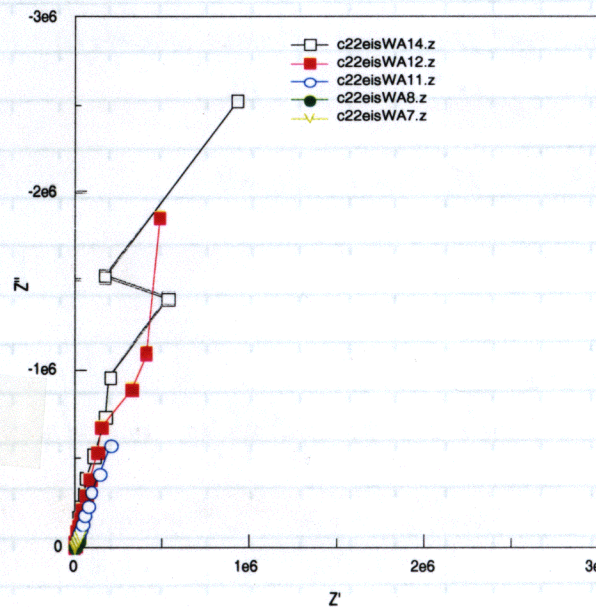
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	7.263	0.03104	0.42737
Rox porous	Free(+)	145.1	4.5169	3.113
CPEporous-T	Free(+)	0.0011188	2.6422E-5	2.3616
CPEporous-P	Free(+)	0.83939	0.0059564	0.70961
Rox barrier	Free(+)	3.0326E5	15320	5.0518
CPE barrier-T	Free(+)	0.0020752	1.6738E-5	0.80657
CPE barrier-P	Free(+)	0.76724	0.0021068	0.27459

Chi-Squared: 0.0008422  
Weighted Sum of Squares: 0.090115

Data File: D:\corrosion tests\alloy c-22\impedance\c22eisWA7.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



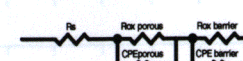
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Element	Freedom	Value	Error	Error %
Rs	Free(+)	8.423	0.040209	0.47737
Rox porous	Free(+)	11.89	0.62439	5.2514
CPEporous-T	Free(+)	0.00082122	8.9993E-5	10.958
CPEporous-P	Free(+)	1.089	0.025116	2.3495
Rox barrier	Free(+)	85456	10940	12.802
CPE barrier-T	Free(+)	0.0050688	3.0734E-5	0.60634
CPE barrier-P	Free(+)	0.59604	0.0019714	0.33075

Chi-Squared: 0.0010346  
Weighted Sum of Squares: 0.11071

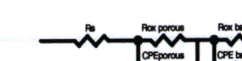
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	10.17	0.035477	0.34884
Rox porous	Free(+)	62.53	1.4169	2.296
CPEporous-T	Free(+)	0.0008427	2.5823E-5	3.0643
CPEporous-P	Free(+)	0.91707	0.0078826	0.85954
Rox barrier	Free(+)	2.6829E5	55502	20.687
CPE barrier-T	Free(+)	0.0042599	2.8995E-5	0.67995
CPE barrier-P	Free(+)	0.63469	0.0019054	0.30021

Chi-Squared: 0.0058225  
Weighted Sum of Squares: 0.062301

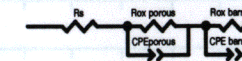
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	13.49	0.059836	0.44356
Rox porous	Free(+)	310.3	6.5618	2.1147
CPEporous-T	Free(+)	0.00067231	1.207E-5	1.7953
CPEporous-P	Free(+)	0.85005	0.0052831	0.6215
Rox barrier	Free(+)	7.1863E5	2.380E5	33.311
CPE barrier-T	Free(+)	0.0033769	4.5301E-5	1.3415
CPE barrier-P	Free(+)	0.69863	0.0032348	0.46289

Chi-Squared: 0.0010373  
Weighted Sum of Squares: 0.111

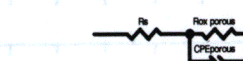
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	18.02	0.059038	0.32782
Rox porous	Free(+)	65.08	16.634	25.559
CPEporous-T	Free(+)	0.0070445	0.00058244	8.268
CPEporous-P	Free(+)	0.55235	0.013988	2.5325
Rox barrier	Free(+)	1.732E6	21243	1.2265
CPE barrier-T	Free(+)	0.00034073	1.1952E-6	0.35078
CPE barrier-P	Free(+)	0.86655	0.00091973	0.10614

Chi-Squared: 0.00028144  
Weighted Sum of Squares: 0.030115

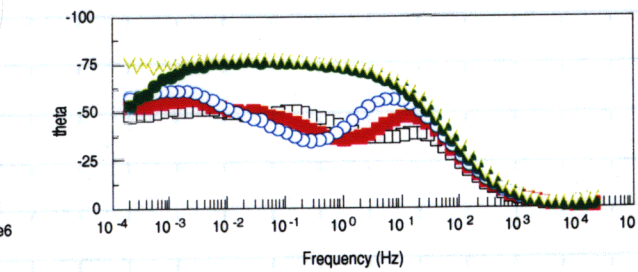
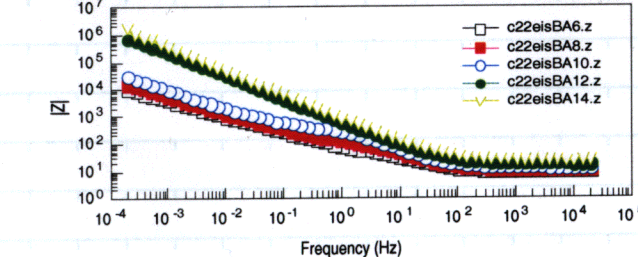
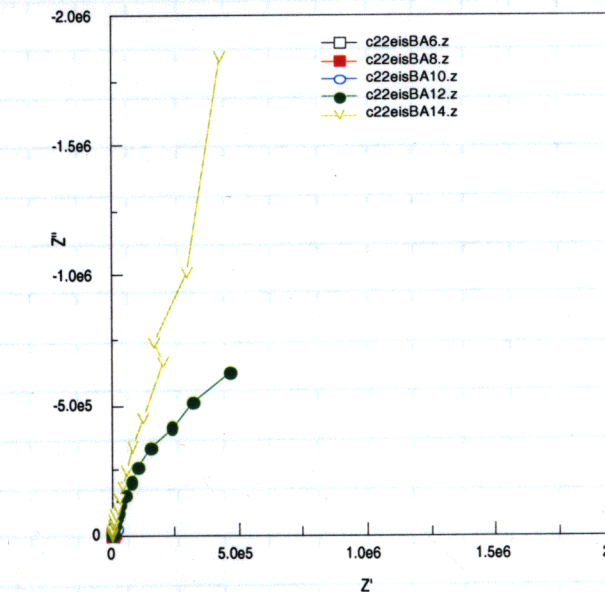
Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA12.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	24.07	0.25033	1.04
Rox porous	Free(+)	162.8	1097	673.83
CPEporous-T	Free(+)	0.0030484	0.00052071	17.081
CPEporous-P	Free(+)	0.62057	0.025741	4.148
Rox barrier	Free(+)	1.1943E7	3.1488E6	26.385
CPE barrier-T	Free(+)	0.00023638	9.9828E-6	3.9739
CPE barrier-P	Free(+)	0.88374	0.0075487	0.85418

Chi-Squared: 0.0059173  
Weighted Sum of Squares: 0.63315

Data File: D:\corrosion tests\alloy c-22\impedance\c22eisBA14.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



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Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

5/22/03

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Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

5/22/03



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ELECTROCHEMICAL IMPEDANCE SYSTEM CHECK

INSTRUMENTS

SOLARTRON 1260 IMPEDANCE / GAIN PHASE ANALYZER  
SERIAL NUMBER 400722

SOLARTRON 1287A ELECTROCHEMICAL INTERFACE  
SERIAL NUMBER 00148300

SOLARTRON 12861 TEST MODULE USED TO  
TEST SYSTEM

SOFTWARE ZVIEW VERSION 2.2 SN 1052-1  
ZPLOT VERSION 2.19 SN 1052-1

IMPEDANCE SPECTRUM OBTAINED FROM 20,000 TO  
0.0002 HZ

DATA PLOTS PAGE 33

SYSTEM FUNCTIONING CORRECTLY.

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

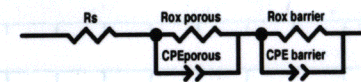
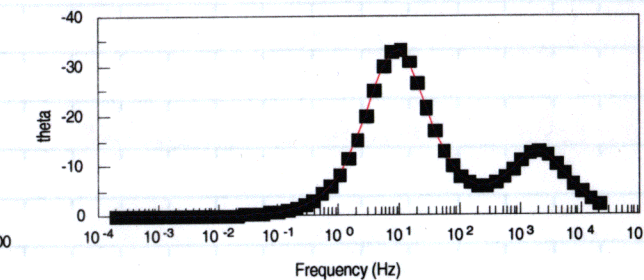
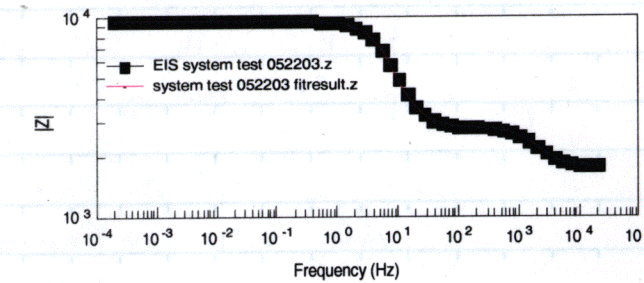
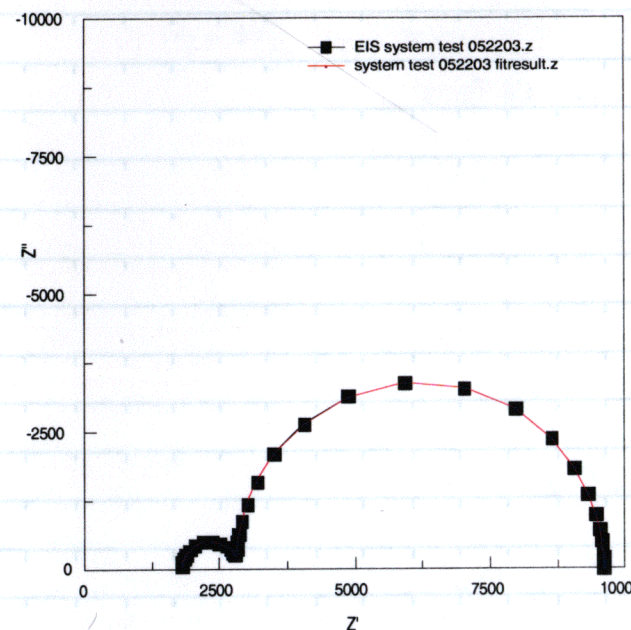
Date \_\_\_\_\_

Recorded by \_\_\_\_\_

5/23/03

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From Page No. \_\_\_\_\_



Element	Freedom	Value	Error	Error %
Rs	Free(+)	1801	0.60292	0.033477
Rox porous	Free(+)	1001	1.0295	0.10285
CPEporous-T	Free(+)	9.9295E-8	8.4667E-10	0.85268
CPEporous-P	Free(+)	1.001	0.00099368	0.099269
Rox barrier	Free(+)	6787	1.4002	0.020631
CPE barrier-T	Free(+)	4.7448E-6	5.5662E-9	0.11731
CPE barrier-P	Free(+)	0.99996	0.00029728	0.029729

Chi-Squared: 1.78E-5  
Weighted Sum of Squares: 0.0019046

Data File: D:\qa\EIS system tests\EIS system test 052203.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Fitting / All Data Points (1 - 57)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

FREQUENCY (HZ)	Z'(a) Spec	Z'(a) Actual	-Z''(b) Spec	-Z''(b) Actual
10,000	1,820	1,824	160	155
1,522	2,320	2,324	520	521
240	2,780	2,782	290	288
5	6,180	6,157	3,400	3,398
1	9,340	9,322	1,310	1,319

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

5/23/03



From Page No. \_\_\_\_\_

Passive film Chemistry - Glove Box -

Specimen - All Tests Run with Test Cell from NB# 485 pg #35  
See Setup on pg #35 And pg #34 from NB#485

Solution = 1000 ppm Cl<sup>-</sup> As KCl  
2.148 g KCl Lot # 605573  
+ DI water to 1000 ml

Temperature = 60°C

Potentiostat = EG & G Versastat SN# 20104

Reference = Fisher 13-620-52 SN# 0249107

Thermocouple Meter = Monogram Box w/omega DP 465 SN# 3130900  
Cal 1/15/03 Due 7/15/03

Calibrated Thermocouple SN# 335 Cal 3/14/03 Due 7/14/03

All measurements Taken with Keithley 614 SN# 0555368  
Cal 3/15/03 Due 10/8/03  
E<sub>corr</sub> = -241mV E<sub>p</sub> = -488mV

Specimen Top PTFE 276 Bolts Torque To 80 In-Oz

User Photo #6104 SN# 139072 Cal 3/6/03 Due 9/6/03

\* Note - Purgon Glove Box with 99.999% N<sub>2</sub>  
then Test Solution And Into Glove Box for 2hr. \*Started 5% Hydrogen  
Put Catalyst Into Glovebox fan Holder - fogged up Glovebox for 15min  
O<sub>2</sub> level slowly decreases to 0 - Hydrogen went from 0.8% to 3.8%  
Turned off Hydrogen/N<sub>2</sub> Gas stabilizes to 3.1%  
Started Heating Cell To 60°C 5/16/03 @ 2:30 pm

\* Note Gas mixture 5% Hydrogen - And N<sub>2</sub>

data pfcgbl

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	5/20/03

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.36  
Filename: A:\pfcgbl.dat  
Pstat: VStat[] Ver 2  
PS POTENTIOSTATIC  
Date Run: 05-01-03  
File Status: NORMAL  
Time Run: 13:53:38

Cond. Time	CT	pass	s	Initial Pot.	IP	100.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	604.0E3	s
Initial Delay	ID	5	s	Stop On	SO	Pass	

Time/Pt.	TP	201.3	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					

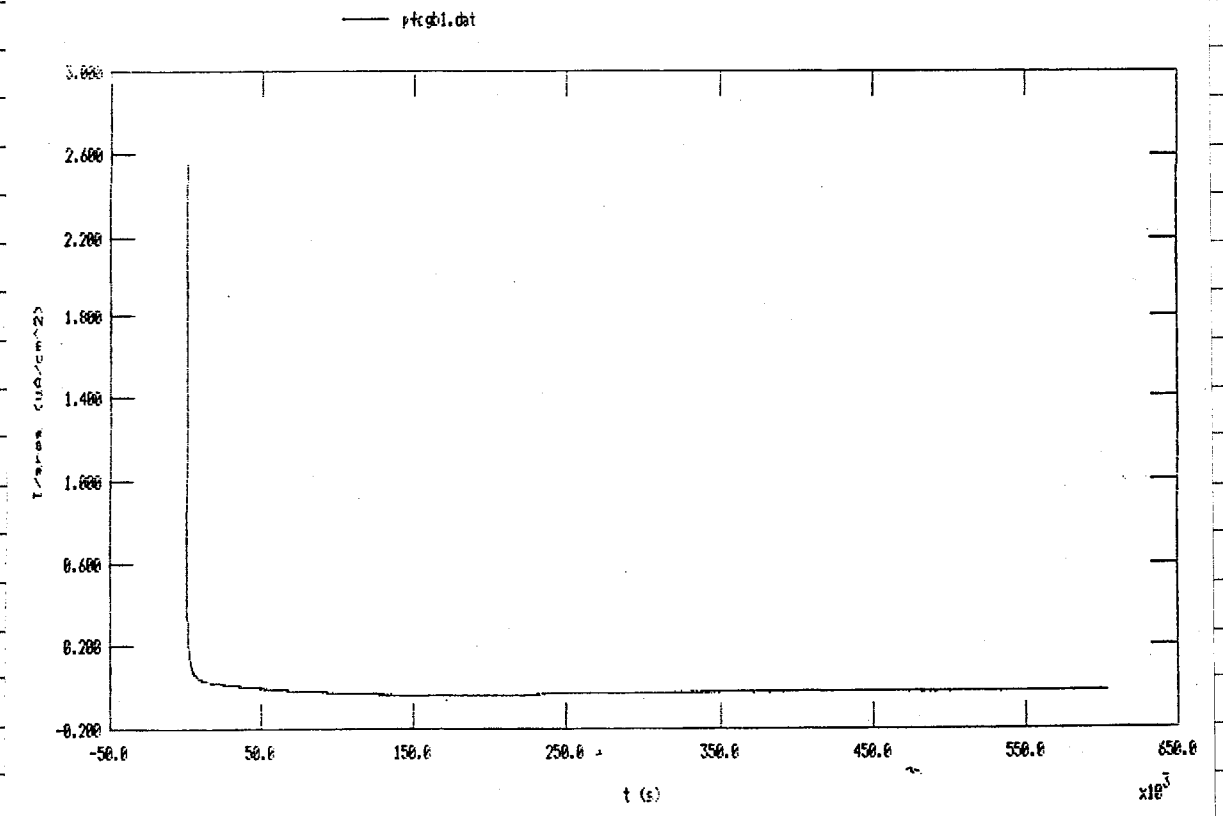
Line Sync.	LS	yes		IR Mode	IR	none	
Rise Time	RT	high stability		Filter	FL	1.5.3Hz	
Working Elec.	WE	Solid		Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	AR	8.162	cm <sup>2</sup>	Equiv. Wt.	EW	26.04	g
Density	DE	8.696	g/ml	AUX A/D	AU	no	
Open Circuit	OC	-232.0E-3	V				

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Model 352/252 Corrosion Analysis Software, v. 2.36  
PS POTENTIOSTATIC  
CP PASS vs. R  
CR AUTO  
REF 0.24150 SCE  
DC -0.232  
Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Filename: A:\pfcgbl.dat  
File Status: NORMAL  
Date Run: 05-01-03  
Time Run: 13:53:38

TP	2.013E+02	T1	6.04E+05
ID	5	RT	HIGH STABILITY
IR	NONE	EW	26.04E+01
LS	YES	EN	8.696E+00
		AU	NO



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	5/27/03



From Page No. \_\_\_\_\_

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY DUPLICATE SUMMARY

Sample ID: **PFC GB #1 CELL SOLUTION**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227900D

Analysis	Sample Result (mg/L)	Duplicate Result (mg/L)	RPD
Chromium	<0.167	<0.167	0.00%
Cobalt	<0.167	<0.167	0.00%
Iron	<1.67	<1.67	0.00%
Molybdenum	0.520	0.409	23.75%
Nickel	3.57	3.58	0.35%
Potassium	9806	9752	0.55%
Sodium	707	703	0.56%
Tungsten	<0.333	<0.333	0.00%

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Sample ID: **PFC GB #1 CELL SOLUTION**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227900

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	0.520	0.167
Nickel	3.57	0.167
Potassium	9806	3.33
Sodium	707	1.67
Tungsten	<0.333	0.333

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Sample ID: **PFC GB #1 20% HCL**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227900

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	0.348	0.167
Nickel	512	3.33
Potassium	3.83	1.67
Sodium	<0.333	0.333
Tungsten	<0.333	0.333

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY MATRIX SPIKE SUMMARY

Sample ID: **PFC #2 CELL GLOVE BOX**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227901S

Analysis	Sample Result (mg/L)	Spike Result (mg/L)	Spike Added (mg/L)	Recovery
Chromium	<0.167	12.5	13.3	93.5%
Cobalt	<0.167	32.2	33.3	96.5%
Iron	<1.67	67.2	66.7	100.9%
Molybdenum	<0.167	133	133	99.5%
Nickel	0.616	31.6	33.3	92.9%
Potassium	7416	8089	667	100.9%
Sodium	4.63	660	667	98.3%
Tungsten	<0.333	133	133	99.7%

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Sample ID: **PFC #2 CELL GLOVE BOX**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227901

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	0.616	0.167
Potassium	7416	3.33
Sodium	4.63	1.67
Tungsten	<0.333	0.333

### SOUTHWEST RESEARCH INSTITUTE PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Sample ID: **PFC #2 PT FLQ 20% HCL**

Lab Name: Southwest Research Institute  
Client: Division 20  
Lab Code: SwRI  
Date Received: 06/19/03  
Matrix: Liquid  
Project No.: 20.06002.01.081  
Task Order: 030619-6  
SRR: 24481  
Lab System ID: 227903

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	<0.167	0.167
Potassium	279	3.33
Sodium	<1.67	1.67
Tungsten	<0.333	0.333

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by B. K. D.

Date 6/30/03

From Page No. \_\_\_\_\_

### FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: Danzell Dorn Request Date: 6-19-03  
Project No.: 20.06002.01.081 Phone No.: X 6051  
Description of Work Requested:  
ICP Analysis - at Solving Sea  
Chromium - Cobalt - Iron - Molybdenum - Nickel - Potassium - Sodium  
Asa Truysten

Optical Microscopy  SEM  Hardness  Profilometer  Auger  Other

QUALITY REQUIREMENTS: The work requested is governed by the CNWRA Quality Assurance Program which addresses requirements of 10CFR50, Appendix B. Personnel performing this work shall be qualified under the CNWRA QA program or equivalently under the SwRI Nuclear QA program. Test and analysis methods shall be documented by approved procedures or recognized, standard methods. Measuring and test equipment shall be calibrated and controlled according to CNWRA and SwRI Nuclear QA program requirements.

Sample Identification	Description
PFC #1	
PFC #2	
20% HCL #1	
20% HCL #2	

B. TO BE COMPLETED BY DIVISION PERFORMING WORK

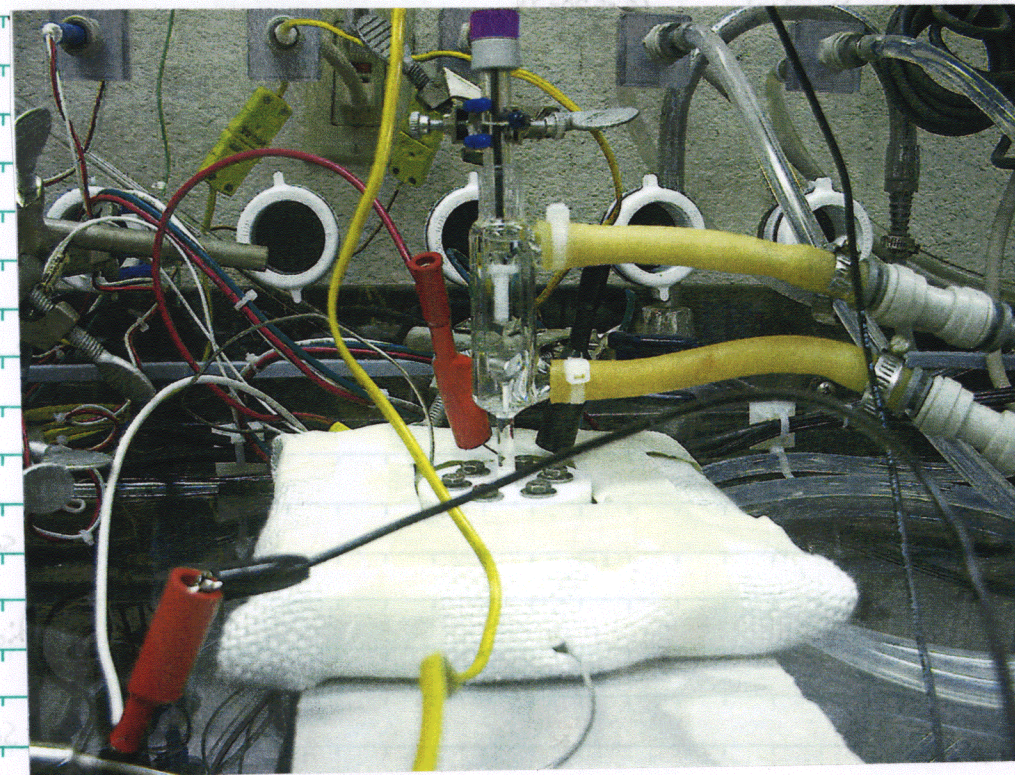
Optical Microscopy  SEM  Hardness  Profilometer  Auger  Other

Person Assigned: \_\_\_\_\_ Signature: \_\_\_\_\_  
Division: \_\_\_\_\_ Date: \_\_\_\_\_

Make, Model & Serial No. of Equipment Used (attach list if necessary): \_\_\_\_\_

Software Used (if any): \_\_\_\_\_  
Standards Used (if any): \_\_\_\_\_  
Photographic Negative Numbers (if applicable): \_\_\_\_\_

Please sign and date any hardcopy of analysis or list of photographs (The photographs themselves need not be signed). If error occurred during entry, do not erase or overwrite, but strikeout with single line, initial and date, and then reenter correct information.



SAMPLE LIST/CHAIN OF CUSTODY

Client: Danzell Dorn CNWRA

Requested Turnaround:  
 2 Weeks  
 3 Weeks  
 Other

Client Purchase Order Other ID: \_\_\_\_\_ Site Zone ID: \_\_\_\_\_

Analyses Requested: \_\_\_\_\_

Sample ID: \_\_\_\_\_  
Sample Collection Date (mm/dd/yyyy): \_\_\_\_\_  
Sample Collection Time: \_\_\_\_\_  
Matrix Type: \_\_\_\_\_  
Sample Type: \_\_\_\_\_  
# of Containers: \_\_\_\_\_

Matrix Types:  
A - Air  
B - Biotin  
D - Dust  
E - Emission/Stack  
L - Liquid  
P - Product  
Sd - Solid  
S - Soil  
SED - Sediment  
T - Tissue  
W - Water  
WP - Wipe

Sample Types:  
D - Duplicate  
ER - Equipment Rinseate  
ES - Environmental Sample  
FB - Field Blank  
FD - Field Duplicate  
MS - Matrix Spike  
MSD - Matrix Spike Dup  
TB - Trip Blank

Relinquished by (Print/Signature): Brian K. Dorn Date: 6/19/03 Time: \_\_\_\_\_  
Received by (Print/Signature): \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

Relinquished by (Print/Signature): \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_  
Received by (Print/Signature): \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

Relinquished by (Print/Signature): \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_  
Received by (Print/Signature): \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

Div 01 COC Form 01-01-001, Rev 8/02

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by B. K. D.

Date 6/30/03



From Page No. \_\_\_\_\_

### Open Circuit Potential Measurements

Specimen C-22 #1 + C-22 #2 + C-22 #3 Cylinders Specimens In cell #1  
See Notebook # 541 pg #22-23 for thermally Aged procedure 200°C for 30 days

C-22 #1  
Start wt = 12.59390g Sartorius Genius SN#12509099 cal 5/15/03 due 1/15/03

End wt = 12.59989g  
C-22 #2  
Start wt = 12.45590g Sartorius Genius SN#12509099 cal 5/15/03 due 1/15/03

End wt = 12.45682g  
C-22 #3  
Start wt = 12.32291g Sartorius Genius SN#12509099 cal 5/15/03 due 1/15/03

End wt = 12.32674g

Solution 0.014 M  $MgCl_2 \cdot 6H_2O$   
4.582g  $MgCl_2 \cdot 6H_2O$  lot #004768  
+ DI water To 2000ml

pH start = 5.821 Fisher Accumet 950 meter SN#3340 cal 5/1/02 due 8/1/03  
pH probe 13-620-296 SN#2291257 Pb

pH Adjusted To 4.261 with 35ul of 20% HCl solution lot #023844

pH End = 5.347

Reference: Fisher 13-620-52 SN# 520 5244

Counter Electrode = Pt Fla

Temperature = 95°C Hg Thermometer SN#115749 cal 1/13/03 due 1/13/04

Solution Bubbles with Zero Air

Specimen Examination:  
C-22 #1: No sign of corrosion - slight bulup of material on Specimen solution level

C-22 #2: No sign of corrosion - slight bulup of material on Specimen @ solution level

C-22 #3: No sign of corrosion - slight bulup of material on Specimen @ solution level

Test started 3-12-03 Test Ended 5/27/03 To Page No. \_\_\_\_\_

Witnessed & Understood by me, Date Invented by Date Recorded by Date  
B. K. [Signature] 5/28/03

TITLE \_\_\_\_\_

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### Open Circuit Measurement.

Cell Computer

C-22 #1 -49mv -53mv

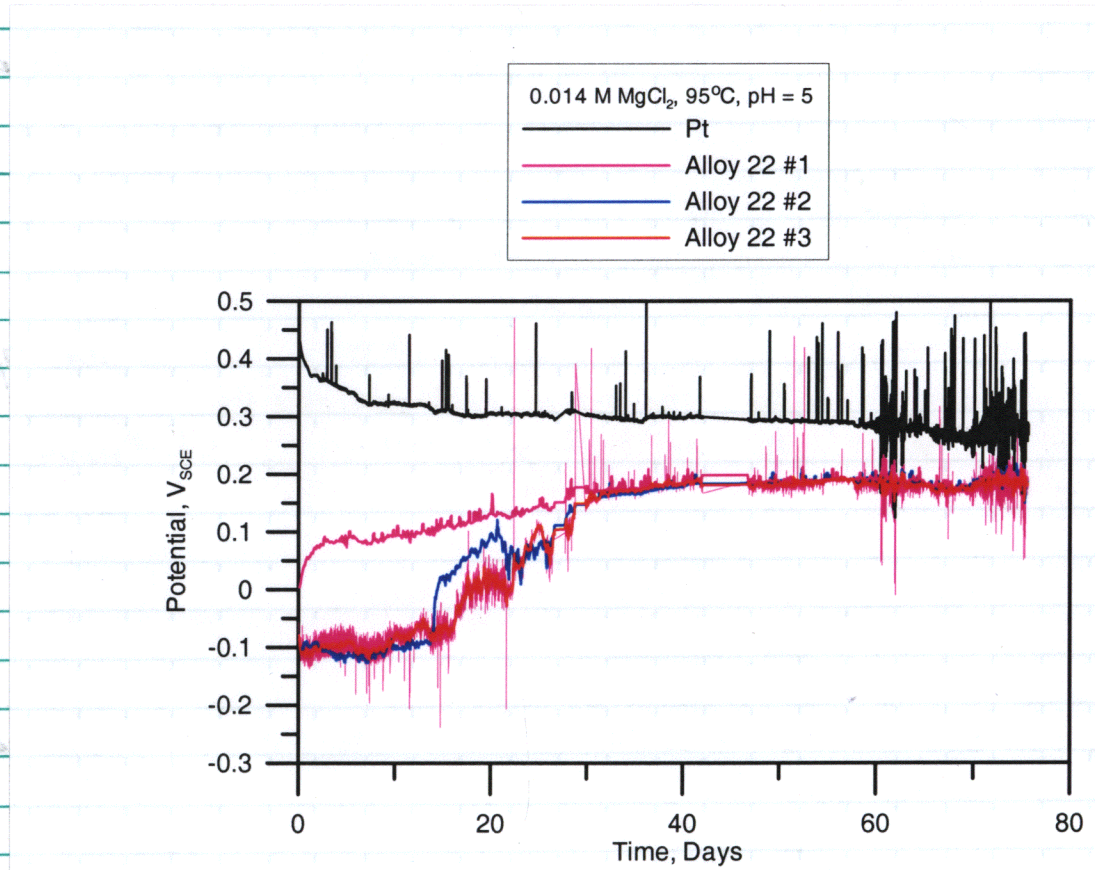
C-22 #2 -59mv -46mv

C-22 #3 +5 +6

Pt: +447mv +446mv

Measurements Taken with  
Keithley 614 SN#0704934 cal 5/26/02  
Due 5/26/03

monitored pH thru testing procedure Ranges from 4.261 - 5.343



Witnessed & Understood by me, Date Invented by Date Recorded by Date  
B. K. [Signature] 5/26/03



From Page No. \_\_\_\_\_

### Open Circuit Potential Measurement

Specimen: C-22#1 + C-22#2 + C-22#3 Cylindrical Specimens from Cell #2  
 All specimens used in previous testing. 600 Grit polished

C-22#1  
 start wt: 12.3219g, Santorius Genius sn# 12809099 cal 5/15/03 due 4/15/03  
 End wt: 12.27640g

C-22#2  
 start wt: 12.47436g, Santorius Genius sn# 12809099 cal 5/15/03 due 11/15/03  
 End wt: 12.43449g

C-22#3  
 start wt: 12.48140g, Santorius Genius sn# 12809099 cal 5/15/03 due 4/15/03  
 End wt: 12.47863g

Solution  
 0.014 M <sup>slightly</sup> MgCl<sub>2</sub> · 6H<sub>2</sub>O  
 0.028 M MgCl<sub>2</sub> · 6H<sub>2</sub>O lot# 004768  
 4.891g MgCl<sub>2</sub> · 6H<sub>2</sub>O lot# 004768  
 + DI water to 2000mls

pH start: 5.749 Fisher Accumet 950 meter sn# 3340 cal 5/7/02 due 5/7/03  
 pH probe 13-620-296 sn# 2291257 P6  
 pH Adjusted to 4.186 with 50ml of 20% HCl solution lot # 028544  
 pH End = 5.189

Reference: Fisher 13-620-52 sn# 0042119  
 Counter Electrode: Pt Flag

Temperature: 95°C Hg Thermometer sn# 1196-1090 cal 1/13/03 due 7/13/03  
 solution bubbles with zero Air

Specimen Examination  
 C-22#1: No Sign of Corrosion - slight build up of material at solution level  
 C-22#2: No Sign of Corrosion - slight build up of material at solution level  
 C-22#3: No Sign of Corrosion - slight build up of material at solution level

Test starts 3-12-03 Test Ends 5/27/03 Data Test #14 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	5/29/03
		<i>B. K. [Signature]</i>	

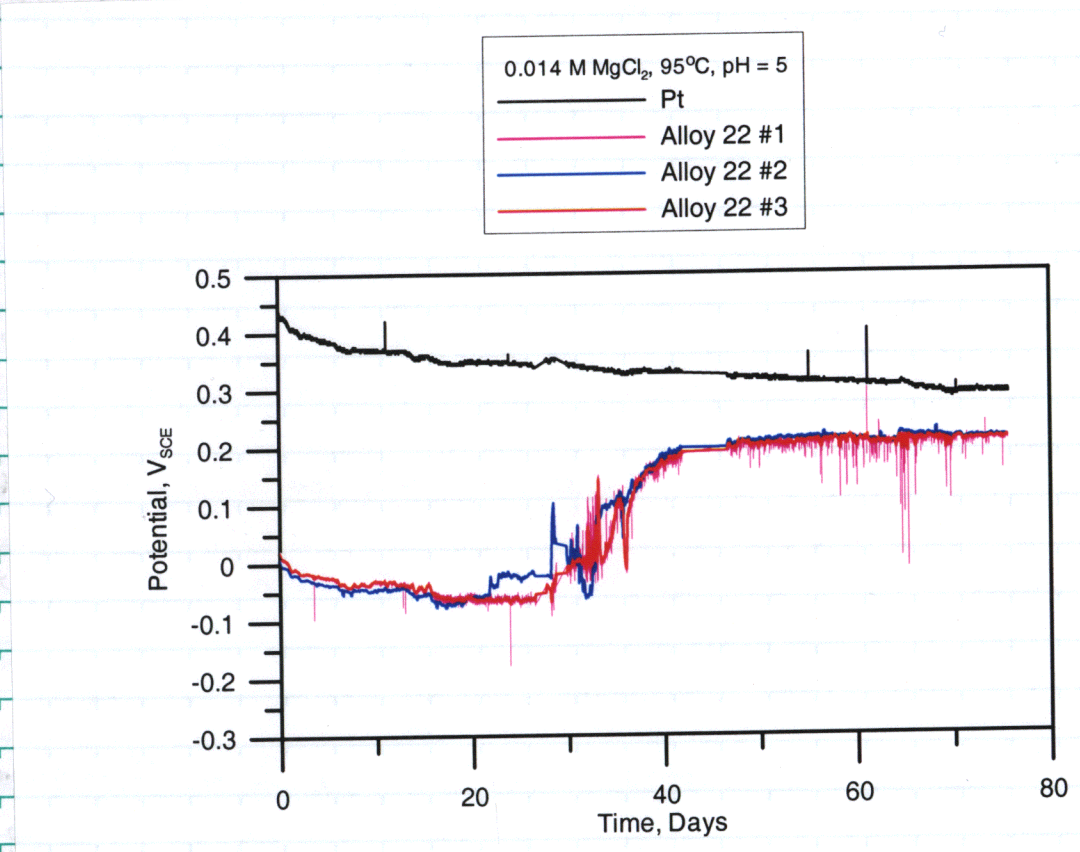
From Page No. \_\_\_\_\_

### Open Circuit Measurements

Cell	Compton	Measurement
C-22#1	+25mv	+27mv
C-22#2	+16mv	+16mv
C-22#3	+75mv	+69mv
Pt	+43mv	+43mv

Measurements taken with Keithley 614 sn# 0704924 cal 5/26/02 due 5/26/03

Monitored pH thru testing procedure Ranges from 4.186 - 5.173



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	5/29/03
		<i>B. K. [Signature]</i>	



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### Electrochemical Impedance Spectroscopy Alloy 22

Objective: same as pg #2

Specimen: C-22 weld Alloy 0590242 DOE Material Plate 062X  
Sillen XX 204866 (See NB #505 pg #2-25)

Weld Alloy  
Start wt = 12.73238g Santaricus Genius SN# 12809099 cal 5/1/02 due 11/13/02  
End wt = 12.73237g

Solution: 0.028 M NaCl \* Used 900 ml in Autoclave  
3.285g NaCl  
+ DI water to 2000ml

pH Start = 5.977 Fisher Accumet 950 Meter SN# 3340 cal 8/7/02 due 8/7/03  
pH End = 6.333 pH probe # 15-620-246 SN# 2291257 PL

Cell Info Area = 8 cm<sup>2</sup> Density = 8.69 g/cm<sup>3</sup> Eq wt = 26.64

Impedance Analyzer = Solartron 1260 - Par 4843 w/Autoclave

Counter Electrode: Pt Flag

Reference: ~~Fisher 13-620-52 SN# 0052132~~ <sup>6/1/03</sup> Sillen - Silver chloride

Temperature: 95°C - 150°C Hg thermometer SN# 598-393 <sup>6/1/03</sup>  
Fluke #2190 Digital Thermometer SN# 3133004 cal 1/31/02 due 1/31/04  
thermocouple # 334 cal 5/1/03 due 11/1/03

Ecorr -198mV Keithley 614 SN# 0555368  
Ept +71mV cal 4/8/03 due 10/8/03

Solution Deaerates then shut off when up to Temperature  
Used 95.99% N<sub>2</sub> for Deaeration

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

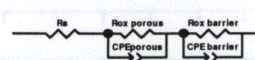
Recorded by \_\_\_\_\_

6/4/03

TITLE

From Page No. \_\_\_\_\_

Specimen Examination: No Sign of Corrosion or Pitting will Repolish Specimen  
for Further Testing - No Staining on Specimen Surfaces



Element	Freedom	Value	Error	Error %
Rs	Free(+)	3.137	0.13603	4.401
Rox porous	Free(+)	1.5789E5	73428	46.565
CPEporous-T	Free(+)	0.0003219	2.2168E-5	6.8966
CPEporous-P	Free(+)	0.84684	0.006287	1.0189
Rox barrier	Free(+)	8.2304E5	1.1396E5	13.846
CPEbarrier-T	Free(+)	0.0013368	0.00041117	30.758
CPEbarrier-P	Fixed(X)	1	N/A	N/A

Chi-Squared: 0.10495  
Weighted Sum of Squares: 16.371

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62x  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barr  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	3.137	0.13603	4.401
Rox porous	Free(+)	1.5789E5	73428	46.565
CPEporous-T	Free(+)	0.0003219	2.2168E-5	6.8966
CPEporous-P	Free(+)	0.84684	0.006287	1.0189
Rox barrier	Free(+)	8.2304E5	1.1396E5	13.846
CPEbarrier-T	Free(+)	0.0013368	0.00041117	30.758
CPEbarrier-P	Fixed(X)	1	N/A	N/A

Chi-Squared: 0.10495  
Weighted Sum of Squares: 16.371

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62x  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barr  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	2.422	0.20048	8.2775
Rox porous	Free(+)	1.435E5	1.7192E19	1.1832E16
CPEporous-T	Free(+)	0.00075272	0.00019074	25.34
CPEporous-P	Free(+)	0.76191	0.035683	4.707
Rox barrier	Free(+)	1.6519E6	1.246E8	75.428
CPEbarrier-T	Free(+)	0.00053601	8.854E-5	16.519
CPEbarrier-P	Fixed(X)	1	N/A	N/A

Chi-Squared: 0.37894  
Weighted Sum of Squares: 59.114

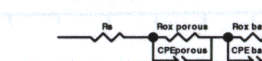
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barr  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	2.385	0.12565	5.3129
Rox porous	Free(+)	4946	18242	368.82
CPEporous-T	Free(+)	0.0010074	0.0011086	110.05
CPEporous-P	Free(+)	0.74554	0.089481	9.3209
Rox barrier	Free(+)	4.1394E5	66313	16.02
CPEbarrier-T	Free(+)	0.00043595	0.00020601	47.255
CPEbarrier-P	Free(+)	0.94985	0.077171	8.1263

Chi-Squared: 0.10567  
Weighted Sum of Squares: 16.38

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62x  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barr  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

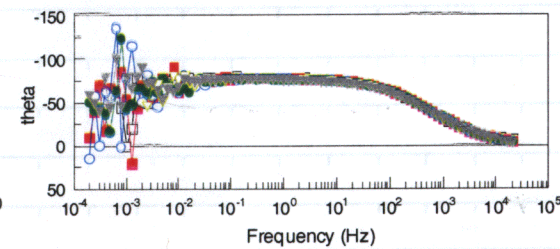
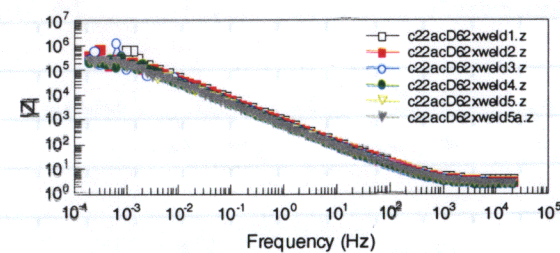
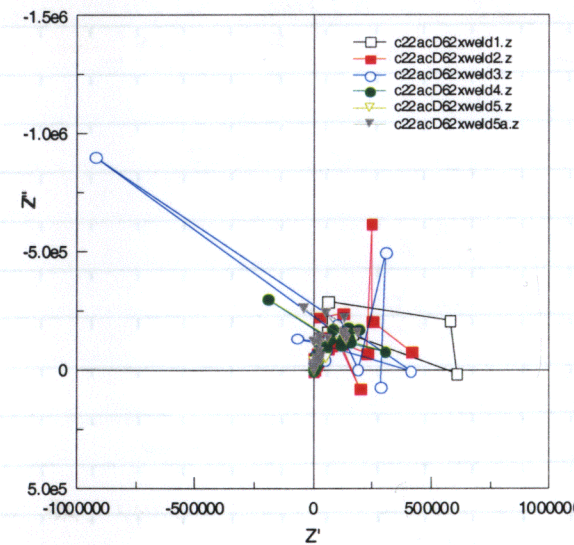


Element	Freedom	Value	Error	Error %
Rs	Free(+)	2.749	0.045179	1.6435
Rox porous	Free(+)	37940	18643	49.138
CPEporous-T	Free(+)	0.001011	0.00035579	35.192
CPEporous-P	Fixed(X)	1	N/A	N/A
Rox barrier	Free(+)	1.3295E9	4.4394E18	3.3392E11
CPEbarrier-T	Free(+)	0.00054625	9.2778E-5	16.984
CPEbarrier-P	Free(+)	0.78165	0.013682	1.7258

Chi-Squared: 0.011011  
Weighted Sum of Squares: 1.4644

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62x  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barr  
Mode: Run Batch Fitting / All Data Points (1 - 70)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

DATA FILES  
C22AC D62X WELD 1.Z  
C22AC D62X WELD 2.Z  
C22AC D62X WELD 3.Z  
C22AC D62X WELD 4.Z  
C22AC D62X WELD 5.Z  
95, 107, 120, 135, 150°C



DATA NOISY at f < 10<sup>-2</sup> Hz. Data is not useful for the determination of passive corrosion rate. High impedance of Ag/AgCl reference electrode may be the source of noise

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

7/31/03



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box -

Specimen: All Tests Run with Test Cell from NB #485 pg #35  
See set up on pg #35 Anode pg #34 Sawm NB #485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.14g KCl Lot# 005573  
+ DI water To 100ml

Temperature: 60°C

Potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter = Monogram Box w/Omega DP465 SN# 3130900  
cal 1/15/03 due 7/15/03

Calibrated Thermocouple SN# 335 cal 1/14/03 due 7/14/03

All measurements Taken with Keithley 617 SN# 537416 cal 4/2/03 due 10/2/03  
E<sub>corr</sub> = 90 mV E<sub>pt</sub> = -183 mV

Specimen Top PTFE 276 Bolts Torque To 80 In-Oz  
Useo Photo #6104 SN# 139072 cal 3/6/03 due 9/6/03

\* Note: Purged Glove Box with 99.999% N<sub>2</sub>  
then Test Solution And Into Glove Box overnight  
started 5% Hydrogen gas w/N<sub>2</sub> - Put Catalyst Into Glovebox  
fogged up some condensation build up. Box went clean  
Hydrogen level went to 2.6% O<sub>2</sub> level dropped  
to A level of 0 After About 20 min.  
Started To Heat Cell To 60°C @ 9:30 a.m.

\* Note: See pg #36 for ICP Analysis of well solution

Data PFCGB2

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>B. E. D.</i>	6/15/03

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.30  
 Filename: a:\pfcbb2  
 Pstat: VStat[] Ver 2  
 PS POTENTIOSTATIC  
 Date Run: 06-04-03  
 File Status: NORMAL  
 Time Run: 13:22:59

Cond. Time	CT	pass	s	Initial Pot.	IP	100.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	604.0E3	s
Initial Delay	ID	5	s	Stop On	SO	Pass	

Time/Pt.	TP	201.3	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					

Line Sync.	LS	yes		IR Mode	IR	none	
Rise Time	RT	high stability		Filter	FL	1 5.3Hz	
Working Elec.	WE	Solid		Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	AR	8.162	cm <sup>2</sup>	Equiv. Wt.	EW	26.04	g
Density	DE	8.690	g/ml	AUX A/D	AU	no	
Open Circuit	OC	83.00E-3	V				

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Model 352/252 Corrosion Analysis Software, v. 2.30  
 File Status: NORMAL Date Run: 06-04-03 Time Run: 13:22:59  
 CP PASS vs. R CT PASS IP 0.100 vs. R ID 5 s TP 2.013E+02 T1 6.040E+05  
 CR AUTO NP 3000 SO Pass IR NONE LS YES FL 1 5.3Hz RT HIGH STABILITY  
 REF 0.24150 SCE WRK SOLID AR 8.162E+00 RR 8.690E+00 EN 2.604E+01 DEH 8.690E+00 AU NO  
 OC 0.083  
 Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

a:\pfcbb2

Witnessed & Understood by me, Date Invented by Date

Recorded by *B. E. D.* 6/15/03

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>B. E. D.</i>	6/15/03



From Page No. \_\_\_\_\_

# Electrochemical Impedance Spectroscopy Alloy 22

Objective: same as pg #2

Specimen: C-22 weld Alloy 05990242 DOE material Plate D62X  
filler XX 204866 (See NB#505 pg#2-25)

weld Alloy  
Start wt = 12.72607g Santorians Genius SN# 12809099 cal 5/15/03 due 11/15/03  
End wt = 12.72579g

Solution 4.0 M NaCl \* Note: Used 900 ml in Autoclave  
467.56g NaCl lot# 03019x  
+ DI water to 2000ml

pH Start = 8.334 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/7/03  
pH End = 4.186 pH probe #13-620-296 SN# 2291257 P6  
pH Adjusted to 3.148 Using a 6 M HCl Solution lot# 023844

Cell Info Area = 8 cm<sup>2</sup> Density = 8.69 g/cm<sup>3</sup> Eg wt = 26.04

Impedance Analyzer = Solartron 1260  
Counter Electrode: Pt Flay  
Reference: Tungsten 1.0 mm Rod thermally Oxidized In A 2M MgCl<sub>2</sub> Solution  
Cell Info: PAR 4843 with Autoclave  
Temperature: 40°C - 125°C Fluke #2190 Digital Thermometer SN# 3133004  
cal 1/31/03 due 1/31/04  
Thermocouple SN# 334 cal 5/1/03 due 11/1/03

E<sub>corr</sub> = -99 mV  
E<sub>pot</sub> = +580 mV Keithley 617 SN# 617537418 cal 4/2/03 due 10/2/03

Solution Degassed with 99.999% N<sub>2</sub>. Degasses overnight then shut off then starts  
to bring cell up to Temp

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

6/19/03

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

8/1/03

TITLE \_\_\_\_\_

From Page No. \_\_\_\_\_

Specimen Examination: No sign of corrosion or pitting - No staining on any surface  
\* Note: will Repolish for further testing



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.21336	0.002194	1.0295
Rp	Free(+)	10699	4294.2	40.515
CPEporous-T	Free(+)	0.002223	0.00083223	37.437
CPEporous-P	Free(+)	0.86317	0.034205	3.9627
Rox barrier	Free(+)	9.9378E5	42839	4.3107
CPE barrier-T	Free(+)	0.00058113	5.0523E-5	8.6939
CPE barrier-P	Free(+)	0.89396	0.013418	1.501

Chi-Squared: 0.0017659  
Weighted Sum of Squares: 0.27387

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62weld6.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.17332	0.0017069	0.98483
Rp	Free(+)	39981	7584.8	18.971
CPEporous-T	Free(+)	0.0012563	0.00012906	10.254
CPEporous-P	Free(+)	0.84115	0.0028773	0.34207
Rox barrier	Free(+)	8.758E5	13136	1.9438
CPE barrier-T	Free(+)	0.00069326	4.0276E-5	5.8097
CPE barrier-P	Free(+)	0.919	0.0064793	0.70504

Chi-Squared: 0.0013347  
Weighted Sum of Squares: 0.20687

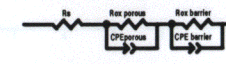
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Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.15329	0.0006564	1.34
Rp	Free(+)	47162	9078.7	19.25
CPEporous-T	Free(+)	0.0011118	0.00011585	10.42
CPEporous-P	Free(+)	0.83297	0.0029383	0.35275
Rox barrier	Free(+)	4.7532E5	13049	2.7453
CPE barrier-T	Free(+)	0.00098099	0.00067E-5	9.1812
CPE barrier-P	Free(+)	0.91333	0.0099918	1.094

Chi-Squared: 0.0019514  
Weighted Sum of Squares: 0.30247

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62weld8.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.14469	0.0033008	2.2781
Rp	Free(+)	20619	20.571	9.8004
CPEporous-T	Free(+)	0.002172	0.00014056	6.3395
CPEporous-P	Free(+)	0.77361	0.0088219	1.1533
Rox barrier	Free(+)	2.6862E5	23226	8.6496
CPE barrier-T	Free(+)	0.0013276	2.1072E-5	1.5872
CPE barrier-P	Free(+)	0.83651	0.0043002	0.51406

Chi-Squared: 0.0029922  
Weighted Sum of Squares: 0.51029

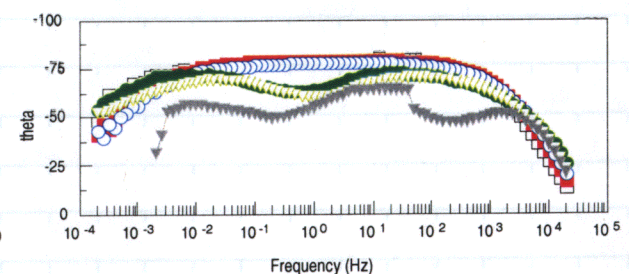
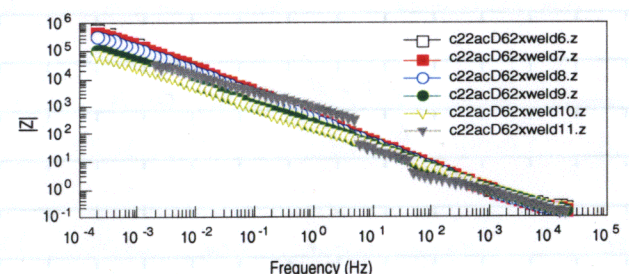
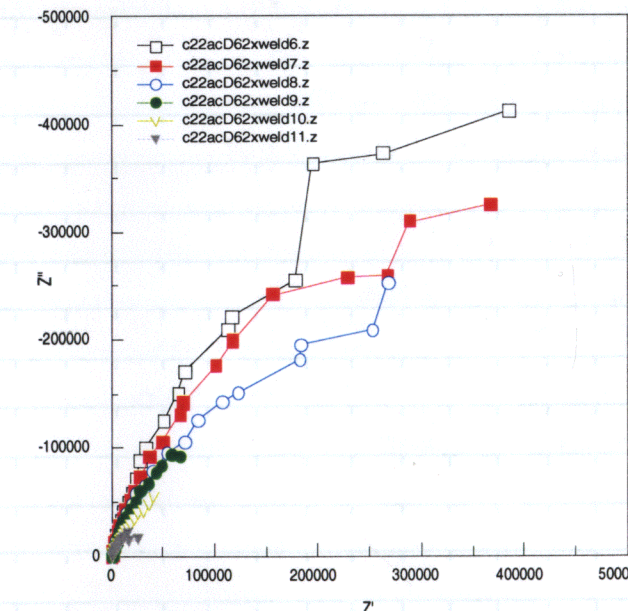
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62weld9.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.13085	0.0028032	2.1423
Rp	Free(+)	36.88	3.9627	10.192
CPEporous-T	Free(+)	0.0022262	0.00017674	7.9391
CPEporous-P	Free(+)	0.88019	0.024659	2.7701
Rox barrier	Free(+)	1.6273E5	7906.5	4.8587
CPE barrier-T	Free(+)	0.0016553	1.5157E-5	0.91566
CPE barrier-P	Free(+)	0.77094	0.0026659	0.33283

Chi-Squared: 0.0025088  
Weighted Sum of Squares: 0.38887

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62weld10.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus



Data could not be analyzed above 105°C. Noise may be due to a problem with the reference electrode.

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

8/1/03



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glovebox -

specimen: All Test Run with Test Cell from NO# 485 pg #35  
See Set up on pg #35 And pg #34 from NO# 485

Solution: 1000 ppm  $Cl^-$  As KCl  
2.14% KCl Lot #005573  
+ DI water To 1000ml

Temperature: 60°C

Potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0245107

Thermocouple Meter: Monogram Box w/ Omega DP 465 SN# 3130900  
cal 1/15/03 due 7/15/03

Calibrated Thermocouple SN# 335 cal 1/14/03 due 7/14/03

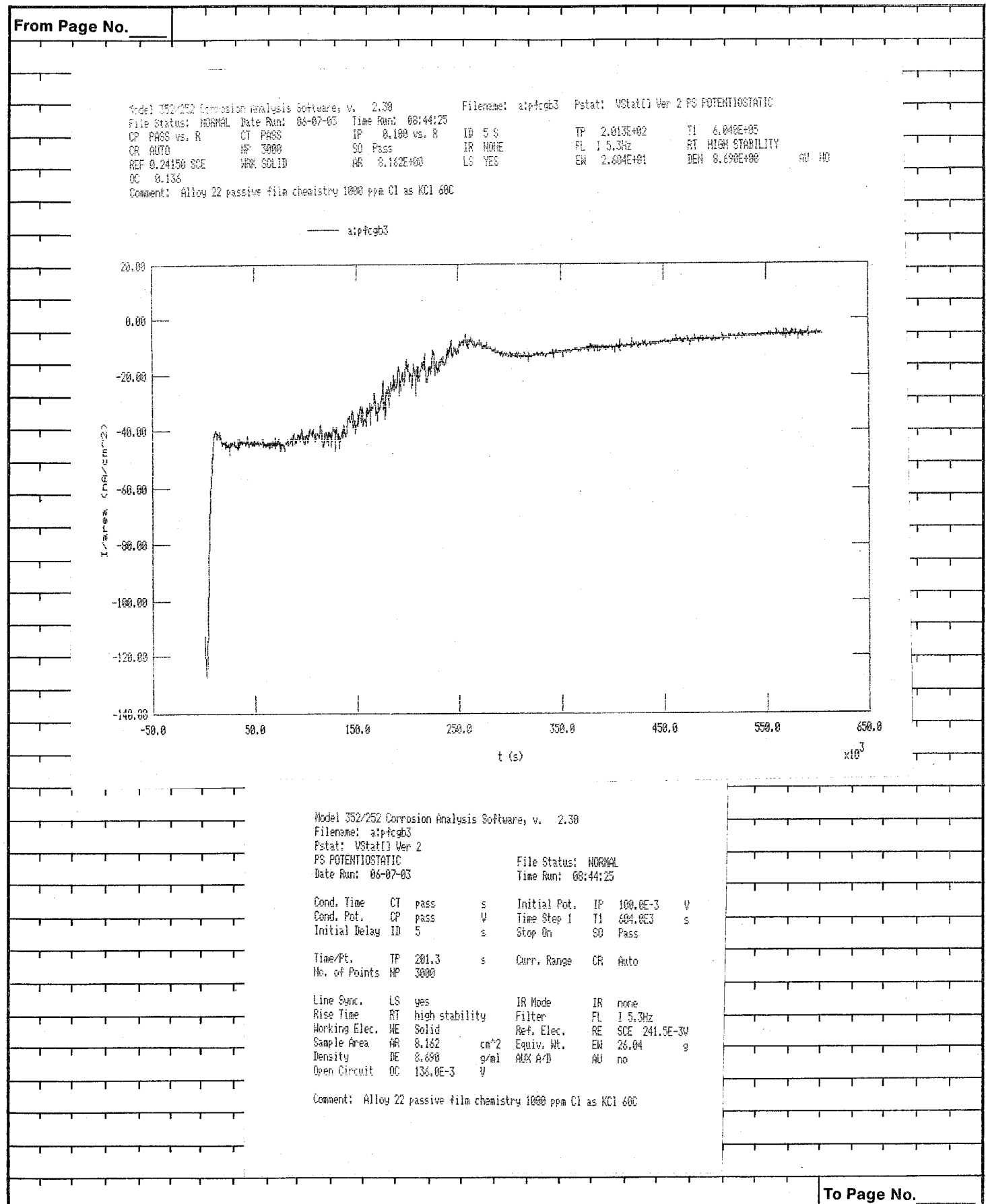
All measurement Taken with Keithley 614 cal 6/9/03 due 6/9/04  
Econn = +152 Epts -160

specimen Top PTFE 276 Bolts Torque To 80 In-Oz  
Usco Proto #6104 SN# 139072 cal 3/6/03 due 9/6/03

\* Note: Purged Glove Box with 99.999%  $N_2$   
thru Test Solution And Into Glove Box for 2 hrs.  
Started 5% Hydrogen gas w/  $N_2$  - Put catalyst Into Glovebox  
while glovebox fogged up some condensation build up Box went clean  
Used about 400 lbs of Gas mixture - started to heat cell once  
 $O_2$  level reaches zero Hydrogen level 3.6%

data PFC gb 3

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 6/23/03  
Recorded by *B. R. J.*



Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 6/30/03  
Recorded by *B. R. J.*





From Page No. \_\_\_\_\_

# Electrochemical Impedance Spectroscopy Alloy 22

Objective: same as pg #2

Specimen - C-22 uelo Alloy 059507662 DOE material plate D62X  
Filler XY 204806 (See NB#505 pg #2-25)

Uelo Alloy  
Start wt: 12.72003g Santorionic Genius SN#12809099 cal 5/15/03 due 11/15/03  
End wt: 12.72049g

Solution: 4.0M NaCl \*Note: Use 900ml in Autoclave  
467.56g NaCl #030192  
+ DI water to 2000ml

pH Start: 8.334 Fisher Accuret 950 meter SN#3340 cal 8/7/02 due 8/7/03  
pH End: 3.857 pH probe #13-620-296 SN#2291257P6  
pH Adjusted To 3.027 using a 6M HCl solution Lot #023844

Cell Info Area = 8cm<sup>2</sup> Density = 8.69g/cm<sup>3</sup> Eq wt = 26.04

Impedance Analyzer: Solartron 1260  
Counter Electrode: Pt Flag  
Reference: Tungsten 1.0mm Rod thermally oxidized in a 2M MgCl<sub>2</sub> solution  
Cell Info: PAR 4843 with Autoclave  
Temperature: 95°C - 175°C Fluke #2190 Digital thermometer SN#3133004  
cal 1/31/03 due 1/31/04  
Thermocouple SN#334 cal 5/1/03 due 10/2/03

E<sub>corr</sub> = +302 mV vs W/W<sub>02</sub> Keithley 619 SN#537418 cal 4/2/03 due 10/2/03  
E<sub>pt</sub> = +495 mV vs W/W<sub>02</sub>

Specimen Examination: No sign of corrosion.

See pg #53 for Auto Files

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

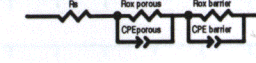
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Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.1162	0.0040507	3.47
Rporous	Free(+)	42673	12675	29.54
CPEporous-T	Free(+)	0.0008909	0.00013896	15.629
CPEporous-P	Free(+)	0.77418	0.010677	1.3791
Rbarrier	Free(+)	1.75855	2.8143E5	16.004
CPEbarrier-T	Free(+)	0.0011952	0.00026337	22.036
CPEbarrier-P	Free(+)	0.97179	0.032613	3.356

Chi-Squared: 0.0040501  
Weighted Sum of Squares: 0.62776  
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62xweld12.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus  
**95°C**



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.11497	0.0031949	2.7789
Rporous	Free(+)	55.48	10.65	19.196
CPEporous-T	Free(+)	0.0051609	0.00096524	18.703
CPEporous-P	Free(+)	1.221	0.085522	7.0043
Rbarrier	Free(+)	2.1041E6	1.9543E6	92.861
CPEbarrier-T	Free(+)	0.00098481	5.215E-6	0.52954
CPEbarrier-P	Free(+)	0.76884	0.00077655	0.10099

Chi-Squared: 0.0029427  
Weighted Sum of Squares: 0.45612  
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62xweld13.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus  
**105°C**



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.099492	0.0020773	2.0879
Rporous	Free(+)	45.39	4.2885	9.4503
CPEporous-T	Free(+)	0.0042287	0.00041376	4.1091
CPEporous-P	Free(+)	0.71756	0.0045107	0.62862
Rbarrier	Free(+)	4.6439E5	17015	3.6639
CPEbarrier-T	Free(+)	0.0012884	8.9931E-6	0.69601
CPEbarrier-P	Free(+)	0.83828	0.0019499	0.23261

Chi-Squared: 0.0014605  
Weighted Sum of Squares: 0.22638  
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62xweld14.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus  
**125°C**



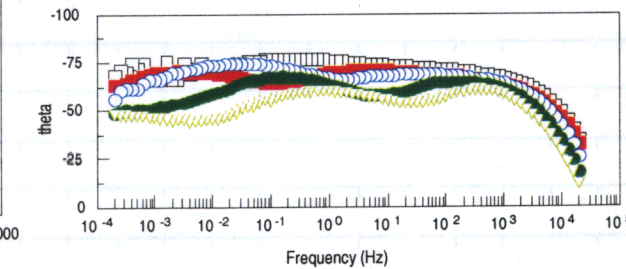
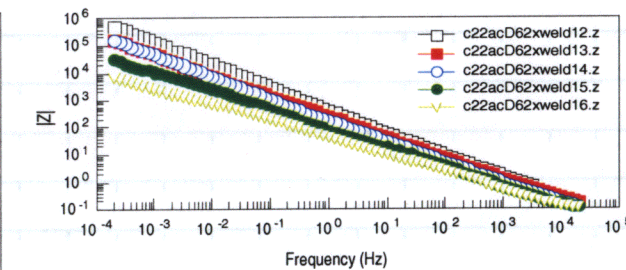
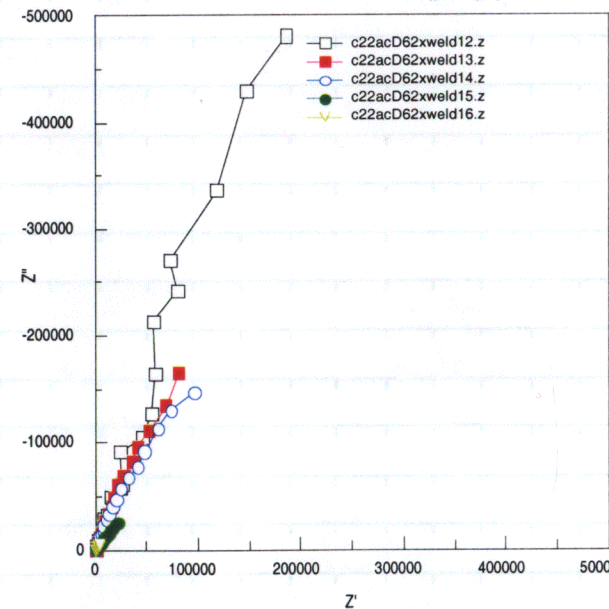
Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.096506	0.0034935	3.62
Rporous	Free(+)	3.317	0.70477	21.247
CPEporous-T	Free(+)	0.0023752	0.00059026	25.64
CPEporous-P	Free(+)	0.96506	0.055058	5.7034
Rbarrier	Free(+)	63811	3619.8	5.6728
CPEbarrier-T	Free(+)	0.0029197	2.8046E-5	0.96058
CPEbarrier-P	Free(+)	0.72848	0.003211	0.44078

Chi-Squared: 0.0058582  
Weighted Sum of Squares: 0.90802  
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62xweld15.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus  
**150°C**



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.066515	0.0064291	9.6656
Rporous	Free(+)	0.50451	0.19826	39.298
CPEporous-T	Free(+)	0.00038814	0.00052136	130.95
CPEporous-P	Free(+)	1.313	0.17815	13.416
Rbarrier	Free(+)	10349	968.73	9.3666
CPEbarrier-T	Free(+)	0.0072385	0.00011177	1.5441
CPEbarrier-P	Free(+)	0.64476	0.0043057	0.6678

Chi-Squared: 0.022099  
Weighted Sum of Squares: 3.4253  
Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62xweld16.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus  
**175°C**



To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

8/1/03



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB#485 pg#35  
See Set up on pg#34-35 from NB#485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.148g KCl lot #005573  
+ DI water to 1000ml

Temperature: 60°C

potentiostat: FG + G Versastat SN#20104

Reference: Fisher 13-620-52 SN#0249107

Thermocouple Model = Monogram Box w/Omega DP465 SN#3130900  
cal 1/15/03 due 7/15/03

Calibrated thermocouple SN#335 cal 1/14/03 due 7/14/03

All measurements taken with Keithley 614 SN#0704934 cal 6/9/03 due 6/9/04

E<sub>corr</sub> = +166mV  
E<sub>pt</sub> = +217mV

Specimen Top PTFE 276 Bolts Torque to 80 In-Oz  
Used Photo 6104 SN#139072 cal 3/6/03 due 9/6/03

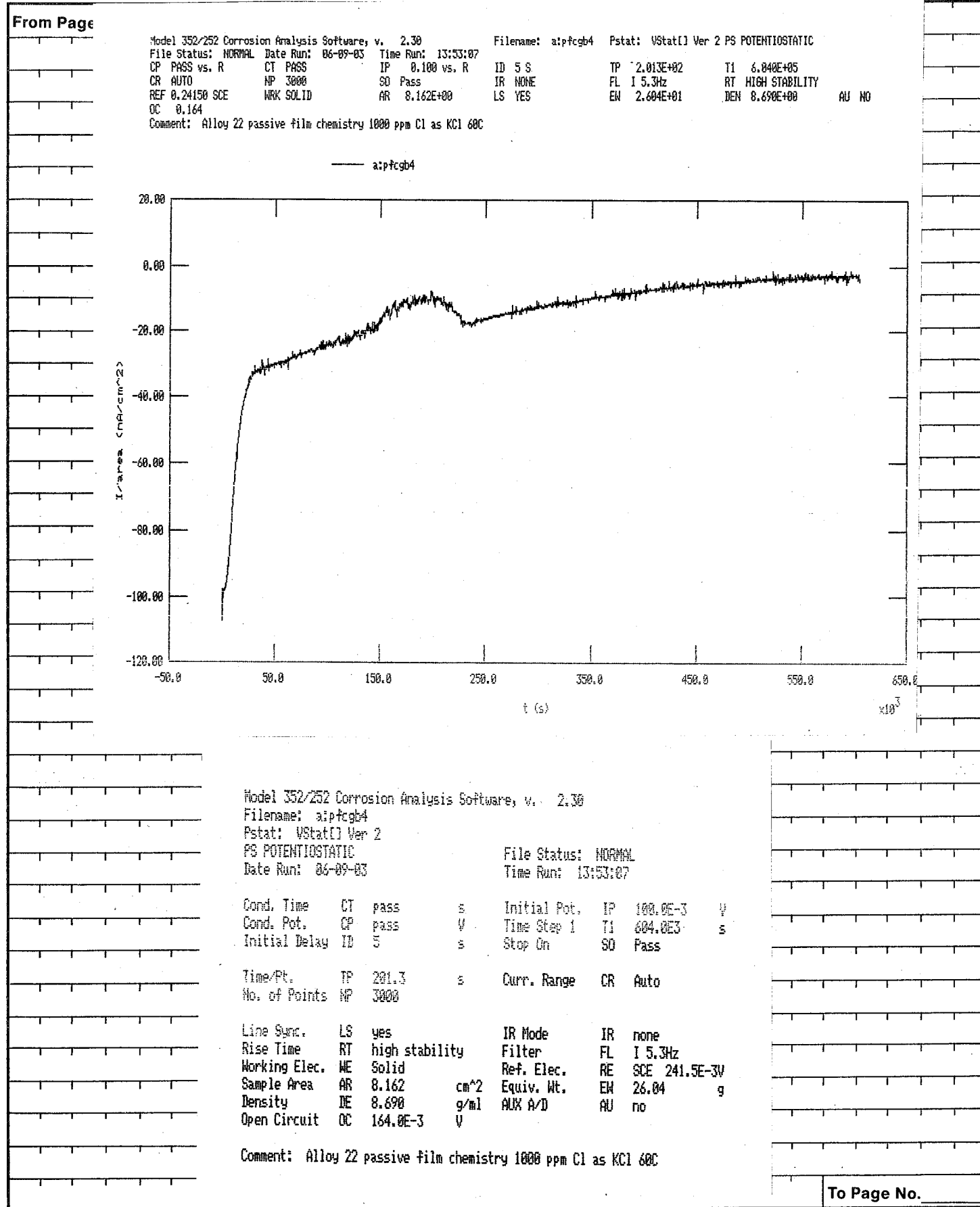
\* Note: Purged Glove Box with 99.999% N<sub>2</sub> then Test Solution  
And Glove Box for 15 hrs then started 5% Hydrogen gas w/N<sub>2</sub>  
Put catalyst in some convection bulbup on surfaces  
waiter till O<sub>2</sub> level was at 000 ppm for 30 min - Building Specimen  
Test cell in 3.8% Hydrogen zero O<sub>2</sub> Environment. Placed Solution  
In well And started Heating to 60°C @ 10:00 a.m.

\* Note See pg#51 for ICP Analysis

Data PFCGB4

To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date	
		Recorded by	7/9/03	
		<i>B. [Signature]</i>		



Witnessed & Understood by me,	Date	Invented by	Date	
		Recorded by	7/9/03	
		<i>B. [Signature]</i>		

From Page No. \_\_\_\_\_

### Electrochemical Impedance Spectroscopy Alloy 22

Objective: Same as pg #2

Specimen: C-22 Base Alloy 059902LL2 ODE material date D62X  
Sillen KY 204886 (See NB#505 pg# 2-25)

Start wt: 12.81327g Sartorius Genius SN# 12608099 cal 5/15/03 due 11/15/03

End wt: 12.81297g

Solution: 4.0 M NaCl \* Note: Used 900mls in Autoclave  
468.56g NaCl Lot# 034128  
+ DI water to 2000mls

pH Start: 7.863 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/7/03

pH End: 3.327 pH probe #13-620-296 SN# 2291287P6

pH Adjusted: used 16ml of 20% HCl solution Lot# 623844 pH = 3.012

Cell Info Area = 8 cm<sup>2</sup> Density = 8.69 g/cm<sup>3</sup> Eg wt = 26.04

Impedance Analyzer: Solartron 1260

Counter Electrode: Pt Flag

Reference: Tungsten 1.0mm Rod thermally oxidized in a 2M MgCl<sub>2</sub> solution

Cell Info: Por 4843 with Autoclave

Temperature: 25°C - 150°C Fluke #2150 Digital thermometer SN# 133004

Cal 1/31/03 due 1/31/04  
Thermocouple SN# 334 cal 5/1/03 due 9/1/03

+238 DR 8/1/03

E<sub>corr</sub> = -302 mV w/wox Keithley 614 SN# 0704936 cal 6/9/03 due 6/9/04

E<sub>pt</sub> = +495 mV vs w/wox

+562 DR 8/1/03

Solution Degraded with 99.999% with N<sub>2</sub>

Specimen Examination: No Sign of Crevice Corrosion

See pg# 57-58 for data files

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

7/22/03

From Page No. \_\_\_\_\_



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.28293	0.0093244	3.2957
Rox porous	Free(+)	22161	11282	50.909
CPEporous-T	Free(+)	0.0022374	0.00046222	20.659
CPEporous-P	Free(+)	0.94272	0.036478	3.8694
Rox barrier	Free(+)	4.1128E5	92844	22.574
CPE barrier-T	Free(+)	0.0011626	0.00015696	13.501
CPE barrier-P	Free(+)	0.85963	0.0082628	0.96232

Chi-Squared: 0.031509  
Weighted Sum of Squares: 4.8838

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base1.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

40 DR 8/1/03  
25°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.16729	0.0065628	3.923
Rox porous	Free(+)	1835	1862.2	101.46
CPEporous-T	Free(+)	0.0020955	0.00089916	42.909
CPEporous-P	Free(+)	0.89784	0.071309	7.9423
Rox barrier	Free(+)	1.3198E5	13162	9.9727
CPE barrier-T	Free(+)	0.0011073	0.00027135	24.506
CPE barrier-P	Free(+)	0.88543	0.044529	5.0291

Chi-Squared: 0.036232  
Weighted Sum of Squares: 5.6159

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base3.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

60°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.12711	0.0023858	1.877
Rox porous	Free(+)	7129	92141	12925
CPEporous-T	Free(+)	0.0024646	0.00013653	5.5303
CPEporous-P	Free(+)	0.93708	0.019673	2.0994
Rox barrier	Free(+)	2.1936E5	12422	5.6628
CPE barrier-T	Free(+)	0.0020198	3.3939E-5	1.6803
CPE barrier-P	Free(+)	0.82895	0.0044026	0.53111

Chi-Squared: 0.0057892  
Weighted Sum of Squares: 0.8423

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base5.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

95°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.11769	0.0021459	1.8233
Rox porous	Free(+)	13.16	1.2348	9.383
CPEporous-T	Free(+)	0.0023137	0.00019996	8.6424
CPEporous-P	Free(+)	0.98968	0.020946	2.1601
Rox barrier	Free(+)	1.1289E5	5815.4	5.1514
CPE barrier-T	Free(+)	0.0034719	3.4274E-5	0.98718
CPE barrier-P	Free(+)	0.77719	0.0029668	0.38173

Chi-Squared: 0.0042694  
Weighted Sum of Squares: 0.66176

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base7.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

125°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.2072	0.0053609	2.5873
Rox porous	Free(+)	9619	11334	117.83
CPEporous-T	Free(+)	0.0022116	0.0002897	108.05
CPEporous-P	Free(+)	0.87757	0.11057	12.6
Rox barrier	Free(+)	2.7834E5	58733	21.101
CPE barrier-T	Free(+)	0.00059686	0.00046915	47.063
CPE barrier-P	Free(+)	0.90036	0.073	8.1079

Chi-Squared: 0.014706  
Weighted Sum of Squares: 2.1323

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base2.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / Freq. Range (0.0006 - 20000)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

40°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.14318	0.003029	2.1155
Rox porous	Free(+)	375.3	63.32	16.925
CPEporous-T	Free(+)	0.0025116	0.00012339	4.9128
CPEporous-P	Free(+)	0.89001	0.022252	2.4752
Rox barrier	Free(+)	1.7549E5	9875.9	5.6295
CPE barrier-T	Free(+)	0.0018864	6.9882E-5	3.7335
CPE barrier-P	Free(+)	0.83862	0.0083841	0.9975

Chi-Squared: 0.0076785  
Weighted Sum of Squares: 1.1902

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base4.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

80°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.12981	0.00212	1.6332
Rox porous	Free(+)	43.76	4.0096	9.1627
CPEporous-T	Free(+)	0.0024878	0.00012364	4.9699
CPEporous-P	Free(+)	0.90691	0.013322	1.4689
Rox barrier	Free(+)	1.588E5	7682.5	4.8253
CPE barrier-T	Free(+)	0.002453	3.1536E-5	1.2856
CPE barrier-P	Free(+)	0.82542	0.0036071	0.437

Chi-Squared: 0.0044075  
Weighted Sum of Squares: 0.68316

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base6.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

105°C



Element	Freedom	Value	Error	Error %
Rs	Free(+)	0.087745	0.0043306	4.9354
Rox porous	Free(+)	0.37019	0.10155	27.432
CPEporous-T	Free(+)	0.00054201	0.00046349	85.513
CPEporous-P	Free(+)	1.309	0.12246	9.3552
Rox barrier	Free(+)	5546	468.74	8.4699
CPE barrier-T	Free(+)	0.0129	0.0001724	1.3364
CPE barrier-P	Free(+)	0.64299	0.0046698	0.62925

Chi-Squared: 0.016378  
Weighted Sum of Squares: 2.5385

Data File: D:\corrosion tests\alloy c-22\impedance\c22acD62base8.z  
Circuit Model File: D:\corrosion tests\alloy c-22\impedance\porous barrier oxide.mdl  
Mode: Run Batch Fitting / All Data Points (1 - 81)  
Maximum Iterations: 500  
Optimization Iterations: 200  
Type of Fitting: Complex  
Type of Weighting: Calc-Modulus

150°C

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

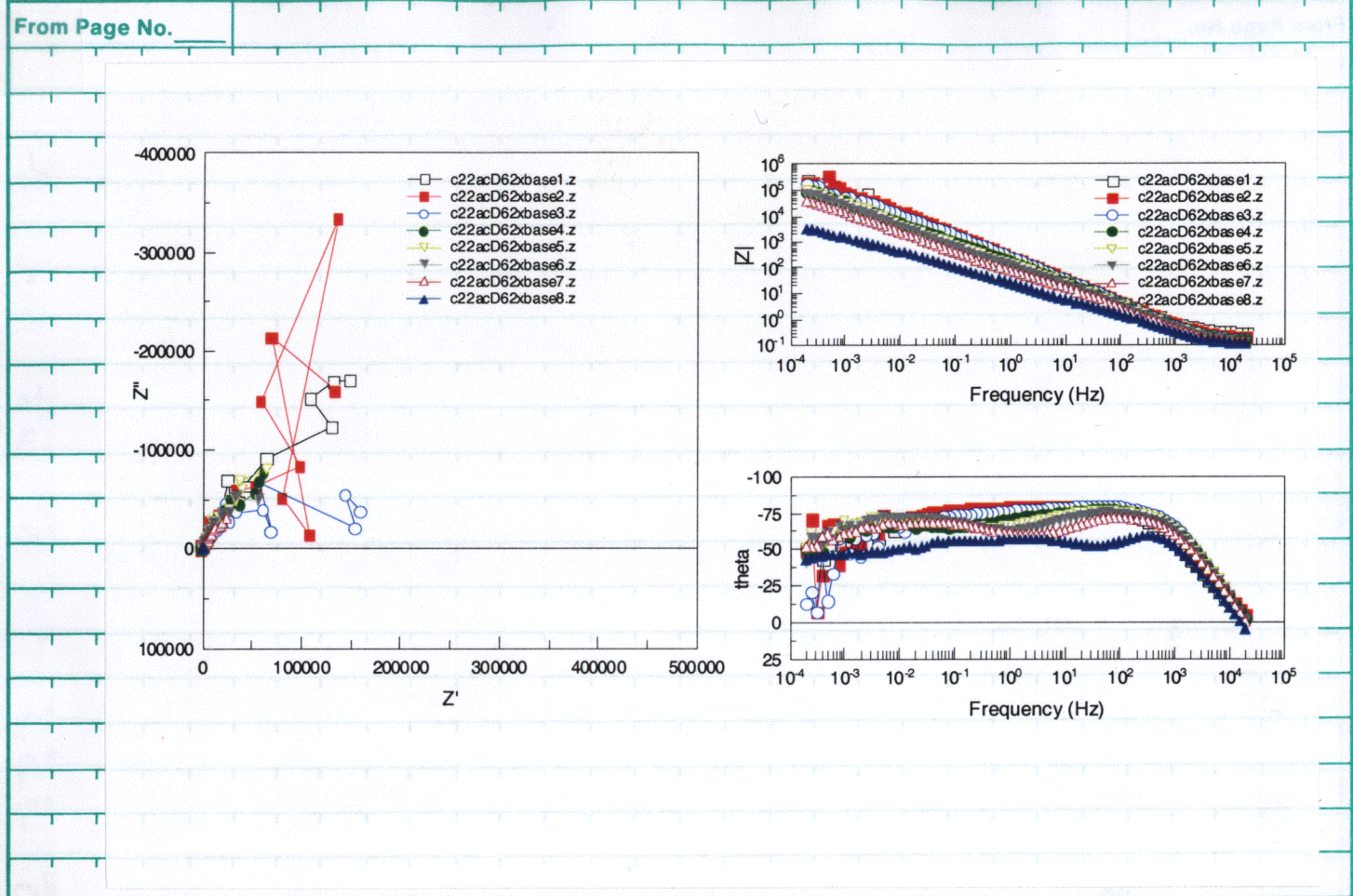
Date

Recorded by

*[Signature]*

8/1/03





Some noise at low frequencies especially at low temperatures

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_ Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by \_\_\_\_\_ Date 8/1/03

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_ Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by \_\_\_\_\_ Date 8/1/03

TITLE \_\_\_\_\_

From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB # 485 pg # 35  
See Set up on pg # 34-35 from NB # 485

Solution 1000 ppm  $Cl^-$  As KCl  
2.101g KCl lot # 005573  
+ DI water To 1000mls  
Temperature: 60°C

potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter: Monogram Box w/omega DP465 SN# 3130900  
cal 7/23/03 due 1/23/04

calibrated thermocouple: SN# 335 cal 7/21/03 due 1/21/04

All measurements taken with Keithley 614 SN# 0704936 cal 6/9/02 due 6/9/04  
Ecorr: +118 mV  
Ept: +216 mV

specimen Top PTFE 276 Bolts Torque To 80 In-oz  
Vaco Photo 6104 SN# 139072 cal 7/6/03 due 9/6/03

\* Note: Purge solution Ana glove box with 99.999%  $N_2$  for 4.5 hrs then started 5% Hydrogen gas w/ $N_2$ . Put catalyst in very mild convection build up of vapor on walls of glovebox - Purge Hydrogen for 35 min very slow flow rate After 0 ppm  $O_2$  was maintained - then assembled cell in that environment started to heat to 60°C  
\* Note Solution Purge with  $N_2$  the whole time until placed into cell

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_ Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by \_\_\_\_\_ Date 8/19/03

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_ Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by \_\_\_\_\_ Date 8/19/03





From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

specimen: All Tests Run with Test cell from NB#485 pg#35  
See set up on pg# 34-35 from NB#485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.101g KCl Lot# 005573  
+ DI water To 1000mls

Temperature: 60°C

potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter: Monogram Box w/ Omega DP465 SN# 3130900  
7/23/03 cal +1/15/03 due 9/23/04 1/23/04

Calibrated thermocouple # 335 cal 7/21/03 due 1/21/04

All measurements Taken with Keithley 614 SN# 0704934 cal 6/9/03 due 6/9/04  
E<sub>corr</sub> = +251 mV  
E<sub>pot</sub> = +293 mV

Specimen Top PTFE 276 Bolts Torque To 80 In-Oz  
Using Proto 6104 SN# 139072 cal 3/6/03 due 9/6/03

\* Note: Purge glovebox then solution with 99.999% slow bubble overnight  
then start 5% Hydrogen N<sub>2</sub> Mix gas - Also Bubbles Hydrogen Mix Gas  
then solution for 2 hrs - start to heat cell to 60°C

Data PFC6B6 To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 8/28/03  
Recorded by *Bif*

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.36  
Filename: a:pfcb6  
Pstat: UStat11 Ver 2  
PS POTENTIOSTATIC  
Date Run: 08-22-03  
File Status: NORMAL  
Time Run: 14:32:47

Cond. Time	CT	pass	s	Initial Pot.	IP	100.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	684.8E3	s
Initial Delay	ID	5	s	Stop On	SO	Pass	
Time/Pt.	TP	201.3	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					

Line Sync. LS yes IR Mode IR none  
Rise Time RT high stability Filter FL 1 S, 3Hz  
Working Elec. WE Solid Ref. Elec. RE SCE 241.9E-3V  
Sample Area SA 8.162 cm<sup>2</sup> Equiv. Wt. EW 26.04 g  
Density DE 8.390 g/ml ADV A/D AU no  
Open Circuit OC 245.0E-3 V

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Model 352/252 Corrosion Analysis Software, v. 2.36  
Filename: a:pfcb6  
Pstat: UStat11 Ver 2 PS POTENTIOSTATIC  
File Status: NORMAL Date Run: 08-22-03 Time Run: 14:32:47  
OP PASS vs. R CT PASS IP 0.100 vs. R IB 5 S TP 2.813E+02 T1 6.848E+05  
CR AUTO NP 3000 SO Pass IR NONE FL 1 S, 3Hz RT HIGH STABILITY  
REF 8.24150 SCE NEW SOLID AR 8.162E+00 LS YES EW 2.604E+01 DEN 8.690E+00 AU NO  
OC 0.245  
Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 9/9/03  
Recorded by *Bif*



From Page No. \_\_\_\_\_

### Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB #485 pg #35  
See Set up on pg # 34-35 from NB #485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.101g KCl lot #005573  
+ DI water To 1000mls

Temperature = 60°C

potentiostat = EG + G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter = Monogram Box w/Omega DP465 SN# 3130900  
cal 7/23/03 Due 1/23/04

calibrated thermocouple SN# 335 cal 7/21/03 Due 1/21/04

All measurements taken with Keithley 614 SN# 0704936 cal 6/9/03  
Due 6/9/04

E<sub>corr</sub> = +178mV

E<sub>pt</sub> = +256mV

Specimen Top PTFE 276 Bolts Tongue To 80 In-O<sub>2</sub>  
Used Photo 6104 SN# 139072 cal 3/6/03 Due 9/6/03

\*Note: Purged N<sub>2</sub> overnight then solution slow bubble. Next AM started  
H Amp N<sub>2</sub> combo gas. till O<sub>2</sub> content in glove box reach 0 ppm then  
left hydrogen level at 3.6 for 1.5hrs. Built Test Cell keep hydrogen N<sub>2</sub>  
Purging the solution that whole time. Placed solution into well started  
Heating to 60°C. Started test

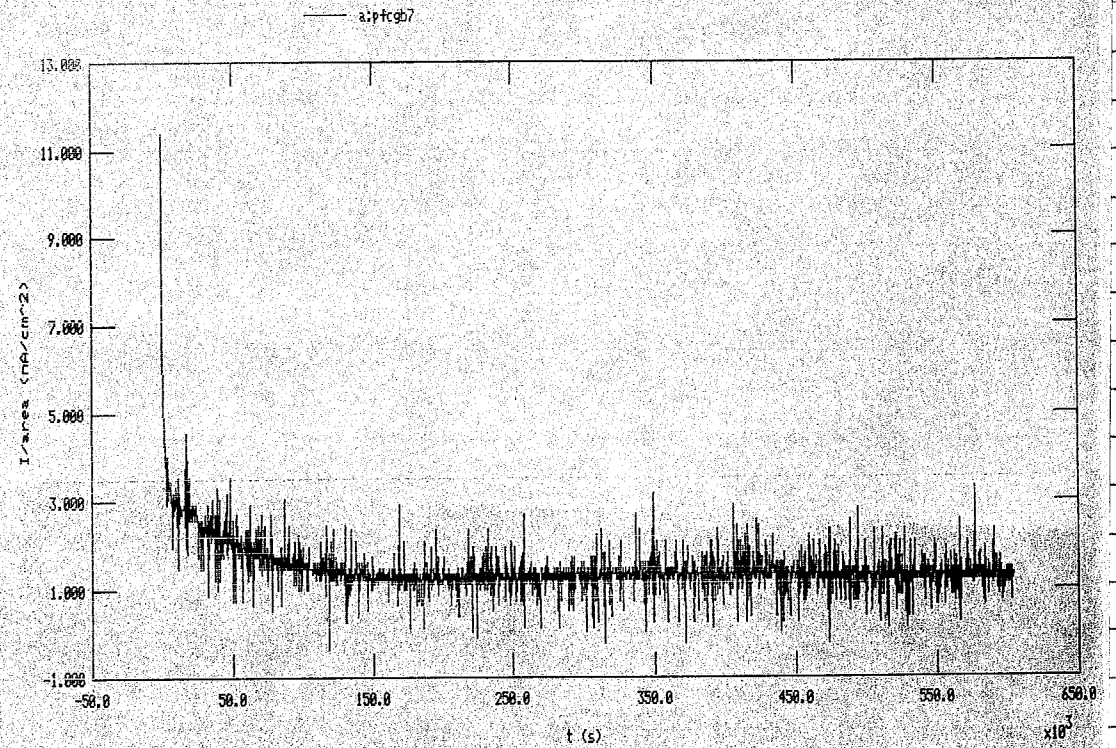
\*Note: ICP Results see pg #78

Data PFCGB7 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	9/2/03

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.38      Filename: a:\pfcgb7      Pstat: VStat11 Ver 2 PS: POTENTIOSTATIC  
 File Status: NORMAL      Date Run: 08-24-03      Time Run: 08:46:47  
 CP: PASS vs. R      CT: PASS      IP: 8.200 vs. R      ID: 5 s      TP: 2.813E+02      TI: 8.048E+05  
 CR: AUTO      NP: 3000      SQ: Pass      IR: NONE      FL: 1.5.3Hz      RT: HIGH STABILITY  
 REF: 0.24150 SCE      WK: SOLID      AR: 8.162E+00      LS: YES      EN: 2.604E+01      DEN: 8.690E+00      AU: NO  
 OC: 8.177  
 Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C



Model 352/252 Corrosion Analysis Software, v. 2.38  
 Filename: a:\pfcgb7  
 Pstat: VStat11 Ver 2  
 PS: POTENTIOSTATIC      File Status: NORMAL  
 Date Run: 08-24-03      Time Run: 08:46:47

Cond. Time	CT	pass	s	Initial Pot.	IP	280.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	884.0E3	s
Initial Delay	ID	5	s	Stop On	SO	Pass	
Time/Pt.	TP	281.3	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					
Line Sync.	LS	yes		IR Mode	IR	none	
Rise Time	RT	high stability		Filter	FL	1.5.3Hz	
Working Elec.	WE	Solid		Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	AR	8.162	cm <sup>2</sup>	Equiv. Wt.	EM	26.04	g
Density	DE	8.698	g/ml	AUX A/D	AU	no	
Open Circuit	OC	177.0E-3	V				

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	9/16/03

From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB #485 pg #35  
See Set up on pg #34-35 from NB #485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
9/19/03 2.10g KCl Lot # 005573  
+ DI water To 1000ml

Temperature 60°C

potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-670-52 SN# 0249107

Thermocouple Meter = Monogram Box w/Omega DP465 SN# 3130700  
Cal 7/23/03 Due 1/23/04

Calibrated thermocouple SN# 335 Cal 7/21/03 Due 1/21/04

All measurements taken with Keithley 614 SN# 0704936 Cal 6/9/03  
Due 6/9/04

E<sub>corr</sub> = +186 mV  
E<sub>pt</sub> = +216 mV

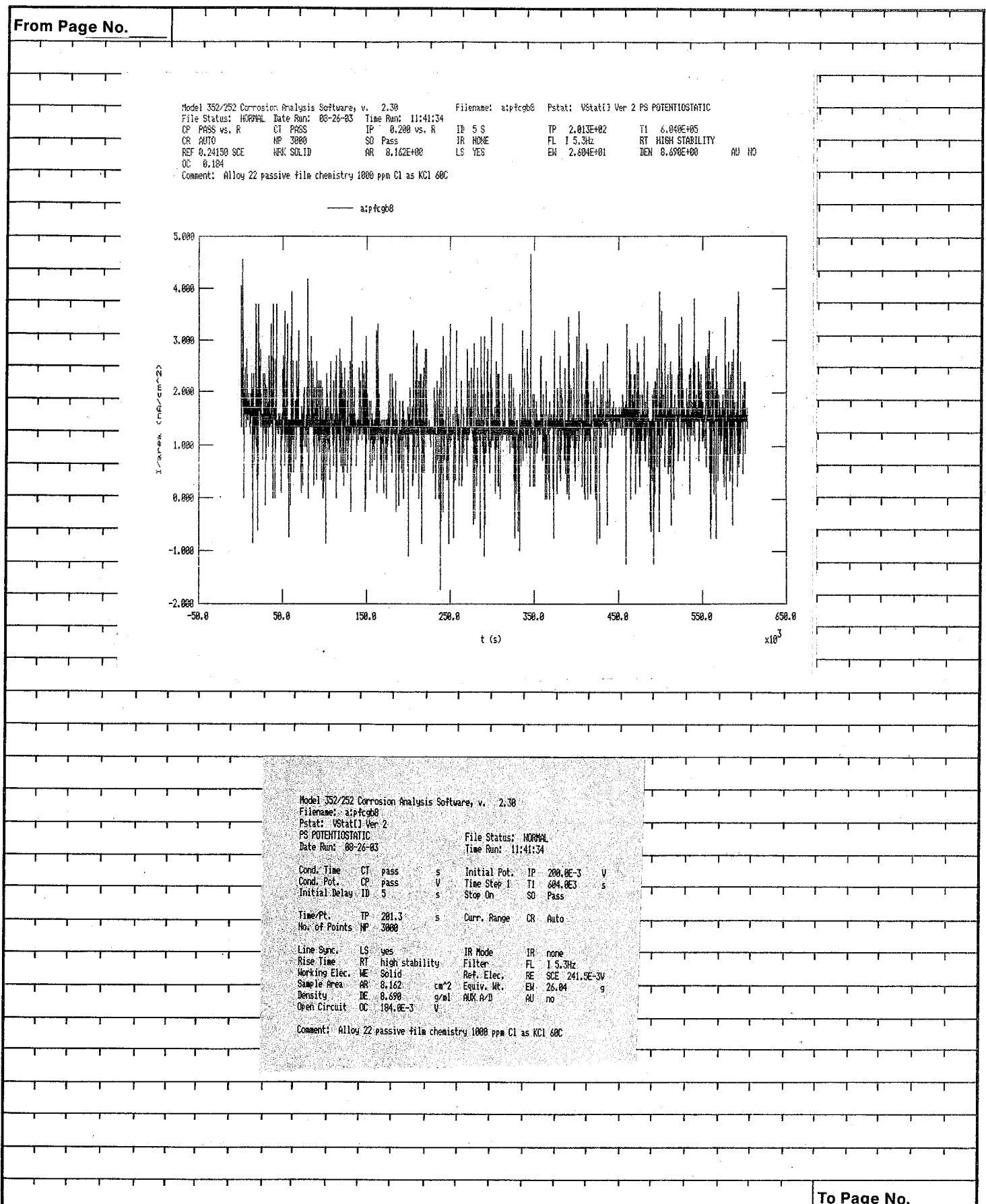
Specimen Top PTFE 276 Bolts Torque To 80 In-Oz  
Uses QDriver 2 SN# 1001200319 Cal Sept 4-03 Due 3/4/04

Note: Puyen glovebox with N<sub>2</sub> overnight through the solution. Next AM  
place in catalyst Inlet Entrance chamber. Startes Hydrogen w/N<sub>2</sub> Gas In Box And  
through the solution waited until O<sub>2</sub> level was 0 ppm then left At that  
concentration with 3.4% Hydrogen level for 30 mins. Assembled test cell waited  
Another 30 min place solution Into well started to heat to 60°C then  
started Test. Hydrogen level was 3.6% O<sub>2</sub> level 00ppm

\* Note: ICP Results See pg # 78

Data: PFC668

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	9/19/03



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	9/29/03

From Page No. _____	Electrochemical Impedance Spectroscopy Alloy 22		
Objective:	Same As pg #2		
Specimen:	Base Alloy c-22 Alloy 059902442 DOE Material Plate 0.62X filler xx 204866 (see NB #505 pg # 2-25)		
Start wt:	12.80938g	Sartorius	Genius sn# 12809099 cal 5/15/03 due 11/15/03
End wt:	12.80892g		
Solution:	4.0 m NaCl * Note! Used 900ml In Autoclave 467.68g NaCl lot # 034103 + DI water to 2000ml		
pH start:	8.396	Fisher Accumet 950 meter sn# 3340	cal 8/11/03 due 8/11/04
pH End:	3.611	pH probe ± 13.620 - 296	sn# 2291257 PL
pH Adjuster To	2.973	using A 20% HCl solution	lot # 023544
Cell Info	Area = 8cm <sup>2</sup> Density = 8.69 g/cm <sup>3</sup> E <sub>s</sub> wt = 26.04		
Impedance Analyzer:	Solartron 1260		
Counter Electrode:	PT Flag		
Reference:	Tungsten Rod 1.0 mm thermally oxidized (Base In solution)		
Cell Info:	Par 4843 with Autoclave		
Temperature:	Fluke # 2190 Digital Thermometer sn# 3133004 cal 1/31/03 due 1/31/04 Thermocouple # 334 cal 5/1/03 due 11/1/03		
E <sub>corr</sub> :	-43mv	Keithley 614	sn# 0704934 cal 6/4/03 due 6/4/04
E <sub>pt</sub> :	+486mv		
Solution Deaerated with	99.999% N <sub>2</sub> overnight		
Specimen Examination:	No sign of corrosion. No staining on specimen will Repolish for further testing		
To Page No. _____			

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	9/9/03
		<i>[Signature]</i>	

From Page No. _____	C22ACD62XBASE 9 - 17 25 - 150 °C		
Objective:	Same As pg #2		
Specimen:	Base Alloy c-22 Alloy 059902442 DOE Material Plate 0.62X filler xx 204866 (see NB #505 pg # 2-25)		
Start wt:	12.80938g	Sartorius	Genius sn# 12809099 cal 5/15/03 due 11/15/03
End wt:	12.80892g		
Solution:	4.0 m NaCl * Note! Used 900ml In Autoclave 467.68g NaCl lot # 034103 + DI water to 2000ml		
pH start:	8.396	Fisher Accumet 950 meter sn# 3340	cal 8/11/03 due 8/11/04
pH End:	3.611	pH probe ± 13.620 - 296	sn# 2291257 PL
pH Adjuster To	2.973	using A 20% HCl solution	lot # 023544
Cell Info	Area = 8cm <sup>2</sup> Density = 8.69 g/cm <sup>3</sup> E <sub>s</sub> wt = 26.04		
Impedance Analyzer:	Solartron 1260		
Counter Electrode:	PT Flag		
Reference:	Tungsten Rod 1.0 mm thermally oxidized (Base In solution)		
Cell Info:	Par 4843 with Autoclave		
Temperature:	Fluke # 2190 Digital Thermometer sn# 3133004 cal 1/31/03 due 1/31/04 Thermocouple # 334 cal 5/1/03 due 11/1/03		
E <sub>corr</sub> :	-43mv	Keithley 614	sn# 0704934 cal 6/4/03 due 6/4/04
E <sub>pt</sub> :	+486mv		
Solution Deaerated with	99.999% N <sub>2</sub> overnight		
Specimen Examination:	No sign of corrosion. No staining on specimen will Repolish for further testing		
To Page No. _____			

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	9/24/03
		<i>[Signature]</i>	

From Page No. \_\_\_\_\_

**OCP ALLOY C22**

**Objective:** To understand the effect of temperature on Ecorr.

**Alloy / Heat No. :** Alloy C-22 Cylinder / 2277-8-3175

**Specimen Preparation:** Specimen surfaces polished to 600 Grit finish using SiC paper. Specimen cleaned in acetone and rinsed in DI water.

<b>Specimen #1:</b>	<b>Specimen #2:</b>	<b>Specimen #3:</b>
Initial Wt: 12.66552g	Initial Wt: 12.29633g	Initial Wt: 12.69017g
Final Wt: 12.66652g	Final Wt: 12.31719g	Final Wt: 12.69082g
Model: Sartorius Genius	SN: 12809099	
Cal: 5/15/03	Due: 11/15/03	

**Solution:** 1000ppm NaCl + 0.240M NaHCO<sub>3</sub>  
 3.314g NaCl lot# 034103  
 40.35g NaHCO<sub>3</sub> lot# 028924  
 + DI water to 2000mls

<b>Reagents measured with</b>	<b>Model:</b> OHAUS	<b>SN:</b> 2883
	<b>Cal:</b> 7/29/03	<b>Due:</b> 1/29/03

**Initial pH:** 8.013

**Final pH:** 10.970

<b>Model:</b> Fisher Accumet 950 Meter	<b>SN:</b> 3340
<b>Cal:</b> 8/11/03	<b>Due:</b> 8/11/04
<b>pH Probe:</b> #13-620-296	<b>SN:</b> 2291257P6

**Test Temperature:** 95°C

**Measured with Hg Thermometer** SN: C94-106  
 Cal: 5/1/03 Due: 5/1/09

**Counter Electrode:** Platinum Flag

**Reference Electrode:** SN: 8205244

**Gas:** Compressed Air

<b>Specimen #1:</b>	<b>Specimen #2:</b>	<b>Specimen #3:</b>
Ecorr: -195mv <small>computer -189mv</small>	Ecorr: -201mv <small>computer -198mv</small>	Ecorr: -260mv <small>computer -255mv</small>
Ept: +227mv <small>+226mv</small>		

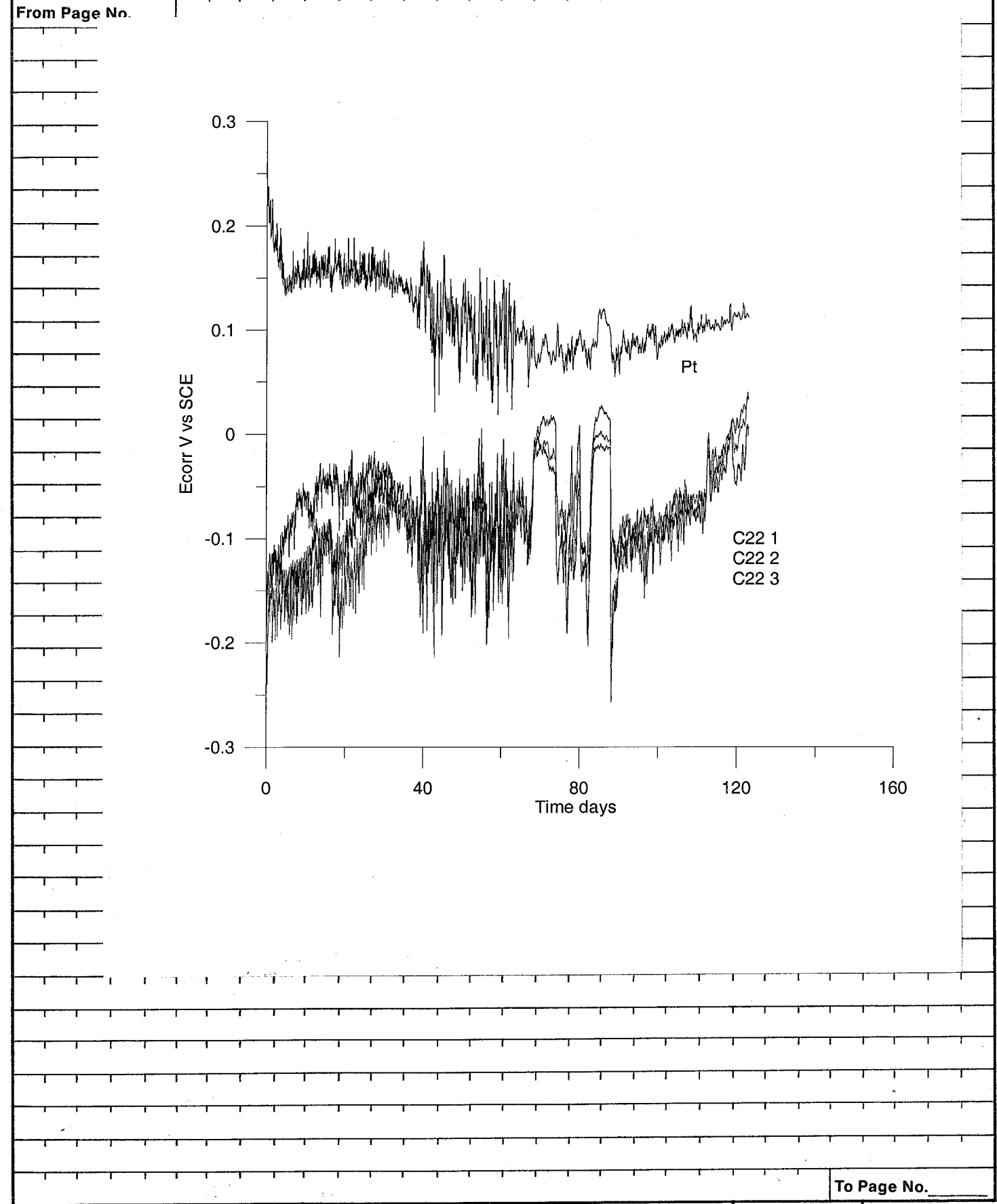
**Model:** Keithley 614 **SN:** 467374  
**Cal:** 10/28/02 **Due:** 10/28/03

No Visible Signs of Corrosion or Pitting - m/o Buildup of material  
 Along solution level - Very m/o surface staining  
*Bit of 2/12/04*

\* will polish for further testing

Data Test #15 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>Bit</i>	10/3/03



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>David D</i>	6/8/04

From Page No. \_\_\_\_\_

**OCP ALLOY C22**

**Objective:** To understand the effect of temperature on Ecorr.

**Alloy / Heat No. :** Alloy C-22 Cylinder / 2277-8-3175

**Specimen Preparation:** Specimen surfaces polished to 600 Grit finish using SiC paper. Specimen cleaned in acetone and rinsed in DI water.

<b>Specimen #4:</b>	<b>Specimen #5:</b>	<b>Specimen #6:</b>
Initial Wt: 12.69140g	Initial Wt: 12.18589g	Initial Wt: 12.38045g
Final Wt: <del>12.689g</del> 12.69131g <sup>8/10</sup> 2/11/04	Final Wt: 12.17611g	Final Wt: 12.41716g
Model: Sartorius Genius	SN: 12809099	
Cal: 5/15/03	Due: 11/15/03	

**Solution:** 1000ppm NaCl + 0.240m NaHCO<sub>3</sub> + 0.028m NaNO<sub>3</sub>  
 3.287g NaCl Lot # 034103  
 40.34g NaHCO<sub>3</sub> Lot # 028924  
 4.781g NaNO<sub>3</sub> Lot # 020809  
 + DI water to 2000ml

<b>Reagents measured with</b>	<b>Model:</b> OHAUS	<b>SN:</b> 2883
	<b>Cal:</b> 7/29/03	<b>Due:</b> 1/29/03

<b>Initial pH:</b> 7.967	
<b>Final pH:</b> 10.995	<b>Model:</b> Fisher Accumet 950 Meter <b>SN:</b> 3340
	<b>Cal:</b> 8/11/03 <b>Due:</b> 8/11/04
	<b>pH Probe:</b> #13-620-296 <b>SN:</b> 2291257P6

<b>Test Temperature:</b> 95°C	<b>Measured with Hg Thermometer</b> <b>SN:</b> H98-170
	<b>Cal:</b> 4/29/03 <b>Due:</b> 4/29/04

<b>Counter Electrode:</b> Platinum Flag	
<b>Reference Electrode:</b>	<b>SN:</b> 0642119
<b>Gas:</b> Compressed Air	

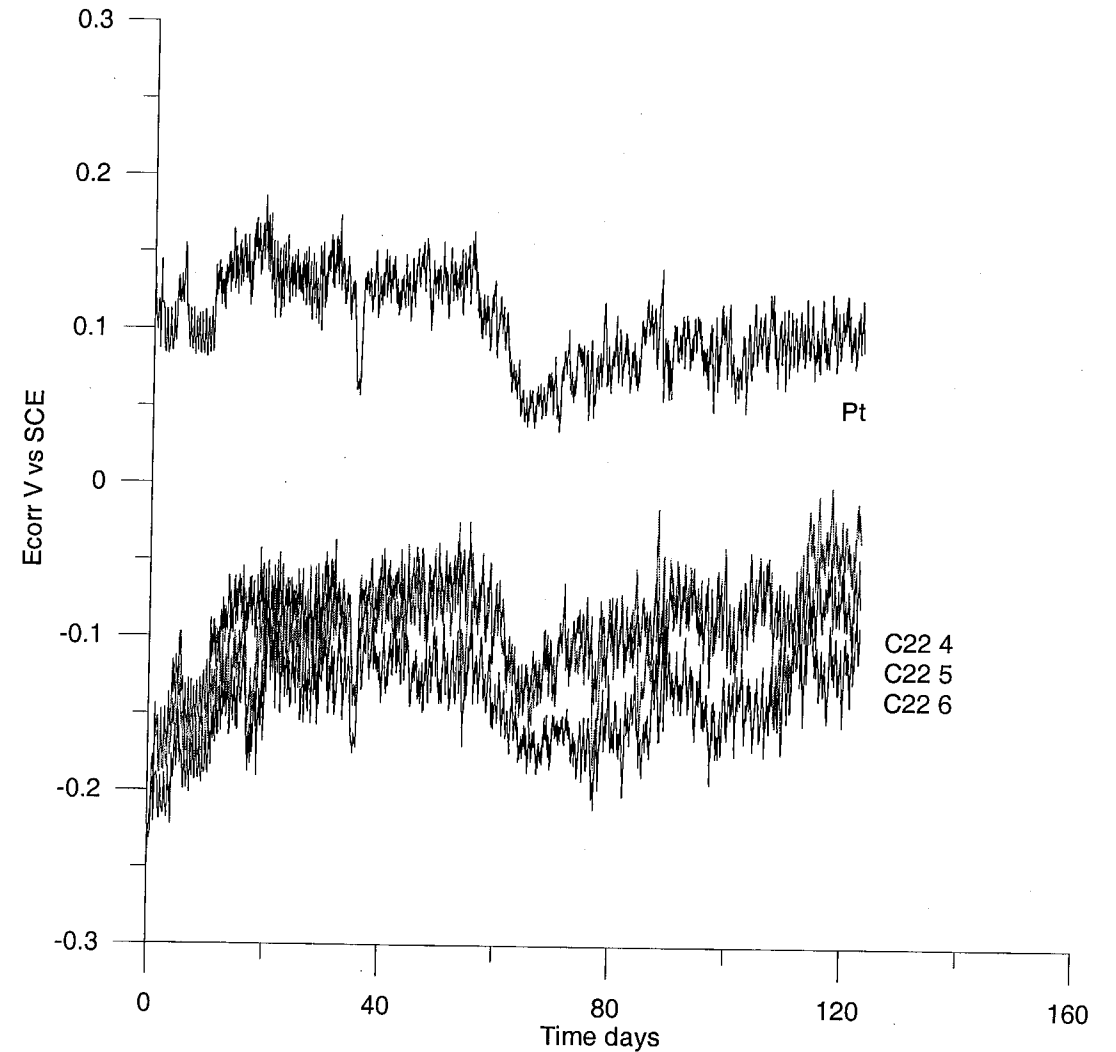
<b>Specimen #4:</b>	<b>Specimen #5:</b>	<b>Specimen #6:</b>
Ecorr: -254mV <sup>computer</sup> -265mV	Ecorr: -271mV <sup>computer</sup> -226mV	Ecorr: -267mV <sup>computer</sup> -189mV
Ept: +135 <sup>+225mV</sup>		
<b>Model:</b> Keithley 614	<b>SN:</b> 467374	
<b>Cal:</b> 10/28/02	<b>Due:</b> 10/28/03	

No Visible Signs of Corrosion or Pitting on Specimen  
 Mild Buildup of Material Along Solution Level - Very Mild Staining  
*B-D* 2/11/04  
 \* will be polished for Next Test

Data Test # 15 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>B-D</i>	10/3/03

From Page No. \_\_\_\_\_



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	5/8/04



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove box.

Specimen: All Tests Run with Test Cell from NB #485 pg #35  
See Set up on pg #35<sup>old</sup> 34-35 from NB #485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.101g KCl lot #005573  
+ DI water To 1000mls

Temperature: 60°C

Potential: EG + G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter = Mporgram Box w/omega DP465 SN# 3130900  
cal 7/23/03 Due 1/23/04

Calibrated Thermocouple SN# 335 cal 7/21/03 Due 1/21/04

All measurements Taken with Keithley 614 SN# 0704936 cal 6/9/03 Due 6/9/04

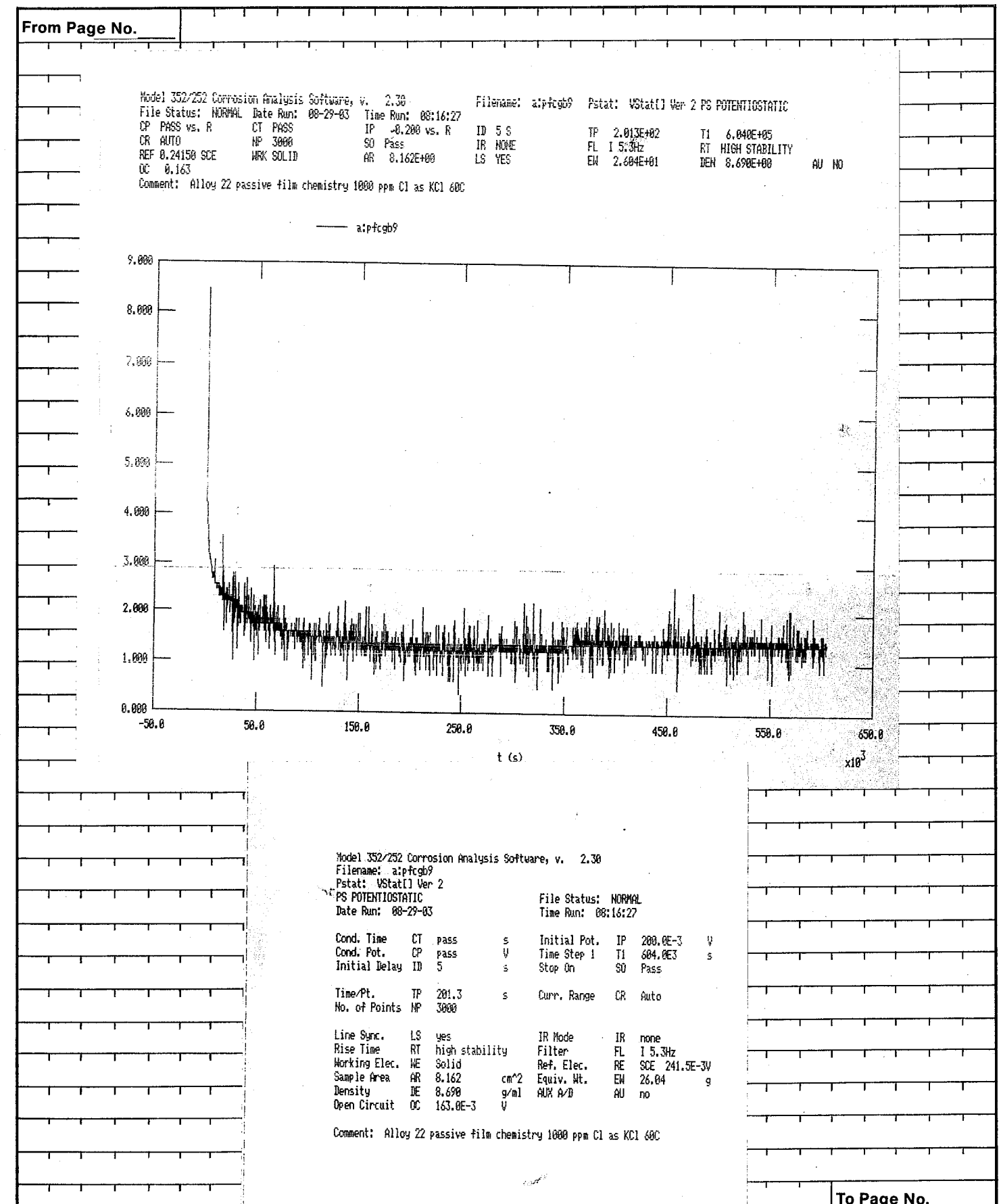
E<sub>corr</sub> = +164mv  
E<sub>pt</sub> = +220mv

Specimen Top PTFE 276 Bolts Torque To 50 In-Oz.

Note: Purge Glove box And Solution with N<sub>2</sub> for 3hrs then start  
Hyrogen N<sub>2</sub> Mix gas place catalyst Into chamber After O<sub>2</sub> started To  
Drop - Purge Mix gas Also through Test Solution - waites till O<sub>2</sub>  
level reaches 000ppm Hold for 30min @ 9.0% Hyrogen Built up  
Test Cell - place solution Into well started To Run Test @ 60°C  
O<sub>2</sub> level still 0ppm And Hyrogen 3.6%

Data PFC g b 9 To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 10/3/03  
Recorded by *[Signature]*



Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 10/13/03  
Recorded by *[Signature]*

From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB #485 pg #35  
See Set up on pg #34-35 from NB #485

Solution: 1000ppm Cl<sup>-</sup> As KCl  
2.10% KCl Lot #005573  
+ DI water To 1000mls

Temperature: 60°C

Potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter: Monogram Box w/omega DP465 SN# 3130900

Calibrated thermocouple SN# 335 Cal 7/21/03 Due 1/21/04

All measurements Taken with Keithley 614 SN# 0764936  
Cal 6/9/03 Due 6/9/04

E<sub>corr</sub> = +137mV  
E<sub>pt</sub> = -480mV

Specimen Tap PTFE 276 Bolts Torque to 80 In-Oz  
Usep Q Drive 2 SN# 1001200219 Cal 9/4/03 Due 3/4/04

Note: Purged Glove box And Solution with N<sub>2</sub> for 3 hrs then started  
Hydrogen / N<sub>2</sub> mix - placed catalyst into chamber After O<sub>2</sub> levels started  
to drop - continued to purge gas then test solution - O<sub>2</sub> level  
000ppm Hydrogen 3.9% Built cell - Placed solution into well  
started to heat to 60°C

Date PFC job 10 \_\_\_\_\_ To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.30  
Filename: a1pfcjob10  
Pstat: VStat() Ver 2  
PS POTENTIOSTATIC  
Date Run: 08-31-03  
File Status: NORMAL  
Time Run: 12:00:16

Cond. Time	CT	pass	s	Initial Pot.	IP	200.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	TI	604.0E3	s
Initial Delay	ID	5	s	Stop On	SO	Pass	

Time/Pt.	TP	201.3	s	Curr. Range	CR	Auto
No. of Points	NP	3000				

Line Sync.	LS	yes	IR Mode	IR	none	
Rise Time	RT	high stability	Filter	FL	1 5.3Hz	
Working Elec.	WE	Solid	Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	SA	0.162	cm <sup>2</sup>	Equiv. Wt.	EW	26.04
Density	DE	8.698	g/ml	AUX A/D	AU	no
Open Circuit	OC	135.0E-3	V			

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Model 352/252 Corrosion Analysis Software, v. 2.30  
File Status: NORMAL Date Run: 08-31-03 Time Run: 12:00:16  
CP PASS vs. R CT PASS IP 0.200 vs. R ID 5 S IP 2.013E+02 TI 6.040E+05  
CR AUTO NP 3000 SO Pass IR NONE FL 1 5.3Hz RT HIGH STABILITY  
REF 0.24150 SCE WRK SOLID AR 0.162E+00 LS YES EW 2.604E+01 GEN 8.698E+00 AU NO  
OC 0.135  
Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

\_\_\_\_\_ a1pfcjob10

To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010001

Sample ID: PT 7 - Container 1  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 030930-7  
Lab System ID: 235410  
Client: Division 20  
Date Received: 09/30/03  
Project No.: 20.06002.01.081  
SRR: 25032

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Cobalt	<0.167	0.167
Chromium	<0.167	0.167
Iron	<1.67	1.67
Potassium	489	3.33
Molybdenum	<0.333	0.333
Sodium	<1.67	1.67
Nickel	<0.167	0.167
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010004

Sample ID: PT 8 - Container 1  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 030930-7  
Lab System ID: 235411  
Client: Division 20  
Date Received: 09/30/03  
Project No.: 20.06002.01.081  
SRR: 25032

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Cobalt	<0.167	0.167
Chromium	<0.167	0.167
Iron	<1.67	1.67
Potassium	584	3.33
Molybdenum	<0.333	0.333
Sodium	36.8	1.67
Nickel	<0.167	0.167
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010007

Sample ID: W 7 - Container 1  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 030930-7  
Lab System ID: 235412  
Client: Division 20  
Date Received: 09/30/03  
Project No.: 20.06002.01.081  
SRR: 25032

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Cobalt	<0.167	0.167
Chromium	<0.167	0.167
Iron	<1.67	1.67
Potassium	19364	20.0
Molybdenum	<0.333	0.333
Sodium	7.33	1.67
Nickel	0.385	0.167
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010009

Sample ID: W 8 - Container 1  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 030930-7  
Lab System ID: 235413  
Client: Division 20  
Date Received: 09/30/03  
Project No.: 20.06002.01.081  
SRR: 25032

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Cobalt	<0.167	0.167
Chromium	<0.167	0.167
Iron	<1.67	1.67
Potassium	25795	20.0
Molybdenum	<0.333	0.333
Sodium	10.4	1.67
Nickel	0.175	0.167
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID: W 9  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 031025-1  
Lab System ID: 236895  
Client: Division 20  
Date Received: 10/24/03  
Project No.: 20.06002.01.081  
SRR: 25150

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	0.214	0.167
Potassium	25451	25.0
Sodium	21.6	3.33
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID: PT 10  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 031025-1  
Lab System ID: 236895  
Client: Division 20  
Date Received: 10/24/03  
Project No.: 20.06002.01.081  
SRR: 25150

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	0.183	0.167
Potassium	639	3.33
Sodium	52.5	3.33
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID: PT 9  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 031025-1  
Lab System ID: 236895  
Client: Division 20  
Date Received: 10/24/03  
Project No.: 20.06002.01.081  
SRR: 25150

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	<0.167	0.167
Potassium	1052	3.33
Sodium	50.7	3.33
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Sample ID: W 10  
Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 031025-1  
Lab System ID: 236894  
Client: Division 20  
Date Received: 10/24/03  
Project No.: 20.06002.01.081  
SRR: 25150

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.167	0.167
Nickel	<0.167	0.167
Potassium	24764	25.0
Sodium	90.6	3.33
Tungsten	<0.333	0.333

**SAMPLE LIST/CHAIN OF CUSTODY**  
Southwest Research Institute  
Chemistry and Chemical Engineering Division  
8220 Calbra Road  
San Antonio, Texas 78238-5166

Shipment Name: Dorell Dunn  
Client: Dorell Dunn - CNUWA  
Client Purchase Order/Other ID: X6090  
Site/Zone ID: X6090

Requested Turnaround:  
 2 Weeks  
 3 Weeks  
 Other

SWRI Contact: Dorell Dunn  
SWRI Project: 20.06002.01.081

Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	ICP Analysis Sheet	Remarks
W 7	10/20/03	9:40	L	L	1	✓	Intact
PT 9	10/20/03	10:20	L	L	1	✓	
W 10	10/20/03	9:30	L	L	1	✓	
PT 10	10/20/03	10:20	L	L	1	✓	

Relinquished by (Print/Signature): Brian K. Dealy / B.K. Dealy (10/20/03)  
Received by (Print/Signature): [Signature] / [Signature] (10/20/03)

Temp: 22°C  
Therm #: 026

**SAMPLE LIST/CHAIN OF CUSTODY**  
Southwest Research Institute  
Chemistry and Chemical Engineering Division  
8220 Calbra Road  
San Antonio, Texas 78238-5166

Shipment Name: Dorell Dunn  
Client: Dorell Dunn - CNUWA  
Client Purchase Order/Other ID: X6090  
Site/Zone ID: X6090

Requested Turnaround:  
 2 Weeks  
 3 Weeks  
 Other

SWRI Contact: Dorell Dunn  
SWRI Project: 20.06002.01.081

Sample ID	Sample Collection Date (mm/dd/yyyy)	Sample Collection Time	Matrix Type	Sample Type	# of Containers	ICP Analysis Sheet	Remarks
W 7	10/20/03	9:40	L	L	1	✓	Intact
PT 9	10/20/03	10:20	L	L	1	✓	
W 10	10/20/03	9:30	L	L	1	✓	
PT 10	10/20/03	10:20	L	L	1	✓	

Relinquished by (Print/Signature): Brian K. Dealy / B.K. Dealy (10/20/03)  
Received by (Print/Signature): [Signature] / [Signature] (10/20/03)

Temp: 22°C  
Therm #: 026

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by [Signature] 11/12/03

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date \_\_\_\_\_  
Recorded by [Signature] 11/12/03



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Test Run with Test Cell From NB #485 pg #35  
See Setup on pg #34-35 from NB #485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.101g KCl Lot #005573  
+ DI water To 1000mls

Temperature: 60°C

potentiostat: EG&G Versastat SN#20104

Reference: Fisher 13-620-52 SN#0249107

Thermocouple Meter = Monaghan Box w/Omega DP465 SN#3130900  
Cal 7/23/03 Due 1/23/04

Calibrated thermocouple SN# 335 Cal 7/21/03 Due 1/21/04

All measurements taken with Keithley 614 SN# 6704936 cal 6/9/03  
Due 6/9/04

Ecorr = +174 mV  
EPT = +265 mV

Specimen Top PTFE 276 Bolts Torqued To 80 In Oz  
Used Q Drive 2 SN#1001200319 Cal 9/4/03 Due 3/4/04

Note: Purges Glove Box same As All previous Test Use N<sub>2</sub>  
then Hydrogen - N<sub>2</sub> Mix till O<sub>2</sub> level reaches 0 ppm then  
Assembles Test Cell - Solution Purges with Mix Gas Also - Heats  
Cell To 60°C - started Test  
\* Note Catalyst And Desiccant Cooked In Oven @ 200°C for 2 hrs  
Prior to Test.  
\* First 2 wk Test

Test PFC6B11

To Page No. \_\_\_\_\_

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

10/31/03

From Page No. \_\_\_\_\_

Model 352/252 Corrosion Analysis Software, v. 2.30  
Filename: a:\pfc6b11  
Pstat: VStat[] Ver 2  
PS POTENTIOSTATIC  
Date Run: 09-03-03  
File Status: NORMAL  
Time Run: 15:18:39

Cond. Time	CT	pass	s	Initial Pot.	IP	200.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	1.200E6	s
Initial Delay	ID	5	s	Stop On	SO	Pass	
Time/Pt.	TP	402.7	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					

Line Sync.	LS	yes		IR Mode	IR	none	
Rise Time	RT	high stability		Filter	FL	1 5.3Hz	
Working Elec.	WE	Solid		Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	SA	8.162	cm <sup>2</sup>	EQUIV. WT.	EW	26.04	g
Density	DE	8.690	g/ml	AUX A/D	AU	no	
Open Circuit	OC	173.0E-3	V				

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

Model 352/252 Corrosion Analysis Software, v. 2.30  
File Status: NORMAL Date Run: 09-03-03 Time Run: 15:18:39  
CP PASS vs. R CT PASS IP 0.200 vs. R ID 5 s TP 4.027E+02 T1 1.200E+06  
CR AUTO NP 3000 SO Pass IR NONE FL 1 5.3Hz RT HIGH STABILITY  
REF 0.24150 SCE WRK SOLID AR 8.162E+00 LS YES EW 2.604E+01 DEN 8.690E+00 AU NO  
OC 0.173  
Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

a:\pfc6b11

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

11/14/04

From Page No. \_\_\_\_\_

### Electrochemical Impedance Spectroscopy - Alloy 22

Objective: Same As pg #2

Specimen: C-22 Alloy 059902LL2 - DOE Base Material Plate 062X  
Filler xx 204866 (Sec NB #505 pg #2-25)

Base Alloy - Cylinder

Start wt = 12.8083g Santarions Genius SN# 12809099 cal 5/15/03 due 11/15/03  
End wt = 12.8125g

Solution: 62X UZ = CaCl<sub>2</sub> = 6.522g Lot# 025259  
MgSO<sub>4</sub>·7H<sub>2</sub>O = 3.970g Lot# 034816  
KNO<sub>3</sub> = 0.721g Lot# 014648A  
NaF = 0.086g Lot# 991559  
NaHCO<sub>3</sub> = 0.020g Lot# 025478  
MgCl·6H<sub>2</sub>O = 1.507g Lot# 035212  
NaCl = 0.988g Lot# 034103  
NaNO<sub>3</sub> = 0.212g Lot# 0.20809  
+ 0.001M NaOH Lot# 033972 0.324g  
In 2000 ml DI

pH Start = 6.21 Orion EA920 cal 1/9/03 due 1/9/04  
pH End = 7.08 pH probe # 13-620-296 SN# 2251257PL6  
Cell Info Area = 8 cm<sup>2</sup> Density = 8.69 g/cm<sup>3</sup> E<sub>g</sub> wt = 26.04

Impedance Analyzer = Solartron 1260  
Counter Electrode: Pt Flay  
Reference: ~~Fuquay 1.0mm Ras thermally oxidized Fe An 1/10/03~~  
Fisher SN# 006612

E<sub>corr</sub> = -180 mV Keithley 614 SN# 0704936 cal 6/9/03 due 6/9/04  
E<sub>pot</sub> = +141 mV

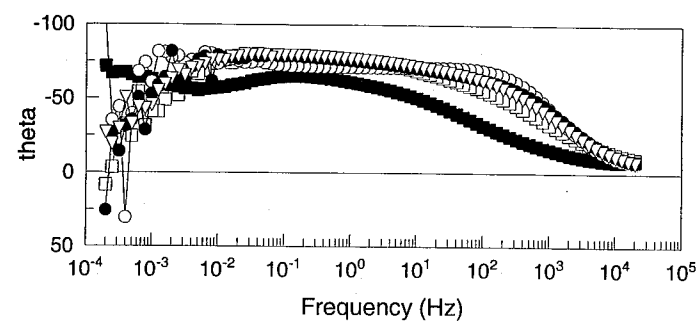
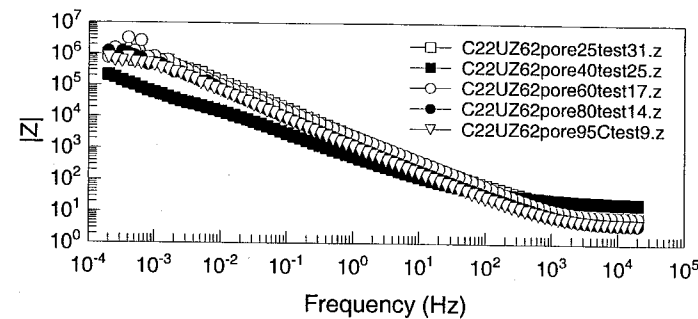
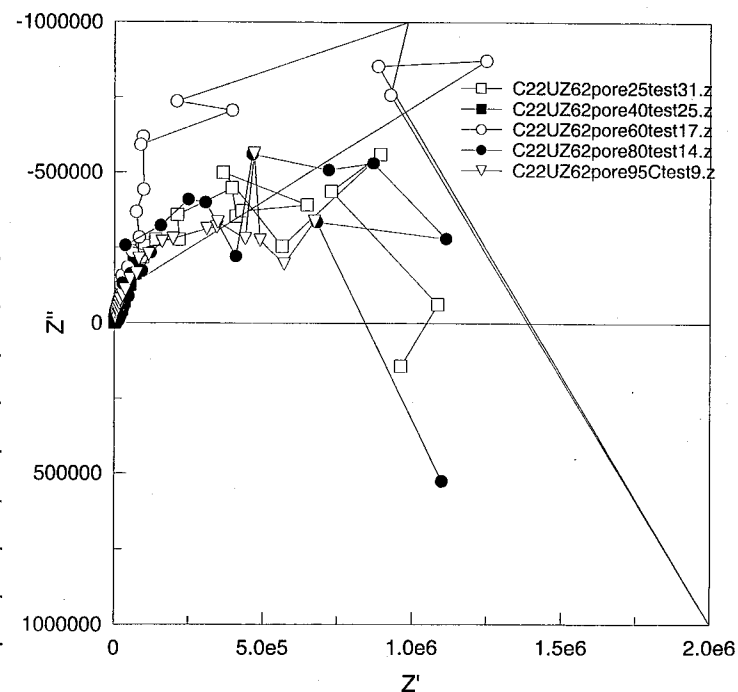
Solution Deaerates with 99.999% N<sub>2</sub>  
Temperature Range 25°C - 95°C  
Thermometer SN# C96-783 cal 10/30/03 due 4/30/04

Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 11/3/03  
Recorded by *B. D. J.*

From Page No. \_\_\_\_\_

Specimen Examination: No sign of pitting. Dull tint staining on surface. Some darker staining towards bottom of specimen but no pitting.  
\* Note: will polish specimen for further testing

\* Note: Solution was made at 31X UZ



Witnessed & Understood by me, \_\_\_\_\_ Date \_\_\_\_\_  
Invented by \_\_\_\_\_ Date 1/12/04  
Recorded by *B. D. J.*



From Page No. _____			
			To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

1/12/04



From Page No. _____			
			To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

1/12/04



From Page No. \_\_\_\_\_

Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB#485 pg #35  
See Set up on pg # 34-35 from NB#485

Solution: 1000 ppm Cl<sup>-</sup> As KCl  
2.104g KCl Lot#006242  
+ DI water To 1000mls

Temperature: 60 °C

Potentiostat: EG & G Versastat SN# 20104

Reference: Fisher 13-620-52 SN# 0249107

Thermocouple Meter = Monogram Box w/omega DP465 SN# 3130900  
cal 7/23/03 due 1/22/04

Calibrated thermocouple SN# 335 cal 7/21/03 due 1/21/04

All measurements taken with Keithley 614 SN# 467374  
cal 11/6/03 due 11/6/04

E<sub>corr</sub> = 7225 mV  
E<sub>pt</sub> = +295 mV

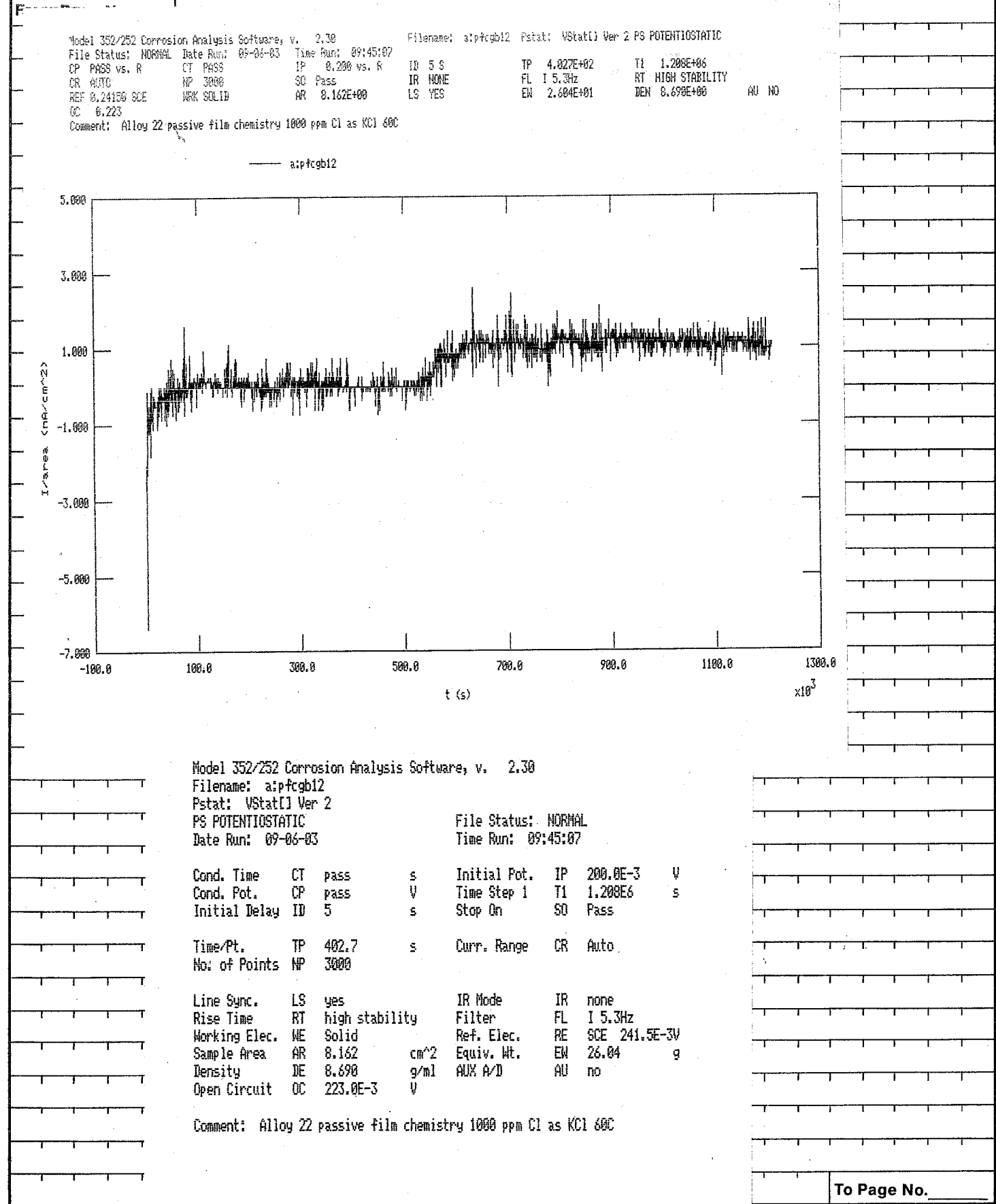
Specimen Top Torques To 80 In-Oz (276 Bolts) with  
Q Paiver SN# 1001200315 cal 9/4/03 due 3/4/03

Note: Purged glovebox with N<sub>2</sub> for 3hrs then solution;  
Started hydrogen N<sub>2</sub> mix gas - Placed in catalyst Ana Desiccant  
In Test Glovebox fan; water till O<sub>2</sub> level reached 0ppm - Assembled  
Test Cell - Solution Also purged with mix gas - started heating to  
60 °C After solution was placed into well (1800ml) - started test

\*Note: will Recharge Catalyst Ana Desiccant After this Test

Data pfcgb12 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
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Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	

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Passive Film Chemistry - Glove Box

Specimen: All Tests Run with Test Cell from NB # 485 pg # 35  
See Set up on pg # 34-35 from NB # 485

Solution: 1000ppm Cl<sup>-</sup> As KCl  
2.101g KCl Lot # 005573  
+ DI water To 1000mls

Temperature: 60°C

potentiostat: EG & G Vstatat SN# 20104

Reference: Fisher 13-62052 SN# 0249107

Thermocouple Meter Monogram Box w/omega DP465 SN# 3130260  
cal 9/23/03 Due 1/23/04

Calibrated thermocouple SN# 335 cal 7/21/03 Due 1/21/04

All Measurements Taken with Keithley 614 SN# 467374 Due 6/9/04 + 1/5/04  
cal 11/6/03 Due 1/6/04

E<sub>corr</sub> = +190 mV  
E<sub>pt</sub> = +229 mV

Specimen: Top PTFE 276 Bolts Torque To 80 In-Oz Used Ozeirun 2  
SN# 1001200319 cal 9/4/02 Due 3/4/04

Note: Recharge Catalyst And Descent Before Test.  
Set up Exactly like Test # 12 on pg # 86

Data PFCgb13 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	

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Model 352/252 Corrosion Analysis Software, v. 2.30  
File Status: NORMAL Date Run: 09-08-03 Time Run: 15:40:39  
CP PASS vs. R CT PASS IP 0.200 vs. R ID 5 S TP 4.027E+02 T1 1.200E+06  
CR AUTO HP 3000 SO Pass IR NONE FL 15.3Hz RT HIGH STABILITY  
REF 0.24150 SCE WRK SOLID AR 8.162E+00 LS YES EN 2.604E+01 DEN 8.698E+00 AU NO  
OC 0.168  
Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

a:pfcb13

Model 352/252 Corrosion Analysis Software, v. 2.30  
Filename: a:pfcb13 Pstat: Vstat11 Ver 2 PS POTENTIOSTATIC  
File Status: NORMAL  
Time Run: 15:40:39

Cond. Time	CT	pass	s	Initial Pot.	IP	200.0E-3	V
Cond. Pot.	CP	pass	V	Time Step 1	T1	1.200E6	s
Initial Delay	ID	5	s	Stop On	SO	Pass	
Time/Pt.	TP	402.7	s	Curr. Range	CR	Auto	
No. of Points	NP	3000					
Line Sync.	LS	yes		IR Mode	IR	none	
Rise Time	RT	high stability		Filter	FL	15.3Hz	
Working Elec.	WE	Solid		Ref. Elec.	RE	SCE 241.5E-3V	
Sample Area	AR	8.162	cm²	Equiv. Wt.	EW	26.04	g
Density	DE	8.698	g/ml	AUX A/D	AU	no	
Open Circuit	OC	188.0E-3	V				

Comment: Alloy 22 passive film chemistry 1000 ppm Cl as KCl 60C

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Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by <i>[Signature]</i>	



**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010011

Sample ID  
W13

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241728

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	0.238	0.167
Potassium	58775	100
Sodium	34.5	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010012

Sample ID  
W14

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241729

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	0.907	0.167
Potassium	53185	100
Sodium	32.3	1.67
Tungsten	<0.333	0.333

Project No.  
Book No.

To Page No.

Date

Invented by

Recorded by

Date

Witnessed & Understood by me,

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010006

Sample ID  
pt13

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241724

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	<0.167	0.167
Potassium	2305	20.0
Sodium	8.60	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010007

Sample ID  
pt14

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241725

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	<0.167	0.167
Potassium	1290	20.0
Sodium	4.25	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010009

Sample ID  
W11

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241726

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	<0.167	0.167
Potassium	62343	100
Sodium	29.6	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010010

Sample ID  
W12

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241727

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	0.464	0.167
Potassium	65602	100
Sodium	28.5	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010003

Sample ID  
pt11

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241722

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	<0.167	0.167
Potassium	748	20.0
Sodium	2.14	1.67
Tungsten	<0.333	0.333

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET 010004

Sample ID  
pt12

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Task Order: 040304-12  
Lab System ID: 241723

Client: Division 20  
Date Received: 03/04/04  
Project No.: 20.06002.01.081  
SRR: 25612

Analysis	Sample Result (mg/L)	Reporting Limit (mg/L)
Chromium	<0.167	0.167
Cobalt	<0.167	0.167
Iron	<1.67	1.67
Molybdenum	<0.333	0.333
Nickel	0.266	0.167
Potassium	1515	20.0
Sodium	3.91	1.67
Tungsten	<0.333	0.333

Project No.  
Book No.

TITLE

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Date

Invented by

Recorded by

Date

Witnessed & Understood by me,

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### Galvanic Corrosion / I-E Noise Test

Objective: Same As pg #1-3

Specimen: Alloy C-22 thermally Aged for 5 min @ 870°C see NB# 607, pg #52  
Heat # 2277-8-3175 Polished to A 600 grit Finish with a PTFE Crevice Washer  
Attached At 50 In-Oz Using @ Deiver SN# 1001700319 cal 9/4/03 due 2/4/04  
C-22 Alloy Plate

Crevice Specimen

Start wt: 40.98741g Santorius genius SN# 12509099 cal: 11/14/03 due: 5/14/04  
End wt: 40.90319

Solution: 5 M Cl<sup>-</sup>  
572.72g NaCl Lot# 035421  
4 mls 37% HCl Lot# 020427  
+ DI water To 2000ml

pH Start: 0.86 Orion E940 Meter SN# 2230 cal 7/15/03 due 7/15/04  
pH End 0.76

Test Temperature: 95°C Hg Thermometer SN# E98-273 cal: 12/22/03 due: 6/22/04  
Counter Electrode Pt Flag DC measurement Only  
Reference Electrode: Fisher 13-620-52 SN# 8077268  
Potentiostat: EG + G 263A SN# 66105  
Decorated with 99.999% N<sub>2</sub> for 3hrs then pulled into Vapor phase of cell  
E<sub>corr</sub>: -249 mV Keithley 617 SN# 537418 cal 10/7/03 due 4/7/04  
E<sub>pot</sub>: -199 mV

Spike Solution: 1 M FeCl<sub>3</sub> · 6H<sub>2</sub>O + HCl 37%  
27.074g FeCl<sub>3</sub> · 6H<sub>2</sub>O Lot# + 10 mls HCl Lot# 020427 37%  
+ DI water To 100ml

then Diluted 1000ml of Spike Solution with 99 mls DI water of straight FeCl<sub>3</sub> · 6H<sub>2</sub>O \* then Added 10 mls 37% HCl to 90 mls FeCl<sub>3</sub> · 6H<sub>2</sub>O Solution for New Spike Solution \*

Continued on pg #93

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Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

1/13/04

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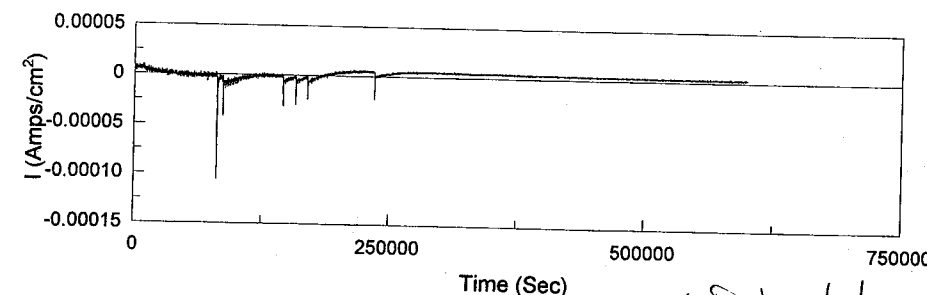
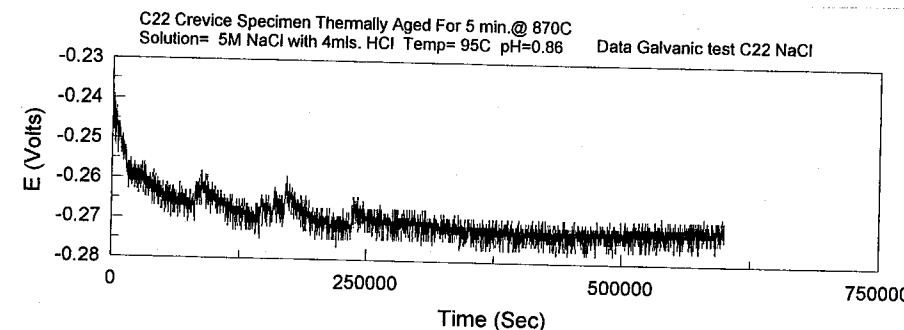
Specimen Examination: No Corrosion will Repolish Specimen And Continue Testing - Plate Specimen No Corrosion

### Spike Test Solution

Date	Time	Point #	Quantity	
1/12/04	1700 sec	18	1000µl	Diluted FeCl <sub>3</sub> Solution
1/12/04	3500 sec	36	1000µl	"
1/12/04	5900 sec	60	1000µl	"
1/13/04	56500 sec	556	1000µl	"
1/13/04	70900 sec	710	6000µl	"

\* New Spike Solution See Bottom of pg #92 - (straight FeCl<sub>3</sub> + HCl Solution)

1/13/04	80900 sec	810	500µl	1 M FeCl <sub>3</sub> + 37% HCl
1/13/04	86500 sec	866	500µl	"
1/14/04	145000 sec	1451	500µl	"
1/14/04	157500 sec	1576	800µl	"
1/14/04	169100	1692	500µl	"
1/15/04	233100	2352	500µl	"



To Page No. \_\_\_\_\_

Witnessed & Understood by me, \_\_\_\_\_

Date \_\_\_\_\_

Invented by \_\_\_\_\_

Date \_\_\_\_\_

Recorded by \_\_\_\_\_

1/12/04

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Galvanic Corrosion / I-F Noise Test

Objective: Same As pg #1-3

Specimen: Alloy C-22 thermally Aged for 5 min @ 870°C See NB# 607 pg #52  
Heat 2277-8-3175 - Polished To A 600 Grit Finish - with 2 PTFE Crevice Washers  
Attached At 50 In-Oz Using Q Deven SN# 1001200319 cal 9/4/03 Due 3/4/04  
C-22 Alloy Plate

Crevice Specimen:  
Start wt: 40.7772g Sartorius genius SN# 12909099 cal 11/14/03 Due 5/14/04  
End wt: 40.7780g

Solution: 5 M NaCl  
584.46g NaCl Lot# 035421  
+ DI water To 2000ml

pH Start = 5.89 Orion E940 Meter SN# 2230 cal 7/15/03 Due 7/15/04  
pH End = 5.91

Test Temperature: 95°C Hg thermometer SN# E98-273 cal 12/22/03 Due 6/22/04  
Counter Electrode: Pt Flag for DC measurement Only  
Reference Electrode: Fisher 13-620-52 SN# 8077268  
Potentiostat: EG&G 263A SN# 66165  
Aerated the Solution with Zero Air  
C-22 TA Plate

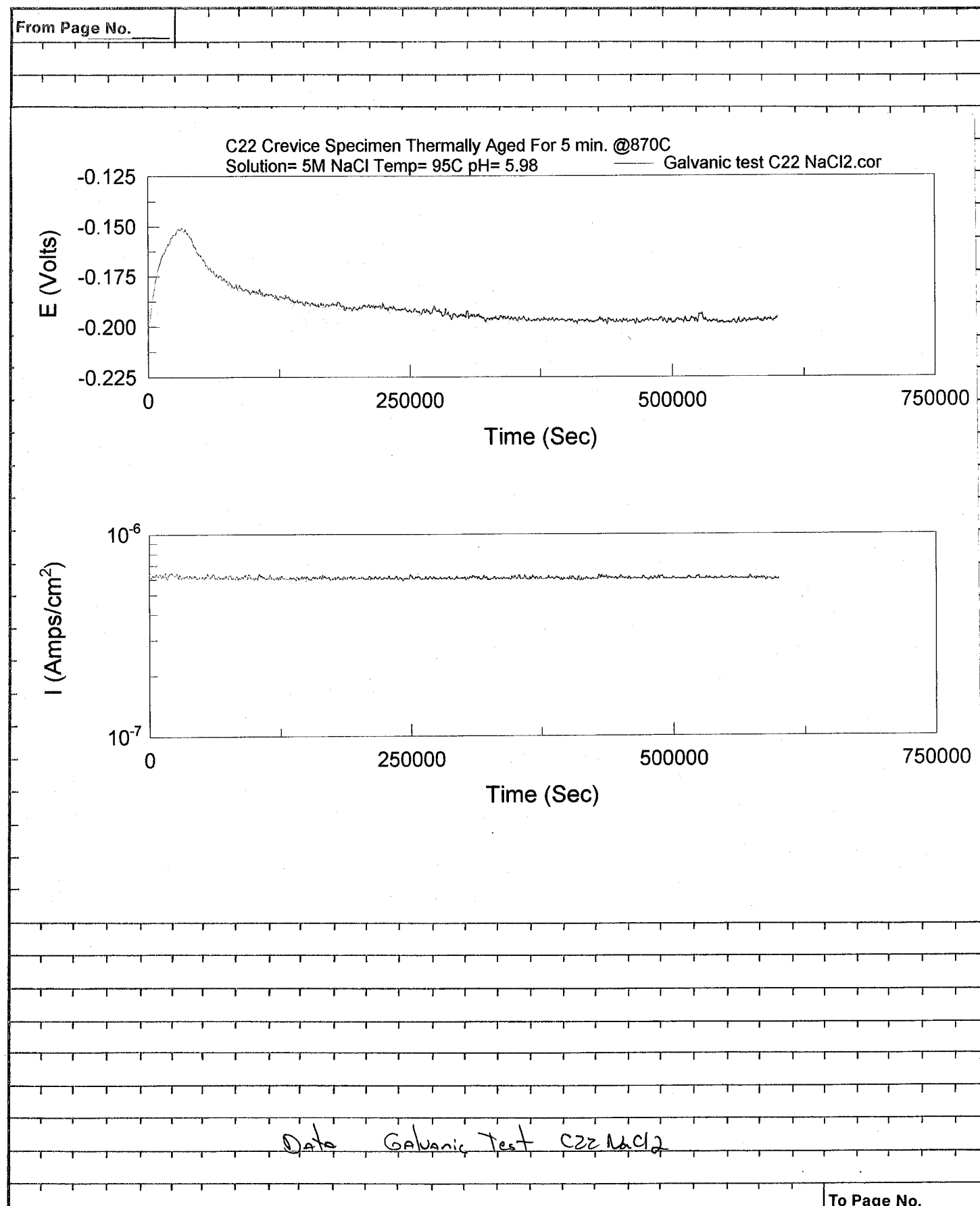
Ecorr = -155mV Eirr = -211mV Keithley 617 SN# 537418  
Ept = +182mV Ept = +183mV cal 10/7/03 Due 9/7/04

Specimen Examination: No sign of corrosion 1/24 feet of crevice washer  
Very mild surface staining

\* Note: Will Repolish for Next Test

Date Galvanic Test C22 NaCl2 To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	1/25/03



Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	1/28/03





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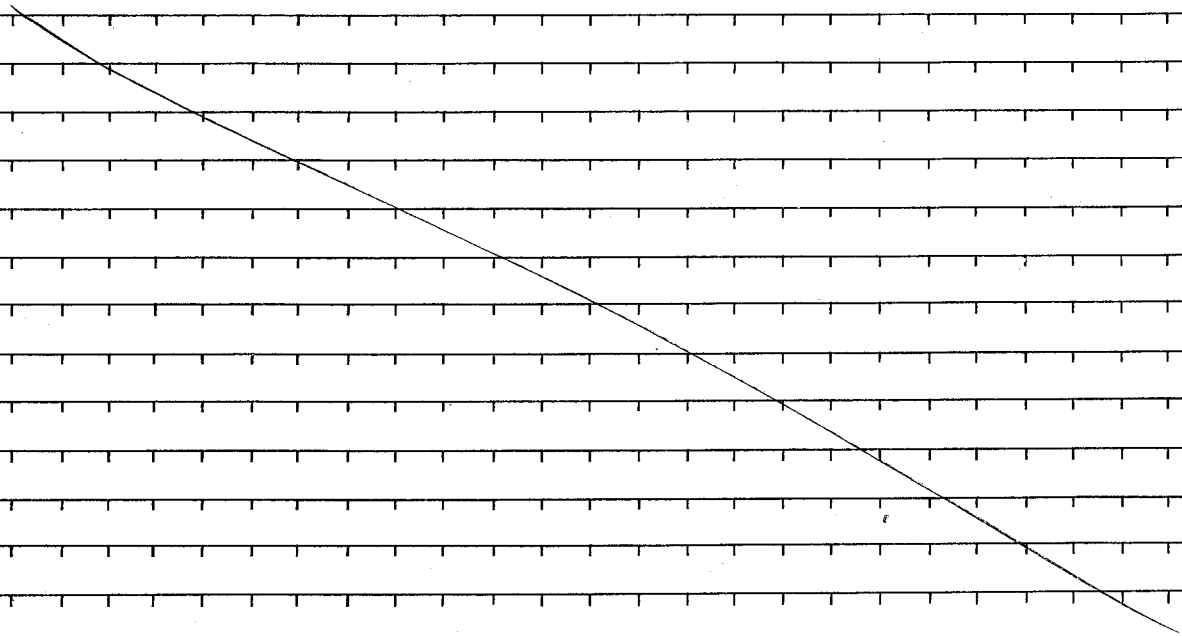
Passive Film Chemistry And EIS

Continue Testing IN

Notebook

# 637

Galvanic Testing, Continued In NB 611



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Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*[Signature]*

3/3/04

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.

*[Signature]* 9/29/04