

308

---

Q200512060002

Scientific Notebook No. 465: Effect of Trace  
Elements Tests (05/24/2001 through  
09/23/2003)

# CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES



CNWRA  
CONTROLLED  
COPY 465

Yi-Ming Pan

Ext. 6640

Beian K. Derby - B.K. Derby - OKD

Yiming Pan *Yim Pan mp*





	Page
Initial Entry for the Effect of Trace Elements Study	5
Initial Test Matrix for the Effect of Trace Elements	7
Cyclic Polarization of 825 with $PbCl_2$ , 3100 ppm $Pb^{2+}$ , 1100 ppm $Cl^-$	14
Cyclic Polarization of 825 with NaCl, 426 ppm $Cl^-$	16
Cyclic Polarization of C22 with $PbCl_2$ , 1800 ppm $Pb^{2+}$ , 700 ppm $Cl^-$	18
Cyclic Polarization of C22 with NaCl, 700 ppm $Cl^-$	22
Cyclic Polarization of C22 with $PbCl_2$ , 3100 ppm $Pb^{2+}$ , 1100 ppm $Cl^-$	26
Electrochemical test at -375 mV, C22 in $PbCl_2$ , 3400 ppm $Pb^{2+}$ , 1200 ppm $Cl^-$	30
Cyclic Polarization using Pt Flag in $PbCl_2$ , 2800 ppm $Pb^{2+}$ , 1000 ppm $Cl^-$	36
Cyclic Polarization using Pt Flag in NaCl, 1000 ppm $Cl^-$	38
Cyclic Polarization using Pt Flag in NaCl, 1000 ppm $Cl^-$	40
Cyclic Polarization of C22 1500 ppm $Pb^{2+}$ , 8600 ppm $Cl^-$ , pH=0.5, sat soln	46
Cyclic Polarization of C22 3600 ppm $Pb^{2+}$ , 1400 ppm $Cl^-$ , pH=3.0, sat soln	50
Cyclic Polarization of C22 16300 ppm $Pb^{2+}$ , 15990 ppm $Cl^-$ , pH=0.5, <sup>super</sup> sat soln	52
ISE Free $Cl^-$ Analysis	54
Chloride Concentration Measurements using Capillary Electrophoresis	57
Cyclic Polarization of C22 5475 ppm $Pb^{2+}$ , 1875 ppm $Cl^-$ , pH=2.9, <sup>sup</sup> sat soln	60
Potentiostatic test of C22 in $PbCl_2$ at 0 mV	64
Potentiostatic test of C22 in $PbCl_2$ at 50 mV	70
Potentiostatic test of C22 in $PbCl_2$ at 100 mV	72
Potentiostatic test of C22 in $PbCl_2$ at 150 mV	74
Potentiostatic test of C22 in $PbCl_2$ at 200 mV	76
Cyclic polarization of C22 in NaCl, $Cl^- = 16,000$ ppm	78
Photograph of Alloy 22 Crevice Specimens	80
SEM Micrographs of C22 & 10 specimen	81
Potentiostatic Test of C22 In $PbCl_2$ @ -100 mV - super sat. solution	82
Potentiostatic Test of C22 In $PbCl_2$ @ -100 mV - super sat. solution	84



## INDEX

Potentiostatic test of Alloy 22 in NaCl at -100 mV	86
Potentiostatic test of Alloy 22 in NaCl at -100 mV	88
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	90
SEM/EDS analysis of C22+32 specimen	93
New Solution Preparation Procedure - Super Saturated	97
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	98
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	100
Solution Preparation for ICP Analysis And Testing	102
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	104
ICP Results from Pg #102	106-107
Solution Preparation for ICP Analysis And Testing	108
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	112
Solution Preparation for ICP Analysis And Testing	114
Potentiostatic test of Alloy 22 in PbCl <sub>2</sub> at -100 mV	118
U-Bend Solution Preparation And ICP Analysis	120
U-Bend Test C22+40 Single And Double U-Bend Specimens	122
U-Bend Solution Preparation - Super Saturated PbCl <sub>2</sub>	126
U-Bend Test C22+41 Single and Double U-Bend Specimens	128
U-Bend Test C22+41 Results	129
U-Bend Test C22+41b Results	130
U-Bend Test C22+41c Results	131
U-Bend Test C22+41d Results	132
U-Bend Test C22+41e Results	133
U-Bend Test C22+41f Results	134
U-Bend Test C22+40 (Restart)	135
U-Bend Test C22+40d	136
U-Bend Test C22+40e	137

## INDEX

PbCl <sub>2</sub> Solution Preparation - Super Saturated	138
Cyclic Polarization of Thermally Aged C22 cylinder in PbCl <sub>2</sub> = C22+42	140
Cyclic Polarization of PT Flg in PbCl <sub>2</sub> = PT08 + PT09	142
Cyclic Polarization of Thermally Aged C22 cylinder in PbCl <sub>2</sub> = C22+43	144
Cyclic Polarization of C22 cylinder in PbCl <sub>2</sub> = C22+44	146
ICP Results of Solution	148
U-Bend Test C22+45 Double U-Bend Specimens	150
Photographs of Alloy specimens	155
SEM Micrographs of Alloy 22 U-Bend Specimens	156
New U-Bend Test Specimen Diagrams from D62X plate (DOE)	158
Solution Preparation for Super Saturated PbCl <sub>2</sub>	160
Cyclic Polarization of C-22 Alloy Cylinder in PbCl <sub>2</sub>	162
Cyclic Polarization of C-22 Alloy Crevice Specimen in PbCl <sub>2</sub>	164
Solution Preparation for Super Saturated PbCl <sub>2</sub> (4 Liters)	166
ICP Analysis for Solution on pg #166	168
ICP Analysis for Solution on pg #160	169
Cyclic Polarization of C-22 Alloy Crevice Specimen in PbCl <sub>2</sub>	170
Cyclic Polarization of C-22 Alloy Crevice Specimen in PbCl <sub>2</sub>	172
Cyclic Polarization of C-22 Alloy Crevice Specimen in PbCl <sub>2</sub>	174
Cyclic Polarization of C-22 Alloy Crevice Specimen in PbCl <sub>2</sub>	176
Solution Preparation for Super Saturated PbCl <sub>2</sub> (5L)	178
Cyclic Polarization of C-22 Alloy DOE weld Material	180
Cyclic Polarization of C-22 Alloy DOE weld Material	182
U-Bend Test C22+55 And C22+56 @ 0 And -100 mV DOE Specimen	184
Solution Preparation for Super Saturated PbCl <sub>2</sub> (5L)	188
U-Bend Test C22+57 And C22+58 @ 0.05 And -0.05 mV DOE Specimen	190
Solution ICP Analysis (Recheck)	194



## INDEX

U Bend Test C22+59 And C22+60 @ -100mV x2 DOE material

thermally Aged And Solution Annealed. 196

Potentiostatic Test of Alloy C22 U-Bend Specimens at -100mV 197

## Initial Scientific Notebook Entry for the Effect of Trace Elements Study

**Title:** Effect of trace elements tests

**Tests Performed by:** Yi-Ming Pan, Darrell Dunn, Brian Derby, Samuel Ireland.

**Objectives:** Determine the effect of trace elements on localized corrosion and stress corrosion cracking of waste package materials

**Equipments:** Polarization test cell, Solartron SI 1287 potentiostat, Keithley 617 electrometer, CorrWare V2.2 data acquisition software.

**Materials:** Alloy 22 Heat #2277-8-3175 and Alloy 825 Heat #HH4371F<sup>G</sup><sub>SI</sub>  
#P4461

**Measurement Parameters:** Current, potential, temperature.

**Required Level of Accuracy:** Current:  $\pm 100$  pA, potential:  $\pm 100$   $\mu$ V, temperature:  $\pm 2.0^{\circ}\text{C}$ .

**Uncertainty and Sources of Error:** The concentration of free lead ion and chloride complexes may not be experimentally determined. The solution chemistry will be calculated using OLI Systems Environmental Simulation Program, Version 6.2e.

*Yi-Ming Pan*  
5/24/01

*B. Ireland* 5/24/01



Specimen Dimensions for following Testing

Drawing no: 20.01402.571.006

S. Brossia

8/2/2000

N. Sridhar

8/2/00

S. Brossia

8/2/00

N. Sridhar

8/2/00

S. Brossia

8/2/00

N. Sridhar

8/2/00

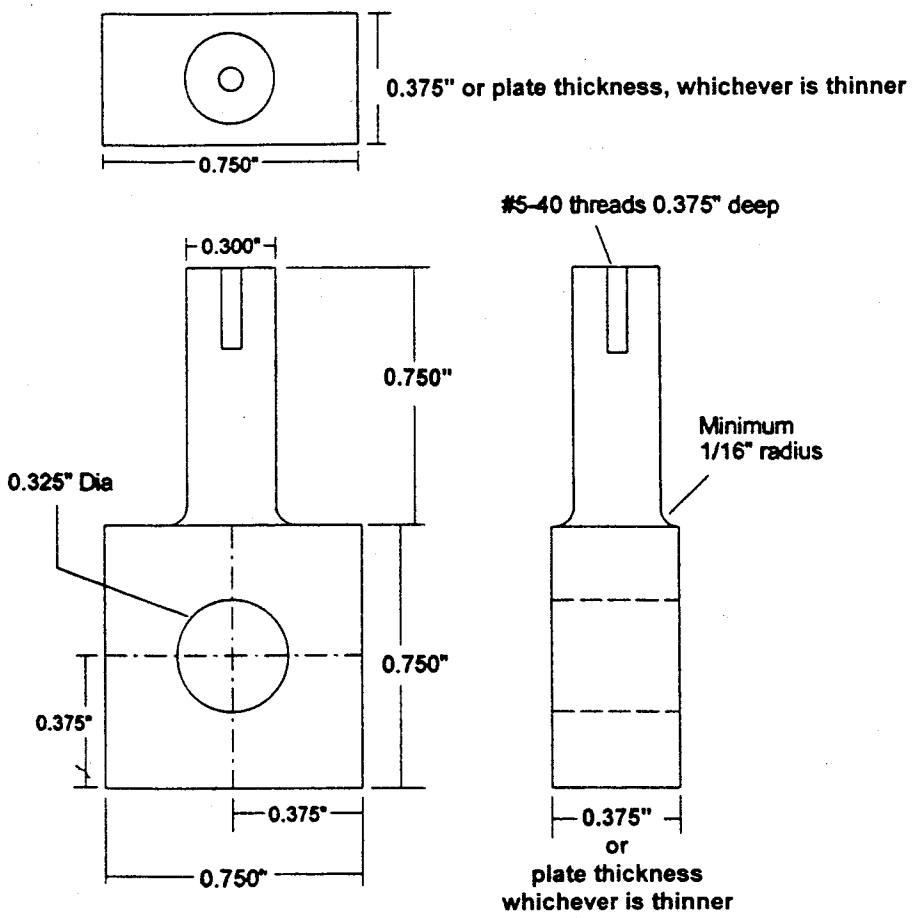
S. Brossia

8/2/00

N. Sridhar

8/2/00

CREVICE REPASSIVATION SPECIMEN  
All Dimensions +/- 0.003"



All C-22 Material Ht# 2277-8-3175

C-22 U-Bend Specimens 2277-1-3133

B. K. Duf 05/24/01

Initial Test Matrix for the Effect of Trace Elements  
(Polarization Curve Measurements)

Test #	Solution	Material	Temperature
1	A - 5000 ppm Pb as PbCl <sub>2</sub>	Alloy 22	95 °C
2	A - 5000 ppm Pb as PbCl <sub>2</sub>	Alloy 825	95 °C
3	B - 0.0349 molal Cl <sup>-</sup> as NaCl	Alloy 22	95 °C
4	B - 0.0349 molal Cl <sup>-</sup> as NaCl	Alloy 825	95 °C

Note: Solution preparation  
Solution A - 6.7578 g PbCl<sub>2</sub> (or 0.0243 mole) in 1 liter DI water  
Solution B - 2.0396 g NaCl (or 0.0349 mole) + 0.0052 g HCl (0.000142 mole) in 1 liter DI water  
Measure pH for both solutions (pH = 3.94 from simulation).

Solution A = 13.5156 g PbCl<sub>2</sub> In 2 Liter DI water  
Solution B = 4.0792 g NaCl + .0104 g HCl In 2 Liter DI water

1) upon preparing solution A we found PbCl<sub>2</sub> to be insoluble with 13.516g PbCl<sub>2</sub> in 2 L of DI water. Lot #L13K01  
OHAUS Precision Standard SN# 2883 cal 3/2/01  
2) Heated solution A for 72 hours in cell at 60°C - deaerated with 99.999% N<sub>2</sub>. Thermometer SN# 115814 cal JAN 29/01. Solution continued to be insoluble.  
Sara Ireland 5/24/01

B. K. Duf 05/24/01



extracted insoluble portion of  $PbCl_2$  and sent solution to be Analyzed  
by ICP *Sent 5/31/01*

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 05/31/01

Matrix: Liquid

Project No.: 20.01402.571

Work Order: 20293

Sample ID	Lab System ID	Lead Result (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.508
True Value	----	0.500
Recovery	----	102%
PBCL2 LOT #L13K01	162173	3098
Duplicate result	162173	3167
RPD	162173	2.20%
Spike result	162173	3618
Spike added	162173	500
Recovery	162173	104%

Reporting Limit:

0.005 mg/L

*6/14/01*

*Sent 6/14/01*

**FORM FOR REQUESTING WORK FROM OTHER DIVISIONS**

**A. TO BE COMPLETED BY DIVISION 20 PERSONNEL**

Requester: SAMUEL FREEMAN Request Date: 05/31/01  
Project No.: 20.01402.571 Phone No.: 254.571.2513  
Description of Work Requested: ICP Analysis of Pb in solution (intercomparison)

☐ 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**

PbCl<sub>2</sub> solution

Lot # L13K01

**B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>**

☐ Optical Microscopy ☐ SEM ☐ Hardness ☐ Profilometer ☐ Auger ☒ Other

Person Assigned: MARK MAYNARD Signature: Mark W. Maynard  
Division: 01 Date: 6/16/01

Make, Model & Serial No. of Equipment Used (attach list if necessary):  
THERMO JARRELL ASH TRACE ANALYZER model #13685000

Software Used (if any): WTR00WS NT

Standards Used (if any): ATTACHED

Photographic Negative Numbers (if Applicable):

<sup>1</sup> 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.

*Mark W. Maynard 5-31-01 @ 13:30*  
*6/14/01*

*Sent 6/14/01*

3) Tried to make solution A using 13.516g  $PbCl_2$  with 95°C DI water 500ml  
added 1400ml DI water at 60°C. Lot # L13K01  
thermometer SN# H98-170 cal 5/3/01  
filled solution to 2000ml. heated up to 85°C and held there for 2 hours  
while deaerating with 99.999%  $N_2$   
- solution continued to be insoluble.

$PbCl_2$  measurements taken with OHAUS Precision Standard SN# 2883 cal 3/2/01

4) heated 2000ml of DI water to 95°C and held there for 24 hours  
while deaerating with 99.999%  $N_2$

thermometer SN# 115814 cal 29 JAN 01

- added 13.516g  $PbCl_2$ , continued deaerating, maintained temperature at 95°C  
for 5 hours. Lot # L13K01, OHAUS Precision Standard SN# 2883 cal 3/2/01  
- solution continued to be insoluble

*Sent 6/5/01*



- filtered solution and sent to be analyzed by ECP

will log results when return

Samuel  
6/6/01

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 20325  
Client: Division 20  
Date Received: 06/06/01  
Project No.: 20.01402.571

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.517
True Value	----	0.500
Recovery	----	103.4%
PBCL2 LOT# L13K01	162405	3127
Duplicate result	162405	3159
RPD	162405	1.02%
Spike result	162405	3628
Spike added	162405	500
Recovery	162405	100.2%

Reporting Limit: 0.005 mg/L

6/28/01  
Samuel

SwRI ENV CHEM ID:5225938 JUL 11 '01 15:06 NO.012 P.03

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 20325  
Client: Division 20  
Date Received: 06/06/01  
Project No.: 20.01402.571

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	201
True Value	----	200
Recovery	----	100.5%
PBCL2 LOT# L13K01	162405	1089
Duplicate result	162405	1100
RPD	162405	1.01%
Spike result	162405	1393
Spike added	162405	400
Recovery	162405	76.0%

Reporting Limit: 0.1 mg/L

7/16/01  
Samuel

7/16/01  
Samuel

Solution MV TEST - Free Cl Concentration Analysis

Objective: measure Cl concentration using NaCl standard solutions

Meter: Accumet 950 Fisher Scientific SN# 3340 cal 7/20/2000

Electrode: Microelectrodes Inc MI-200 SN# 42992

Reference: Fisher Lot# 13-620-52 SN# 8238325

Stock solution - 7.100g NaCl and 500ml DI H<sub>2</sub>O 8613.79 ppm Cl<sup>-</sup>

Consecutive 1/2 ratio dilutions

Soln	ppm Cl <sup>-</sup>	mV Run 1	mV Run 2
1	8613.79	20.5	35.9
2	4306.90	36.3	43.9
3	2153.45	57.8	54.5
4	1076.72	69.6	65.9
5	538.36	83.8	77.5
6	269.18	99.4	89.1
7	134.59	116.5	100.3
8	67.30	132.7	113.8

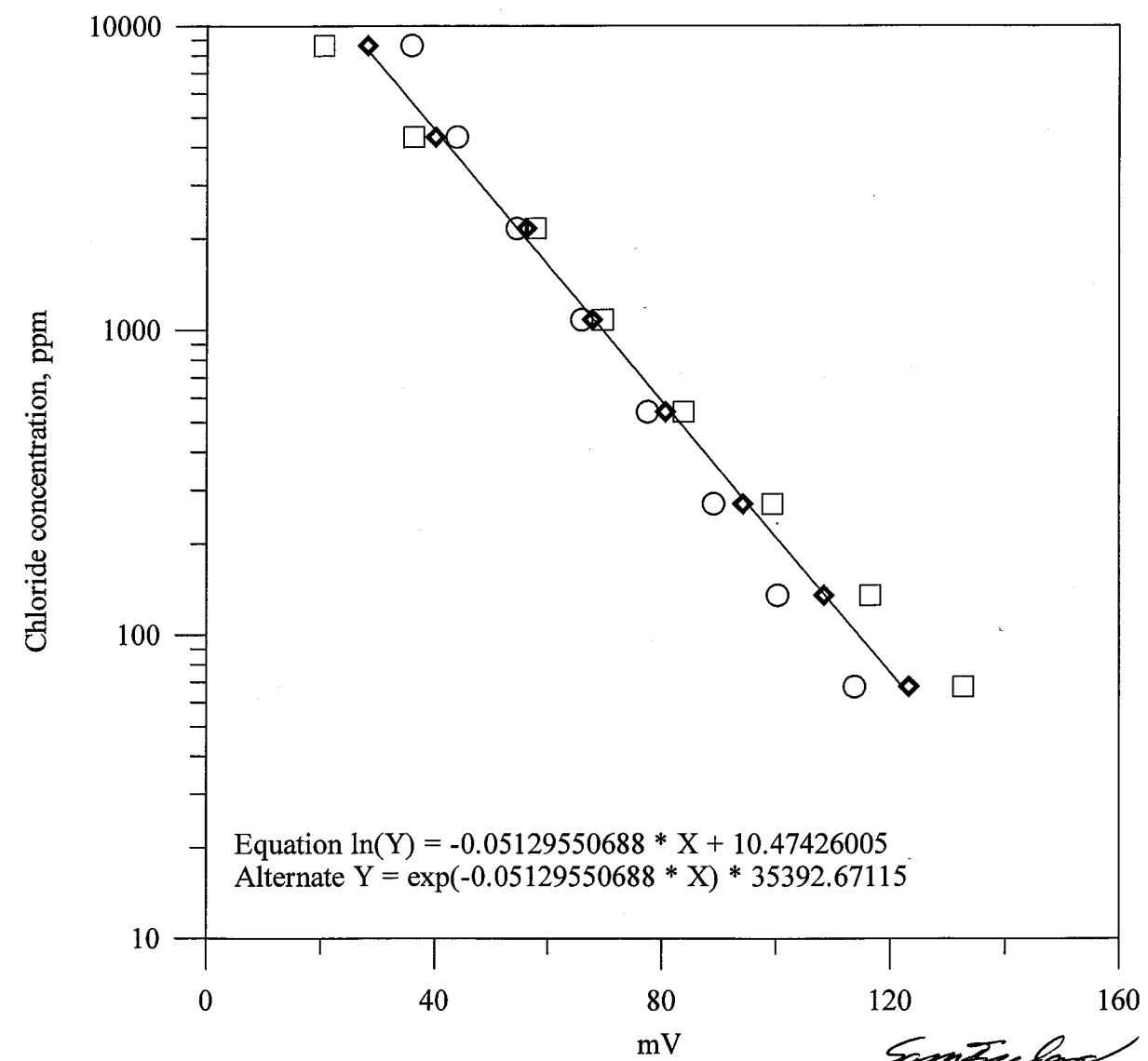
Unknown PbCl<sub>2</sub> solution 79.3 73.6

pH = 4.584

Solution: measurements taken with OHAUS precision standard

SN# 2883 cal 2/3/01 SF 6/6/01 3/2/01

Samuel  
6/6/01



*Samuel*  
6/6/01

- Using above equation to calc  $Cl^-$  conc

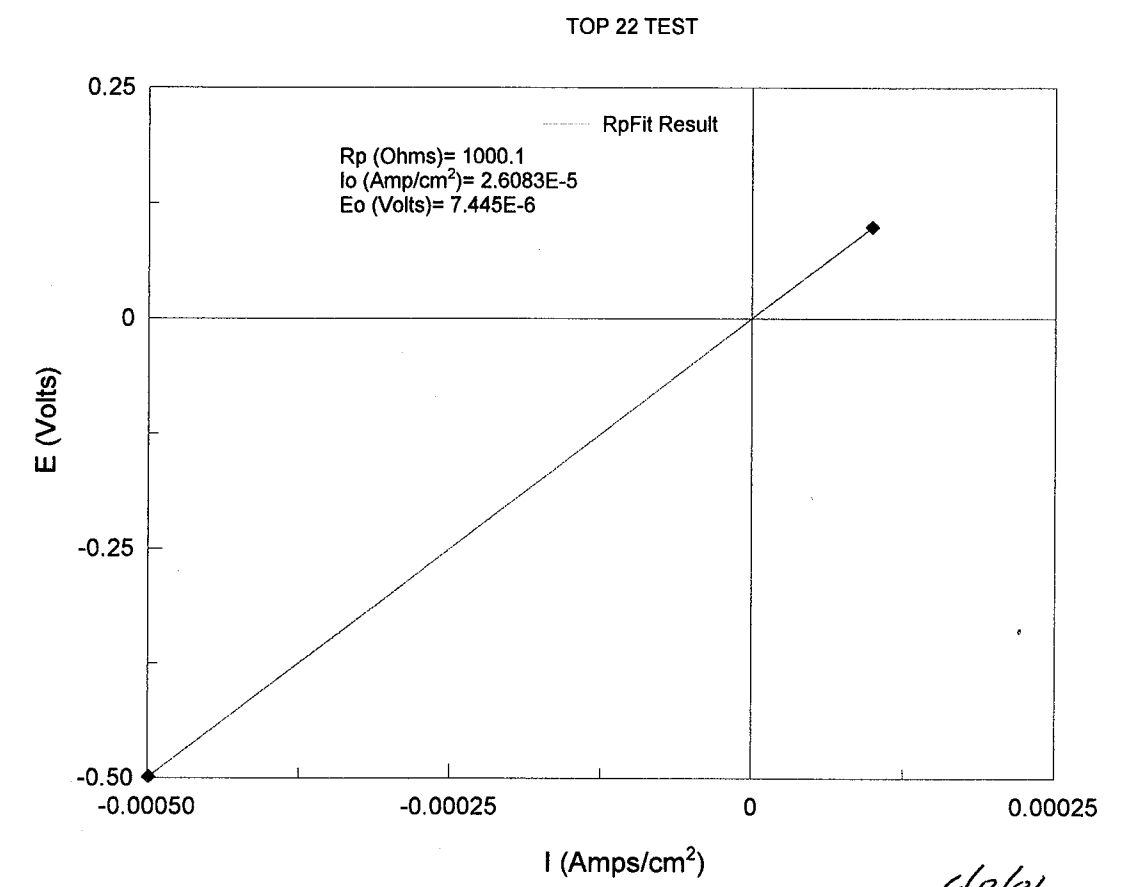
$Cl^-$  ppm = 701.15

*Samuel*  
6/6/01

Run Top-22 test

Verification of the performance of Potentiostat & Data Acquisition Software

1000 ohm Resistor SN# 171001 cal 04/27/01



6/2/01  
*Samuel*

6/12/01  
*Samuel*



Cyclic Polarization of 825 with  $\text{PbCl}_2$ , 3100 ppm  $\text{Pb}^{2+}$ , 1100 ppm  $\text{Cl}^-$

Cyclic Polarization of 825 on SE map 7/5/02

Objective: See pg #5

Specimen: Alloy 825 600 Grit Finish Specimen Dimensions on Pg #6

with 2 PTFE Crevice Washers Attached at 50 in.-O<sub>2</sub> using

Proto 6104 SN# 314047 cal 6/4/01

Start wt = 33.908g OHAUS Precision Standard

End wt = 33.882g SN# 2883 cal 3/2/01

Solution prep on pg #9, #4, Analysis on pg #10-12

Start pH = 4.584 Fisher Accumet 950 meter

End pH = 4.513 SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 8238325

Temperature: 95°C Thermometer SN# 115814 cal 1/29/01

$E_{\text{corr}} = -254 \text{ mV}$  measurement taken with

$E_{\text{pr}}$ : not measured Solotron 1287

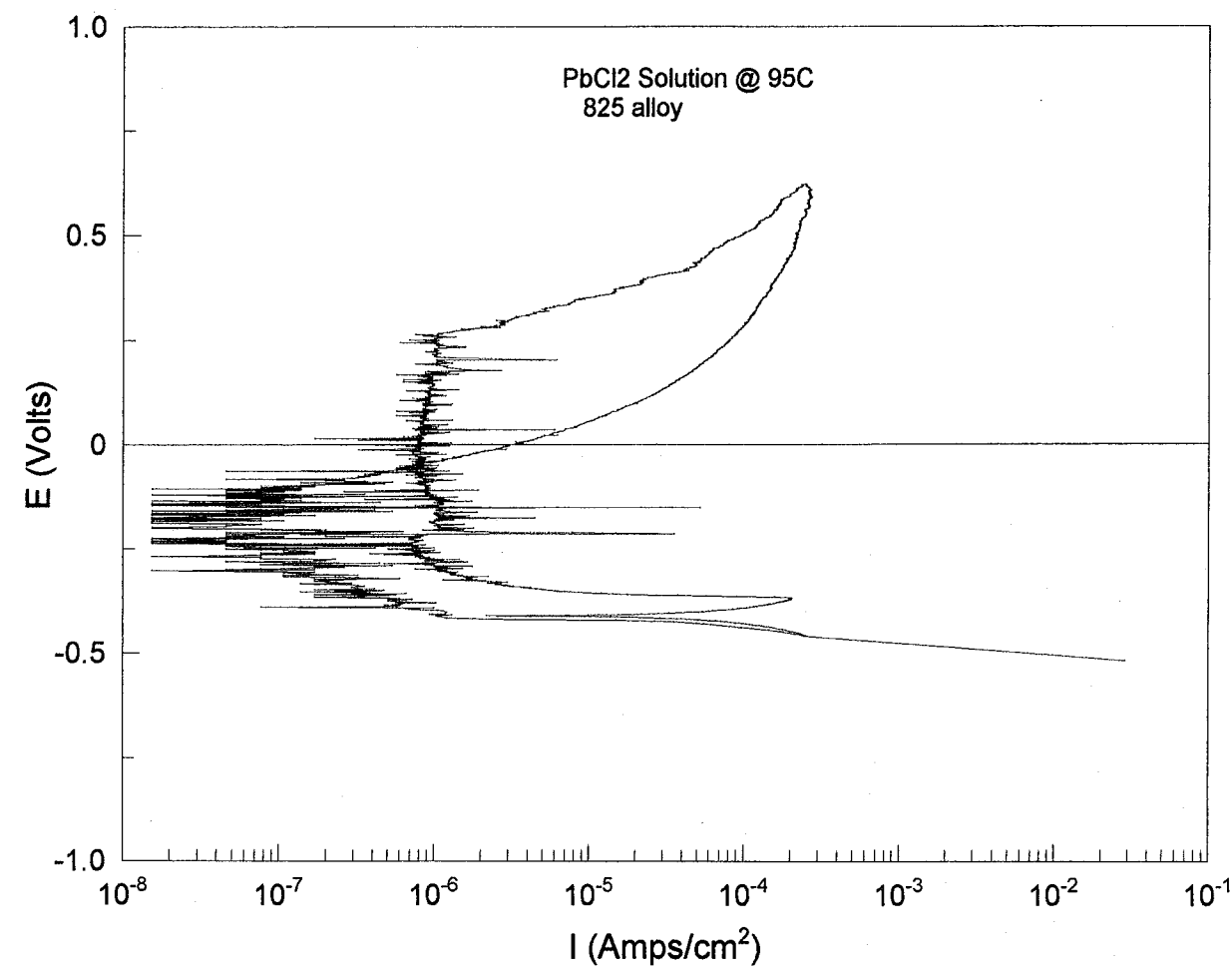
Solution Deaerated with 99.999%  $\text{N}_2$

Specimen Examination: Crevice corrosion on 23 of 24 crevice points

Data File: A825+1.cor

6/13/01

Sam Fickel



6/13/01

Sam Fickel

6/13/01

Sam Fickel

Cyclic Polarization of 825 with NaCl, 426 ppm  $\text{Cl}^-$ 

objective: See page # 5

Specimen: Alloy 825 600 grit Finish Specimen Dimensions on pg # 6

with 2 PTFE Crevice Washers attached at 50 in-O<sub>2</sub> using

Proto 6104 SN# 314047 cal 6/4/01

Start wt = 37.690g OHAUS Precision Standard

End wt = 37.664g SN# 2883 cal 3/2/01

Solution: 1.44 ml of 0.1M HCl stock solution OHAUS Precision Standard

1.399g NaCl Lot# 010166 SN# 2883 cal 3/2/01

+ 2000 ml DI water + Deaerated with 99.999%  $\text{O}_2$ 

Start PH = 4.616 Fisher Accumet 950 meter

End PH = no reading taken Dump Solution SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: PT Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp: 95°C Thermometer H98-170 SN# cal 5/3/01

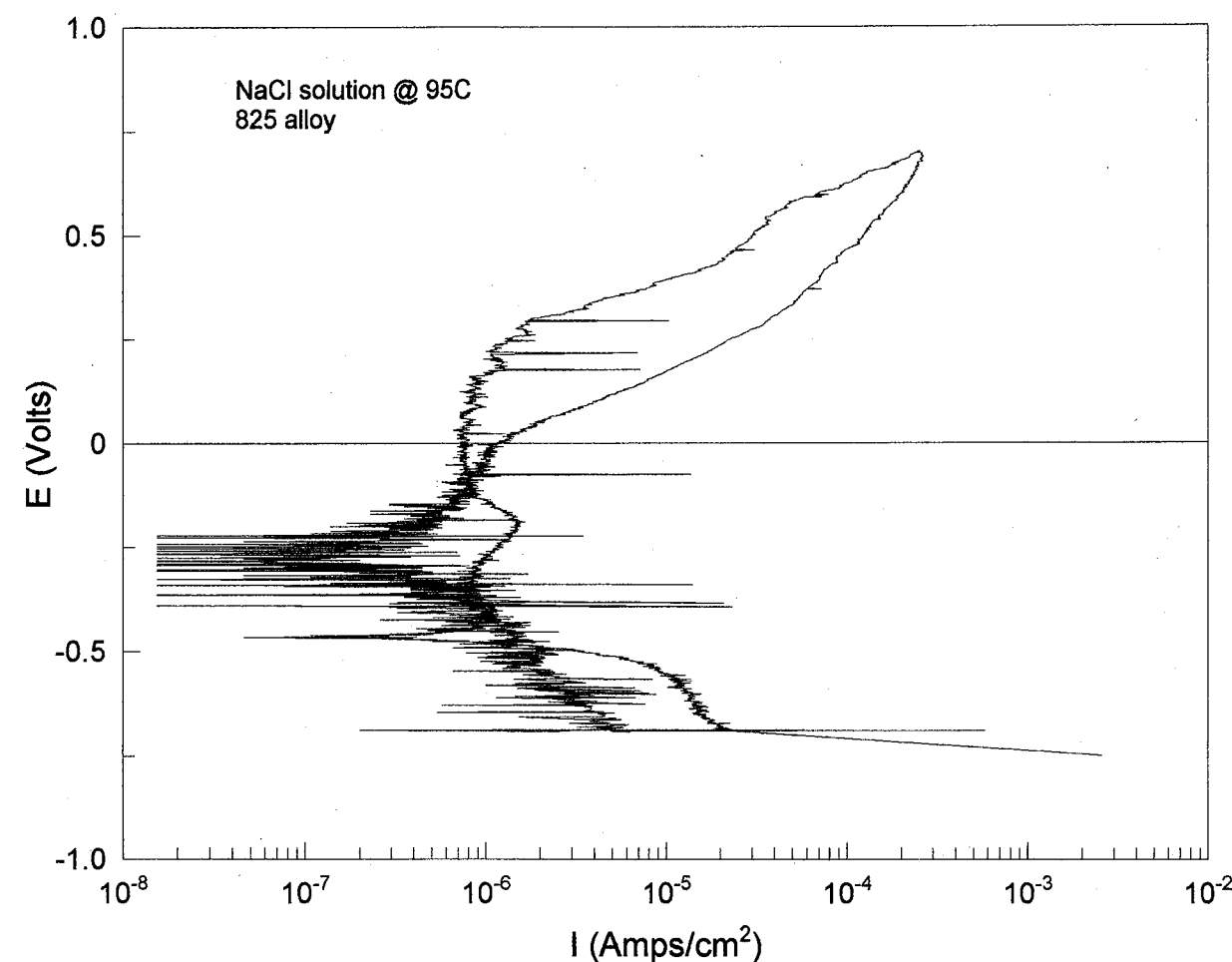
 $E_{\text{con}} = -401 \text{ mV}$  Keithley 617 SN# 537418 $E_{\text{PT}} = -398 \text{ mV}$  cal 2/22/01Solution Deaerated with 99.999%  $\text{O}_2$ 

Specimen Examination: Crevice corrosion on 17/24 feet.

Data File: A825+2.cor

6/4/01

Santoluc

426.82 ppm  $\text{Cl}^-$ 

6/14/01

Santoluc

6/14/01

Santoluc



Cyclic Polarization of C-22 with  $PbCl_2$ , 1800 ppm  $Pb^{2+}$ , 700 ppm  $Cl^-$

objective: See pg# 5

Specimen: Alloy C-22 600 grit Finish Specimen Dimension on pg# 6

with 2 PTFE Crevice Washers attached at 50 in-oz using

Proto 6104 SN# 314047 cal 6/4/01

Start wt = 39.01g OHAUS Precision Standard

End wt = 38.985g SN# 2883 cal 3/2/01

Solution: Saturated  $PbCl_2$  - prep by filtering excess  $PbCl_2$  in 2000ml DI  $H_2O$

Lot# L13K01 Deaerated with 99.999%  $O_2$

Start pH = 4.905 Fisher Accumet 950 meter

End pH = 5.586 SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: PT Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp: 95°C Thermometer SN# F98-393 cal 12/21/00

$E_{corr} = -343mV$  Keithley 617 SN# 537418

$E_{pr} = -278mV$  cal 2/22/01

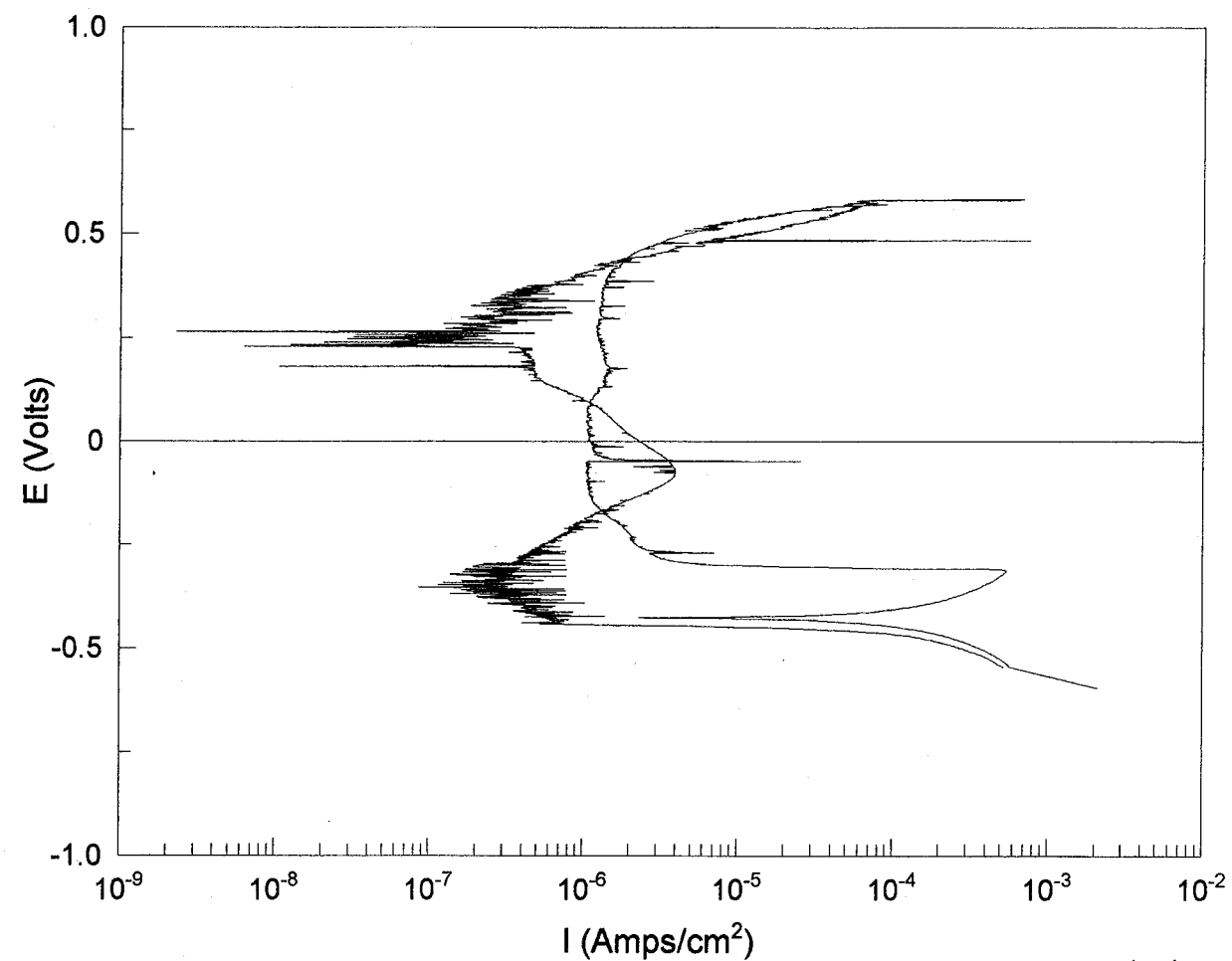
Specimen Examination: No Visible Corrosion

Data File: C22.tl.com

6/14/01

Samuelson

Alloy C22  
 $PbCl_2$  solution  
@ 95°C



6/14/01

Samuelson

6/14/01

Samuelson

# Solution mV test - free Cl conc Analysis

Objective: measure Cl concentration using NaCl standard solutions

Meter: Accumet 950 Fisher Scientific SN# 3340 cal 7/20/00

Electrode: Microelectrodes Inc MI-200 SN# 42992

Reference: Fisher 13-620-51 SN# 8077248

Stock solution: 3.55g NaCl + 500ml DI H<sub>2</sub>O 4306.9 ppm Cl<sup>-</sup>  
consecutive 1/2 ratio dilutions, Lot# D10166

Soln	ppm Cl <sup>-</sup>	mV Run 1	mV Run 2
1	4306.9	27.3	31.0
2	2153.45	43.5	43.8
3	1076.72	58.6	58.2
4	538.36	73.3	73.3
5	269.18	89.5	88.5
6	134.59	106.1	106.3

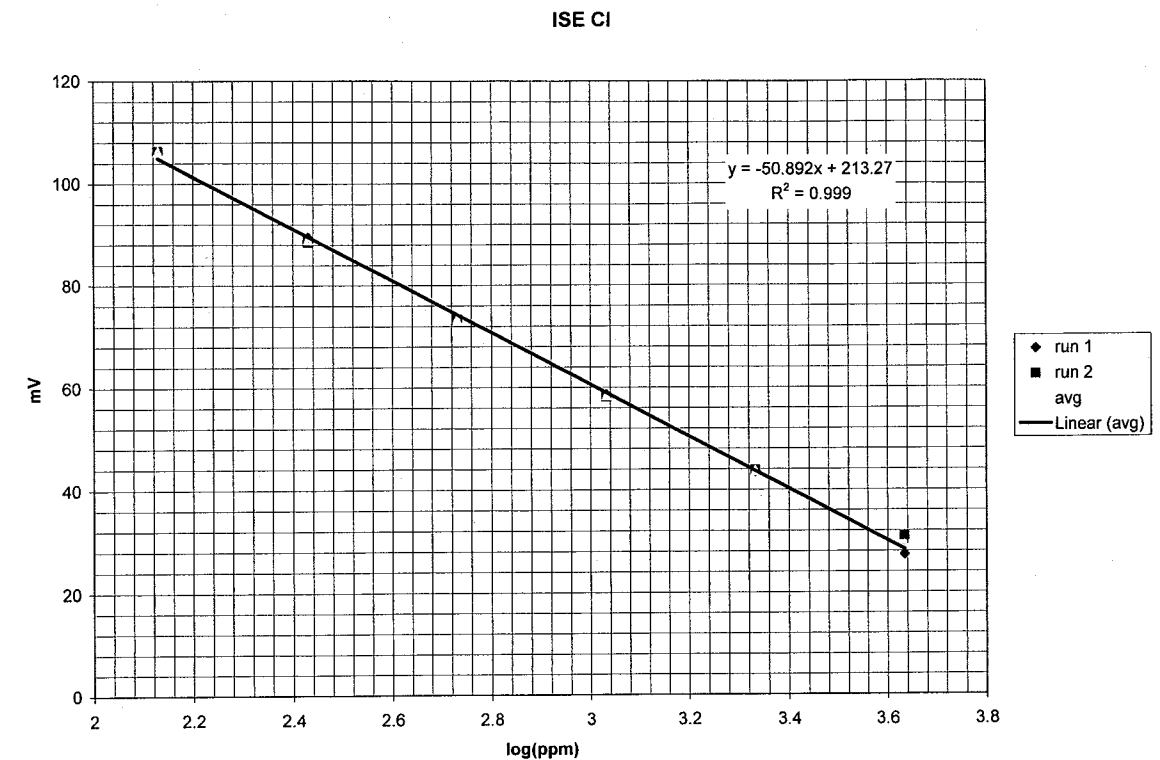
unknown PbCl<sub>2</sub> solution 80.2 80.1

prep on 7g #18

Solution measurements taken with OHAUS precision standard

SN# 2883 cal 3/2/01

6/14/01  
Santana



6/14/01  
Santana

Cl<sup>-</sup> ppm = ~~415~~ 414.66

6/14/01

Santana



Cyclic Polarization of C-22 with NaCl, 700 ppm Cl<sup>-</sup>

Objective: See pg # 5

Specimen: Alloy C-22 600 grit Finish Specimen Dimensions on pg # 6

with 2 PTFE crevice washers attached at 50 psi using

Proto 6104 SN# 314047 cal 6/4/01

Start wt = 38.59g OHAUS Precision Standard

End wt = 38.565g SN# 2883 cal 3/2/01

Solution: ~~Saturated PbCl<sub>2</sub> sol~~ 2.311g NaCl Lot# 010166+ 1 ml of .1M HCl stock solution + 2000 ml DI H<sub>2</sub>O

Start pH = 4.725 Fisher Accumet 950 meter Probe 13-620-296

End pH = 5.902 SN# 3440 cal 7/20/00 SN# 1100208

measurements taken with OHAUS Precision Standard SN# 2883 cal 3/2/01

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp: 95°C Thermometer SN# H98-170 cal 5/3/01

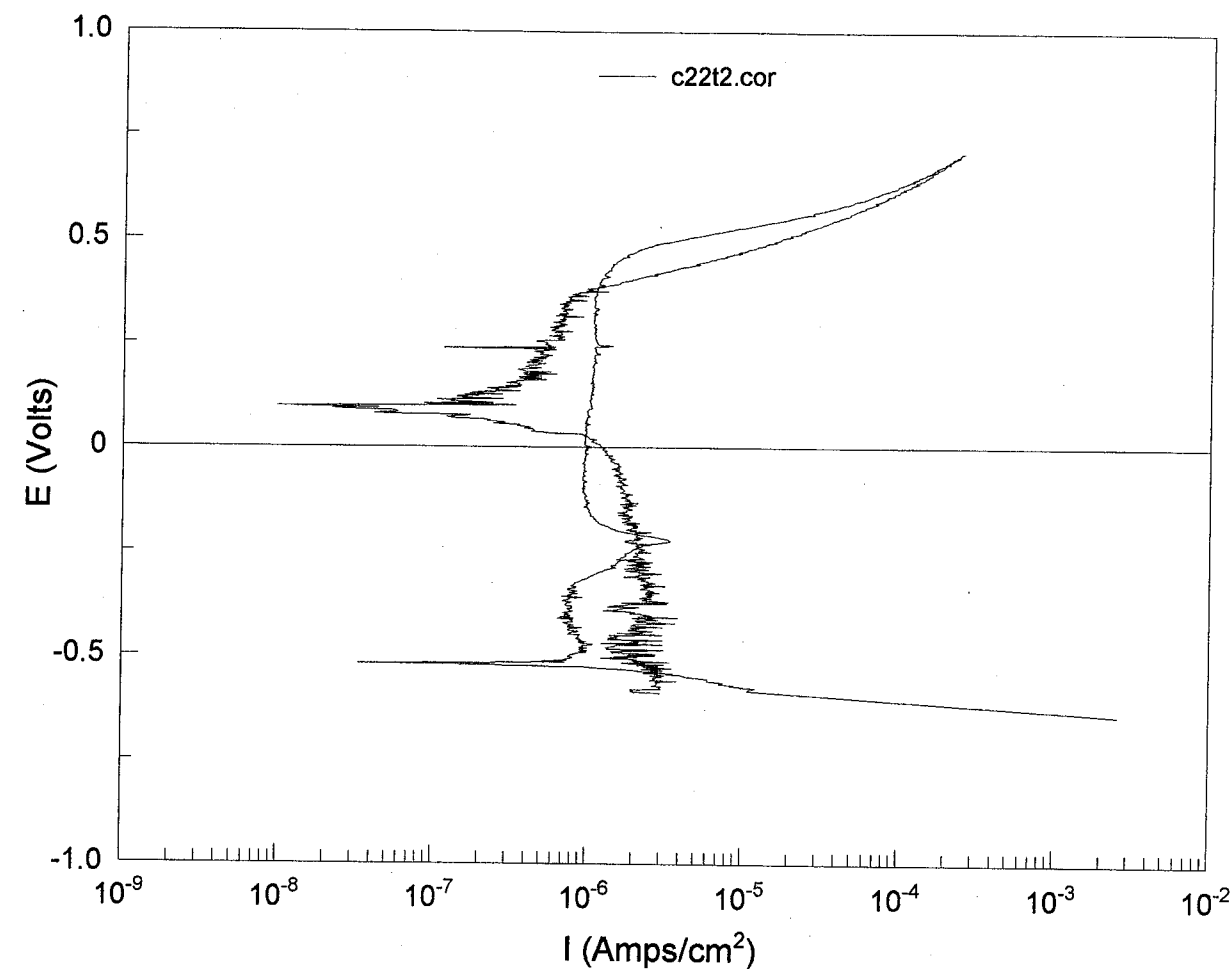
E<sub>corr</sub> = -372 mV Keithley 617 SN# 537418E<sub>pt</sub> = -378 mV cal 2/22/01Solution Deaerated with 99.999% N<sub>2</sub>

Specimen Examination: No Visible Corrosion

Data File: C22t2.cor

6/18/01

Sam F. Wilson

702.7 ppm Cl<sup>-</sup>

6/18/01

Sam F. Wilson

6/18/01

Sam F. Wilson

Solution prep: heated 2000ml of DI to 95°C and held there for 24 hours while deaerating with 99.999 N<sub>2</sub>, Thermometer SN# F98-393 cal 12/21/00  
 - added 13.500g PbCl<sub>2</sub> Lot# L13K01, OHAUS Precision Standard SN# 2883 cal 3/2/01, continued deaerating while maintaining temp at 95°C for 5 hours, then filtered solution at R.T

### Solution ISE Test - Free Cl<sup>-</sup> concentration

Objective: measure Cl<sup>-</sup> concentration using NaCl standard solutions

Meter: Accumet 950 Fisher Scientific SN# 3340 cal 7/20/00

Electrode: Microelectrodes Inc MI-200 SN# 42992

Reference: Fisher 13-620-51 SN# 8077248

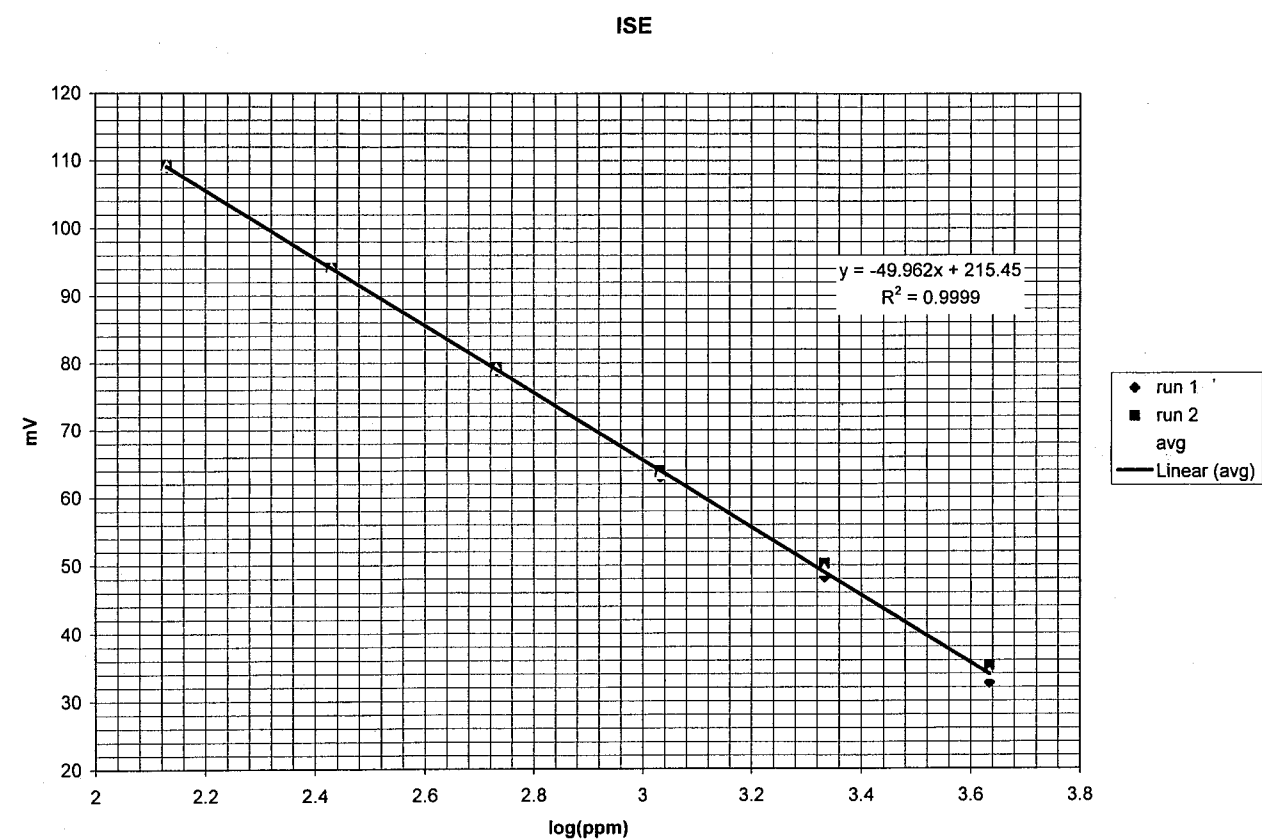
Stock solution: 3.55g NaCl + 500 ml DI H<sub>2</sub>O 4306.9 ppm Cl<sup>-</sup>

consecutive 1/2 ratio dilutions, Lot# 010166

Scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

Soln	ppm Cl <sup>-</sup>	Run 1 mV	Run 2 mV
1	4306.90	32.6	35.2
2	2153.45	48.2	50.2
3	1076.72	63.0	64.0
4	538.36	78.9	79.3
5	269.18	94.0	94.1
6	134.59	109.0	109.3
PbCl <sub>2</sub> solution		73.4	75.2

6/19/01  
 Sam Trubee



6/19/01  
 Sam Trubee

Cl<sup>-</sup> ppm = 692.62 ppm

6/19/01  
 Sam Trubee

Cyclic Polarization of C22 with  $\text{PbCl}_2$ , 3100 ppm  $\text{Pb}^{2+}$ , 1100 ppm  $\text{Cl}^-$ 

Objective: See pg # 5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg # 6

with 2 PTFE Crevice Washers Attached at 50 in-oz using

Proto 6104 SN# 314047 cal 6/4/01

Start wt = 38.67g OHAUS Precision Standard

End wt = 38.646g SN# 2883 cal 3/2/01

Solution: prep on pg # 24 solution Deaerated with 99.999%  $\text{N}_2$ 

Start pH = 4.484 Fisher Accumet 950 meter

END pH = 5.098 SN# 3440 cal 7/20/00

Probe 13-G20-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: PT Flag

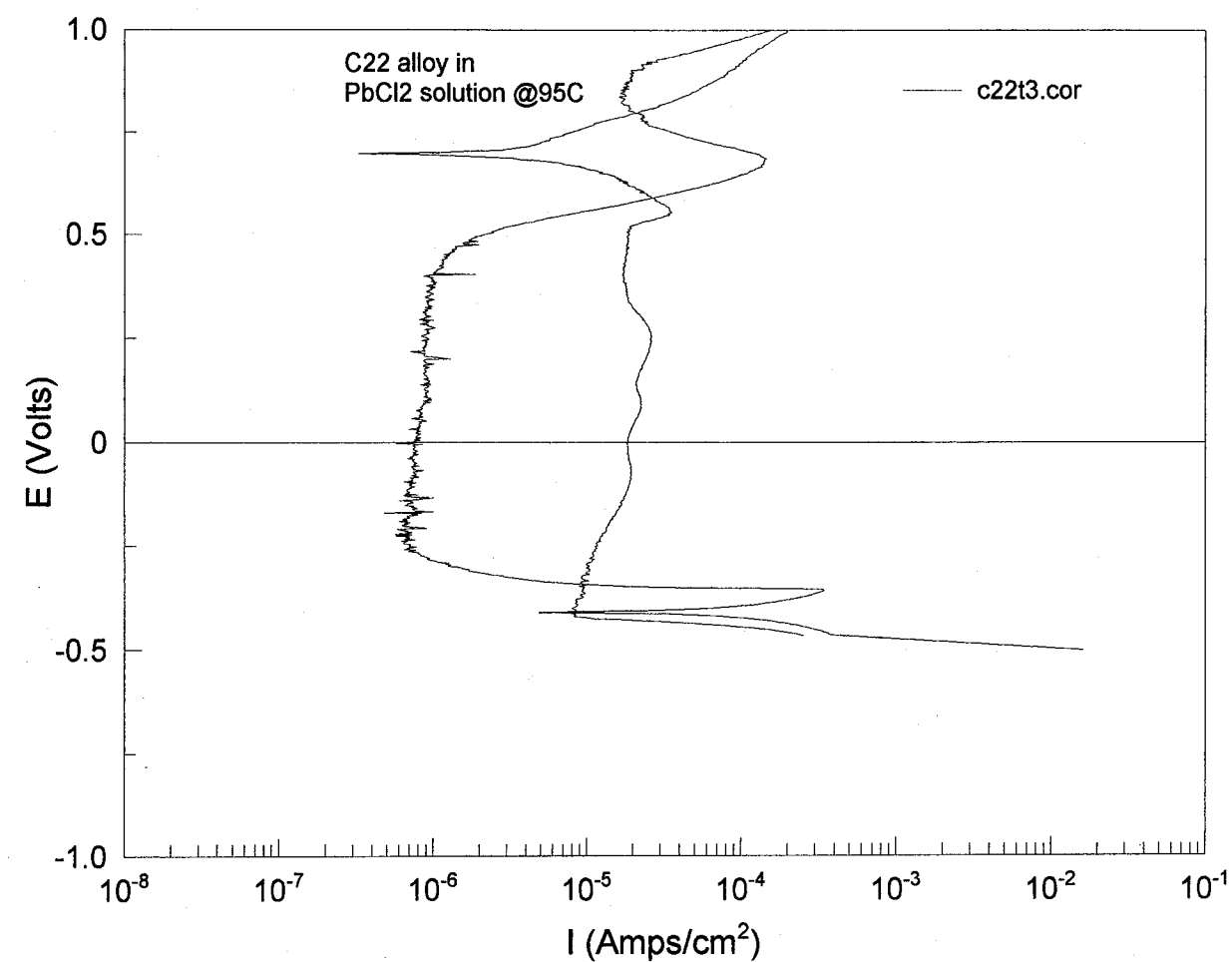
Reference Electrode: Fisher 18-620-52 SN# 8238325

Temp: 95°C Therm H98-170 cal 5/3/01

 $E_{\text{corr}}$ : -243 mV Keithley 617 SN# 537418 $E_{\text{pt}}$ : -677 mV cal 2/22/01

Specimen Exam: Discolorization &amp; pitting on 24 of 24 feet.

Data File: C22\*3.cor

6/20/01  
San Jula6/20/01  
San Jula6/20/01  
San Jula



~~Electrochemical Test at 375 mV SI PP 10/15/01~~

~~Solution Prep: 1000 ml DI H<sub>2</sub>O + 1 ml of 1M HCl stock solution, heated to 95°C  
and held for 24 hours while deaerating with 99.999% N<sub>2</sub>, added 6.746g PbCl<sub>2</sub>  
Lot# L13K01, continued deaerating while maintaining temp at 95°C for 6 hours  
Then filtered solution~~

~~Scale: Fisher Accumeter OHAUS Precision standard SN# 2883 cal 3/2/01~~

~~Thermometer: SN# 9253474 cal 5/23/01~~

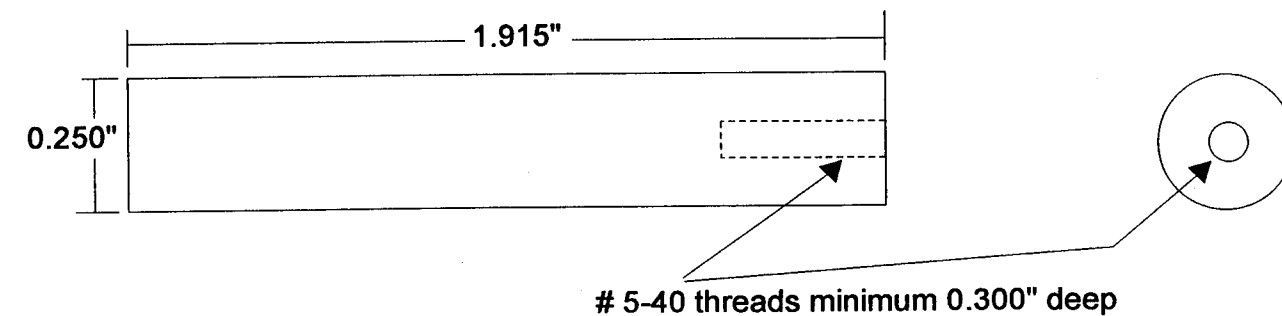
6/21/01

Sam Tuller

Darrell S. Dunn  
SwRI-CNWRA  
Bldg 57.  
Ext. 6090

### Cyclic Polarization Specimen

All dimensions +/- 0.003'



### Special Instructions:

1. Machine perpendicular to rolling direction
2. Use low stress machining procedure.
3. Return unused material.

6/22/01

Sam Tuller

Sam Tuller  
6/22/01

Electrochemical Test at  $-375\text{ mV}$  of C22 in  $\text{PbCl}_2, 3400\text{ ppm Pb}^{2+}$

1200 ppm  $\text{Cl}^-$

Objective: see pg #5

Specimen: Alloy C22 600 spit Finish Specimen Dimensions on pg #29

Start wt: 12.512g OHAUS Precision Standard

END WT: 12.500g SN# 2883 cal 3/2/01

Solution: Prep on Pg #28 Deaerated with 99.999%  $\text{N}_2$

Start PH = 4.439 Fisher Accumet 950 meter

END PH = 5.098 SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solotrom 1287

Counter Electrode: PT Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp:  $95^\circ\text{C}$  thermometer SN# 9253474 cal 5/23/01

$E_{\text{corr}} = -251\text{ mV}$  Keithley 617 SN# 537418

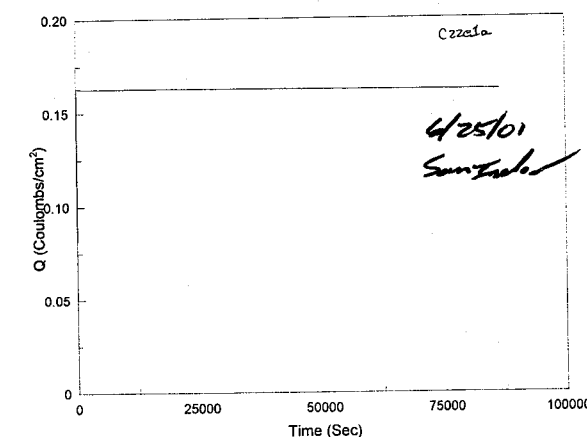
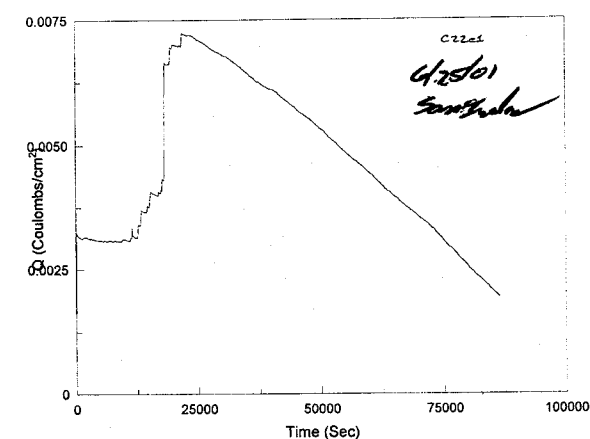
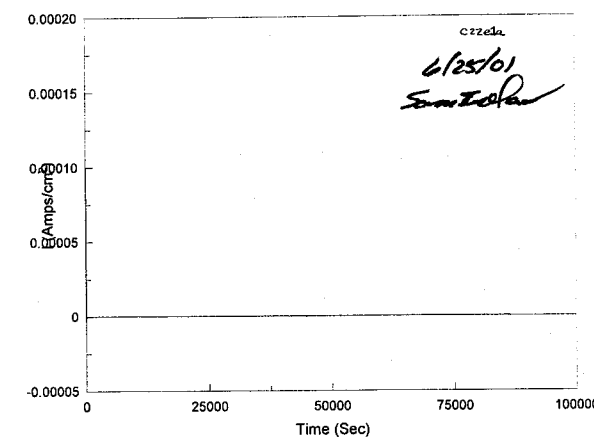
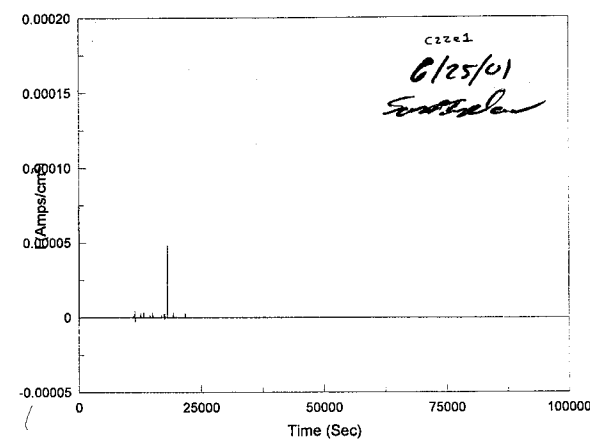
$E_{\text{PT}} = -111\text{ mV}$  cal 2/22/01

Specimen Exam: No visible corrosion

Data File: C22e1.cor } 48 hours total  
C22e1a.cor }

6/22/01

Sanjiv



6/25/01

Sanjiv

Solution Prep: heated 2000 ml DI H<sub>2</sub>O to 95°C and held there for 24 hours while deaerating with 99.999% N<sub>2</sub>, Thermometer H98-170 cal 5/3/01

- added 11.634 g PbCl<sub>2</sub> Lot # L13K01, OHAUS Precision Standard SN# 2883 cal 3/2/01, continued deaerating while maintaining temp at 95°C for 5 hours

- filtered 1000 ml of solution ⇒ called soln A

- adjusted pH with 1 M HCl stock solution to pH = 2.905 then filtered remaining solution ⇒ soln B

Fisher Accumet 950 meter SN# 3440 ~~SN# 346052~~ cal 7/20/00, Probe 13-620-296 SN# 1100208

Solution ISE Test, Free Cl<sup>-</sup>, soln B

Objective: measure Cl<sup>-</sup> conc using NaCl standard soln

Meter: Accumet 950 Fisher Scientific SN# 3340 cal 7/20/00

Electrode: Microelectrodes INC MI-200 SN# 42992

Reference: Fisher 13-620-51 SN# 8077248

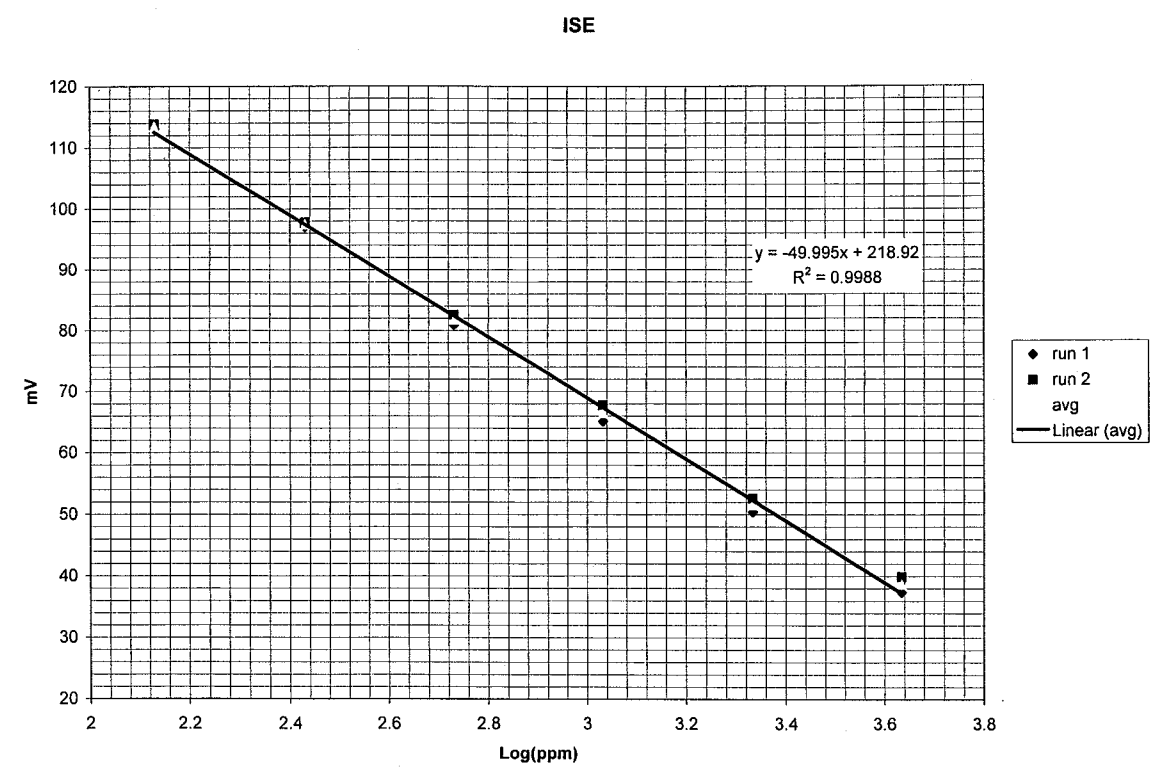
Stock solutions: 3.550g NaCl + 500 ml DI H<sub>2</sub>O Lot # 010166

consecutive 1/2 ratio dilutions

scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

Soln	PPM Cl <sup>-</sup>	Run 1 mV	Run 2 mV
1	4306.90	37.4	39.9
2	2153.45	50.4	52.6
3	1076.72	65.1	67.8
4	538.36	80.9	82.6
5	269.18	97.0	97.8
6	134.59	113.0	113.8
PbCl <sub>2</sub> solution		71.0	72.7

6/25/01  
Sam Tolan



Cl<sup>-</sup> ppm = 874.38

6/25/01  
Sam Tolan



Chloride Concentration Measurements using Capillary Electrophoresis

Objective: Measure chloride concentrations of the PbCl<sub>2</sub> solutions for CPP tests

Instrument: Waters Capillary Ion Analyzer

Method: Anion Single Method

Data Files: Data acquisition using Millennium System

Sample Set: PbCl<sub>2</sub> solution - dilution (Test ID A825t1)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	PbCl <sub>2</sub> , 100:1 dilution	Unknown	PbCl <sub>2</sub> solution_dilution	6/13/01 2:10:44 PM	Anion Single Method Set

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	1	spike Cl in PbCl <sub>2</sub> _1:100	Unknown	PbCl <sub>2</sub> solution spike Cl	6/13/01 3:53:03 PM	Anion Single Method Set
2	1	10 ppm Cl std	Unknown	PbCl <sub>2</sub> solution spike Cl	6/13/01 4:03:15 PM	Anion Single Method Set

Sample Set: PbCl<sub>2</sub> solution for alloy 22 (Test ID C22t1)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 11:25:54 AM	Anion Single Method Set
1	1	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 11:15:42 AM	Anion Single Method Set
2	2	spike Cl in 1:100 dilution	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 11:46:21 AM	Anion Single Method Set
2	1	spike Cl in 1:100 dilution	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 11:36:08 AM	Anion Single Method Set
3	2	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 12:06:44 PM	Anion Single Method Set
3	1	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution for alloy 22	6/14/01 11:56:33 AM	Anion Single Method Set

Sample Set: PbCl<sub>2</sub> solution of 061901 (Test ID C22t3)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 3:34:26 PM	Anion Single Method Set
1	1	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 3:24:13 PM	Anion Single Method Set
2	2	spike Cl in 1:100	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 3:54:50 PM	Anion Single Method Set
2	1	spike Cl in 1:100	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 3:44:38 PM	Anion Single Method Set
3	2	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 4:15:16 PM	Anion Single Method Set
3	1	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution of 061901	6/19/01 4:05:04 PM	Anion Single Method Set

Sample set: PbCl<sub>2</sub> solution with pH 3

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 10:20:36 AM	Anion Single Method Set
1	1	PbCl <sub>2</sub> , 1:100 dilution	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 10:10:24 AM	Anion Single Method Set
2	2	Spike Cl in 1:100	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 10:40:59 AM	Anion Single Method Set
2	1	Spike Cl in 1:100	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 10:30:48 AM	Anion Single Method Set
3	2	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 11:01:25 AM	Anion Single Method Set
3	1	10 ppm Cl	Unknown	PbCl <sub>2</sub> solution with pH 3	6/25/01 10:51:11 AM	Anion Single Method Set

Results: Chloride concentration was calculated by integrating the area of the chloride peak and comparing with that of the standard chloride solution.

Test Solution	Chloride Concentration (ppm)	
	1:100 Dilution Sample	Spike Sample
A825t1	1,017	962
C22t1	632	705
C22t3	962	929
pH 3	1242	1152

*[Signature]*  
6/25/01

Cyclic Polarization using Pt Flag in  $PbCl_2$ , 2800 ppm  $Pb^{2+}$ , 100 ppm  $Cl^-$

Objective: See pg #5

Specimen: Pt Flag 7.48 cm<sup>2</sup> surface Area

Solution: Prep on pg #32 soln A, Deaerated with 99.999%  $N_2$

Start pH = 4.942      END pH = 4.720

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52      SN# 8238325

Temp: 95°C therm SN# 9253474 cal 5/23/01

pH meter: Fisher Accumet 950 meter SN# 3440 cal 7/20/00, Probe 13-620-296 SN# 1100208

$E_{corr}$ : -202 mV      Keithley 617 SN# 537418

$E_{pr}$ : -169 mV      cal 2/22/01

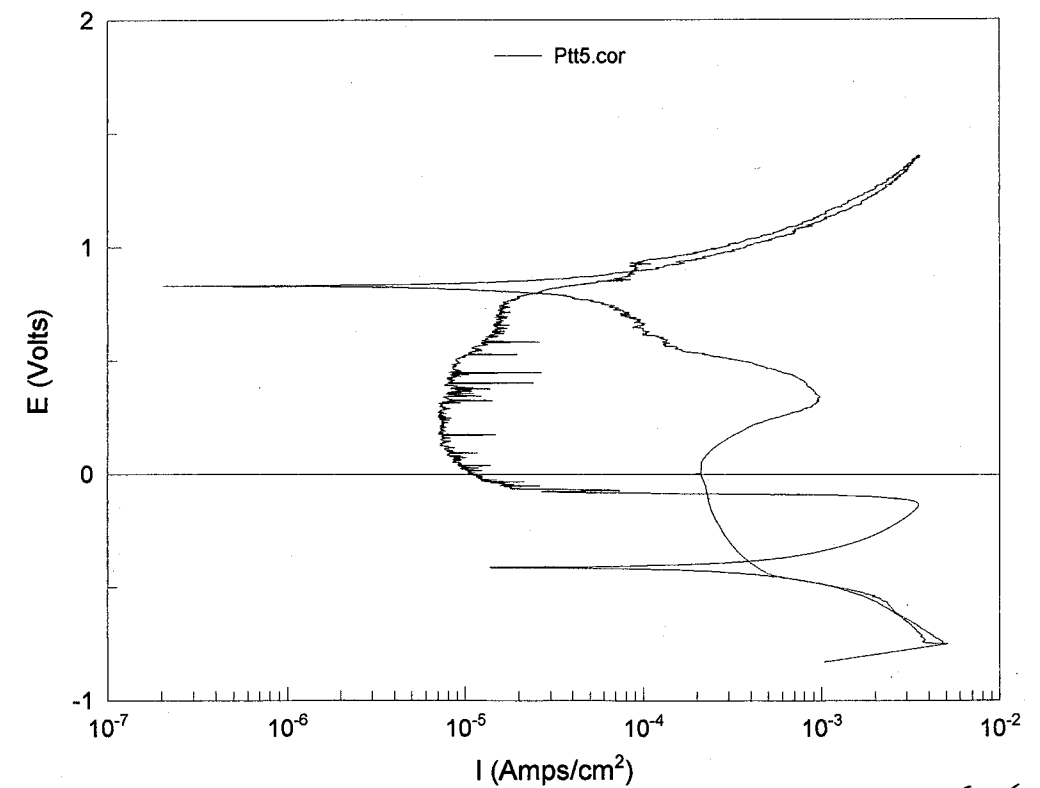
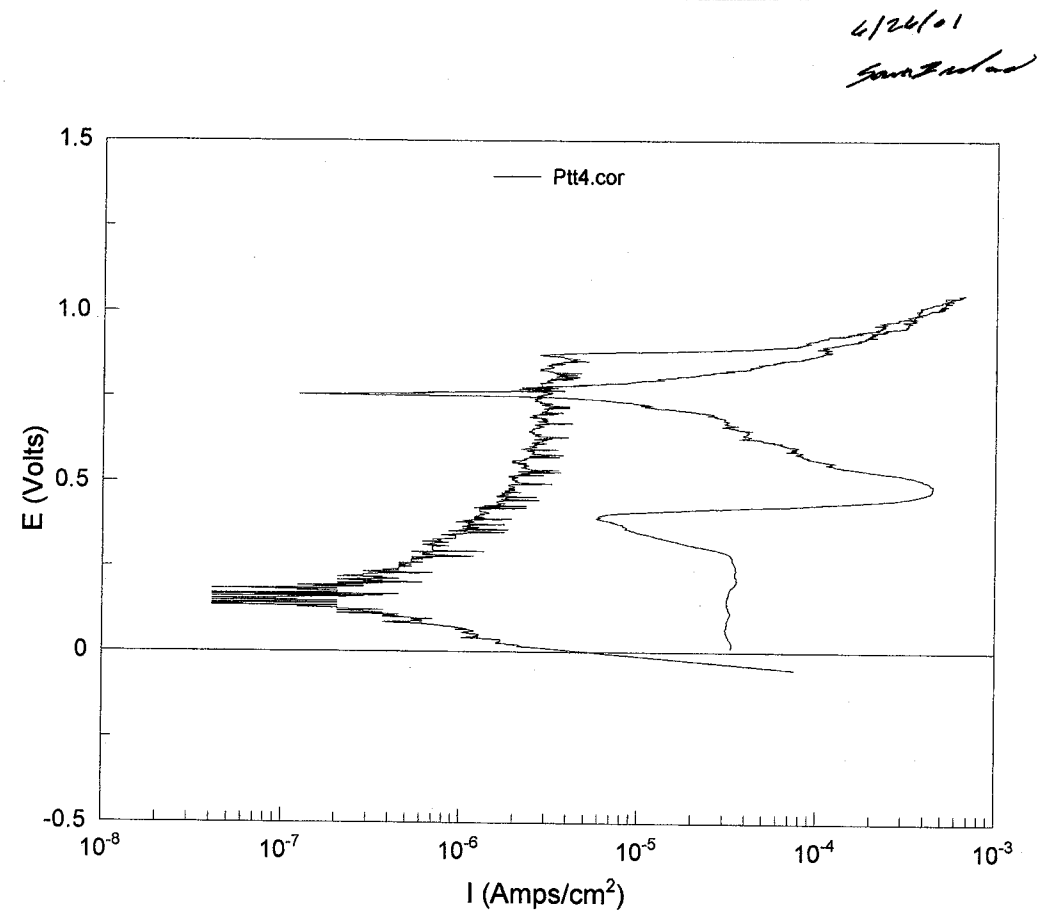
- Repeated Scan, adjusted Range

Data Files: Ptt4.cor

Ptt5.cor

6/26/01

*Samuel*



6/22/01  
*Samuel*

6/27/01  
*Samuel*

Cyclic Polarization using Pt Flag in NaCl, 1000ppm Cl<sup>-</sup>

Objective: see pg#5

Specimen: Pt Flag 7.48cm<sup>2</sup> surface Area

Solution: 1.648g NaCl Lot# 010166 + 1000 ml of DI H<sub>2</sub>O + .12ml (.1M HCl stock soln)

Start PH = 4.942 Fisher Accumet 950 meter Probe 13-620-296

END PH = 4.72<sup>ST</sup> 4.940 SN# 3440 cal 7/20/00 SN# 1100208

Scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

Deaerated with 99.9992 N<sub>2</sub>

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp: 95 °C Therm SN# 9253474 cal 5/23/01

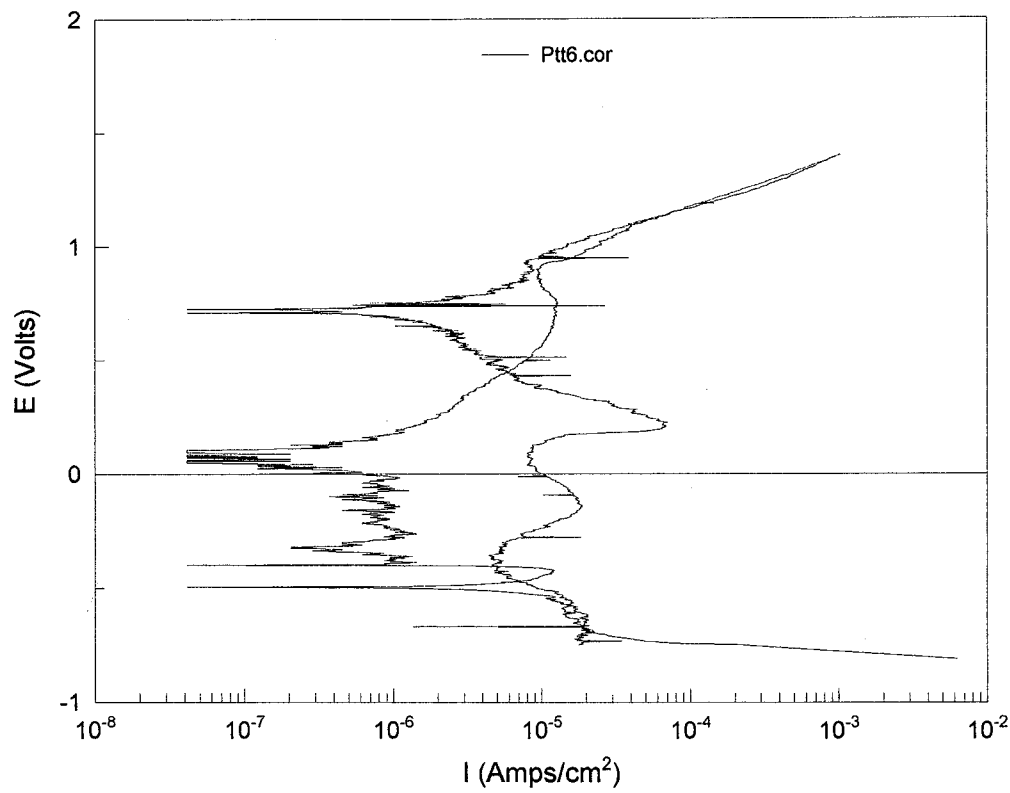
E<sub>corr</sub>: -332mV Keithley 617 SN# 537418

E<sub>PT</sub>: -250mV cal 2/22/01

Data File: Ptt6.cor

-possible contamination of PbCl<sub>2</sub>

6/27/01  
Sam Turelao



6/27/01  
Sam Turelao

6/27/01  
Sam Turelao

Cyclic Polarization using Pt Flag in NaCl, 1000 ppm Cl<sup>-</sup>

objective: See pg#5

Specimen: Pt Flag 7.48cm<sup>2</sup> Surface Area

Solution: 3.296g NaCl Lot# 010166 + 2000 ml DI H<sub>2</sub>O, deaerated with 99.999% N<sub>2</sub>

Start PH = 4.942<sup>SE</sup> 4.940 Fisher Accumet 950 meter | Probe 13-620-296

END PH = 5.857 SN# 3440 cal 7/20/00 | SN# 1100208

Scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

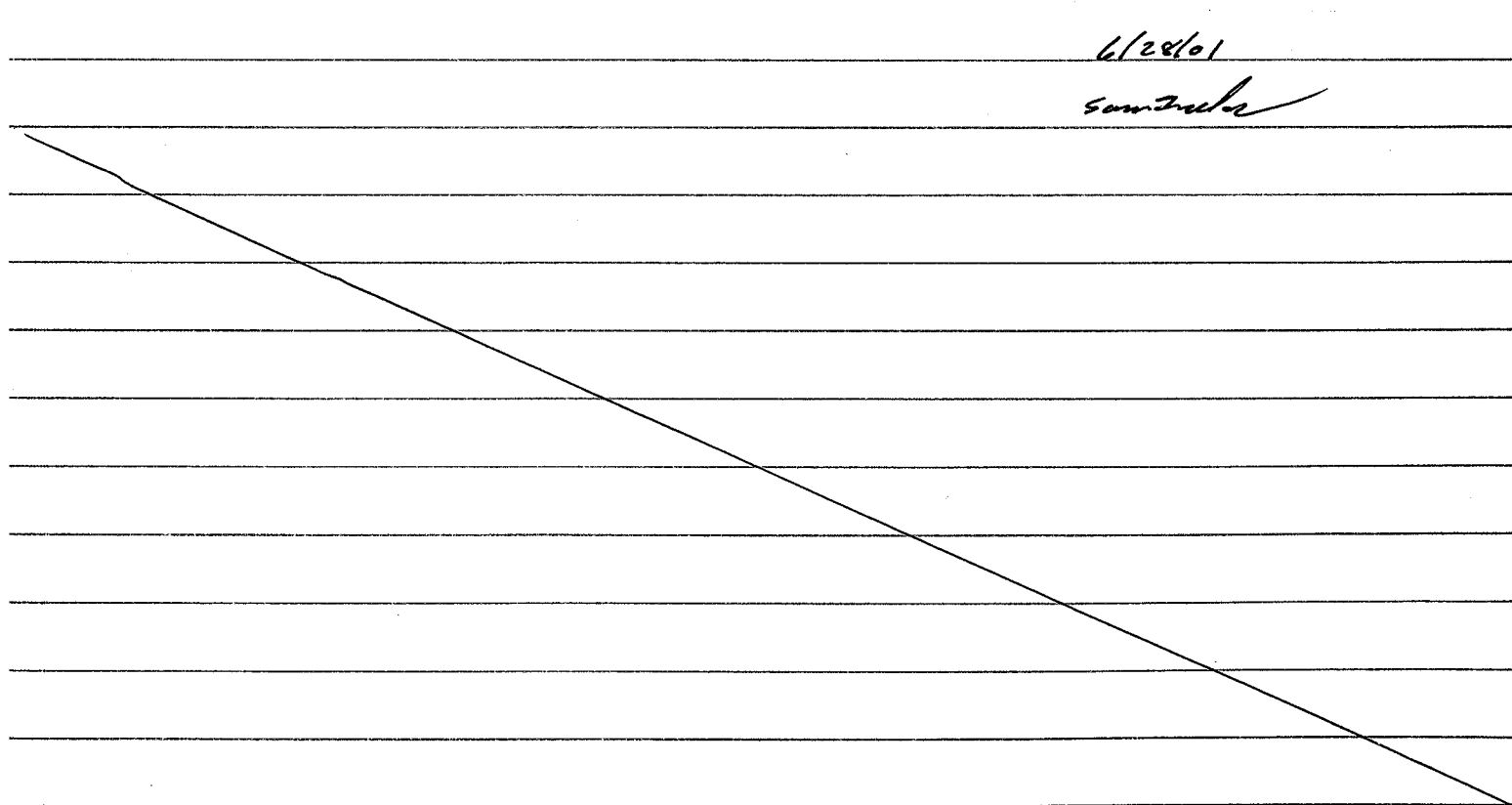
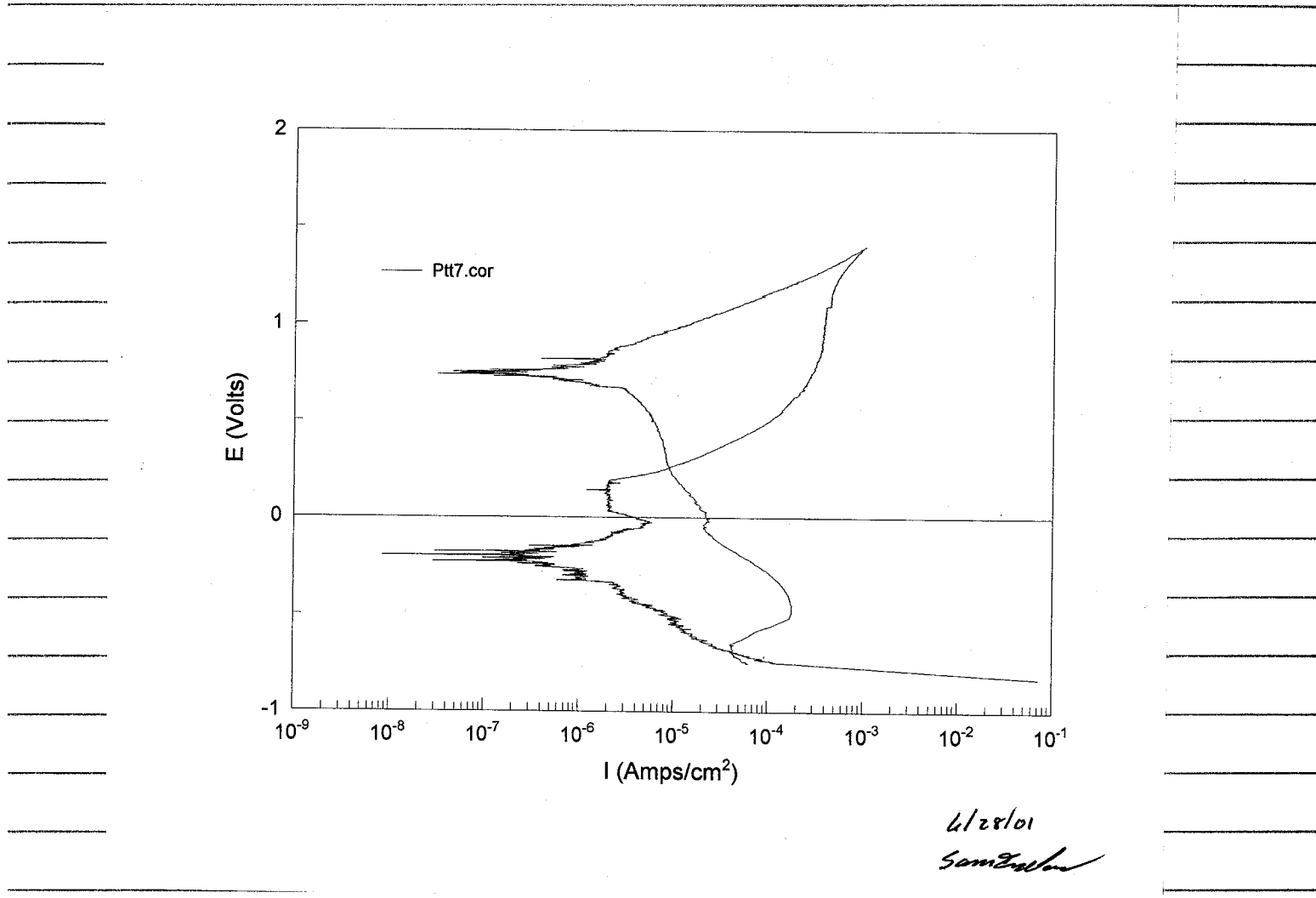
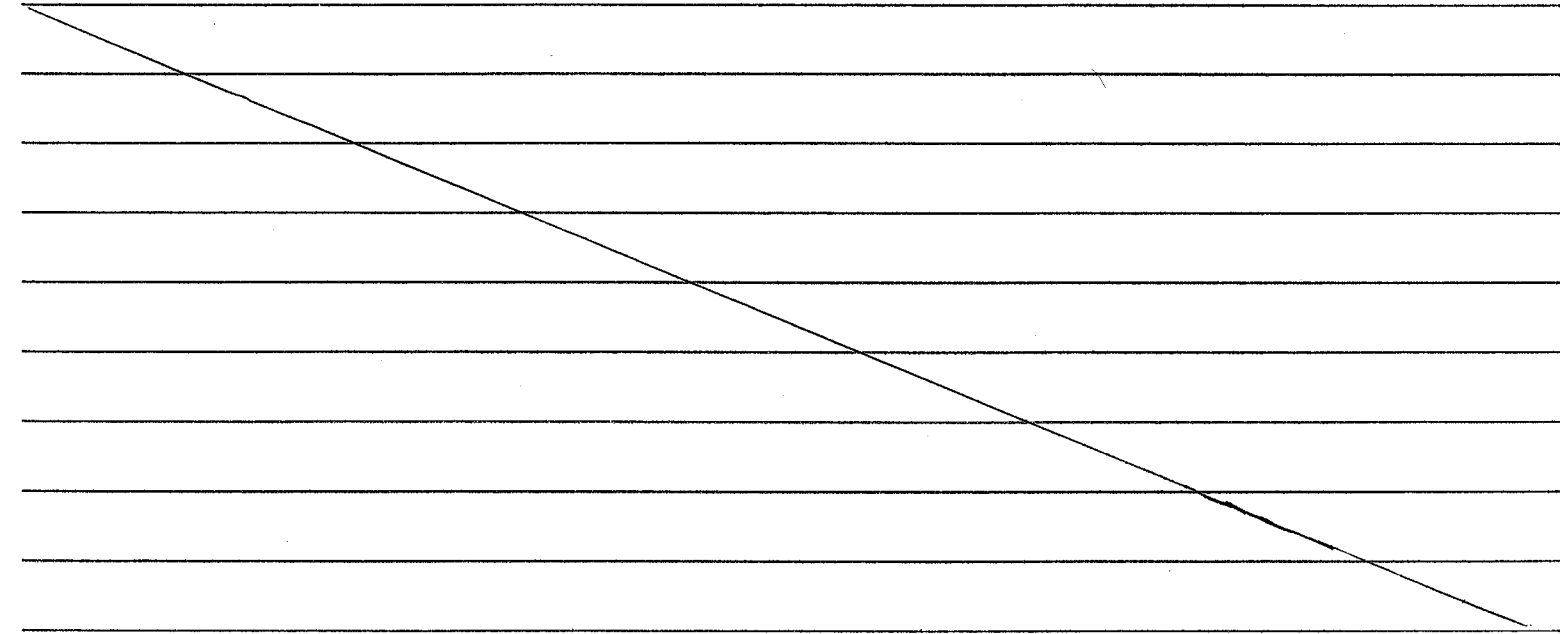
Temp: 95°C therm SN# 9253474 cal 5/23/01

E<sub>corr</sub>: -168mV Keithley 617 SN# 537418

E<sub>pr</sub>: -225mV cal 2/22/01

Data File: Pt+7.COR

6/28/01  
Sam Zuck





solutions sent for lead (ICP) and chlorine (IE) Analysis on 6/25/01

Solution name	Data File	Pg # Prep, Pg # test
A	C22+1.cor	18, 18
B	C22+3.cor	24, 26
C	c22e1.cor c22e1a.cor	28, 30
D	Pt+4.cor Pt+5.cor	32, 36
E	NO File At this time	32, N/A

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SWRI  
Matrix: Liquid  
Work Order: 20458  
Client: Division 20  
Date Received: 06/25/01  
Project No.: 20.01402.571

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.501
True Value	----	0.500
Recovery	----	100.2%
A	163528	1835
Duplicate result	163528	1842
RPD	163528	0.38%
B	163529	3079
Duplicate result	163529	3075
RPD	163529	0.13%
C	163530	3420
Duplicate result	163530	3451
RPD	163530	0.90%
D	163531	2765
Duplicate result	163531	2783
RPD	163531	0.65%
E	163532	3237
Duplicate result	163532	3241
RPD	163532	0.12%
Spike result	163532	3758
Spike added	163532	500
Recovery	163532	104.2%

Reporting Limit: 0.005 mg/L

6/28/01  
6/28/01  
San Pedro

SWRI ENV CHEM ID:5225938 JUL 11 '01 15:26 No.013 P.03  
SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SWRI  
Matrix: Liquid  
Work Order: 20458  
Client: Division 20  
Date Received: 06/25/01  
Project No.: 20.01402.571

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	201
True Value	----	200
Recovery	----	100.5%
A	163528	693
Duplicate result	163528	697
RPD	163528	0.63%
Spike result	163528	1039
Spike added	163528	400
Recovery	163528	86.5%
B	163529	1080
C	163530	1223
D	163531	980
E	163532	1328

Reporting Limit: 0.1 mg/L

7/16/01  
San Pedro

6/28/01 San Pedro  
7/16/01 San Pedro

FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: SAM IRELAND Request Date: 6/25/01  
Project No.: 20.01402.571 Phone No.: 82993  
Description of Work Requested: Lead and Chlorine Concentration Analysis  
57 lead chloride solutions; A,B,C,D,E

☐ 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

B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>

☐ Optical Microscopy ☐ SEM ☐ Hardness ☐ Profilometer ☐ Auger ☐ Other

Person Assigned: M. Maxwell / A. Al-Balcan Signature: M. Maxwell / A. Al-Balcan  
Division: 01 Date: 8/16/01

Make, Model & Serial No. of Equipment Used (attach list if necessary): ICP-TRACE #2  
IC-TRACE BY Sde.

Software Used (If any): WITH DOWS NT FOR ICP, QUATTRO FOR ICP

Standards Used (If any): LOCATED IN FILE.

Photographic Negative Numbers (If Applicable):

San Pedro 8/16/01

<sup>1</sup> 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.

8/16/01  
San Pedro

8/16/01  
San Pedro

Solution prep: heated 2000ml of DI H<sub>2</sub>O to 95°C and held there for 24 hours while deaerating with 99.999% N<sub>2</sub>, thermometer H98-170 cal 5/3/01

- added 13.515 g PbCl<sub>2</sub> Lot# L13K01, OHAUS Precision Standard SN# 2883 cal 3/2/01, continued deaerating while maintaining temp at 95°C for 5 hours, then allowed solution to cool & filtered therm SN# 9253474 cal 5/23/01
- adjusted PH with HCl to PH=0.531, added excess PbCl<sub>2</sub>
- Continued deaerating at 95°C for 24 hours, then filtered excess PbCl<sub>2</sub>

7/3/01  
Sam Fickel

Solution Prep: Soln E from pg# 42

- added excess PbCl<sub>2</sub>
- deaerated at 95°C for 24 hours then filtered excess PbCl<sub>2</sub> therm SN# 9253474 cal 5/23/01
- Sent solns from pg# 44 & pg# 45 for analysis, will log results below

7/5/01  
Sam Fickel

SWRI ENV CHEM ID:5225938 JUL 11'01 15:36 NO.014 P.03

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20  
Lab Code: SwRI Date Received: 07/05/01  
Matrix: Liquid Project No.: 20.01402.571  
Work Order: 20508

Sol'n	Data File
pg# 44	C22x7.COR
pg# 45	C22x8.COR

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	199
True Value	----	200
Recovery	----	99.3%
pg# 44	163915	8559
Duplicate result	163915	8495
RPD	163915	0.75%
Spike result	163915	12872
Spike added	163915	4000
Recovery	163915	107.8%
pg# 45	163916	1418

Reporting Limit: 0.1 mg/L

7/16/01  
Sam Fickel

SWRI ENV CHEM ID:5225938 JUL 11'01 15:36 NO.014 P.02

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20  
Lab Code: SwRI Date Received: 07/05/01  
Matrix: Liquid Project No.: 20.01402.571  
Work Order: 20508

Sample ID	Lab System ID	Lead Result (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.512
True Value	----	0.500
Recovery	----	102.4%
pg# 44	163915	1544
Duplicate result	163915	1532
RPD	163915	0.78%
Spike result	163915	2053
Spike added	163915	300
Recovery	163915	101.8%
pg# 45	163916	3559

Reporting Limit: 0.005 mg/L

7/16/01  
Sam Fickel

7/16/01  
Sam Fickel

Cyclic Polarization of C22 with  $PbCl_2$ , 1500ppm  $Pb^{2+}$ , 8600ppm  $Cl^-$

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #6

with 2 PTFE Crevice Washers attached at 50mm-og using

Proto 6104 SN# 314047 cal 6/4/01

start wt: 38.64g OHAUS Precision Standard

End wt: N/A SN# 2883 cal 3/2/01

Solution: prep on pg #44 Solution deaerated with 99.999%  $N_2$

start PH = 0.531 Fisher Accumet 950 meter

END PH = N/A SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

temp: 95°C therm SN# 9253474 cal 5/23/01

$E_{corr}$ : -92mV Keithley 617 SN# 537418

$E_{pr}$ : -215mV cal 2/22/01

- Computer malfunction

Data Files: C22+4.COR → Computer lock up

C22+5.COR → incomplete scan

C22+6.COR → incomplete scan, discoloration

7/8/01

Samuel

Specimen repolished to 600 grit Finish, Reference Electrode replaced

Lugan probe rebuilt, Crevice washers attached at 50mm-og using, Computer shutdown

Proto 6104 SN# 314047 cal 6/4/01

New Start wt: 38.381g OHAUS Precision Standard

End wt: 38.352g SN# 2883 cal 3/2/01

Solution prep on pg #46, 44 pg 10/15/01

start PH = 0.531 Fisher Accumet 950 meter

END PH = 0.494 SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-51 SN# 8077248

temp: 95°C therm# 9253474 cal 5/23/01

New  $E_{corr}$ : -88mV Keithley 617 SN# 537418

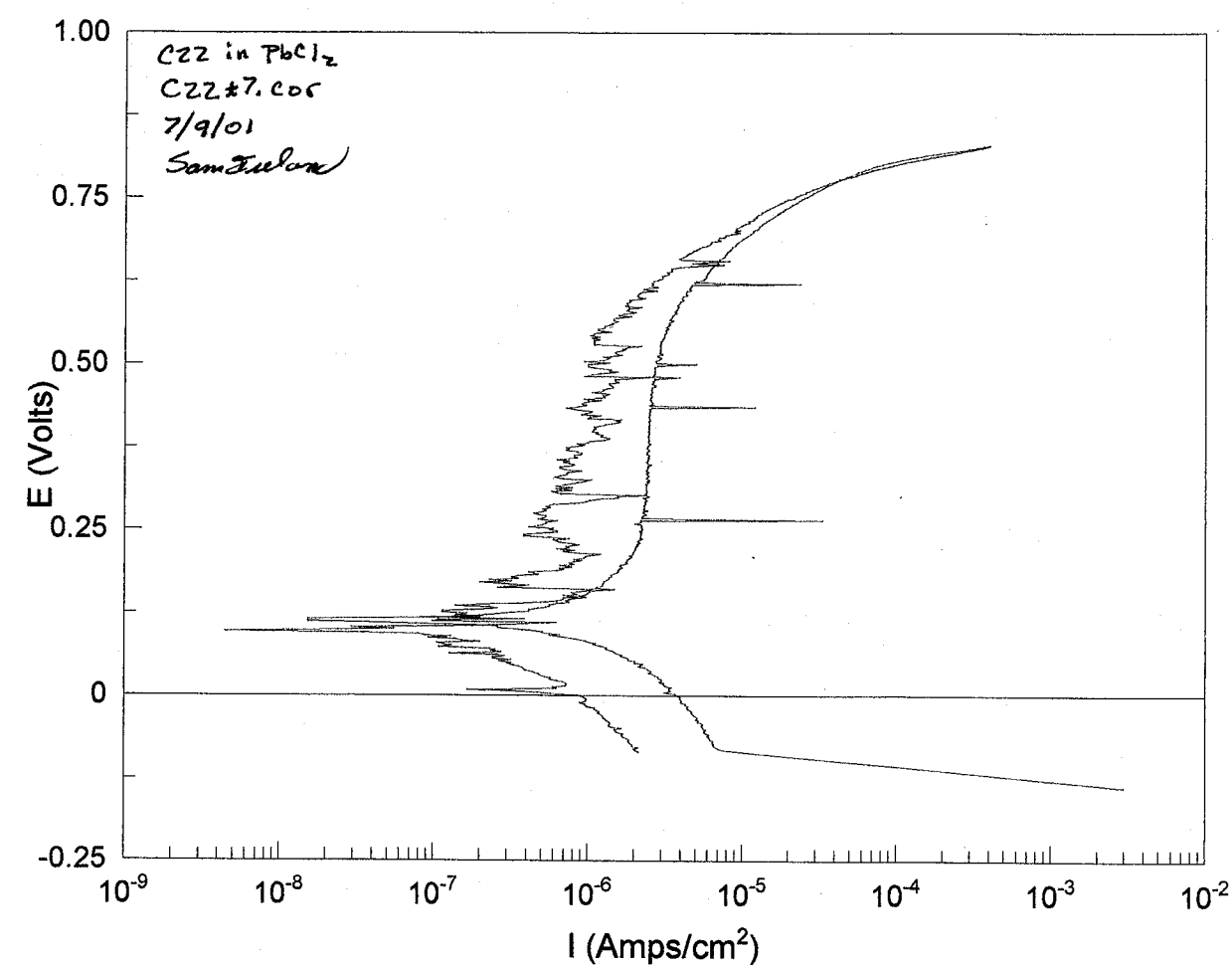
New  $E_{pr}$ : -397mV cal 2/22/01

Data Files: C22+7.COR

Specimen exam: No visible corrosion

7/9/01

Samuel



7/9/01  
Samuelson

Solution prep: heated 1000 ml DI  $\text{H}_2\text{O}$  to  $95^\circ\text{C}$  held there for 24 hours

while deaerating with 99.999%  $\text{N}_2$ , thermometer SN# 9253474 cal 6/23/01

- added 5.432 g  $\text{PbCl}_2$  Lot# L13K01, OHAUS Precision Standard SN# 2883

cal 3/2/01, continued deaerating while maintaining temp for 5 hours

- allowed solution to cool, added 38.02 ml (6M stock HCl) to  
adjust pH = .501

Fisher Accumet 950 meter SN# 3440 cal 7/20/00, probe 13-620-296 SN# 1100208

- filtered remaining solution

7/9/01

Samuelson

- added 10.164 g  $\text{PbCl}_2$  to solution above SE 7/14/01

- heated solution above while deaerating, then added 20.164 g  $\text{PbCl}_2$   
over the course of 4 hours to create a supersaturated solution  
at  $95^\circ\text{C}$

used same thermometer, scale and  $\text{N}_2$  as above

7/10/01

Samuelson



Cyclic Polarization of C22 in  $\text{PbCl}_2$ , 3600 ppm  $\text{Pb}^{2+}$ , 1400 ppm  $\text{Cl}^-$

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimension on pg #6

with 2 PTFE Crevice washers attached ~~using~~ at 50 in-oz using

Proto 6104 SN# 314047 cal 6/4/01

Start wt: 31.211g

OHAUS Precision standard

END wt: 31.210g

SN# 2883 cal 3/2/01

Solution: prep on pg #45

Solution deaerated with 99.999%  $\text{N}_2$

Start PH = 2.905

Fisher Accumet 950 meter

END PH = 4.671

SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 8238325

Temp: 95°C Therm SN# 9253474 cal 5/23/01

$E_{\text{corr}}$ : -286 mV

Keithley 617 SN# 537418

$E_{\text{pr}}$ : -340 mV

cal 2/22/01

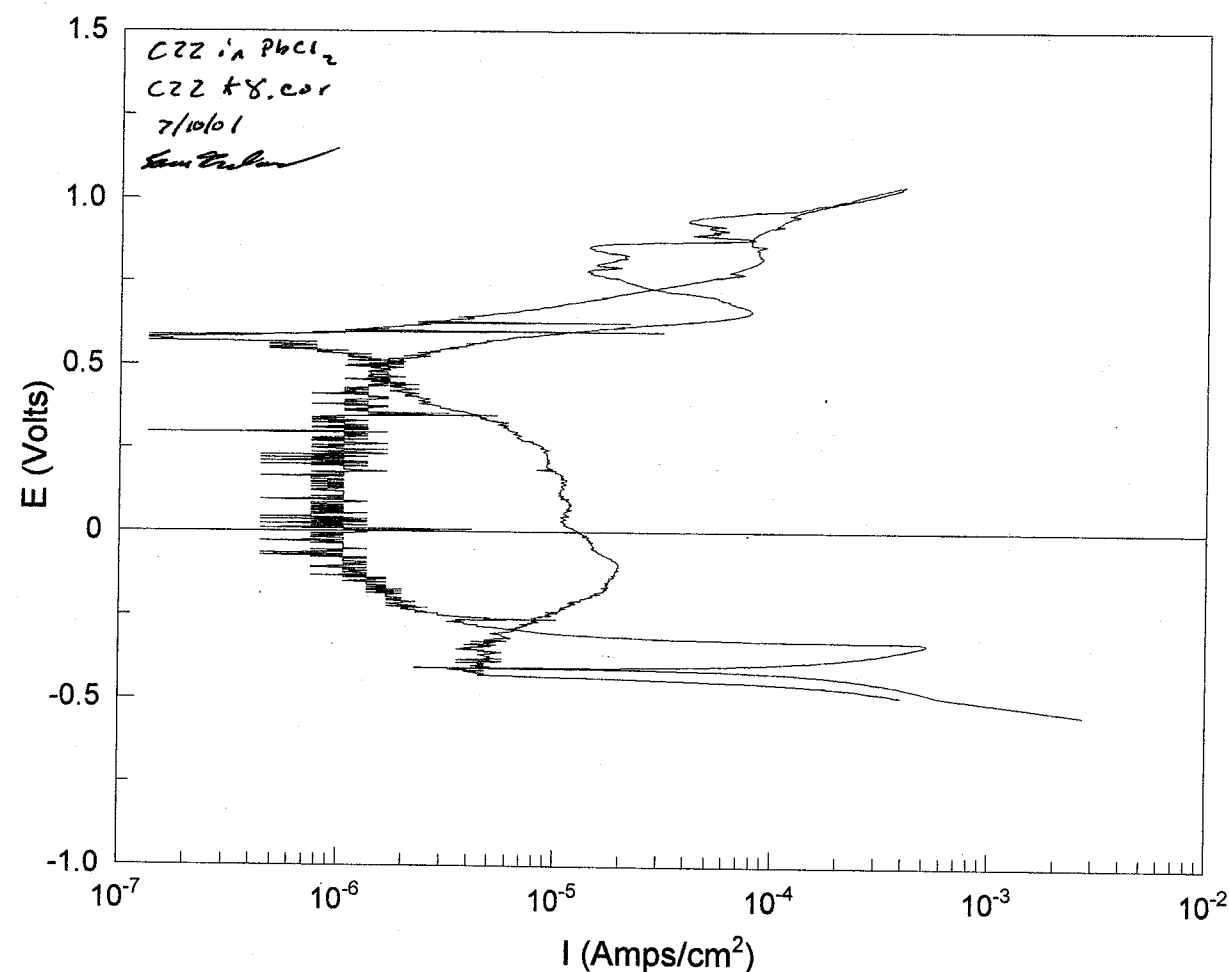
Data File: C22+8.cor

Specimen exam: Crevice corrosion on 24/24 feet

yellow deposit on surface

7/10/01

Sam T. Fisher



7/10/01

Sam T. Fisher

Cyclic Polarization of C22 in PbCl<sub>2</sub> 16300 ppm Pb<sup>2+</sup>, 15990 ppm Cl<sup>-</sup>

Objective: See pg#5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg#6  
with 2 PTFE crevice washers attached at 50µm-oz using  
Proto 6104 SN# 314047 cal 6/4/01

Start wt: 38.68756g Sartorius GENIUS  
END wt: 38.66354g SN# 12809099 cal 5/22/01

Solution: prep on pg#49 solution deaerated with 99.999% N<sub>2</sub>  
Start PH = .501 Fisher Accumet 950 meter  
END PH = <sup>Not</sup> Taken supersat soln SN# 3440 cal 7/20/00  
Taken at Room Temp Probe 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-51 SN# 8077248

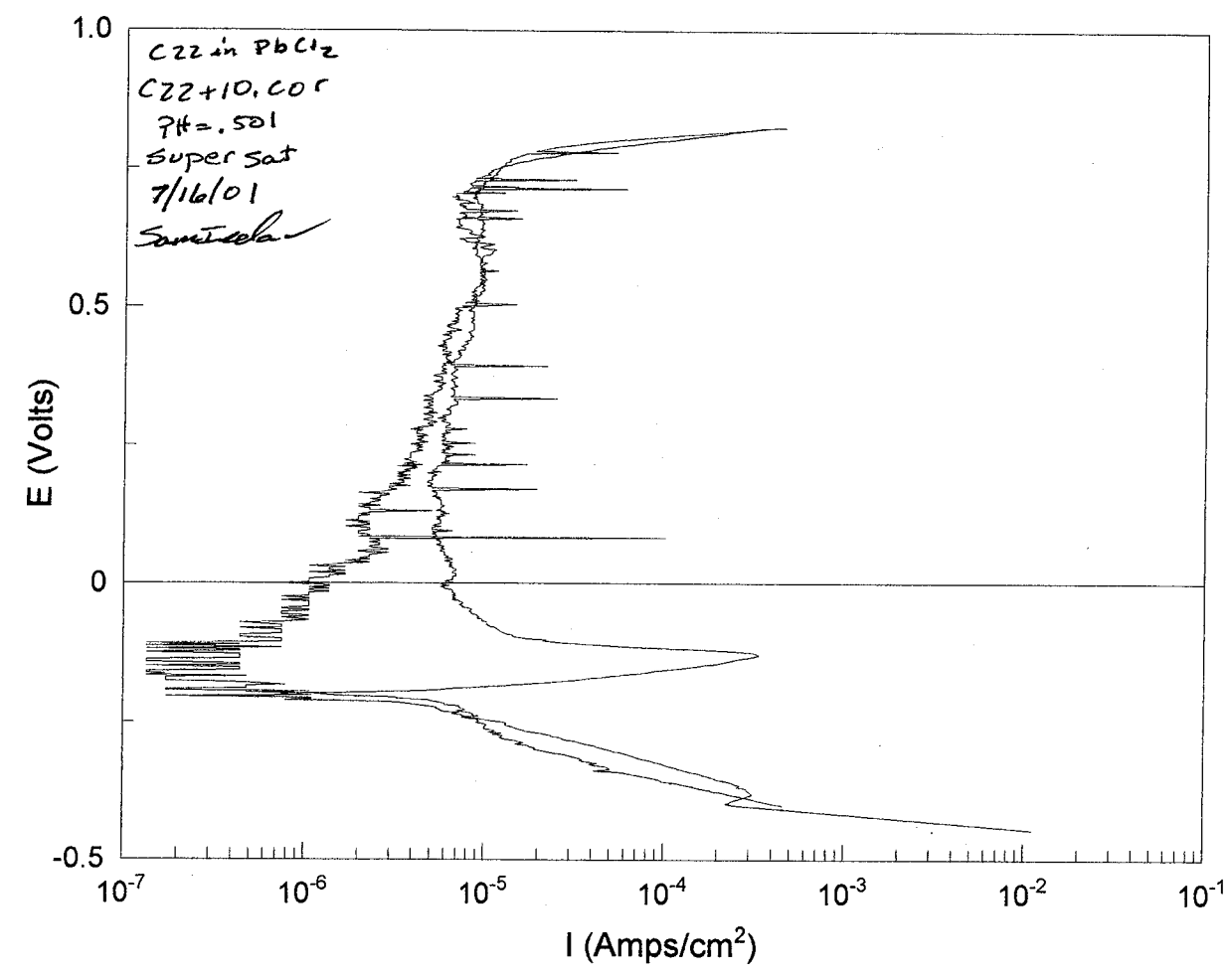
Temp: 95°C Therm SN# 9253474 cal 5/23/01

E<sub>cor</sub>: -204 mV Keithley 617 SN# 537418  
E<sub>pr</sub>: ~~+282 mV~~ -282 mV cal 2/22/01

Data File: C22+10.COR

Specimen Exam: crevice corrosion on 2/24 post  
discolorization

7/16/01  
Sam Pala



7/16/01  
Sam Pala

ISE Free Cl<sup>-</sup> Analysis

objective: measure Cl<sup>-</sup> concentrations using NaCl standard solutions

Meter: Accumet 950 Fisher Scientific SN# 3340 cal 7/20/00

Electrode: Microelectrodes Inc MI-200 SN# 42992

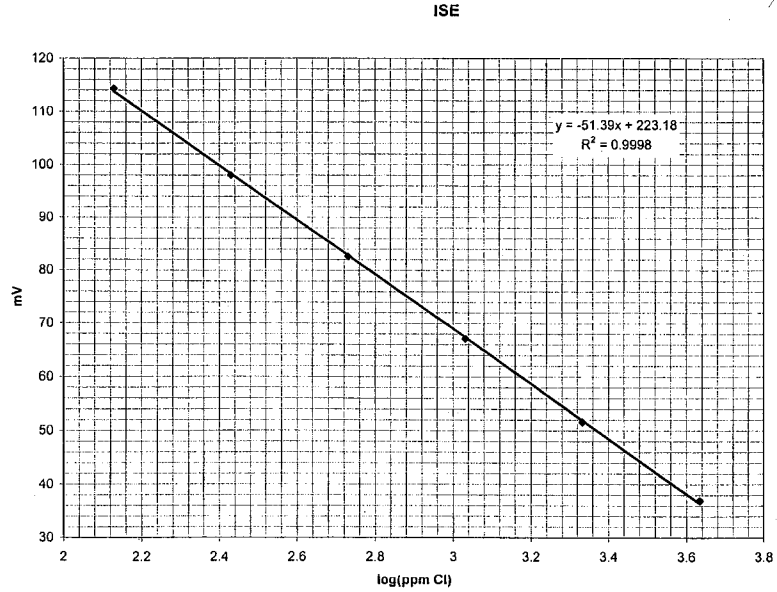
Reference: Fisher 13-620-52 SN# 0052132

Stock solution: 3.55g NaCl + 500 ml DI H<sub>2</sub>O  
consecutive 1/2 ratio dilutions,

Soln PPM Cl<sup>-</sup> SE 7/19/01

soln	ppm	run 1	run 2	avg	log(ppm)
1	4306.9	34.4	39.5	36.95	3.634165
2	2153.45	49.7	53.5	51.6	3.333135
3	1076.72	65.1	69.1	67.1	3.032103
4	538.36	80.5	84.7	82.6	2.731073
5	269.18	96.4	99.6	98	2.430043
6	134.59	112.7	115.9	114.3	2.129013
c22t7		33.1	33.8	33.45	
c22t8		70.9	71.6	71.25	
c22t10		47.6	48.2	47.9	

7/17/01  
Santibanez



7/12/01  
Santibanez

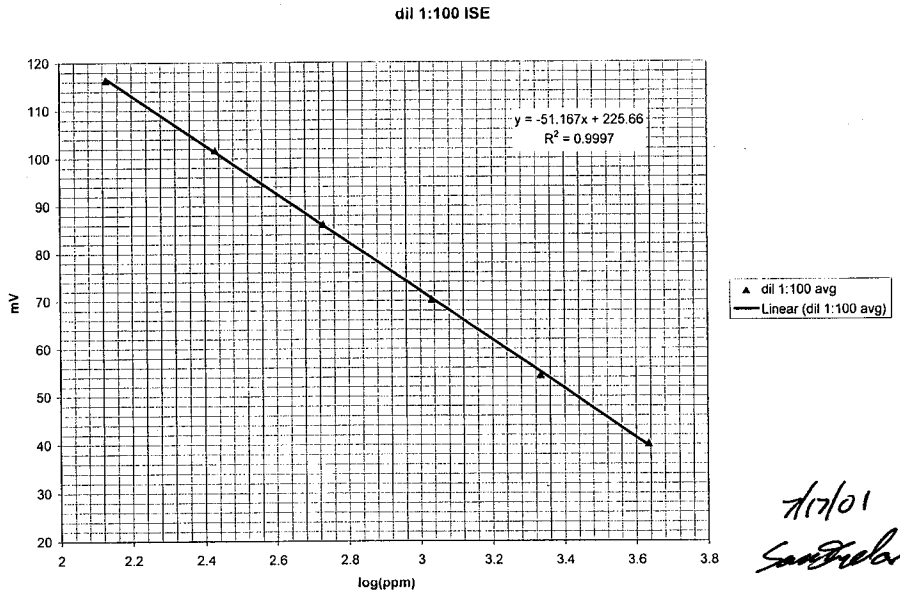
7/18/01  
Santibanez

Sample/test	PPM Cl <sup>-</sup> calc	Remarks
C22+7	4919.98	Sat soln, small amount of precipitate
C22+8	904.51	Sat soln, small amount of precipitate
C22+10	2575.02	Super sat soln, precipitate very large

dilute samples 1/100, after dissolving all precipitate

soln	ppm	run1	run2	avg	log(ppm)
1	4306.9	39.5	40.2	39.85	3.634165
2	2153.45	53.5	54.4	53.95	3.333135
3	1076.72	69.1	70.3	69.7	3.032103
4	538.36	84.7	86.2	85.45	2.731073
5	269.18	99.6	101.8	100.7	2.430043
6	134.59	115.9	116.4	116.15	2.129013
c22t7		122.4	126.4	124.4	
c22t8		154.6	155.3	154.95	
c22t10		112.5	117.2	114.85	

7/17/01  
Santibanez



7/17/01  
Santibanez

7/18/01  
Santibanez

Sample/test	PPM Cl <sup>-</sup>	PH before dilution
C22t7	9528.18	0.531
C22t8	2409.60	3 <sub>st</sub> 2.905 <sup>pp14/1/01</sup>
C22t10	14643.80	0.531, reading taken before super sat condition

Conclusion:

- ISE analysis should not be used for the samples above due to extreme variation in PH. We could not use TISAB to control the pH and bring it to within range of the electrode because of the increased solubility of PbCl<sub>2</sub>.

7/18/01  
S. P. ...

Chloride Concentration Measurements using Capillary Electrophoresis

Objective/ Instrument/ Method/ Data Files : See Page 34

Sample set : Super-saturated PbCl<sub>2</sub> solution with pH 0.5 (Test ID C22t10)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	PbCl <sub>2</sub> , 1:100 dilution	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 3:53:28 PM	Anion Single Method Set
1	1	PbCl <sub>2</sub> , 1:100 dilution	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 3:43:16 PM	Anion Single Method Set
2	2	Spike Cl in 1:100	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:13:53 PM	Anion Single Method Set
2	1	Spike Cl in 1:100	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:03:41 PM	Anion Single Method Set
3	2	PbCl <sub>2</sub> , 1:500 dilution	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:34:19 PM	Anion Single Method Set
3	1	PbCl <sub>2</sub> , 1:500 dilution	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:24:05 PM	Anion Single Method Set
4	2	Spike Cl in 1:500	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:54:42 PM	Anion Single Method Set
4	1	Spike Cl in 1:500	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 4:44:30 PM	Anion Single Method Set
5	2	10 ppm Cl	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 5:15:09 PM	Anion Single Method Set
5	1	10 ppm Cl	Unknown	Supersat PbCl <sub>2</sub> solution low pH	7/16/01 5:04:57 PM	Anion Single Method Set

Sample set : Saturated PbCl<sub>2</sub> solutions (Test ID C22t7, C22t8)

Vial	Injection	SampleName	Sample Type	Sample Set Name	Date Acquired	Acq Method Set
1	2	PbCl <sub>2</sub> , 1:100, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:03:15 AM	Anion Single Method Set
1	1	PbCl <sub>2</sub> , 1:100, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 9:53:03 AM	Anion Single Method Set
2	2	Spike Cl in 1:100, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:23:41 AM	Anion Single Method Set
2	1	Spike Cl in 1:100, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:13:27 AM	Anion Single Method Set
3	2	PbCl <sub>2</sub> , 1:500, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:44:05 AM	Anion Single Method Set
3	1	PbCl <sub>2</sub> , 1:500, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:33:52 AM	Anion Single Method Set
4	2	Spike Cl in 1:500, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:04:30 AM	Anion Single Method Set
4	1	Spike Cl in 1:500, pH0.5	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 10:54:18 AM	Anion Single Method Set
5	2	10ppm Cl	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:24:56 AM	Anion Single Method Set
5	1	10ppm Cl	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:14:44 AM	Anion Single Method Set
6	2	PbCl <sub>2</sub> , 1:100, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:45:23 AM	Anion Single Method Set
6	1	PbCl <sub>2</sub> , 1:100, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:35:10 AM	Anion Single Method Set
7	2	Spike Cl in 1:100, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 12:05:49 PM	Anion Single Method Set
7	1	Spike Cl in 1:100, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 11:55:34 AM	Anion Single Method Set
8	2	PbCl <sub>2</sub> , 1:500, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 12:26:13 PM	Anion Single Method Set
8	1	PbCl <sub>2</sub> , 1:500, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 12:16:01 PM	Anion Single Method Set
9	2	Spike Cl in 1:500, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 12:46:39 PM	Anion Single Method Set
9	1	Spike Cl in 1:500, pH3	Unknown	Saturated PbCl <sub>2</sub> solutions	7/17/01 12:36:27 PM	Anion Single Method Set

J. P.  
7/18/01

Results : Chloride concentration was calculated by integrating the area of the chloride peak and comparing with that of the 10 ppm standard solution

Test Solution	Chloride Concentration (ppm)	
	1:100 Dilution Sample	Spike Sample
C22t10	24,240	26,060
C22t7	7,715	7,784
C22t8	1,277	1,333

*J.P.*  
7/18/01

Solution Prep: heated 2000ml DI H<sub>2</sub>O to 55°C held there for 24 hours while deaerating with 99.999% N<sub>2</sub>  
- added 10.168 g PbCl<sub>2</sub> Lot# L13K01, continued deaerating for 5 hours while maintaining temp at 95°C  
- allowed solution to cool to room temp, then adjusted pH to 2.906 with 6M HCl stock solution Lot# 010166  
- heated solution to 95°C while deaerating with 99.999% N<sub>2</sub> for 4 hours then filtered solution (at 95°C) to create supersaturated solution.

thermometer: SN# 9253474 cal 5/23/01  
scale: OHAUS Precision standard SN# 2883 cal 3/2/01  
meter: Fisher Accumet 950 meter SN# 3440 cal 7/20/00, probe SE *J.P. 7/19/01*  
probe: ~~13-620-92~~ SN# 0089765 *J.P. 7/19/01* 13-620-296 SN# 1100208

7/18/01  
*[Signature]*



Cyclic Polarization of C22 in  $\text{PbCl}_2$  5425 ppm  $\text{Pb}^{2+}$ , 1875 ppm  $\text{Cl}^-$

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #6

with 2 PTFE crevice washers attached at 50 in.-oz using

Proto 6104 SN# 314047 cal 6/4/01

Start wt: 38.389g

Sartorius GENIUS 5T *grip 14/14/01*

END wt: 38.329g

SN# 12809099 cal 5/22/01 *grip 14/14/01*

OHAUS Precision Standard SN# 2883 cal 3/2/01

Solution: prep on pg #59

Solution deaerated with 99.999%  $\text{N}_2$

Start PH = 2.906

Fisher Accumet 950 meter

END PH = <sup>NOT</sup> taken <sup>super sat soln</sup>

SN# 3440 cal 7/20/00

taken at Room Temp

Probe 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 0089765

Temp: 95°C therm SN# 9253474 cal 5/23/01

$E_{\text{corr}}$ : -284 mV

Keithley 617 SN# 537418

$E_{\text{pr}}$ : -80 mV

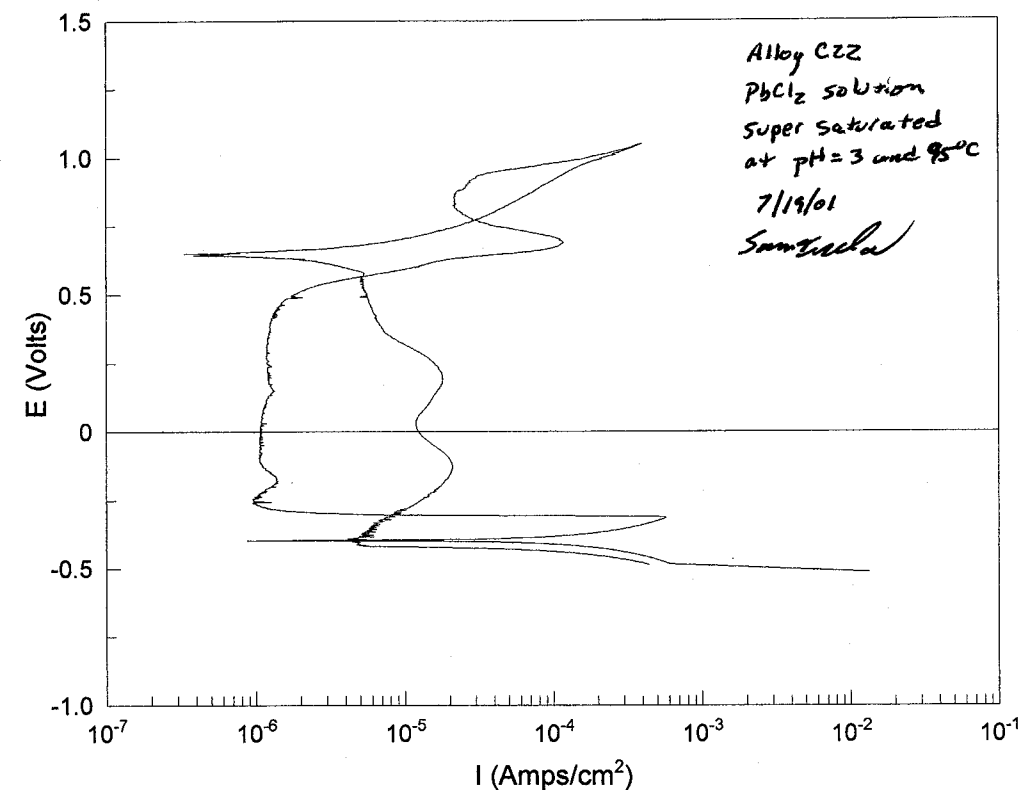
cal 2/22/01

Data File: C22+11.COR

Specimen exam: crevice corrosion on  $\frac{24}{24}$  feet

7/19/01

*Sam Tule*



7/19/01

*Sam Tule*

sent solutions from pg#52 and pg#<sup>60</sup>59 for ICP/IC Analysis. Will log  
Results below. sent on 7/16/01 pg#52 and pg#60 on 7/19/01  
7/20/01 7/20/01  
Santana

Solution pg#52 formed precipitate therefore no dilution. new solution called pg#52A  
Solution pg#60 was a 1/2 dilution  
7/20/01  
Santana

Sample ID	Lab System ID	Lead Result (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.516
True Value	----	0.500
Recovery	----	103.2%
PG # 52A (1:10)	164829	1630
Duplicate result	164829	1635
RPD	164829	0.31%
Spike result	164829	2138
Spike added	164829	500
Recovery	164829	101.6%
PG # 60 (1:2)	164830	2711

Reporting Limit: 0.005 mg/L  
8/5/01  
Santana

Cert 10 (x10)  
Cert 11 (x2)

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	203
True Value	----	200
Recovery	----	101.5%
PG # 52A	164829	1599
Duplicate result	164829	1594
RPD	164829	0.31%
Spike result	164829	1750
Spike added	164829	200
Recovery	164829	75.7%
PG # 60	164830	942

Reporting Limit: 0.1 mg/L  
8/5/01  
Santana

8/5/01  
Santana

Solution prep: heated 4000ml DI H<sub>2</sub>O to 95°C held there for 24 hours  
while deaerating with 99.999% N<sub>2</sub>; 2000ml per cell  
- added 11.504g PbCl<sub>2</sub> to each cell Lot# L13K01, continued deaerating  
for 5 hours while maintaining temp at 95°C  
- allowed solutions to cool to room temp, then adjusted pH to 0.500  
with 6M HCl stock solution Lot# 010166  
- re heated solutions to 95°C and continued deaerating  
- added 20.00g PbCl<sub>2</sub> to each cell and maintained temp for 5 hours  
Thermometer: 7160188 SN# C96-377 cal 7/16/01  
: SN# C96-649 cal 6/26/01

Scale: OHAUS Precision standard  
SN# 2883 cal 3/2/01  
PH Meter: ORION model 720A  
cal 10/25/00 SN# 0033618  
Probe: Fisher 13-620-296 SN# 1100208

7/20/01  
Santana

Potentiostatic test Alloy C22 in  $PbCl_2$  at 0mV

objective: See pg#5

Specimen: Alloy C22 600grit Finish Specimen Dimensions on pg#29

Start wt: 12.440g

OHAUS Precision Standard

END wt: ~~N/A~~ <sup>7/26/01</sup> 12.43152g

SN# 2883 cal 3/2/01

solution: prep on pg#63

start pH = 0.500 at room temp before supersaturated condition

~~Fisher Accumet 950 meter SN# 3440 SE~~ <sup>7/26/01</sup>

Orion model 720A SN# 0033618 cal 10/25/00

Probe: Fisher 13-620-296 SN# 1100208

Potentiostat: Solotion 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-51 SN# 8027166

temp: 95°C Thermometer SN# C96-649 cal 6/26/01

 $E_{corr} = -173mV$ 

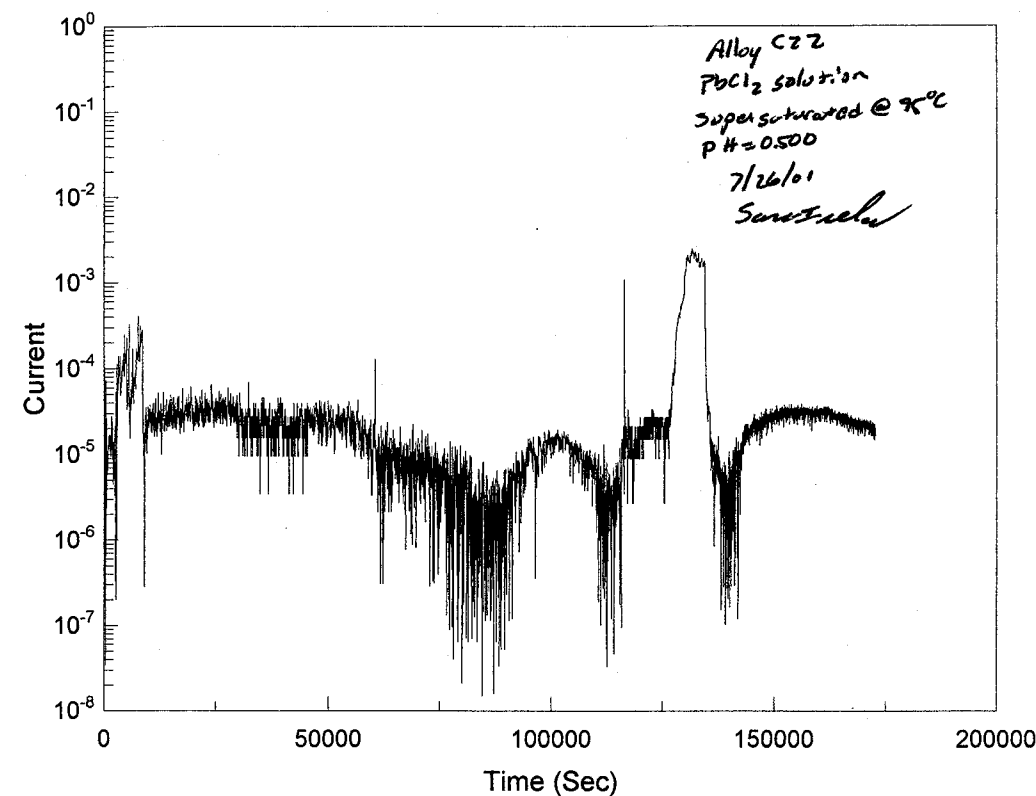
Keithley 617 SN# 537418

 $E_{pr} = -660mV$ 

cal 2/22/01

Specimen exam: ~~N/A~~ <sup>7/26/01</sup> slight discolorization near top of specimen

Data File: C22t20.cor

7/26/01  
Samsuel7/26/01  
Samsuel

Potentiostatic test Alloy C22 in  $\text{PbCl}_2$  at 50 mV

Objective: See pg#5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg#29

St wt: 12.46291 g

Sartorius Genius

END wt: N/A

SN# 12809099 cal 5/22/01

Solution prep on pg#63

Start pH = 0.500 at room temp before supersaturated condition

Orion Model 720A SN# 0033618 cal 10/25/00

Probe: Fisher 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference Electrode: Fisher 13-620-52 SN# 0052132

Temp: 95°C thermometer SN# 9253474 cal 5/23/01

$E_{\text{corr}}$ : -165 mV

Keithley 617 SN# 537418

$E_{\text{PT}}$ : -620 mV

cal 2/22/01

Specimen exam: N/A

Data File: C22t21.cor

Air <sup>prop 10/15/01</sup>

\* ~~Lugan probe format~~ bubble formed in Luggin probe, therefore test was shutdown and restarted see pg# 68

7/27/01

*Sam F. Lee*

7/27/01

*Sam F. Lee*

Potentiostatic test Alloy C22 in  $\text{PbCl}_2$  at 50 mV

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #29

st wt: 12.64667g

Sartorius Genius

END wt: N/A

SN# 12809099 cal 5/22/01

Solution prep on pg #63

Start PH = 0.500 at room Temp before supersaturated condition

Orion Model 720 A SN# 0033618 cal 10/25/00

Probe: Fisher 13-620-296 SN# 1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0052132

Temp: 95°C Thermometer SN# 9253474

$E_{corr} = -161 \text{ mV}$

Keithley 617 SN# 537418

$E_{pr} = -243 \text{ mV}$

cal 2/22/01

Specimen exam: N/A

Data File: C22t21a.cor

\* Potentiostat was not holding potential therefore test was shut down and bandwidth changed, see pg #70

7/28/01

*[Signature]*

7/28/01

*[Signature]*



Potentiostatic test Alloy C22 in  $PbCl_2$  at 50 mV

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #29

St Wt: 12.51341g Sartorius Genius

End Wt: 12.51239g SN# 12809099 cal 5/22/01

Solution prep on pg #63

Start pH = 0.500 at room temp before supersaturated condition

Orion Model 720A SN# 0033618 cal 10/25/00

Probe: Fisher 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-51 SN# 8027166

Temp: 95°C thermometer SN# C96-649 cal 6/26/01

$E_{corr} = -199 mV$

Keithley 617 SN# 537418

$E_{pr} = -520 mV$

cal 2/22/01

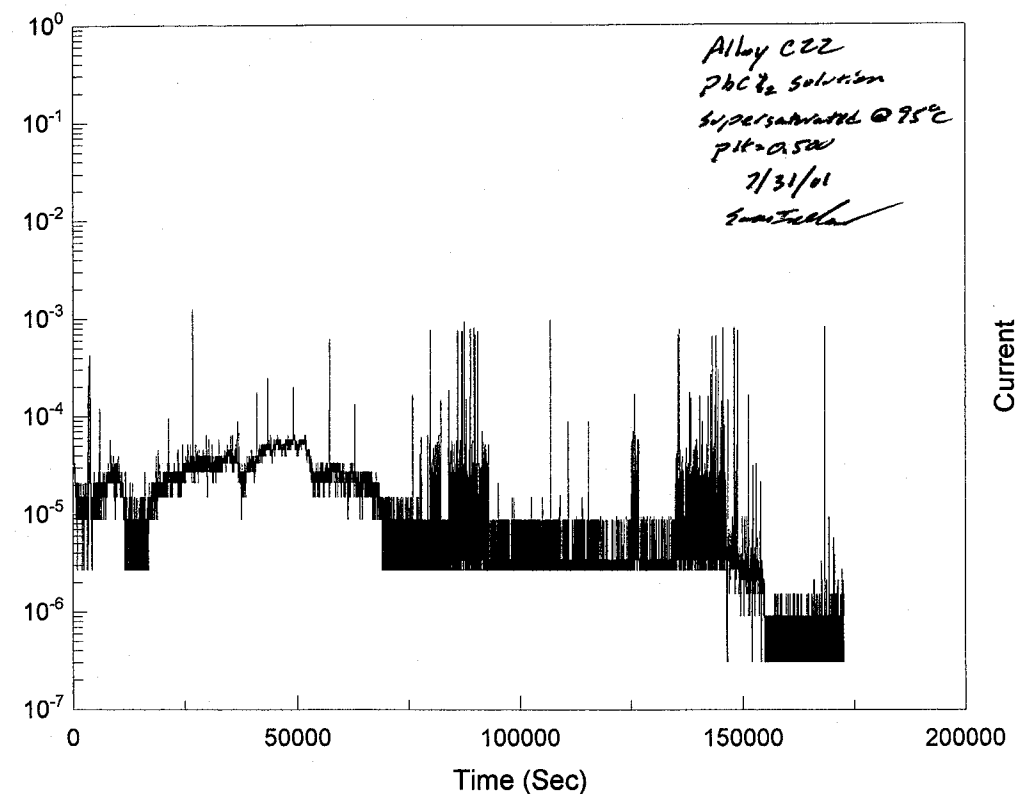
Specimen exam: no visible damage

Data File: C22+22.COR

\* Specimen Repolishes for further Testing 10/10/01 *San Paula*

7/31/01

*San Paula*



Solution prep: heated 2000 ml DI  $H_2O$  to 95°C, held there for 24 hours

while deaerating with 99.999%  $N_2$  thermometer SN# C96-649 cal 6/26/01

- added 10.000g  $PbCl_2$  Lot# L13K01, continued deaerating for 5 hours while maintaining temp at 95°C

- allowed solution to cool to room temp, adjusted pH to 0.506 with 6M HCl (134.4ml Lot# 010166, reheated solution to 95°C and continued deaerating then added 20.000g  $PbCl_2$ , continued heating and deaerating for 24 hours

Scale: OHAUS Precision Standard SN# <sup>ST</sup>225 2883 cal 3/2/01

pH meter: ORION Model 720A cal 10/25/00 SN# 0033618

Probe: Fisher 13-620-296 SN# 1100208

7/31/01

*San Paula*

Potentiostat test Alloy C22 in  $PbCl_2$  at 100 mV

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #29

STWT: 12.52002g Sartorius Genius

ENDWT: 12.51625g SN# 12809099 cal 5/22/01

Solution prep on pg #71

Start pH = 0.506 see pg #71 used 1 liter

potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-51 SN# 8027166

Temp: 95°C thermometer SN# C96-649 cal 2/26/01

$E_{corr} = -233 mV$

Keithley 617 SN# 537418

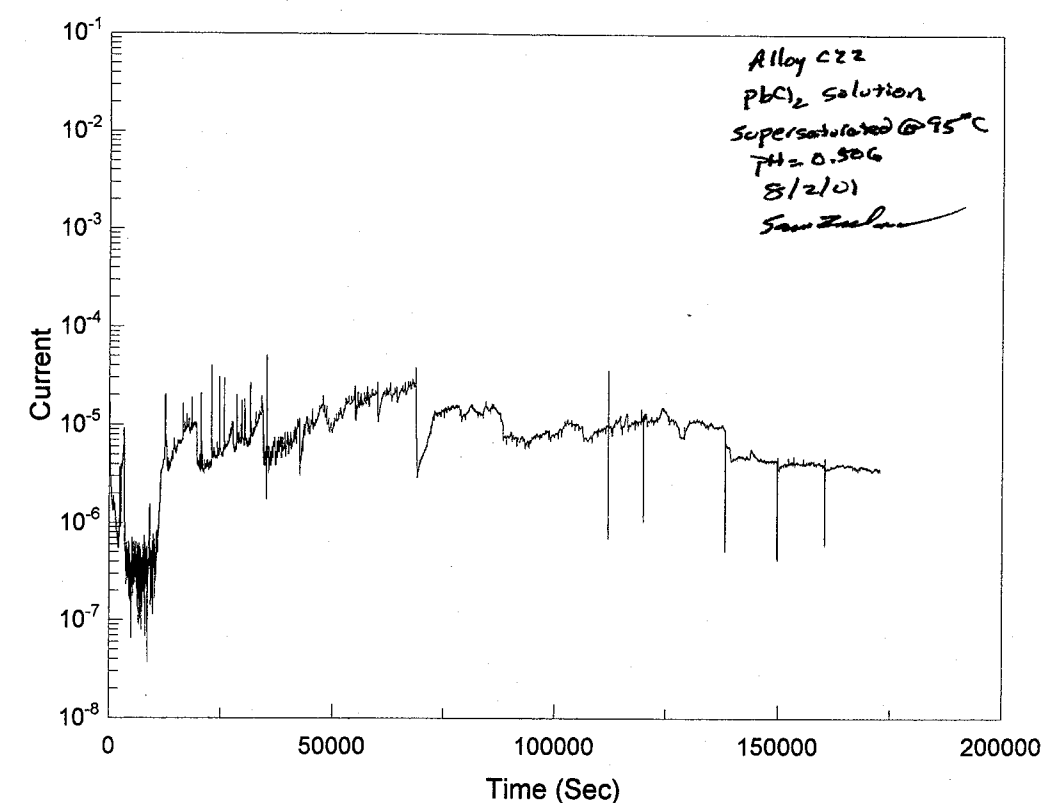
$E_{pr} = -503 mV$

cal 2/22/01

Specimen exam: no visible damage

Data File: C22+23.COR

8/2/01  
San Julian



8/2/01  
San Julian

Potentiostat test Alloy C22 in  $PbCl_2$  at 150 mV

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish

Specimen Dimensions in pg #29

St wt: 12.45279g

Sartorius Genius

End wt: 12.45194g

SN# 12809099 cal 5/22/01

Solution prep on pg #71

Start pH: 0.506 see pg #71 used 1 Liter

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0052132

Temp: 95°C Thermometer SN# 9253474 cal 5/23/01

$E_{corr} = -219 mV$

Keithley 617 SN# 537418

$E_{pt} = -550 mV$

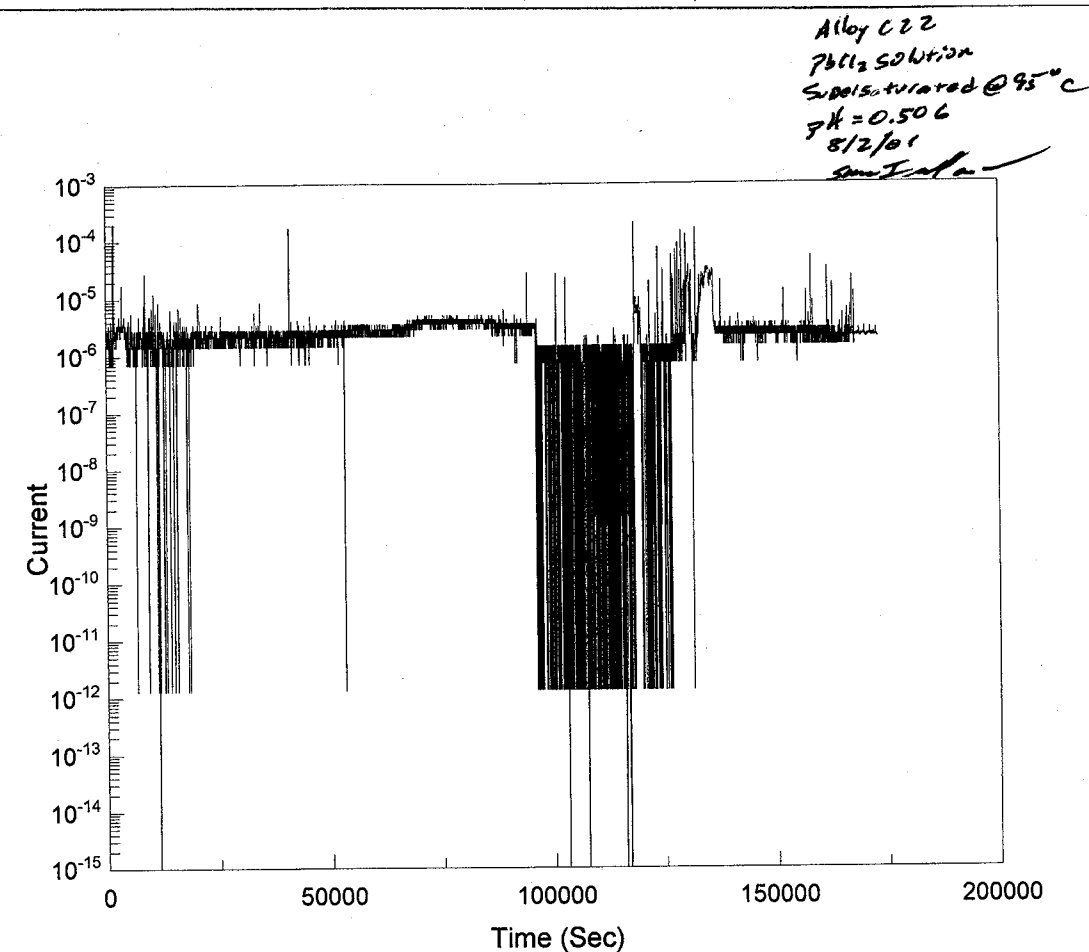
cal 2/22/01

Specimen Exam: No visible damage

Data File: C22+24.COR

\* Specimen Repolishes for further Testing 10/10/01 Bickel

2/2/01  
Bickel



Solution prep: heated 2000 ml DI  $H_2O$  to 95°C, held there for 24 hours while deaerating with 99.9998  $N_2$ , added 10.000g  $PbCl_2$  Lot# L13KD1 continued deaerating for 5 hours while maintaining temp at 95°C - allowed solution to cool to room temp, adjusted pH to 0.504 with 6M HCl (130 ml) Lot# 010166, reheated solution to 95°C and continued deaerating while adding 20.000g  $PbCl_2$  - heated and deaerated for 24 hours thermometer SN# C96-649 cal 6/26/01

Scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

pH meter: DRION Model 720A cal 10/25/00 SN# 0033618

Probe: Fisher 13-620-296 SN# 1100208

8/2/01  
Bickel

Potentiostat test Alloy C22 in  $PbCl_2$  at 200 mV

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish

Specimen Dimensions on pg #29

st wt: 12.63276 g

Sartorius Genius

END wt: 12.63173 g

SN# 12809099 cal 5/22/01

Solution: prep on pg #75

Start pH = 0.584 see pg #75 used 1 Liter

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0052132

Temp: 95°C thermometer SN# 9253474 cal 5/23/01

$E_{corr} = -237$  mV

Keithley 617 SN# 537418

$E_{pr} = -251$  mV

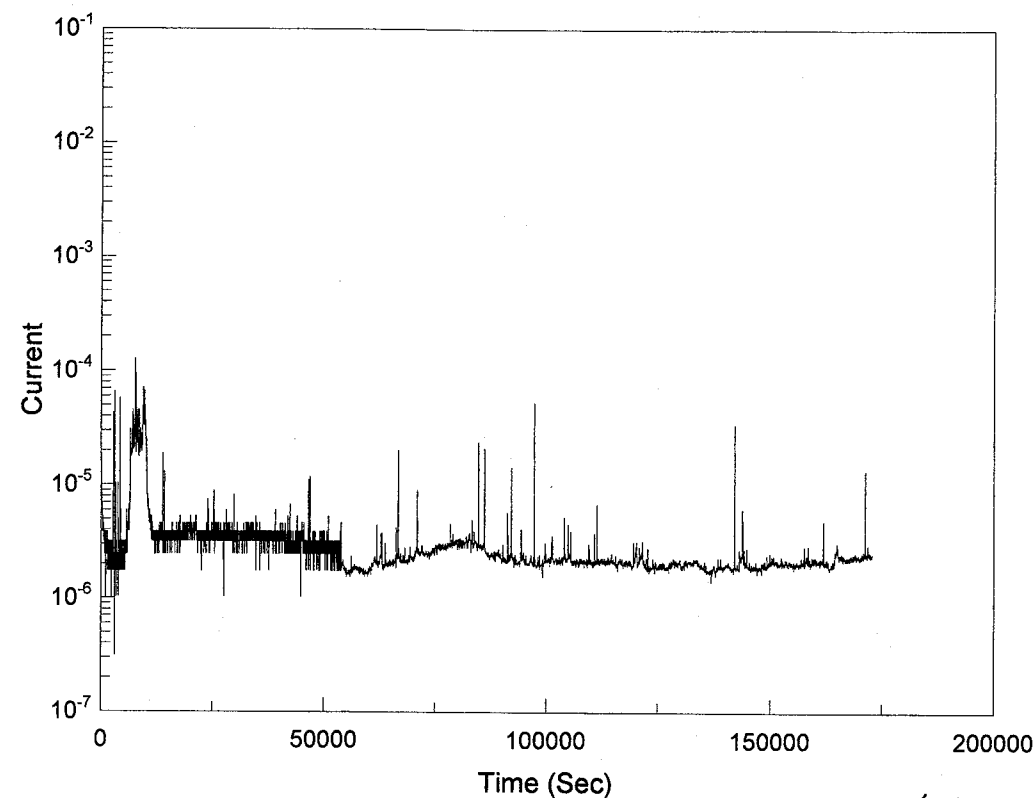
cal 2/22/01

Specimen exam: slight discolorization at top

Data File: C22d25.cor

8/6/01

*SanPaula*



8/6/01

*SanPaula*

8/6/01

*SanPaula*

Cyclic Polarization of C22 in NaCl,  $Cl^- = 16000 \text{ ppm}$

Objective: See pg #5

Specimen: Alloy C22 600 grit Finish Specimen Dimensions on pg #6

with 2 PTFE Crevice washers attached at 50 in. oz using

Proto 6104 SN# 314047 Cal 6/4/01

Start wt: 38.904

OHAUS Precision Standard

End wt:

SN# 2883 cal 3/2/01

Solution: added 93.4 ml 6M HCl Lot# 002560 to 2000 ml DI H<sub>2</sub>O  
to adjust pH to 0.501 then added 20.003g NaCl Lot# 006924 <sup>SN# 11574</sup>  
006945

start pH = 0.501

Fisher Accumet 950 meter

END pH =

SN# 3440 cal 7/20/00

Probe 13-620-296 SN# 1100208

Scale: OHAUS Precision Standard SN# 2883 cal 3/2/01

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference Electrode: 13-620-52 Fisher SN# 0089765

Temp: 95°C Therm SN# C96-649 cal 6/26/01

$E_{corr} = -209 \text{ mV}$

Heithley 617 SN# 537418

$E_{pot} = -370 \text{ mV}$

cal 2/22/01

\* Air in Luggin probe - repeated scan

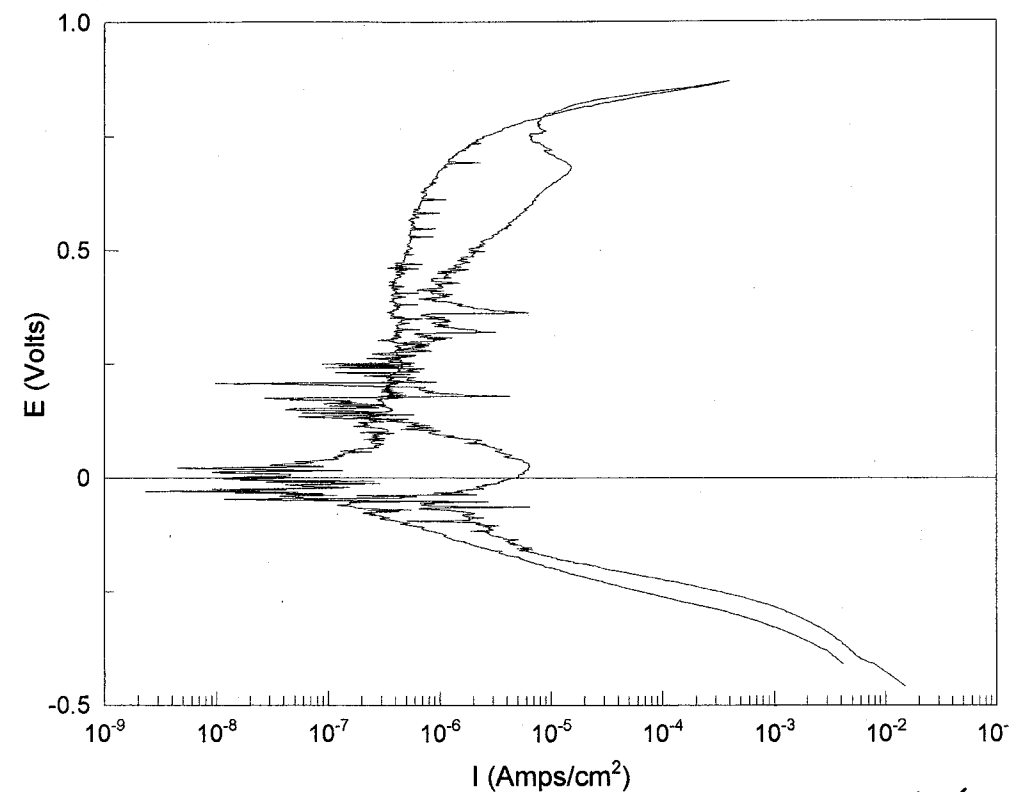
Data File: C22+30.cor

C22+30a.cor

Specimen exam: <sup>unpolished</sup> no visible corrosion / slight crevice corrosion on 2/24 feet

8/10/01

Samuel



8/10/01

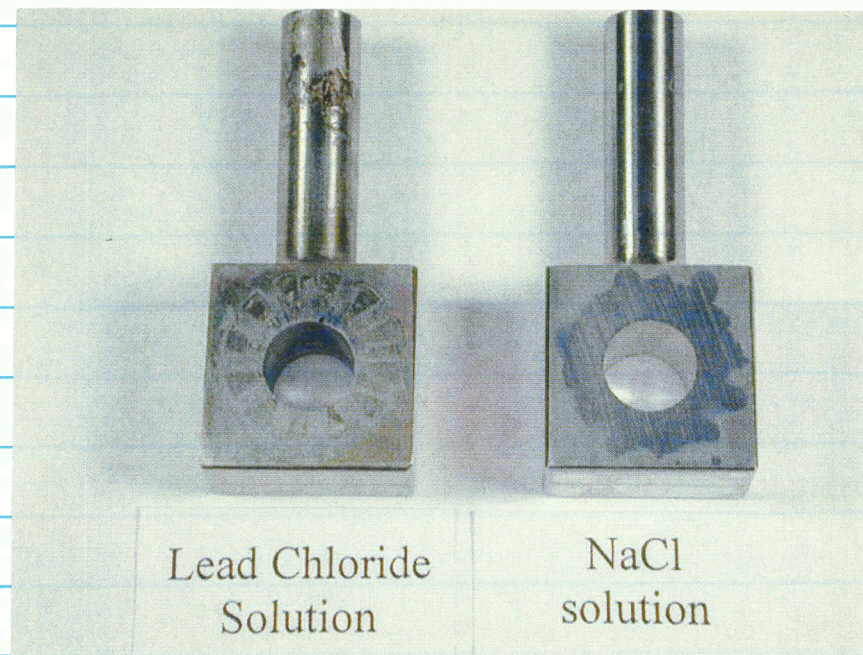
Samuel

8/10/01

Samuel



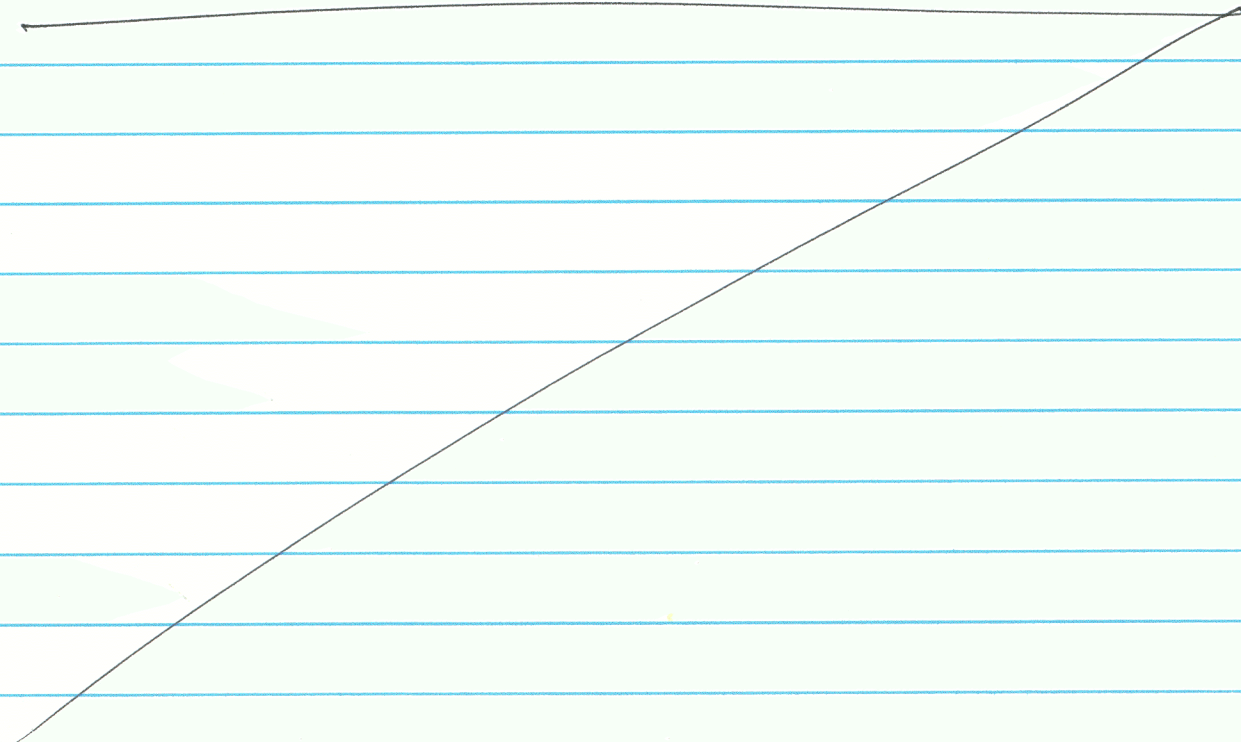
Photograph of Alloy 22 Crevice Specimens



C22t10  
specimen

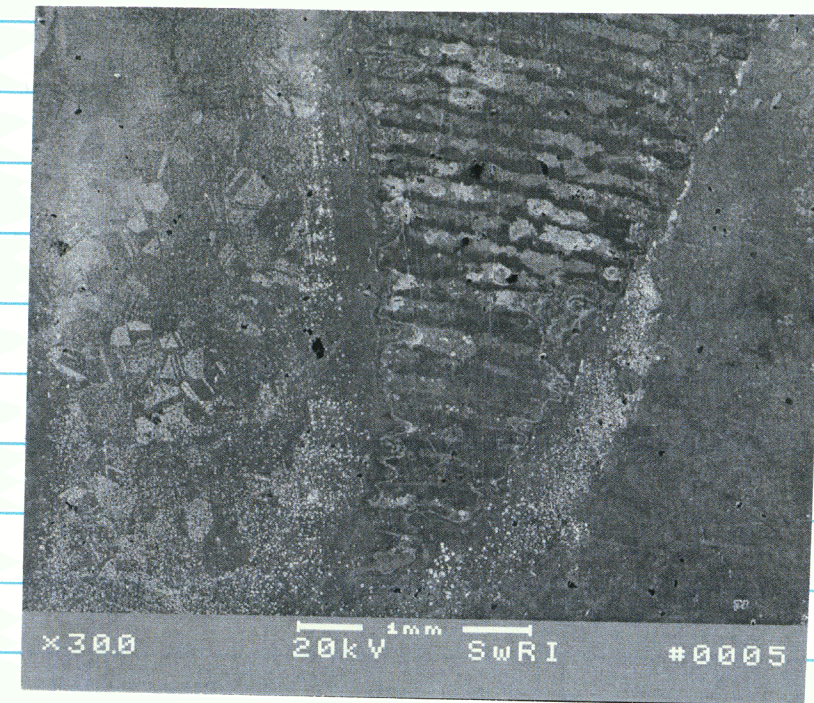
C22t2  
specimen

*Ji Pa*  
8/10/01

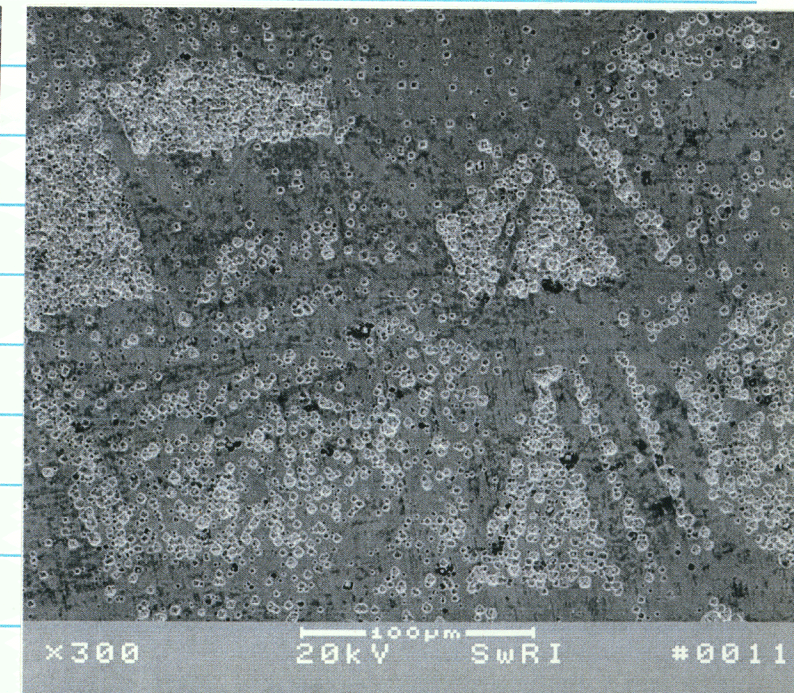
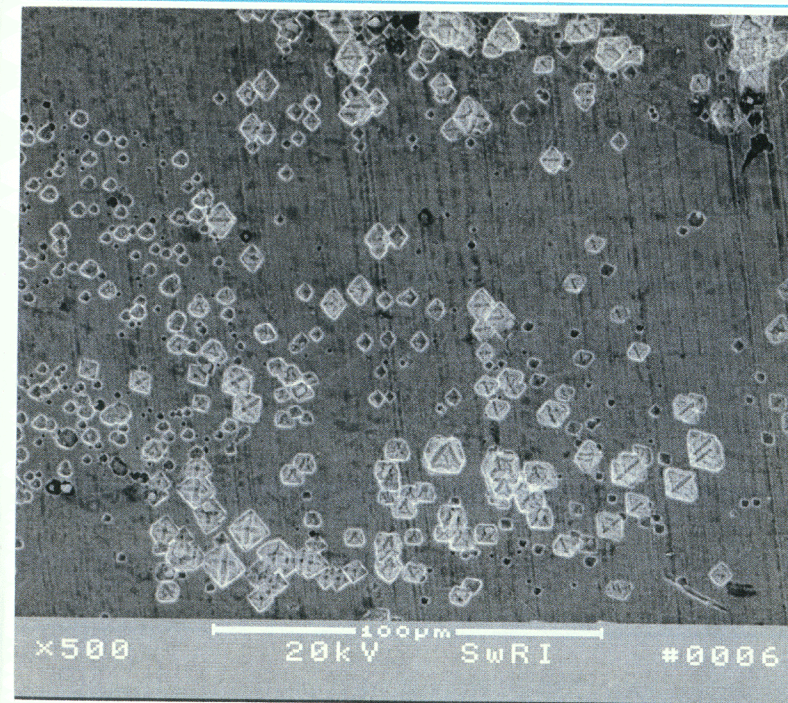


SEM Micrographs of C22t10 specimen

1. Surface attack at one crevice area



2. Enlarged regions showing etch pitting



*Ji Pa*  
8/10/01

6-month Cell  
9/14/01



# Potentiostatic Test Alloy C-22 cylinder in $PbCl_2$ at $-100mV$

Objective: See pg #5

Specimen: Alloy C-22 cylinder Specimen Dimensions on pg #29 600 Grt Finish

Start wt 12.31731 g Santaricus Genus SN#12809099 Cal 5/22/01

End wt 12.28901 g

Solution: Preparation on pg #75 (Solution was 1 month old)

Start PH .493 Fisher Accumet 950 meter SN#3340 Cal 7/24/01

End PH Not Taken Probe 13-620-296 SN#1100208

Potentiostat: Solatron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# <sup>812 9/25/01</sup> 0052132 0042119

Temperature: 95°C Thermometer SN# A2000-123 Cal 2/23/02

Bubbles with 99.999%  $N_2$  for Deaeration

Ecorr = -197 mV Keithley 617 SN#537418 Cal 2/22/02

Ept = +271 mV

E Applied -100 mV

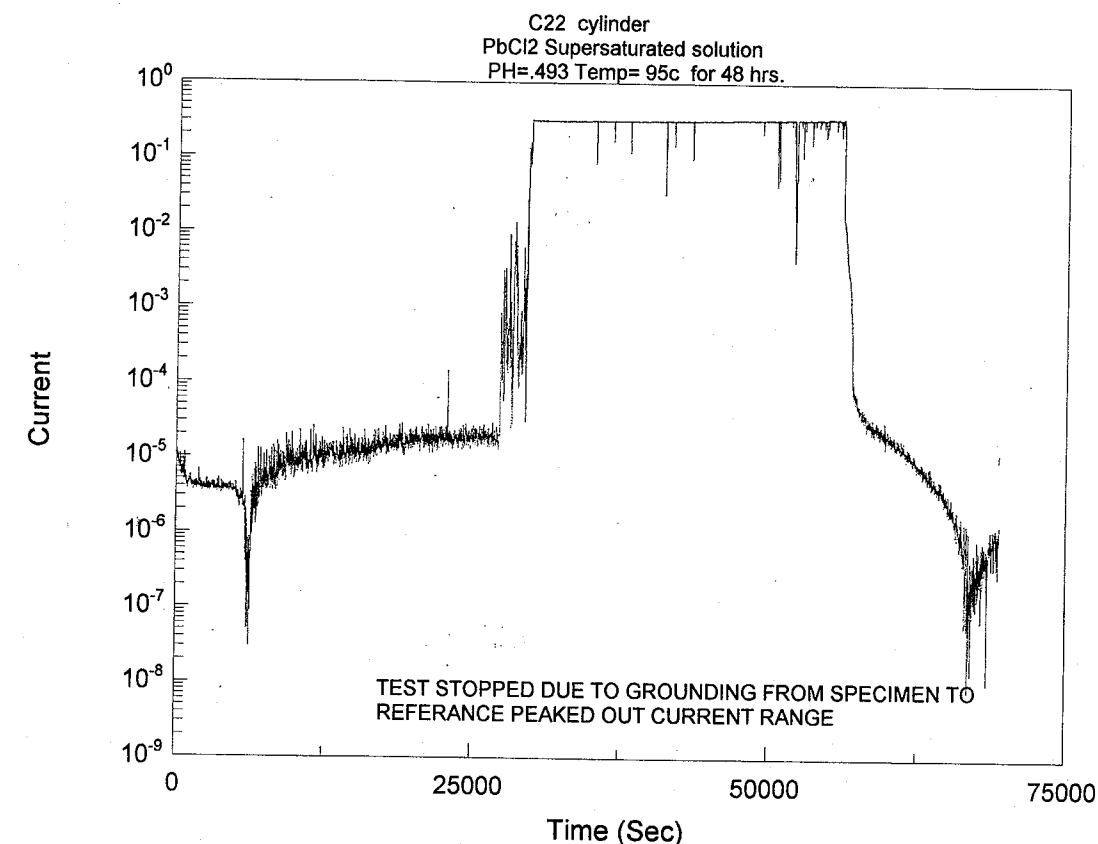
Specimen Examination: slight build up of material at solution level. will Repolish  
And Restart Test with New Solution. Because of problem that develops from  
material buildup on specimen that spikes current on test procedure

Data Saved AS <sup>8/22/01</sup>

Test C22T38 - Bubble In Luggin Probe Restart

Test C22T37a - Buildup of material on specimen shorts Luggin Probe  
Green All the way from Specimen To Surrounds Luggin Probe <sup>8/22/01</sup>

*B. J. D.*  
9/25/01



*B. J. D.*  
9-27-01

New Solution prep: Heated 2000mls DI water To 95°C

Heb @ 95°C for 24 hrs while deaerating with 99.999%  $N_2$

Add 10.000g  $PbCl_2$  Lot# L13K01 - Continues @ 95°C And

deaeration for 5 more hours. - Allowed Solution To Cool To 23°C

Adjusted PH To <sup>812 9/1/01</sup> 0.508 with 6m HCl (85mls of HCl) Lot# 010166

Reheated Solution to 95°C while deaerating with 99.999%  $N_2$

then Add 20.000g  $PbCl_2$  - Continues @ 95°C And deaeration  
for 24 hours.

Thermometer SN# F98-393 Cal 6/26/01

Scale: Ohaus Precision Standard SN#2883 Cal 3/2/01

PH meter: Oton Model #720 <sup>812 10/1/01</sup> Cal Fisher Accumet 950 SN#3340 <sup>Cal</sup> 7/24/01

PH Probe Fisher 13-620-296 SN#1100208

*B. J. D.*  
10/1/01

Potentiostatic Test Alloy C-22 Cylinder in PbCl<sub>2</sub> at -100mV

Objective: See Pg #5

Specimen: Alloy C-22 cylinder - Specimen Dimensions on Pg #29 600 grit Finish

Start wt 12.2805g Sartorius Genius SN#12809099 Cal 5/22/01  
End wt 12.27637g

Solution: Preparation Pg # 83

Super Saturated PbCl<sub>2</sub>

Start PH = 0.508 Fisher Accumet 950 meter SN# 3340 Cal 7/24/01  
End PH = 0.958 Probe PH 13-620-296 SN# 1100208

Potentiostat: Solotek 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0642119

Temperature: 95°C Thermometer SN# A2000-123 Cal 2/23/01

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

<sup>on 10/4/01</sup>  
E<sub>corr</sub> = -197mV -119mV Keithley 617 SN# 537418 Cal 2/22/01

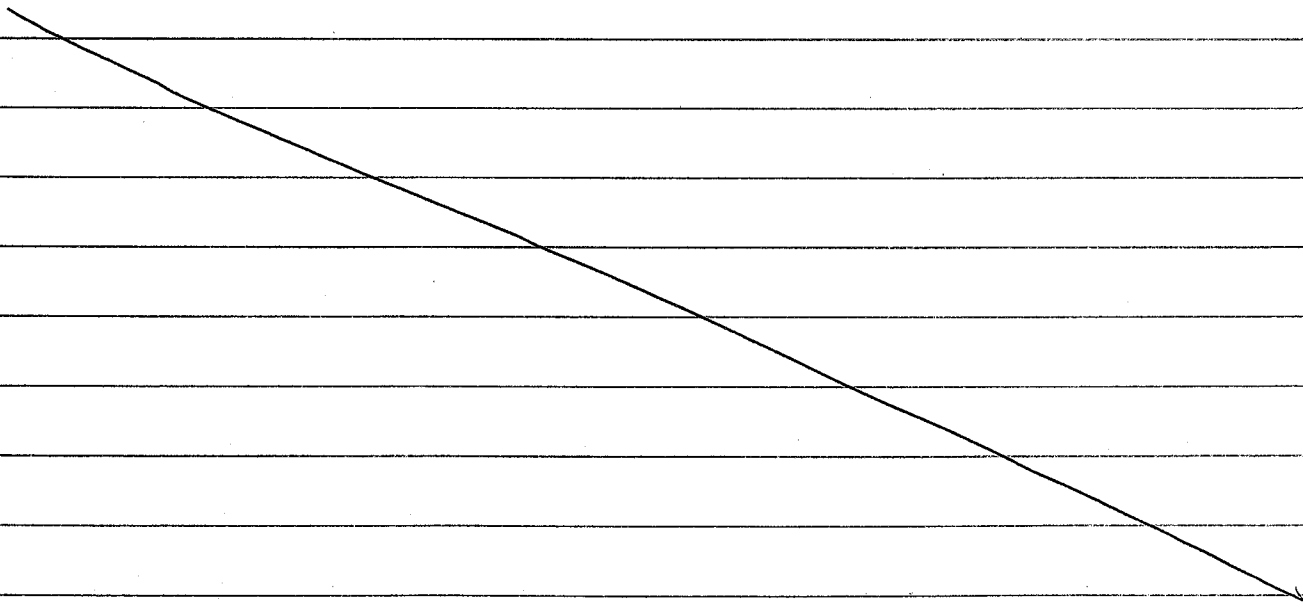
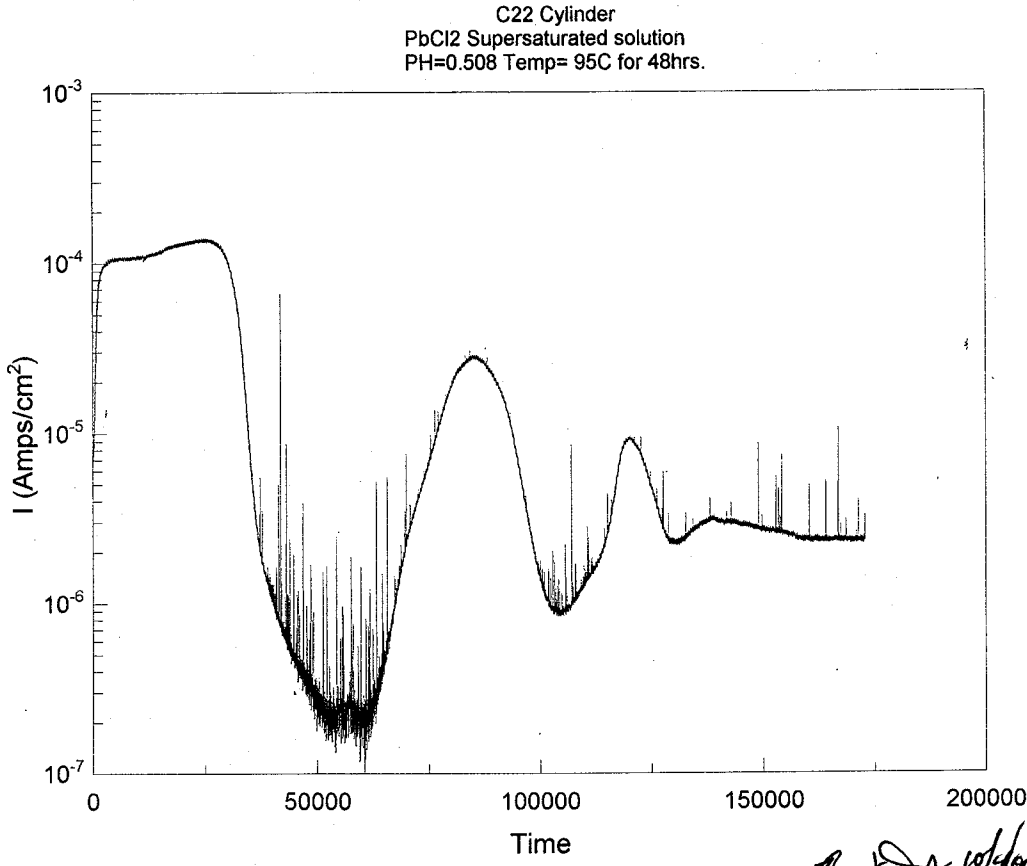
<sup>on 10/4/01</sup>  
E<sub>pt</sub> = +271mV +342mV

E Applied = -100mV

Specimen Examination - Specimen showed lots of Pitting and Corrosion lines  
Down the length of Specimen (Under Microscope)

Data Saved As C22T32

B. E. D. A.  
10-2-01



B. E. D. A.  
10/4/01

## Potentiostatic Test Alloy C-22 Cylinder in NaCl at -100mV

Objective: See Pg #5

Specimen: Alloy C-22 Cylinder Specimen Dimensions on Pg #29 600 Grt Finish

Start wt: 12.31887g Sartorius Genius SN# 12809099 cal 5/22/01

End wt: 12.23123g

Solution: Make NaCl Solution of Equivalent  $\text{Cl}^-$  and PH as C22+32 Test16000 ppm  $\text{Cl}^-$  Solution = 7.83g NaCl Lot # 015568

52.6 mls of Premix 6M HCl Lot # 002560

+ DI water To 1000 mls

Start PH: 0.516 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

End PH: 0.475 Probe 13-620-296 SN# 1100208

Potentiostat: Solotran 1287

Counter Electrode: PT Flag

Reference: Fisher 13-620-52 SN# 0042119

Temperature: 95°C Thermometer SN# A2000-123 cal 2/23/01

Deaerates with 99.999%  $\text{N}_2$ 

Ecorr: -169 mV Keithley 617 SN# 537418 cal 2/22/01

Ept: +472 mV

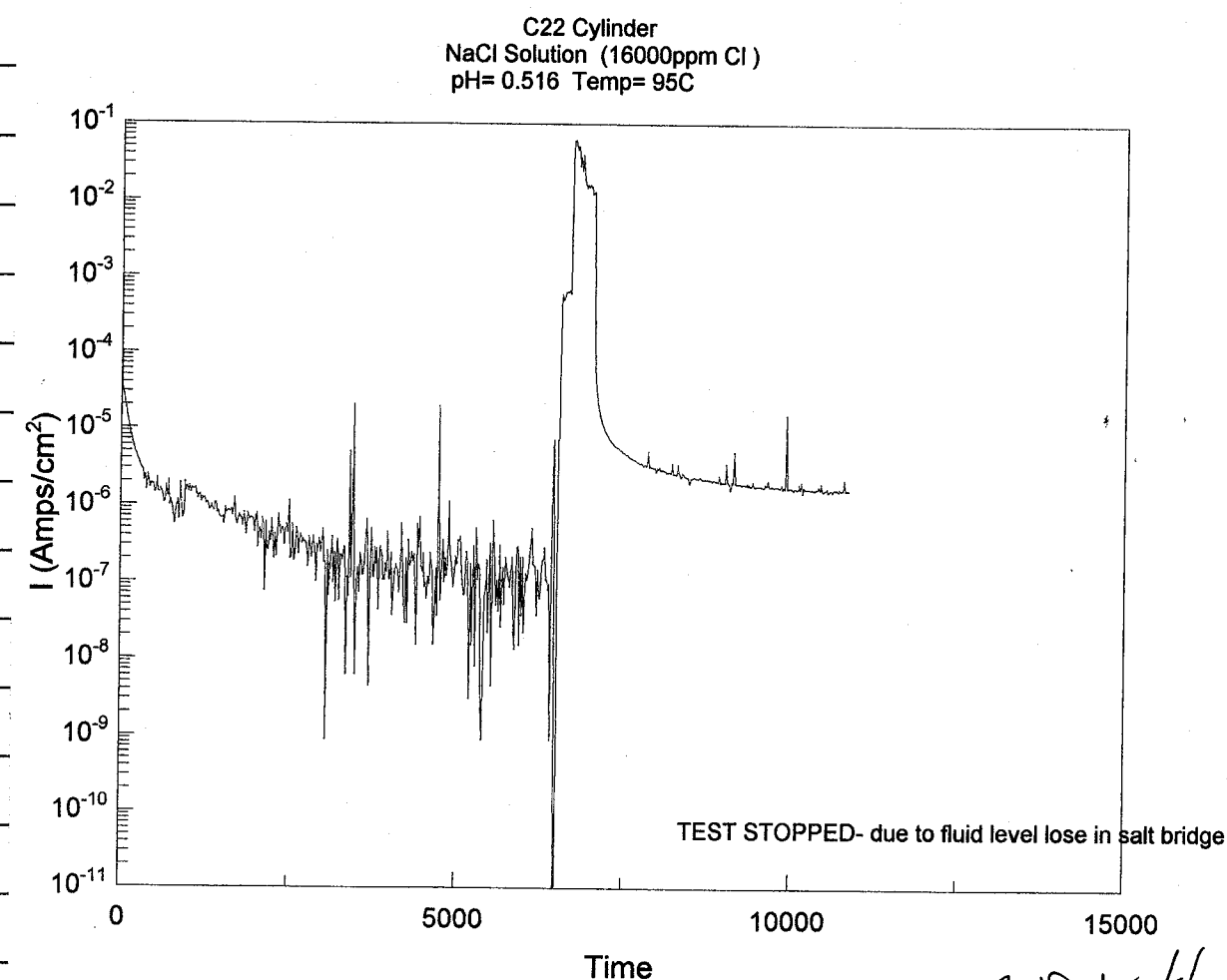
E Applies: -100 mV

Specimen Examination - No Visible Signs of Corrosion - Some Bulge at Material At

Solution level - some signs of lines down length of specimen

C-22 T33.cor - Test stopped due to fluid/solution  
Level Drop In Salt Bridge Lost Connection

B. J. J. 10/9/01



B. J. J. 10/9/01

B. J. J. 10/9/01

## Potentiostatic Test Alloy C-22 Cylinder in NaCl at -100 mV

Objective: See Pg #5

Specimen: Alloy C-22 Cylinder Specimen Dimensions on Pg #29 600 Grit Finish

Start wt = 12.06296g Sartorius Genius SN# 12809099 cal 5/22/01

End wt = 12.23123g <sup>new</sup> 12.05813gSolution = Made NaCl Solution of Equivalent Cl<sup>-</sup> And PH as C22+32 Test16000 ppm Cl<sup>-</sup> Solution Prepared As 7.83g NaCl Lot # 05568

52.6 mls of Inchem 6M HCl Lot #

+ DI water To 1000 mls

Start PH = 0.513 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

End PH = 0.522 Probe 13-620-296 SN# 1100208

Potentiostat: Solotron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0042119

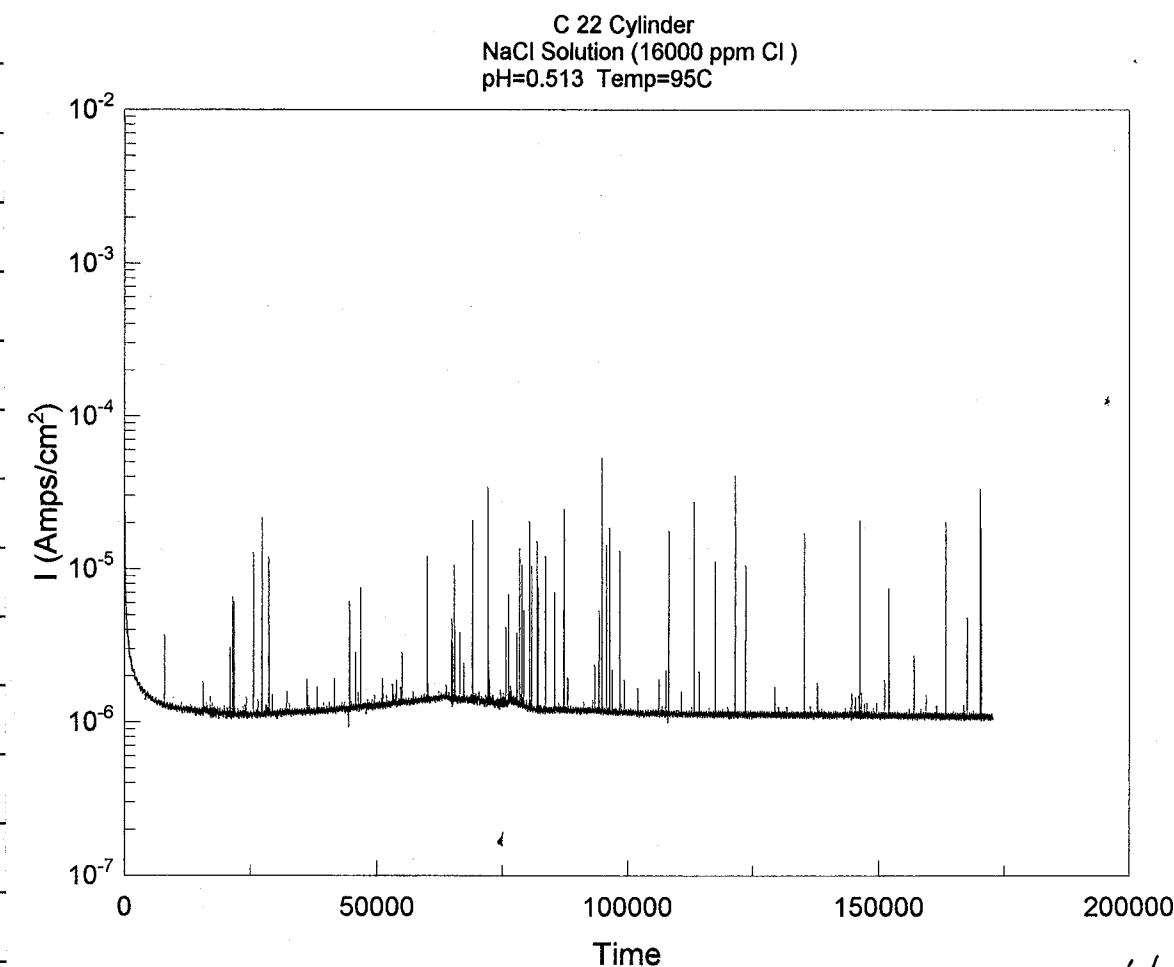
Temperature: 95°C Thermometer SN# A2000-123 cal 2/23/01

Deaerated with 99.999% N<sub>2</sub>E<sub>corr</sub> = -215 mV Keithley 617 SN# 537418 cal 2/22/01E<sub>pt</sub> = +135 mV

E Applied = -100 mV

Specimen Examination: No visible Signs of Corrosion. Bulge of Material @ Solution level

C 22 T 33A.con

B. J. D. J.  
10/10/01

B. J. D. J. 10/12/01

B. J. D. J.  
10/12/01

# Potentiostatic Test Alloy C-22 Cylinder in $PbCl_2$ at $-100mV$

Objective: See Pg #5

Specimen: Alloy C-22 Cylinder Specimen Dimensions on pg #29-600 Grt  
(specimen after +450mV test page 70) & Repolished

Start wt: 12.48651 g Sartorius Genius SN#12809099 Cal 5/22/01

End wt: 12.47976 g

Solution: Preparation on Pg #91

Start PH=0.504 Fisher Accumet 950 meter SN#3340 Cal 7/24/01

End PH=1.021 Probe 13-620-296 SN#1100208

Potentiostat: Solatron 1287

Counter Electrode: PT Flag

Reference: Fisher 13-620-52 SN#0042119

Temperature: 95°C Thermometer SN#A2000-123 Cal 2/23/01

Deaerates with 99.999%  $N_2$

Ecorr = -169mV Keithley 617 SN#537414 Cal 2/22/01

Ept = +218mV

E Applied = -100mV

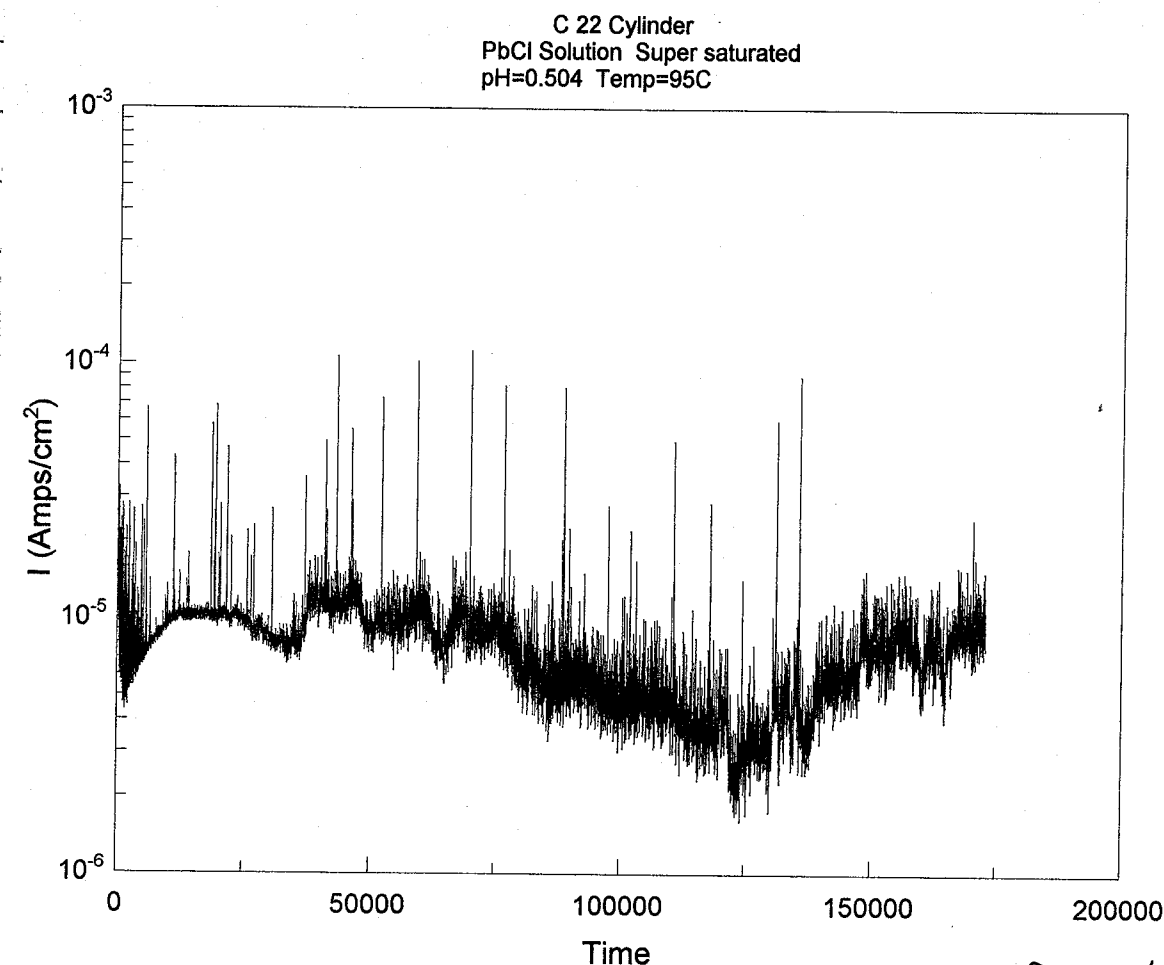
Specimen Examination: Specimen showing No signs of Corrosion Troughs like Previous

Test C22+32: Staining on surface of specimen

Repolished Specimen from Pg #70

C22+34.con

B. K. D. J.  
10/12/01



B. K. D. J.  
10/14/01

Solution Preparation: heated 2000mls DI H<sub>2</sub>O to 95°C Held @ Temp for 24 hours while Deaerating with 99.999%  $N_2$  then Added 10.000g  $PbCl_2$  Lot# L13K01 Continued @ 95°C And Deaeration for 5 hours. Allowed solution To Cool To Room Temp 23°C - Adjusted pH To 0.504 with 113mls of 6m HCl Solution Lot# 956110 Reheated solution to 95°C And Deaerated Solution then Added 20.003g  $PbCl_2$  - Kept @ 95°C And Deaerated for 24 hrs. Thermometer SN# F98-393 Cal 6/26/01

Scale: Ohaus Precision Stannum SN#2883 Cal 3/2/01

pH meter - Fisher Accumet 950 meter SN#3340 Cal 7/24/01

pH probe - Probe 13-620-296 SN#1100208

B. K. D. J.  
10/12/01



Sent Tests: C22+32 for ICP Analysis  
C22+34 for ICP Analysis

\* Note \* that the A solution on Result sheets are from Test solutions which were taken After the Tests were Run

Also All solutions were 1/2 Dilution sent for ICP Analysis

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SwRI      Date Received: 10/16/01  
Matrix: Liquid      Project No.: 20.01402.571  
Work Order: 21118

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.509
True Value	----	0.500
Recovery	----	102%
#32 (1:2)	169470	3739
Duplicate result	169470	3782
RPD	169470	1.14%
Spike result	169470	4183
Spike added	169470	500
Recovery	169470	88.8%
#32A (1:2)	169471	2183
#34 (1:2)	169472	1403
#34A (1:2)	169473	1488

Reporting Limit: 0.005 mg/L

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

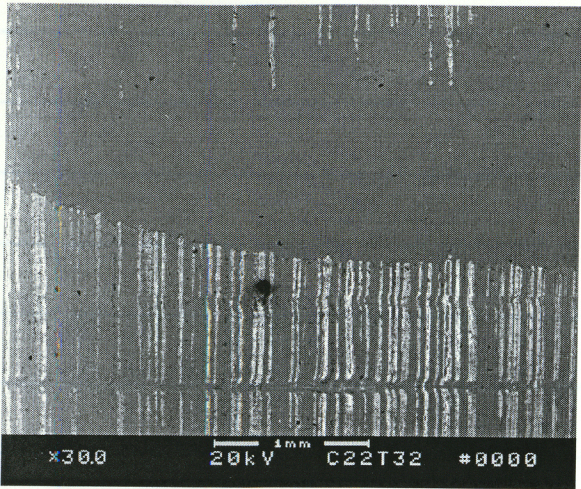
Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SwRI      Date Received: 10/16/01  
Matrix: Liquid      Project No.: 20.01402.571  
Work Order: 21118

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	212
True Value	----	200
Recovery	----	106%
#32	169470	3055
Duplicate result	169470	2990
RPD	169470	2.15%
Spike result	169470	3282
Spike added	169470	200
Recovery	169470	114%
#32A	169471	2782
#34	169472	5497
#34A	169473	7134

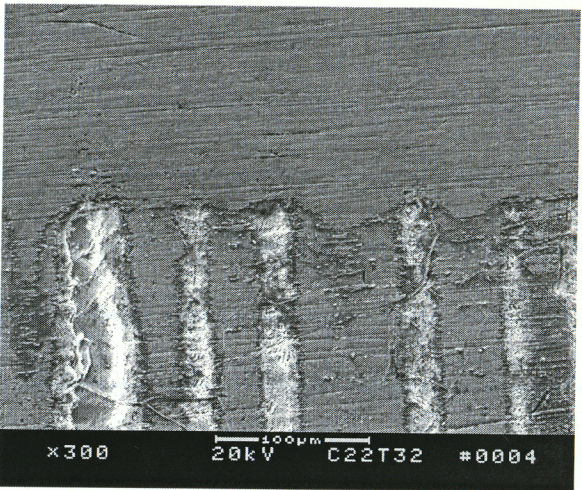
Reporting Limit: 0.1 mg/L

Bi K. [Signature]  
10/24/01

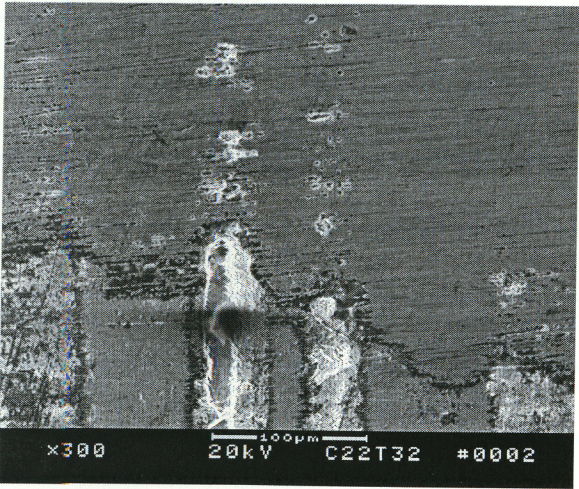
SEM/EDS Analysis of C22+32 specimen



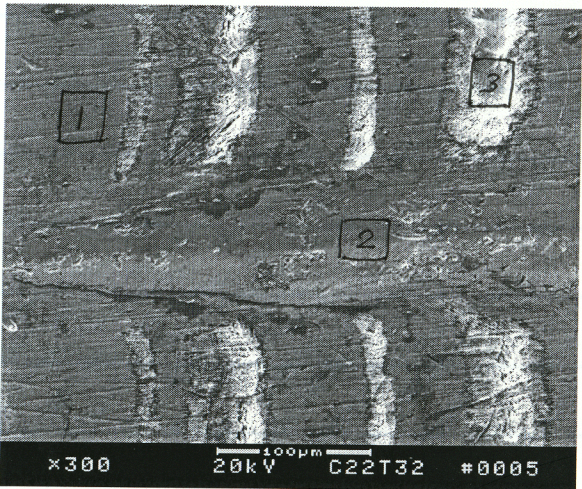
(a) Exposed/unexposed boundary 30x



(b) Enlarged area 1 300x



(c) Enlarged area 2 300x

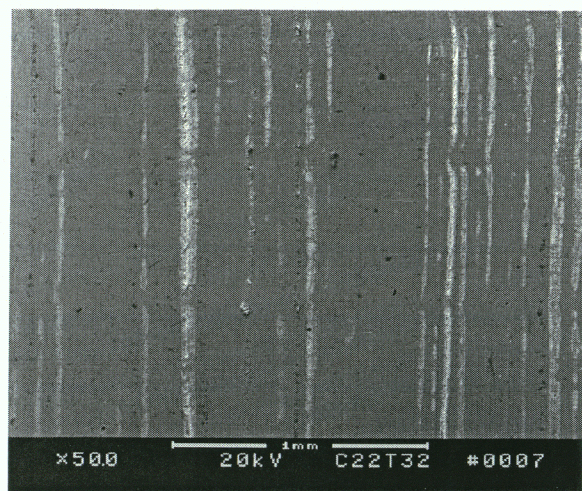


(d) Enlarged area 3 300x

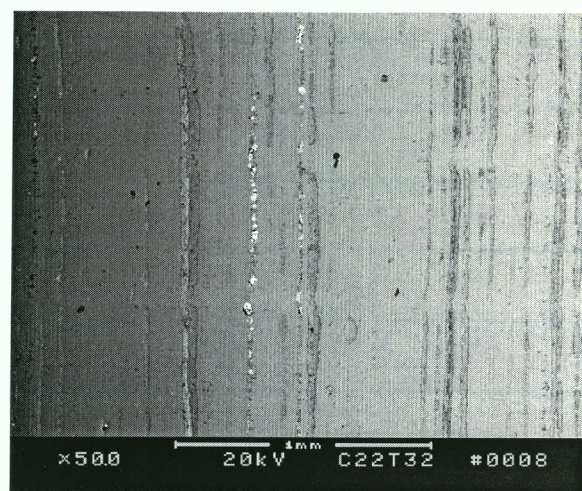
Figure 1. Scanning electron micrographs at the exposed and unexposed boundary of Alloy 22 tested at -100 mV<sub>SCE</sub> in supersaturated PbCl<sub>2</sub> solution at 95 °C for 2 days

[Signature]  
10/25/01

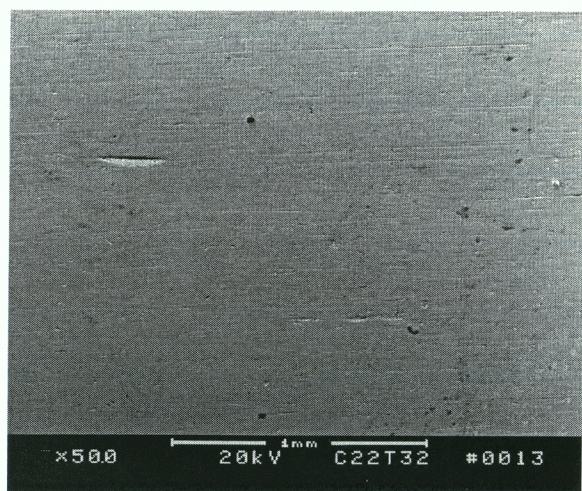




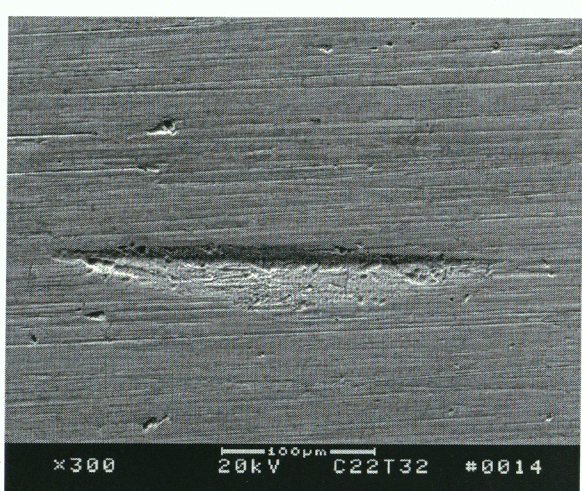
(a) Exposed region 50x



(b) Backscattered image of (a) 50x



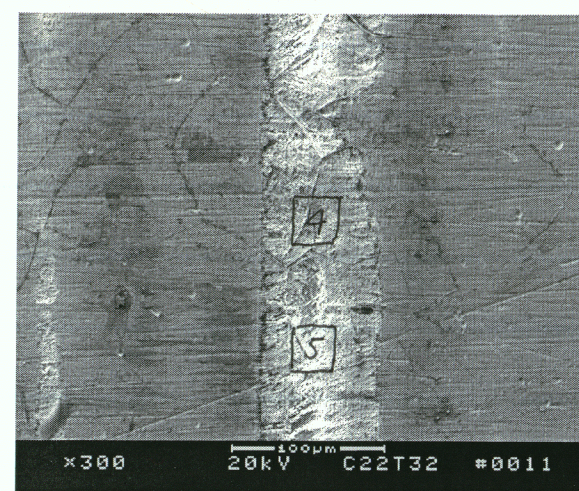
(c) Unexposed region 50x



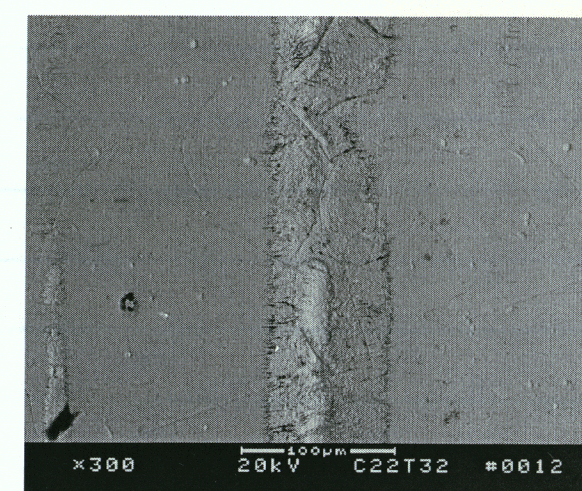
(d) Enlarged area in (c) 300x

Figure 2. Scanning electron micrographs on the exposed region, (a) & (b), and the unexposed region, (c) & (d), of Alloy 22 tested at  $-100 \text{ mV}_{\text{SCE}}$  in supersaturated  $\text{PbCl}_2$  solution at  $95^\circ\text{C}$  for 2 days

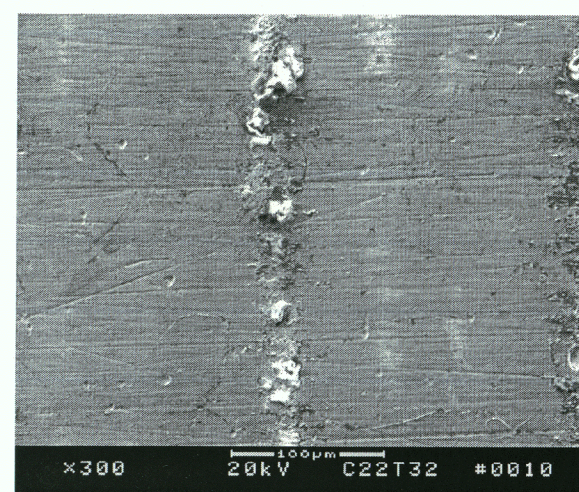
*Yij Pan*  
10/25/01



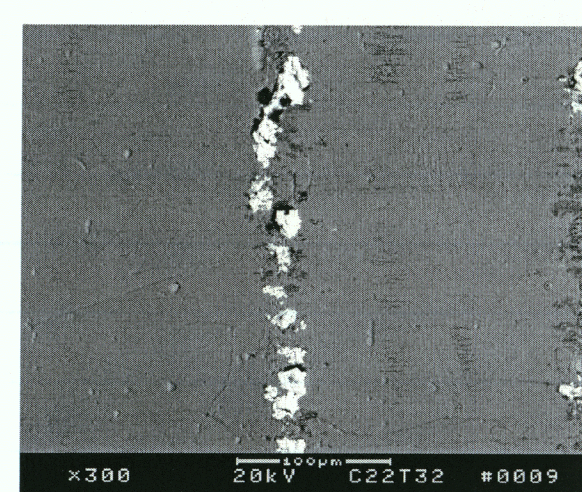
(a) Corrosion trough 300x



(b) Backscattered image of (a) 300x



(c) Pb deposit 300x



(d) Backscattered image of (c) 300x

Figure 3. Scanning electron micrographs on the exposed region of Alloy 22 tested at  $-100 \text{ mV}_{\text{SCE}}$  in supersaturated  $\text{PbCl}_2$  solution at  $95^\circ\text{C}$  for 2 days showing corrosion trough, (a) & (b), and Pb deposit, (c) & (d)

*Yij Pan*  
10/25/01



Table 1. Chemical compositions from various locations on Alloy 22 tested at -100 mV<sub>SCE</sub> in supersaturated PbCl<sub>2</sub> solution at 95 °C for 2 days

Chemical Content (wt%)								Location Remarks
Ni	Cr	Mo	Fe	W	Co	Al	V	
57.6	21.7	12.2	3.93	3.14	0.87	0.32	0.16	Between troughs, location 1 in Fig. 1(d)
57.1	21.8	12.2	3.91	3.45	0.83	0.39	0.21	On machine mark, location 2 in Fig. 1(d)
58.6	21.8	10.8	3.97	3.17	1.02	0.35	0.14	Corrosion trough, location 3 in Fig. 1(d)
65.1	22.1	5.90	3.91	1.50	0.95	0.22	0.19	Corrosion trough, location 4 in Fig. 3(a)
63.9	22.1	6.77	3.81	1.83	0.98	0.33	0.21	Corrosion trough, location 5 in Fig. 3(a)

*Mj Pan*  
10/25/01

New Solution Preparation Procedure - 1000mls 1 Liter Solution

Prec Adjust pH In DI water Start pH = 5.047  
End pH = .500

Adds 59mls of 6m HCl Lot#956110 - pre made solution  
To DI for pH Adjustment

Heats DI water with pH of .500 To 95°C  
while Degenating with 99.999% N<sub>2</sub>  
Also During Heating procedure DI water was stirred

Held At 95°C Degenating And stirring overnight (17 hrs total)  
then Adds 25.00g PbCl<sub>2</sub> Lot# L13K01 all At once  
Held At Temp with PbCl<sub>2</sub> for 6 hrs then Allowed To Cool Down

Solution Cools overnight Re √ PH = .507

\*Note\* Some Small fine Granules still Remain In solution

measurements Taken with Ohaus Precision Standard scale SN# 2883 cal 3/2/01  
pH meter = Fisher Accumat 950 meter SN# 3340 cal 7/24/01  
pH Probe 13-620-296 SN# 1100208  
Thermometer = H<sub>2</sub> thermometer SN# F98-393 cal 6/24/01  
*S. K. J.*  
11/26/01

# Potentiostatic Test Alloy C22 Cylinder In $\text{PbCl}_2$ @ -100mV

Objective: See pg #5

Specimen: Alloy C22 Cylinder Specimen Dimensions on pg #29 600 Grit Finish  
(specimen after the 150 mV test in page 74) & repolished 7/14/01

Start wt: 12.43005g Santaricus Genius SN# 12809099 cal 5/22/01

End wt 12.43388g

Solution: Preparation on pg #97

Start pH = .507 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

End pH = .593 Fisher pH Probe 13-620-296 SN# 1100208

Potentiostat: Solotek 1287

Counter Electrode: PT Flag

Reference: Fisher 13-620-52 SN# 0042119

Temperature: 95°C Thermometer SW# A2000-123 cal 2/23/01

Bubbles with 99.999%  $\text{N}_2$  for Deaeration

Ecorr = -187mV Keithley 614 SN# 467374 cal 10/4/01

Ept = +382mV

E Applies: -100mV

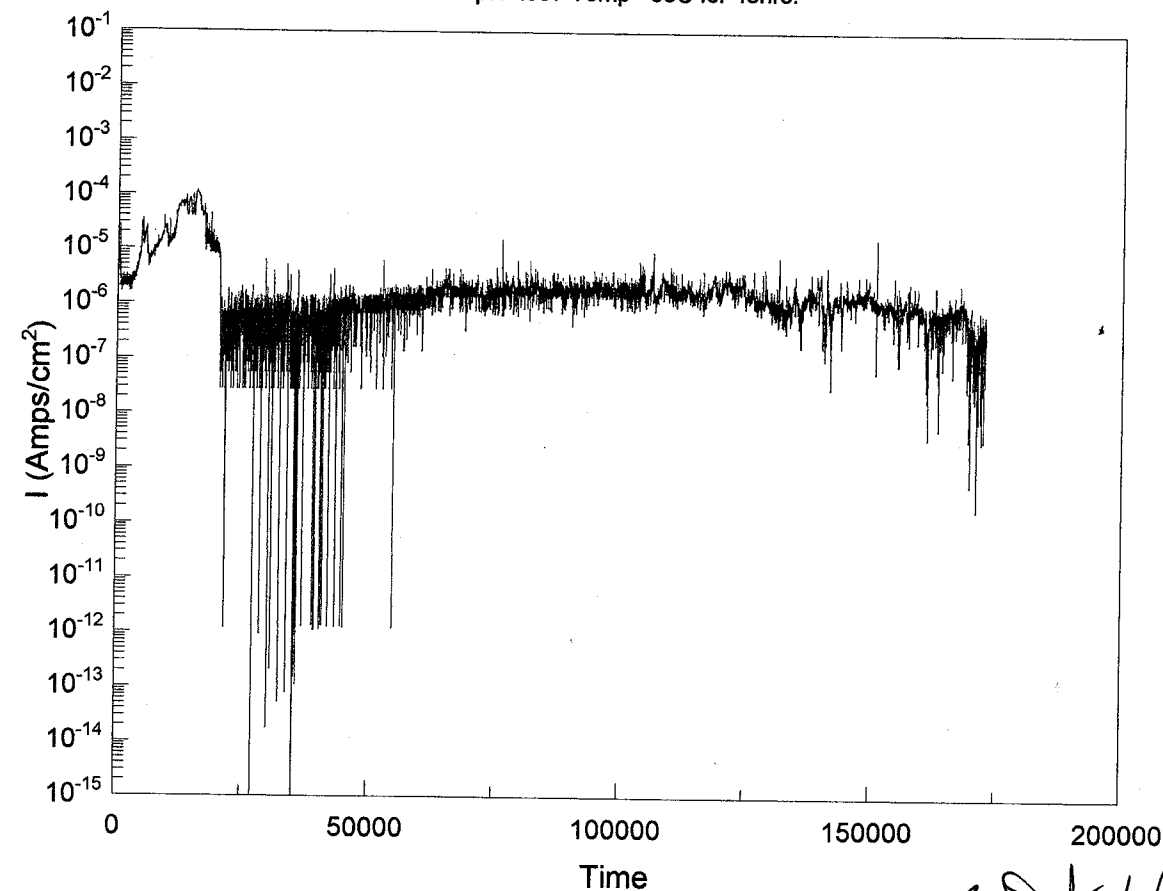
Specimen Examination: Some Buildup of material on specimen - cleaned off  
Some staining on Top Above solution level And on Bottom of Specimen

\* Note lots of evaporation of solution in cell

Test C22-135.con

B. K. Duf  
10/26/01

C22 in  $\text{PbCl}_2$  Super saturated solution  
pH=.507 Temp= 95C for 48hrs.



B. K. Duf  
10/24/01

# Potentiostatic Test Alloy C-22 Cylinder In $PbCl_2$ @ -100mv

Objective: See pg #5

Specimen: Alloy C-22 Cylinder Specimen Dimensions on pg #29 600 Grit Finish

Start wt = 12.33404g Sartorius Genius SN#12809099 cal 5/22/01

End wt = 12.33367g

Solution: Preparation on pg #101

Start pH = .513 Fisher Accumet 950 meter SN#3340 cal 7/24/01

End pH = .437 Fisher pH Probe 13-620-296 SN#1100208

Potentiostat: Solotrac 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN#0042119

Temperature: 95°C Thermometer SN#A2000-123 cal 2/23/01

Deaerated with 99.999%  $N_2$

Ecorr = -215mv Keithley 614 SN#467374 cal 10/4/01

Ept = +393mv

E Applied: -100mv

Specimen Examination: Some Buildup of material on specimen At Solution

line - slight staining on Top of Specimen Above Solution level

Test C22+36.con

B. K. D. J.  
10/31/01

Solution Preparation Procedure - 1000mls DI 1 Liter Solution

Pre Adjust pH In DI water Start pH = 5.047

End pH = .502

Add ed 63mls 6m HCl Lot #956110 - prepare solution

To Adjust pH In DI water

Heats DI water with pH of .502 To 95°C while Deaerating with  $N_2$

Also stirs solution during heating Procedure - Held At Temp for 17hrs

then Added 20.006g  $PbCl_2$  Lot #L13K01 All At Once

Held At Temp with  $PbCl_2$  for 6hrs then Cools To Room Temperature

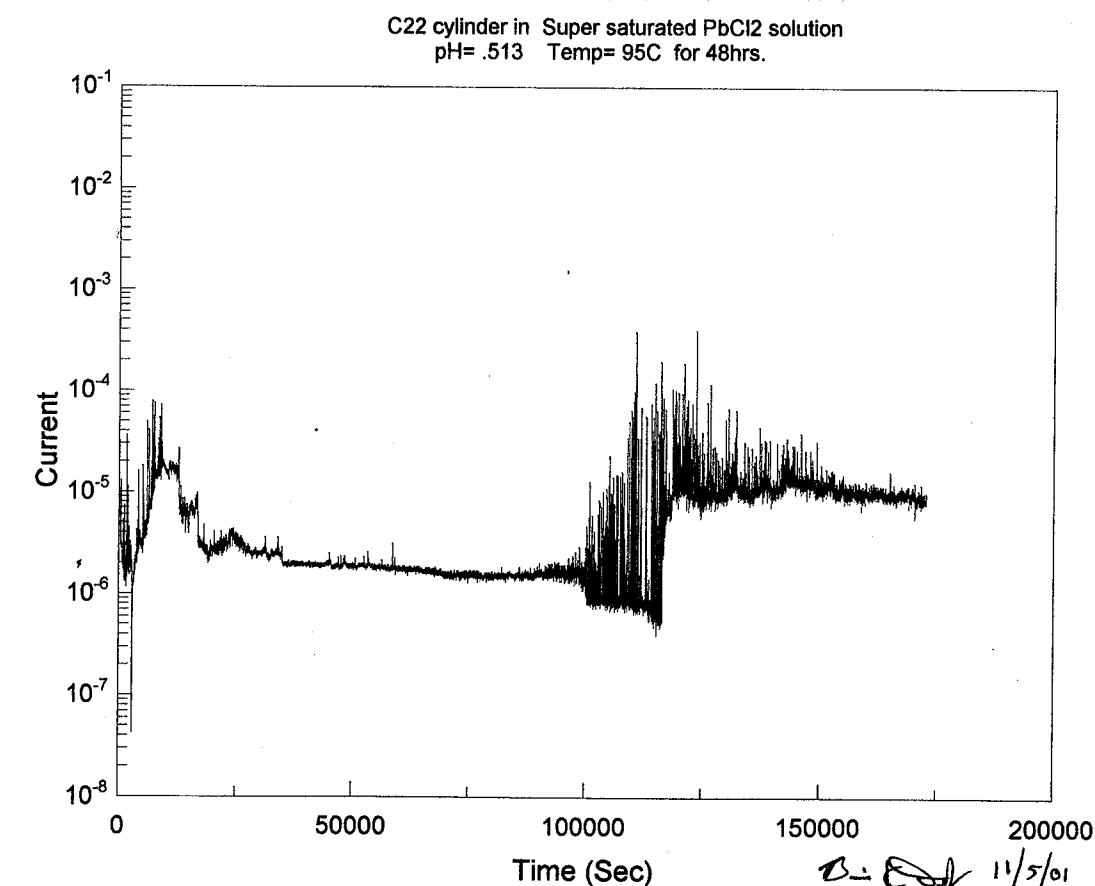
Solution Cools overnight Rev pH = .513

\* Note \* Some small fine Granules still Remain In Solution

measurements Taken with Ohaus Precision Standard scale SN#2883 cal 3/2/01

pH meter - Fisher Accumet 950 meter SN#3340 cal 7/24/01

pH probe 13-620-296 SN#1100208



# Solution Preparation for Testing And ICP Analysis

objective: To check solution concentration measurements of Lead And chloride

2 Test cells with 1000 mls / 1 Liter of DI In Each

Cell #1 - Adjusted pH Paper To Adding  $PbCl_2$  And Heating Solution  
 start pH = 4.097 Final pH = .497 Added 72 mls of  
 Premade 6m HCl Solution - Deaerates And Heated To  $95^{\circ}C$   
 overnight (17 hrs.) Then Added 25.008g  $PbCl_2$  Lot # L13K01  
 @ 8:20 AM Held At Temp for 6 hrs then solution for ICP removed  
 At  $95^{\circ}C$  Temperature then Dilutes To  $\frac{1}{2}$  \* Hold the Rest of Cell #1  
 solution for Testing - Thermometer SN# C96-852 cal 10/30/01

Cell #2 - Adjusted pH Paper To Adding  $PbCl_2$  And Heating Solution  
 start pH = 4.137 Final pH = .497 Added 65 mls of  
 Premade 6m HCl Solution - Deaerates And Heated To  $95^{\circ}C$   
 overnight (17 hrs.) then Added 25.004g  $PbCl_2$  Lot # L13K01  
 @ 8:25 AM Held At Temp for 6 hrs then solution for ICP  
 removed At  $95^{\circ}C$  Temperature then Dilutes To  $\frac{1}{2}$  \* Hold the Rest  
 of Cell #2 solution for Testing - Thermometer SN# C96-816 cal 10/30/01

Solution Deaerates with 99.999%  $N_2$

Measurements - Ohaus Precision Standard Scale SN# 2883 cal 3/2/01

Premade - 6m HCl 252.5 mls HCl Lot # 002545  
 + DI water To 500 mls made 11/16/01

\* 20 g  $PbCl_2$  may be used in solution preparation  
 to avoid the problem of build-up around the probe  
 or specimen during testing gmp 11/21/01

Bi K Duf 11/20/01

# Results from ICP Analysis Analysis on Cell #1 + Cell #2

## FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

### A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: Y. Ming Pan / Brian Dufy Request Date: 11/21/01  
 Project No.: 28 07402.571 Phone No.: x 6640 (Y. Ming) x 5446 (Lab)  
 Description of Work Requested: Lead And Chloride Concentration Analysis

☐ 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
#1	$PbCl_2$ Solution 10 mls
#2	$PbCl_2$ Solution 10 mls

### B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>

☐ 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): \_\_\_\_\_

<sup>1</sup> 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.

B. K. Duf 12/21/01

## Results 1:2 Dilution Lead

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.523
True Value	----	0.500
Recovery	----	105%
#1	171556	2233
Duplicate result	171556	2239
RPD	171556	0.27%
Spike result	171556	2750
Spike added	171556	500
Recovery	171556	103%
#2	171557	2081

Reporting Limit: 0.005 mg/L

Bi K Duf 12/21/01

## Results 1:2 Dilution Chloride

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	191
True Value	----	200
Recovery	----	95.7%
#1	171556	7008
Duplicate result	171556	6972
RPD	171556	0.52%
Spike result	171556	7846
Spike added	171556	1000
Recovery	171556	83.8%
#2	171557	7949

Reporting Limit: 0.1 mg/L

Bi K Duf 12/21/01

\* Some Solids left In Sample Insufficient dilution  
 Bi K Duf 1/9/02 - See pg # 11 Sample #3A

Bi K Duf 12/21/01

Potentiostatic Test Alloy C-22 Cylinder In  $\text{PbCl}_2$  @ -100mV

Objective: See pg #5

Specimen: Alloy C-22 Cylinder Specimen Dimensions on pg #29 600 Grit Finish

Start wt. = 12.69310g Santorionic Genius SN# 12909099 Cal 5/22/01

End wt. = 12.68546g

Solution: Test Solution Cell #1 pg #102

Start pH = .505 Fisher Accumet 950 meter SN# 3340 Cal 7/24/01

End pH .437 Fisher pH Probe 13-620-296 SN# 1100208

Potentiostat: Solotom 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0089777

Temperature: 95°C Thermometer SN# A2000-123 Cal 2/23/01

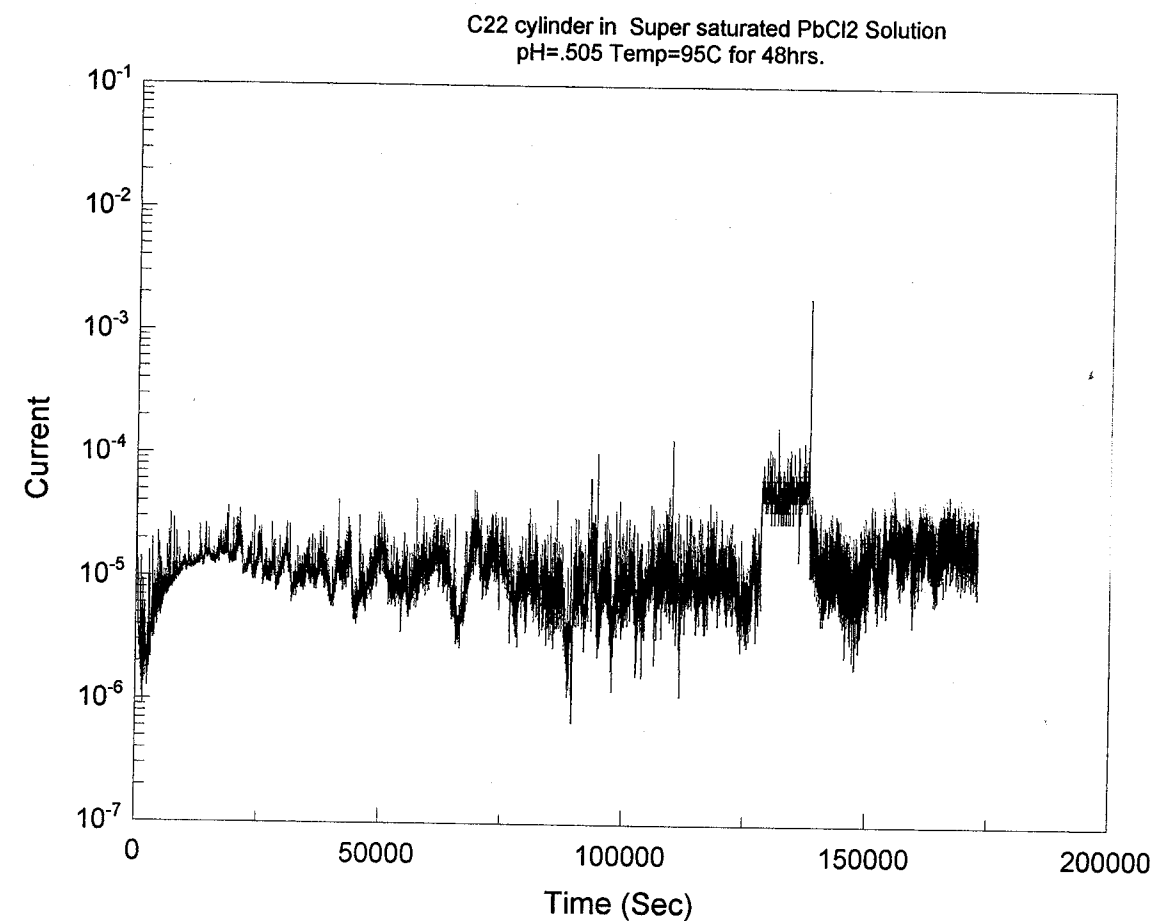
Bubbles with 99.999%  $\text{N}_2$  for Deseration $E_{\text{scan}} = -177\text{mV}$  Keithley 614 SN# 467374 Cal 16/4/01 $E_{\text{pt}} = +441\text{mV}$  $E_{\text{Applied}} = -100\text{mV}$ 

Specimen Examination: Some small pitting noticed upon magnification

All Solis In Bottom of Cell went Into Solution when Cell was up To 95°C  
 And potential was Applied also 11/24/01

Test C22+37.wor

B. R. J.  
 11/26/01



B. R. J. 11/24/01

B. R. J.  
 11/24/01

sent solutions for ICP Analysis on 11/01/01

Test solutions = C22 + 20  $P_g \rightarrow 64 + 65$

C22T35 Pg # 98-99

C22+36  $P_{\gamma} \neq 106-101$

All solutions were  $\frac{1}{2}$  dilution Summation for Lead and Chromium.  
ICP Analysis

**This report may not be reproduced except in its entirety without the written approval of SwRI.**

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

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 11/01/01

Matrix: Liquid

Project No.: 20.01402.571

Work Order: 21205

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	198
True Value	----	200
Recovery	----	98.9%
TEST # 20	170298	1400
Duplicate result	170298	1388
RPD	170298	0.86%
Spike result	170298	1740
Spike added	170298	400
Recovery	170298	85.1%
TEST # 35	170299	5812
TEST # 36	170300	6232

Reporting Limit: 0.1 mg/L

TS: KDF 12/3/01

Bin K. Jaf  
12/03/01

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

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 11/01/01

Matrix: Liquid

Project No.: 20.01402.571

Work Order: 21205

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.501
True Value	----	0.500
Recovery	----	100%
TEST # 20	170298	769
Duplicate result	170298	773
RPD	170298	0.52%
Spike result	170298	1280
Spike added	170298	500
Recovery	170298	102%
TEST # 35 (1:2)	170299	2316
TEST # 36 (1:2)	170300	1650

Reporting Limit: 0.005 mg/L

mkd 12/3/0

B. [Signature]  
12/01/01



## Solution Preparation for Testing And ICP Analysis

objective: To check Solution Concentration Measurements of Lead And Chlorine  
2 Test cells with 1000mls / 1 Liter of DI In Each Cell

12/27/01

Cell #1: follows Pg #49 Instructions for Solution Preparation

Heats Cell To  $95^{\circ}\text{C}$  stirs overnight + Deserates with 99.999%  $\text{N}_2$   
(15 hrs) then Adds 5.008g  $\text{PbCl}_2$  - kept At  $95^{\circ}\text{C}$  + Deserates / stirs  
for 5 hrs. then Cools Solution To Room Temp  $24^{\circ}\text{C}$  (2 hrs 15 min)

Kept Deserating - Then Adjusts pH - No Deseration During pH Adjustment

Begin pH = 4.621 Fisher Accumat 950 meter SN#3340 cal 7/24/01

End pH = .500 pH Probe #13-620-296 SN#1100208

Adds 29.4mls of 6m HCl solution Lot#062545 To Adjust pH

\* Note: After Adding HCl - Solution went from cloudy To Clear

Reheats Solution To  $95^{\circ}\text{C}$  Then Adds 10.047g  $\text{PbCl}_2$  Lot#L13K01

Solution went cloudy then Clear In About 30min - Still up To Temp

$95^{\circ}\text{C}$  And Deserating - Adds Another 10.004g  $\text{PbCl}_2$  Lot#L13K01.

Solution went cloudy And Didn't clear up - Pulls Sample #1A (After 4 hrs)

Turns off Heater controller kept At Room Temp + Deserates over weekend  
while still stirring solution. Mon 12/31/01 Returns Cell To Temp  $95^{\circ}\text{C}$

stirs And Deserates - After 3 hrs solution still slightly cloudy After 5 hrs

Pulls Sample #1B - kept Deserating And <sup>also 11/30/01</sup> stirring At Room Temp Till

Wed 1/2/02 fine powder In cell went To crystal form - Reheats while

stirring And Deserating Cell solution - Looks Clearer Up To Temp  $95^{\circ}\text{C}$

Heb for 5 hrs then Pulls Sample #1C - kept solution

for possible Testing

\* Thermometer A2000-123 cal 8/23/01

B. R. Duff  
1/3/02

## Solution Preparation for Testing And ICP Analysis

objective: To check Solution Concentration Measurements of Lead And Chlorine  
2 Test cells with 1000mls / 1 Liter of DI In Each Cell

12/27/01

Cell #2 - Solution Preparation following Issues Discussed from Pg #102

\* Issue: Adjust pH After  $\text{PbCl}_2$  has been Added To Preparation Cell.  
possible controlling factor that affects Pb Concentration In Solution.

Heats Cell To  $95^{\circ}\text{C}$  stirs And Deserates overnight 99.999%  $\text{N}_2$

(15 hrs.) then Adds 25.036g  $\text{PbCl}_2$  Lot#L13K01 - kept At  $95^{\circ}\text{C}$

Deserates / stirs for 5 hrs - then Cools Solution To Room Temp  $24^{\circ}\text{C}$

(2 hrs 25 min) kept Deserating - Then Adjusts pH - No Deseration During Adjustment

Begin pH = 3.746 Fisher Accumat 950 meter SN#3340 cal 7/24/01

End pH = .497 pH Probe #13-620-296 SN#1100208

Adds 33.5mls of 6m HCl solution Lot#062545 To Adjust pH

\* Note: After Adding HCl - solution Remains cloudy.

Reheats Solution To  $95^{\circ}\text{C}$  pulls Sample #2A (After 4 hrs)

Turns off Heater controller kept At Room Temp And Deserates over the  
weekend still stirring solution - Mon 12/31/01 solution still cloudy Reheat To

$95^{\circ}\text{C}$  - After 3 hrs up To Temp solution seems cleaner - pulls Sample

#2b After 5 hrs. kept Deserating <sup>(without stirring) 1/9/02</sup> At Room Temp Till Wed 1/2/01

Crystal formation In bottom of cell - Re Heats while stirring And

Deserating Cell solution At  $95^{\circ}\text{C}$  solution looks cleaner - Heb for

5 hrs then Pulls Sample #2c - Uses solution for

Test C22+38.con

Thermometer C96-816 cal 10/30/01

B. R. Duff  
1/3/02

\*Note Solution #3A Is Solution from pg #102 cell 2 It was  
Reheated To 95°C And Deserated with 99.999% N<sub>2</sub> for 3 hrs Then Sample  
was Taken. Thermometer E98-191 cal 12/11/01

All Solution Are 1:10 Dilution

### FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

#### A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: Yi-Ming Pan / Brian Deaby Request Date: 1/02/02  
Project No.: 20.1402.571 Phone No.: X 6640 (Yi-Ming) X5448 (Lab)

Description of Work Requested:  
Lead And Chloride Concentration Analysis (ICP)

\* We Need Results As Soon As Possible - Thank You \*

☐ 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
#1A PbCl <sub>2</sub> Solution	#1C PbCl <sub>2</sub> Solution
#2A PbCl <sub>2</sub> Solution	#2C PbCl <sub>2</sub> Solution
#1B PbCl <sub>2</sub> Solution	#3A PbCl <sub>2</sub> Solution
#2B PbCl <sub>2</sub> Solution	

#### B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>

☐ 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): \_\_\_\_\_

<sup>1</sup> 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.

*B. Deaby*  
1/3/02

ICP Results from pg # 110

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 01/02/02

Matrix: Liquid

Project No.: 20.01402.571

Work Order: 21539

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	193
True Value	----	200
Recovery	----	96.5%
#1A PbCL2 Solution	179026	931
Duplicate result	179026	939
RPD	179026	0.86%
Spike result	179026	1097
Spike added	179026	200
Recovery	179026	83.2%
#1B PbCL2 Solution	179027	973
#1C PbCL2 Solution	179028	725
#2A PbCL2 Solution	179029	1009
#2B PbCL2 Solution	179030	980
#2C PbCL2 Solution	179031	795
#3A PbCL2 Solution	179032	1387

Reporting Limit: 0.1 mg/L

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 01/02/02

Matrix: Liquid

Project No.: 20.01402.571

Work Order: 21539

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.488
True Value	----	0.500
Recovery	----	97.6%
#1A PbCL2 Solution	179026	1028
Duplicate result	179026	1015
RPD	179026	1.27%
Spike result	179026	1530
Spike added	179026	500
Recovery	179026	100%
#1B PbCL2 Solution	179027	1247
#1C PbCL2 Solution	179028	598
#2A PbCL2 Solution	179029	877
#2B PbCL2 Solution	179030	1044
#2C PbCL2 Solution	179031	499
#3A PbCL2 Solution	179032	802

Reporting Limit: 0.005 mg/L

*B. Deaby*  
1/9/02

# Potentiostatic Test Alloy C22 Cylinder In $\text{PbCl}_2$ @ -100mV

Objective: See pg #5

Specimen: Alloy C22 Cylinder Specimen Dimensions on pg #29 600 Coat Finish

Start wt: 12.71072 g Sartorius Genius SN# 12809099 cal 5/20/01

End wt: 12.70949 g

Solution: Preparation on pg #109 Cell #2

Start pH: .497 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

End pH: .586 Fisher pH Probe 13-620-296 SN# 1100203

Potentiostat: Solartron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0089741

Temperature: 95°C Thermometer SN# H00-387 cal 11/19/01

Deaerates with 99.999%  $\text{N}_2$

Ecorr: +11 mV Keithley 617 SN# 537418 cal 2/22/01

EPT: +461 mV

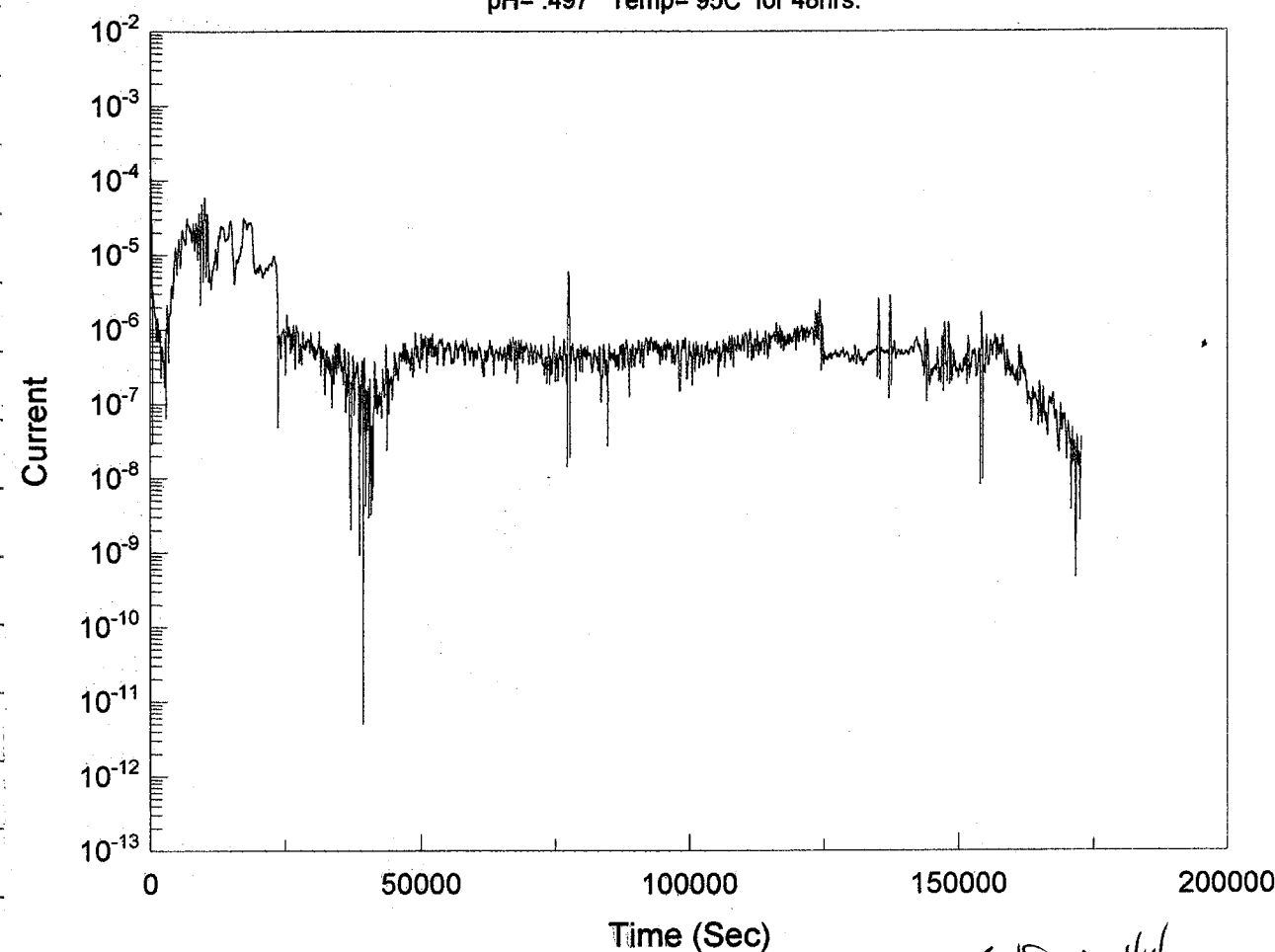
E Applies: -100 mV

Specimen Examination: No Corrosion Noticed - Trans passive Dissolution?

Tot C22+38.con

*[Signature]*  
1/3/02

C22 cylinder in Super Saturated  $\text{PbCl}_2$  solution  
pH= .497 Temp= 95C for 48hrs.



*[Signature]*  
1/4/02

# Solution Preparation for Testing And Tap Analysis

Objective: To check Solution Concentration Measurements of Lead And Chloride

1 Test Cell with 1000mls / 1 Liter of DI water

1-7-02

Heated Cell To  $95^{\circ}\text{C}$  Deaerated And Stirred overnight (15 hrs) 99.999%  $\text{N}_2$

1-10-02 Added 5.007g  $\text{PbCl}_2$  Lot# L13K01. Solution Turned cloudy

Stirred And Deaerated for 4 hrs. Then Cooled Solution To Room Temperature To Adjust pH.

pH I: 4.217 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

pH F: .500/.498 pH probe #13-620-296 SN# 1100205

Added 28.6mls of 6 M HCl Solution Lot# 002545 To Adjust pH

\* Note: solution was still cloudy After HCl was Added then solution went clear

After 20-25 min while reheating At About  $46.5^{\circ}\text{C}$  \* Reheat Cell To  $95^{\circ}\text{C}$

Added 10.041g  $\text{PbCl}_2$  Lot# L13K01 when Cell Reach  $95^{\circ}\text{C}$ . Solution Stayed clear. Kept Deaerating And Stirring. After 1 hr.

Added 2.001g  $\text{PbCl}_2$  Lot# L13K01. Solution got cloudy then clear In 15 min After 30 min

Added 1.002g  $\text{PbCl}_2$  Lot# L13K01. Solution got cloudy then cleared up But some solid Around Bottom outside - Around edge of Cell from stirring

1-11-02

Left Stirring @  $95^{\circ}\text{C}$  And Deaerating with 99.999%  $\text{N}_2$  overnight (15 hrs) Solution clear.

Added 2.003g  $\text{PbCl}_2$  Lot# L13K01. Solution went cloudy then clear In About 30 min - Kept stirring And Deaerating 2 hrs then

Added 3.002g  $\text{PbCl}_2$  Lot# L13K01. Solution went cloudy then clear In About 2 hrs - Kept stirring And Deaerating 2 hrs then  
\* Continue on Pg #115

Bick Duf  
1/17/02

Added 2.004g  $\text{PbCl}_2$  Lot# L13K01. To Reach A Total of 25.060g of  $\text{PbCl}_2$ . Kept stirring And Deaerating @  $95^{\circ}\text{C}$  for 3 hrs Then Pulled Sample #1. Stopped stirring Let Solution Settle Before Sample #1 was pulled (5 min)

Then Pulled Sample #2 - 3 hrs After Sample #1 was pulled Deaerating But still no stirring Cell still @  $95^{\circ}\text{C}$

Shut off Temperature controller After Sample #2 was pulled will Deaerate And Stir over weekends -

1-14-02

Stirred And Deaerated cell over the weekends - Solution cloudy Started To Reheat Cell To  $95^{\circ}\text{C}$  kept stirring And Deaerating  
 $\checkmark \text{O}_2$  Level @  $50^{\circ}\text{C}$  = .09 Omega DO meter DOH-920  
Probe #541/520

Solution cleared up After 1 hr @  $95^{\circ}\text{C}$  - But had A fine Powder floating Around Bottom of the Cell - Kept Deaerating And stirring for 5 hrs.

Pulled Sample #3 - 5 hrs After Cell was up To  $95^{\circ}\text{C}$  - Stopped stirring for 5 min before Sample was pulled - still Deaerating

Pulled Sample #4 - 3 hrs After Sample #3 was pulled. still no stirring - still Deaerating

Kept Deaerating overnight But NO stirring And kept Cell up To Temperature  $95^{\circ}\text{C}$

Pulled Sample #5 - Shut Down Temperature Controller use Solution To Run Test C22T39

Pulled Sample #6 out of Test Cell @  $95^{\circ}\text{C}$  Deaerates After Completion of Test.

Bick Duf 1/17/02

see Pg # 115 for How Sample # 1-6 @ 1:10 Dilutions were Taken  
Sample # 7 Pg # 106 Cell # 1 Solution - Solis Removes from Bottom  
of Beaker/flask evaporation of Liquid And places .02031g of  
Solis In ~~1:10~~<sup>1:100</sup> Dilution 20 mls of DI for Dilution  
Sample # 8 Pg # 102 Cell # 2 Solution - Solis Removes from Bottom  
of Beaker/flask evaporation of Liquid And places .02058g of  
Solis In 20 mls of DI for Dilution - Sent All Samples  
for ICP Analysis

FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: Y. Ming Pan / Brian Derby Request Date: 1/17/02  
Project No.: 20.01402.571 Phone No.: x 6640 (Y.Ming) x 5448 (Lab)  
Description of Work Requested:  
Lead And Chlorine Concentrations Analysis (ICP)  
\* We Need Results As Soon As Possible - Thank You \*

☐ 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
#1	PbCl <sub>2</sub> Solution	#6 PbCl <sub>2</sub> Solution
#2	PbCl <sub>2</sub> Solution	#7 PbCl <sub>2</sub> Solution
#3	PbCl <sub>2</sub> Solution	#8 PbCl <sub>2</sub> Solution
#4	PbCl <sub>2</sub> Solution	
#5	PbCl <sub>2</sub> Solution	

B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>

☐ 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): \_\_\_\_\_

<sup>1</sup> 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.

B. J. Pa  
1/18/02

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 21664

Client: Division 20  
Date Received: 01/17/02  
Project No.: 20.01402.571

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	206
True Value	----	200
Recovery	----	103%
#1 PbCL2 Solution	188026	1036
Duplicate result	188026	1036
RPD	188026	0.03%
Spike result	188026	1251
Spike added	188026	200
Recovery	188026	108%
#2 PbCL2 Solution	188027	976
#3 PbCL2 Solution	188028	966
#4 PbCL2 Solution	188029	1059
#5 PbCL2 Solution	188030	1012
#6 PbCL2 Solution	188031	1092
#7 PbCL2 Solution	188032	220
#8 PbCL2 Solution	188033	243

Reporting Limit: 0.1 mg/L

S. K. J. 1/23/02

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 21664

Client: Division  
Date Received: ☐  
Project No.: 20.0

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.506
True Value	----	0.500
Recovery	----	101%
#1 PbCL2 Solution	188026	1122
Duplicate result	188026	1122
RPD	188026	0.00%
Spike result	188026	1630
Spike added	188026	500
Recovery	188026	102%
#2 PbCL2 Solution	188027	1102
#3 PbCL2 Solution	188028	1114
#4 PbCL2 Solution	188029	1086
#5 PbCL2 Solution	188030	1096
#6 PbCL2 Solution	188031	1160
#7 PbCL2 Solution	188032	597
#8 PbCL2 Solution	188033	662

Reporting Limit: 0.005 mg/L

B. J. Pa 1/23/02

Solid Cl/Pb ratio calculation:

Sample ID	Concentration (mg/L)		Concentration (mM)		Cl/Pb ratio
	Pb	Cl	Pb	Cl	
# 7	597	220	0.0029	0.0062	2.14
# 8	662	243	0.0032	0.0068	2.12

B. J. Pa  
1/23/02

# Potentiostatic Test Alloy C22 cylinder In $\text{PbCl}_2$ @ -100mv

Objective: See pg #5

specimen: Alloy C22 Cylinder Specimen Dimensions on pg #29 600 Grit Finish

Start wt: 12.46823g Santarions Genius SN# 12809099 Cal 5/22/01

End wt: 12.44308g

Solution: See preparation on pg #114-115

Start pH: .498 Fisher Accumet 950 meter SN# 3340 Cal 7/24/01

End pH: .873 Fisher pH Probe #13-620-296 SN# 1100205

Potentiostat: Selatron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 0042119

Temperature: 95°C Thermometer SN# H00-387 Cal 11/19/01

Solution Deaerates with 99.999%  $\text{N}_2$

Ecorr: -153mv Keithley 614 SN# 00555368 Cal 2/23/01

Ept = +386mv

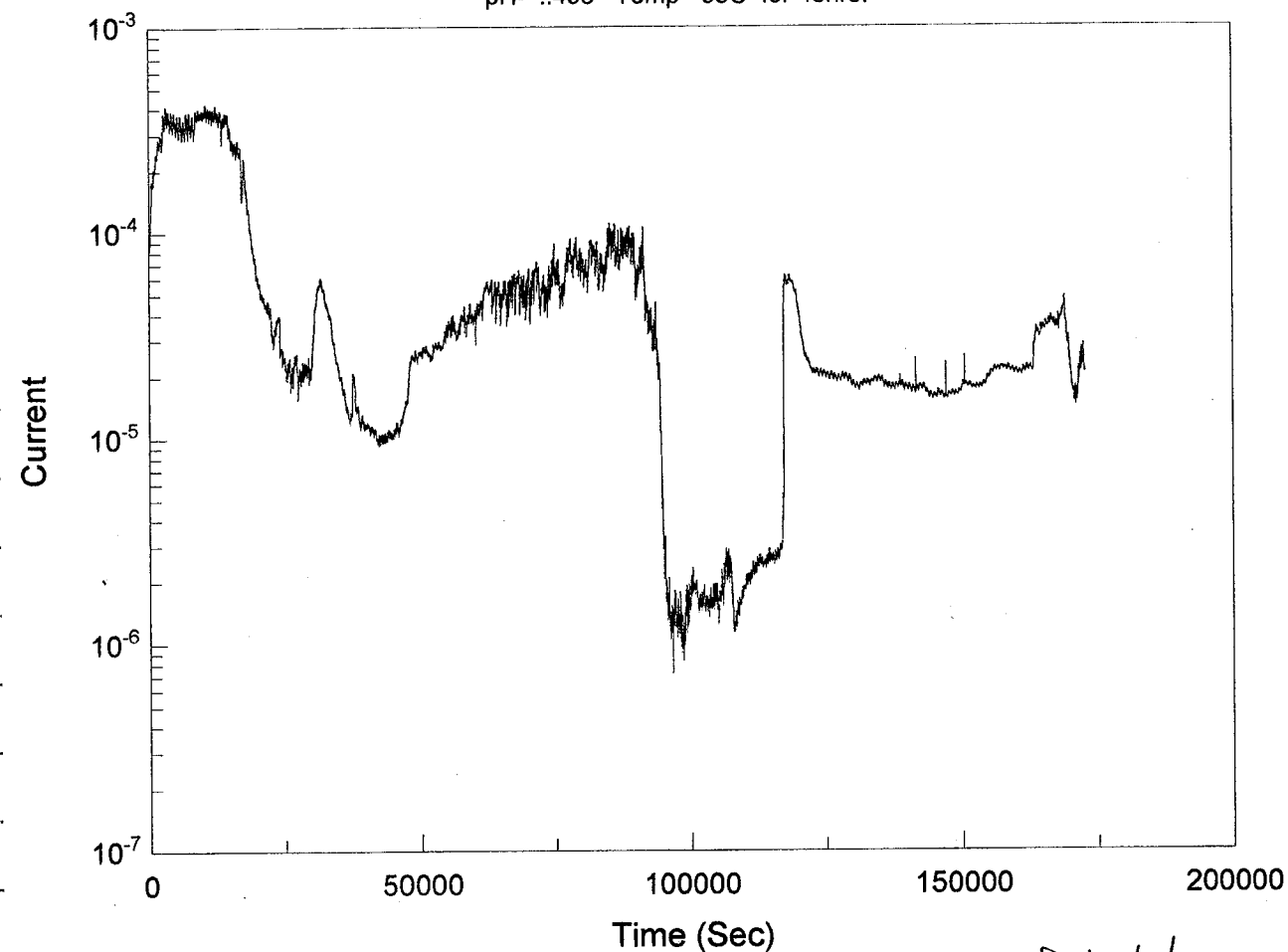
E Applied: -100mv

Specimen Examination:

Test C22+39-CA

B. R. J.  
1/14/02

C22 cylinder in Super Saturated  $\text{PbCl}_2$  solution  
pH= .498 Temp= 95C for 48hrs.



B. R. J.  
1/14/02

## U-Bend Solution Preparation

Solution: 2 Liter / 2000mls prepared for U-Bend Testing  
PbCl<sub>2</sub> Lot # L13K01 Deaeration with 99.999% N<sub>2</sub>  
Total PbCl<sub>2</sub> Added To Solution 50.192g

2/25/02 Deaeration of 2000mls DI water over weekends  
Then Heated Solution To 95°C - Once Cell with Solution  
was @ 95°C - Added 10.041g PbCl<sub>2</sub> Lot # L13K01 Held  
At Temperature Deaerating And stirring for 5 hrs then  
shut off Heat controller To Cool solution overnight  
Solution was cloudy

2/26/02 Adjusted pH of Solution  
pH start: 4.602 Fisher Accumet 950 meter SN# 3340 cal 7/24/01  
pH End: .507 pH probe # 13-620-296 SN# 1100208  
Added 84.5mls of 6m HCl Solution Lot # 62545

Solution Remains cloudy  
Reheated Solution To 95°C Deaerating And stirring  
Solution went clear @ 60°C

Added 15.003g PbCl<sub>2</sub> Lot # L13K01 Cell Temp 68°C  
Solution went cloudy then clear In 15 min (9:38 Time)

Added 10.005g PbCl<sub>2</sub> Lot # L13K01 Cell Temp 80°C  
Solution went cloudy - small Fine Particles on Bottom of Cell (Time 10:30)

Added 5.073g PbCl<sub>2</sub> Lot # L13K01 Cell Temp 91°C (12:30 Time)  
Solution same

Added 5.044g PbCl<sub>2</sub> Lot # L13K01 Cell Temp 95°C (1:35 Time)  
Solution same

(continues pg #121)

BiKDJ 3/4/02

Added 5.026g PbCl<sub>2</sub> Lot # L13K01 Cell Temp 95°C (3:30 pm Time)  
Solution still cloudy with fine particles on Bottom of Cell  
Kept Deaeration And stirring @ 95°C overnight

2/27/02

Pulled Sample for ICP Analysis of Solution 1:10  
for Lead And Chloride Ratios

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 21943

Client: Division 20  
Date Received: 02/27/02  
Project No.: 20.01402.571

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.493
True Value	----	0.500
Recovery	----	98.6%
#1 PbCl <sub>2</sub> Solution	199329	1163
Duplicate result	199329	1146
RPD	199329	1.47%
Spike result	199329	1692
Spike added	199329	500
Recovery	199329	106%

Reporting Limit: 0.005 mg/L

BiKDJ 3/4/02

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 21943

Client: Division 20  
Date Received: 02/27/02  
Project No.: 20.01402.571

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	196
True Value	----	200
Recovery	----	98.0%
#1 PbCl <sub>2</sub> Solution	199329	1649
Duplicate result	199329	1653
RPD	199329	0.22%
Spike result	199329	1835
Spike added	199329	200
Recovery	199329	92.6%

Reporting Limit: 0.1 mg/L

BiKDJ 3/4/02

BiKDJ 3/4/02



Potentiostatic Test Alloy C-22 U-Bend Specimens

PbCl<sub>2</sub> Solution @ -100 mV

Objective: See Pg #5

Specimen: Alloy C22 ASTM G-30 Single U-Bend Specimen Serial # 01

Dimensions 5" (L) x 3.975" (M) x 0.750" (W) x 0.125" (T) with 0.375 mounting Holes

Alloy C22 ASTM G-30 Double U-Bend Specimen Serial # 01

Dimensions (A) 5" (L) x 3.975" (M) x 0.750" (W) x 0.125" (T)

(B) 5.395" (L) x 4.370" (M) x 0.750" (W) x 0.125" (T) with 0.375 mounting Hole x 2

Start wt Single U-Bend: 58.7161g Santarions Genius SN# 12809099 cal 12/21/01

End wt Single U-Bend: 58.2003g

Start wt Double U-Bend: (A) 58.95734g (B) 64.5086g Santarions Genius

End wt Double U-Bend: (A) 58.36243g (B) 63.76478g SN# 12809099 cal 12/21/01

Solution: PbCl<sub>2</sub> Super Saturated See Pg # 120-121

Start pH: .507 Fisher Accuret 950 meter SN# 3340 cal 12/21/01

End pH: .042 (see Pg # 125)

Potentiostat: Solartron 1267

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN# 8238341

Temperature: 95°C H<sub>2</sub> Thermometer SN# 115749 cal 1/10/02

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

E<sub>corr</sub>: -250 mV Keithley 617 SN# 53741R 3/11/02

E<sub>pt</sub>: -224 mV

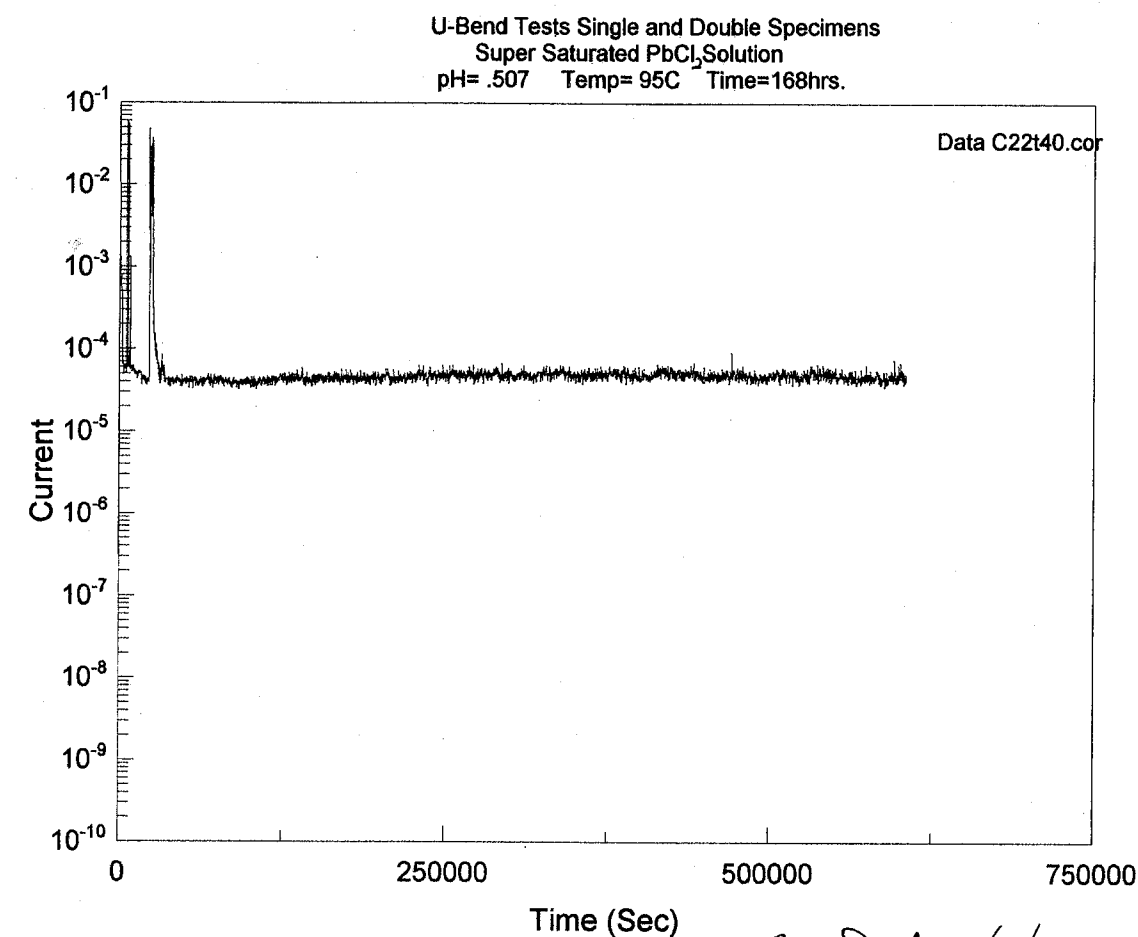
E<sub>Appl</sub>: -100 mV

(Continued Pg # 123)

3/4/02 B. F. J.

Specimen Examination: Test C22+40.con Removed Specimens from Test Cell for Examination. Upon magnification Notices Grain Boundary Etching on Both Single And Double U-Bend Specimens After (168 hrs) In PbCl<sub>2</sub> Solution. Will Continue Test for Another (168 hrs) 1 week In Same Test Solution \* Test Data C22+40B.con

\* Note: Surface Area Estimate for specimen 46 cm<sup>2</sup>



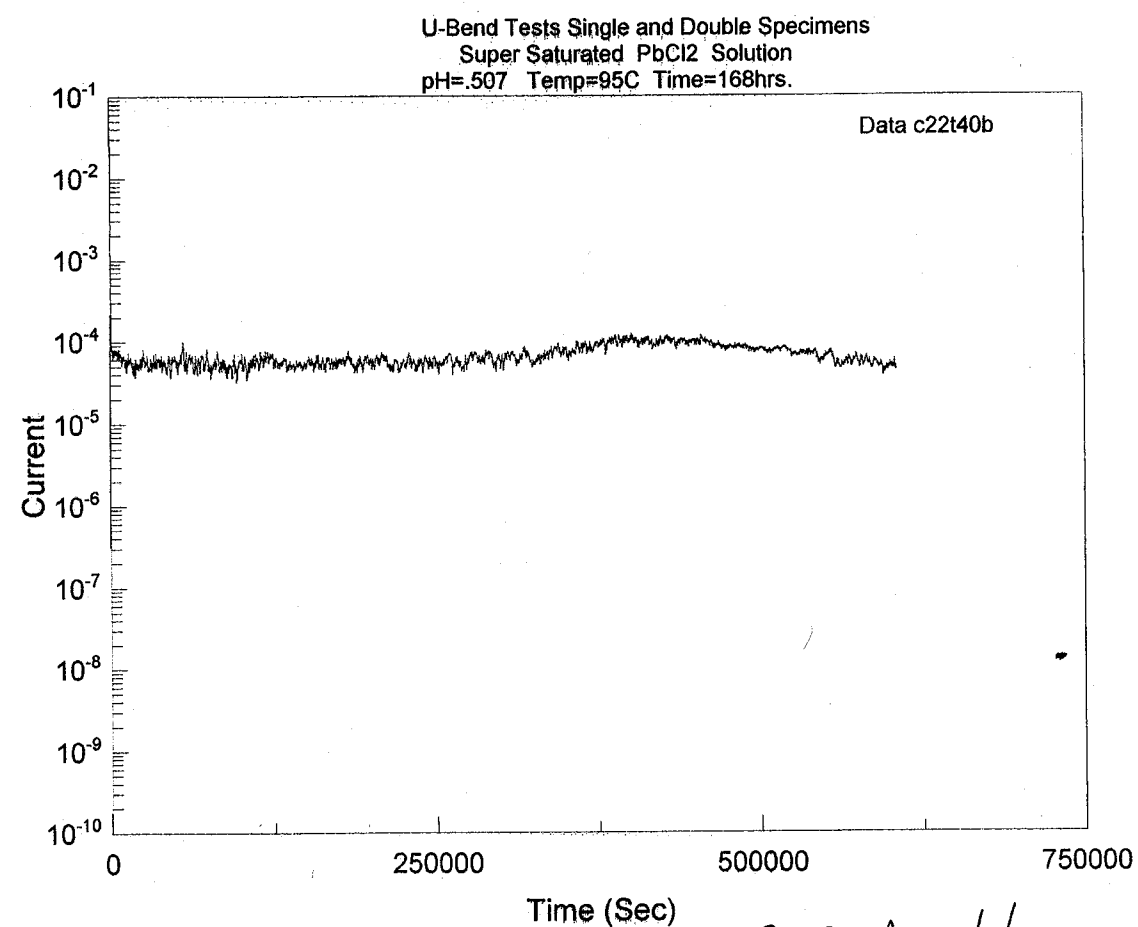
B. F. J. 3/12/02

(Continued Test on Pg # 124)

B. F. J.  
3/12/02

specimen Examination: Test C22+40b - Removed Specimens for Examination from Test cell - Under magnification Noticed grain Boundary Etching on Both single And Double U-Bend Specimens After (168hrs) In  $PbCl_2$  Solution.

Total Test Time So far on Specimen In  $PbCl_2$  Solution 336 hrs. will continue Testing In same Solution And set up for An Additional 336 hrs Test data \* C22+40a



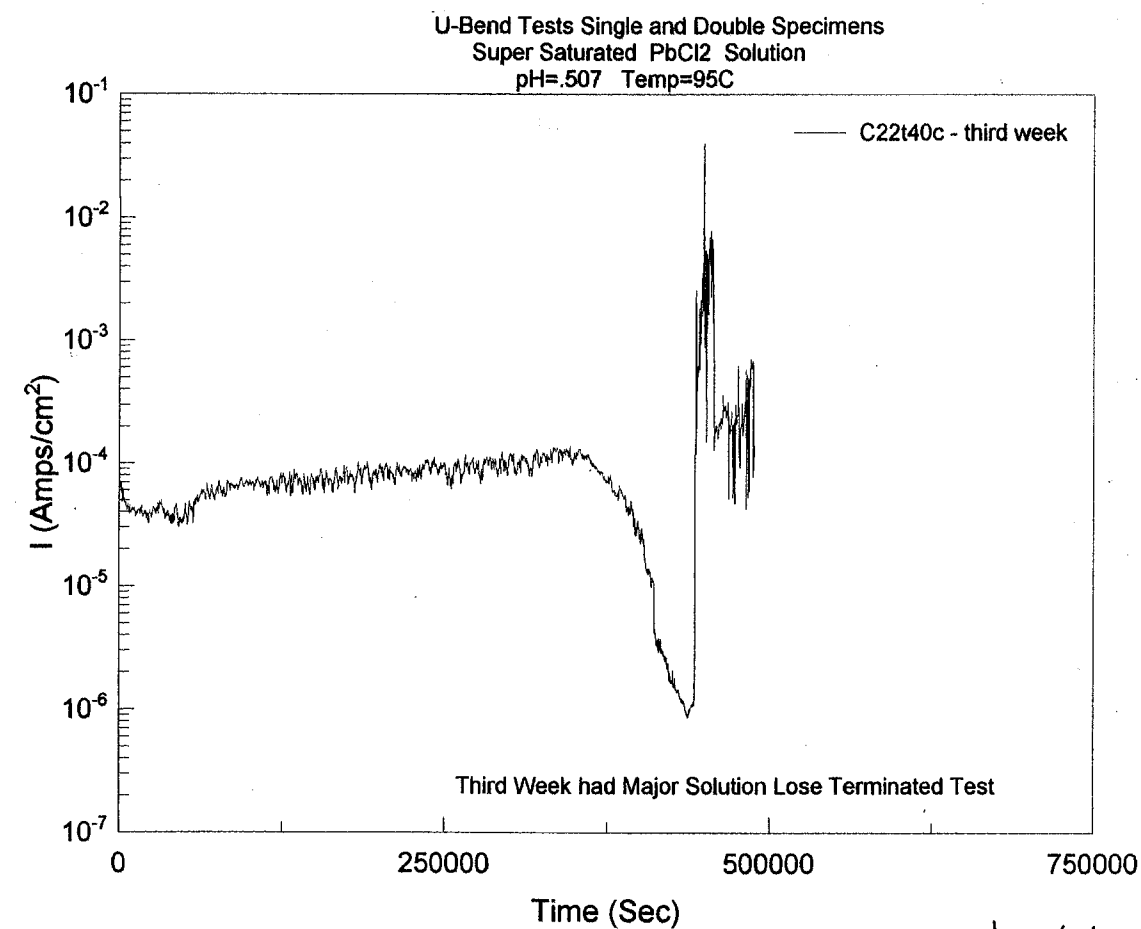
Bill D. 3/20/01

(Continues on pg #125)

Bill D.  
3/20/01

\* Note: Test shut Down on 3/25/02 A.M. - Solution level Drapped In Test Cell. Solution  $PbCl_2$  Became Concentrated And Had A Blue/Green Tint. Took End pH Reading of .042. It Drapped from .502 To .042 Because of solution loss. Will Clean up Specimens And Re weigh see pg #122. Noticed Grain Boundary Etching on Both single And Double U-Bend Specimens.

\* Some small cracks noticed on <sup>one of the</sup> single Double U-Bend Specimen (B) Around Bolt hole



Bill D. 3/25/02

Bill D.  
3/25/02

U-Bend Solution Preparation

Solution: 2 Liter/2000mls prepared for U-Bend Testing  
PbCl<sub>2</sub> Lot# L13K01 Deserates with 99.999% N<sub>2</sub>  
Total PbCl<sub>2</sub> Added To Solution 50.209g

3/25/02 Started Deseration And Stirring of Solution @ 1:30 pm  
Deserates And Stires Solution Overnight

3/26/02 Started To heat Solution To 95°C @ 7:30am  
Continues Deseration And Stirring - Solution Reaches  
And Maintains Temperature At 10:00 am

Added 10.098g PbCl<sub>2</sub> Lot# L13K01 - Solution went cloudy  
Kept Deserating And Stirring @ 95°C for 5 hrs  
Shut off Heat Controller To Cool Solution overnight  
Solution still cloudy

3/27/02 Adjustes pH of Solution  
pH Start: 4.578 Fisher Accuret 950 meter sn#3740 cal 7/24/01  
pH End: .502 pH Probe # B-620296 sn#1100208

Added 89.6mls of 6m HCl Solution Lot# 002585 prepared 5/27/02  
Started To Reheat Solution To 95°C @ 8:50 am  
Continue Deseration And Stirring of Solution - Solution Cloudy  
Solution went clear @ 55.3°C @ 9:36 am

Added 15.001g PbCl<sub>2</sub> Lot# L13K01 Cell Temp 61.7°C - Solution was  
cloudy then Clear In 15min (Time 9:45am)

Added 10.018g PbCl<sub>2</sub> Lot# L13K01 Cell Temp 87.3°C - Solution went  
cloudy (Time 10:40am)

(Continued on pg#127)

B. K. D. J. 3/28/02

Added 5.003g PbCl<sub>2</sub> Lot# L13K01 Cell Temp 91.2°C - Solution Cloudy  
with fine Particals In Bottom of Cell (Time 11:40am)

Added 5.046g PbCl<sub>2</sub> Lot# L13K01 Cell Temp 91.2°C - Solution  
Remains the Same - Cloudy Time (1:00pm)

Added 5.043g PbCl<sub>2</sub> Lot# L13K01 Cell Temp 92.0°C - Solution  
Remains the Same - Cloudy with fine Particals.

will keep @ 95°C Deserating And Stirring overnight

3/28/02 Solution slightly Cloudy with fine particals Drifting on  
Bottom of Cell - Booke up with Glass Rod (7:30am)

Pulled Solution for ICP Analysis At 9:30 am 1:10 Dilution  
for Lead And Chloride Ratios

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 22138

Client: Division 20  
Date Received: 03/28/02  
Project No.: 20.01402.571

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	0.512
True Value	----	0.500
Recovery	----	102%
#1 PbCl <sub>2</sub> Solution	202212	948
Duplicate result	202212	945
RPD	202212	0.32%
Spike result	202212	1423
Spike added	202212	500
Recovery	202212	95.0%

Reporting Limit: 0.005 mg/L

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 22138

Client: Division 20  
Date Received: 03/28/02  
Project No.: 20.01402.571

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	207
True Value	----	200
Recovery	----	104%
#1 PbCl <sub>2</sub> Solution	202212	1475
Duplicate result	202212	1447
RPD	202212	1.92%
Spike result	202212	1633
Spike added	202212	200
Recovery	202212	79.0%

Reporting Limit: 0.1 mg/L

B. K. D. J. 4/8/02

B. K. D. J. 3/28/02

Potentiostatic Test Alloy C22 U-Bend Specimen

PbCl<sub>2</sub> Solution @ -100mV

Objective: See pg #5

Specimen: Alloy C22 ASTM G-30 Single U-Bend Specimen Serial #02

Dimensions: 5"(L) x 3.975"(m) x 0.750"(w) x 0.125"(T) with 0.375 mounting Hole

Alloy C22 ASTM G-30 Double U-Bend Specimen Serial #02

Dimensions: (A) 5"(L) x 3.975"(m) x 0.750"(w) x 0.125"(T)

(B) 5.395"(L) x 4.370"(m) x 0.750"(w) x 0.125"(T) with 0.375 mounting Hole X2

Start wt Single U-Bend = 58.95753g Sartorius Genius SN#12809099 cal 12/21/01

End wt Single U-Bend = 58.11955g

Start wt Double U-Bend: (A) 58.88774g (B) 64.58032g Sartorius Genius

End wt Double U-Bend: (A) 57.45435g (B) 62.95774g SN#12809099 cal 12/21/01

Solution: PbCl<sub>2</sub> Super Saturated See pg #126-127

Start pH = .502 Fisher Accumet 950 meter SN#3340 cal 12/21/01

End pH = .572

Potentiostat: Solartron 1287

Counter Electrode: Pt Flag

Reference: Fisher 13-620-52 SN#8238341

Temperature: 95°C Hg Thermometer SN#115749 cal 1/10/02

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

Ecorr = -273mV Keithley 617 SN#537418 cal 3/11/02

E<sub>pt</sub> = -258mV

E Applied = -100mV

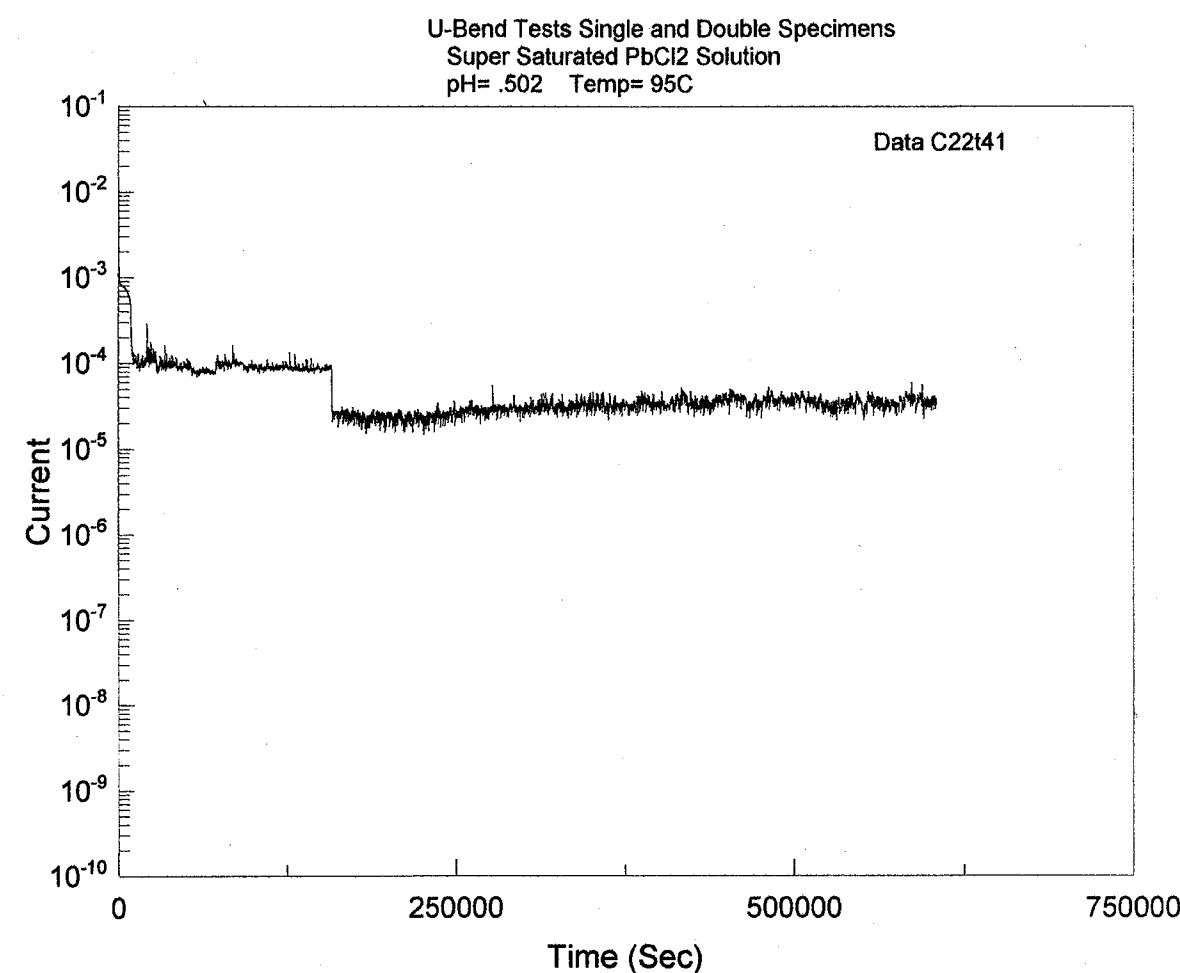
(Continues on pg #129)

B. R. J.  
4/12/02  
Copies sent to QA Records  
J. Pa 4/14/02

Specimen Examination: Test C22+41. Removed Specimens from Test cell for Examination. Under magnification Grain Boundary Etching noticed on Both Single And Double U-Bend Specimens After 168 hrs of Testing.

Also Noticed Some Staining on Both Specimens. Will continue with Same Solution for Another 168 hrs under same Testing Conditions.

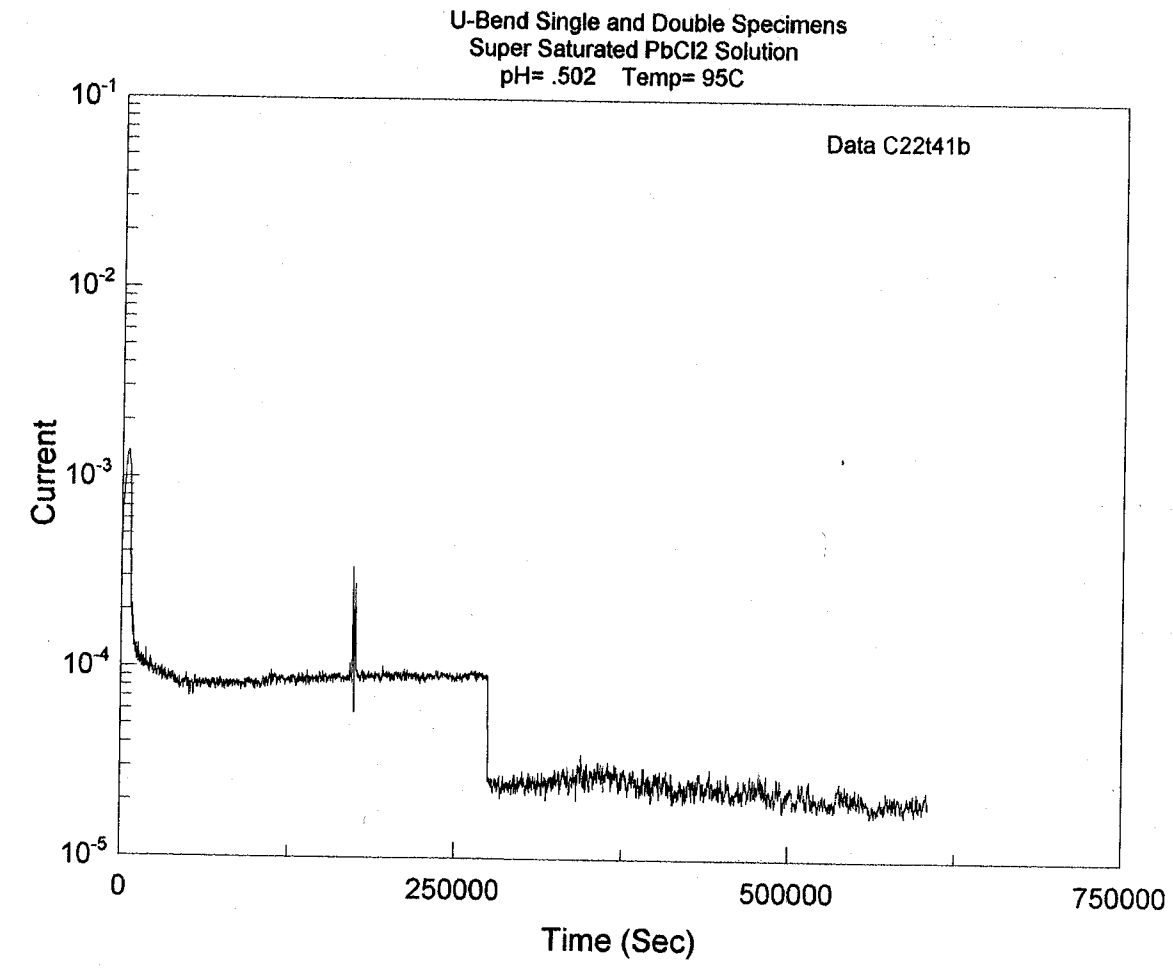
\* New Test Data C22+41b



\* Note: Surface Area Estimate for Specimen 46 cm<sup>2</sup>

B. R. J.  
4/8/02

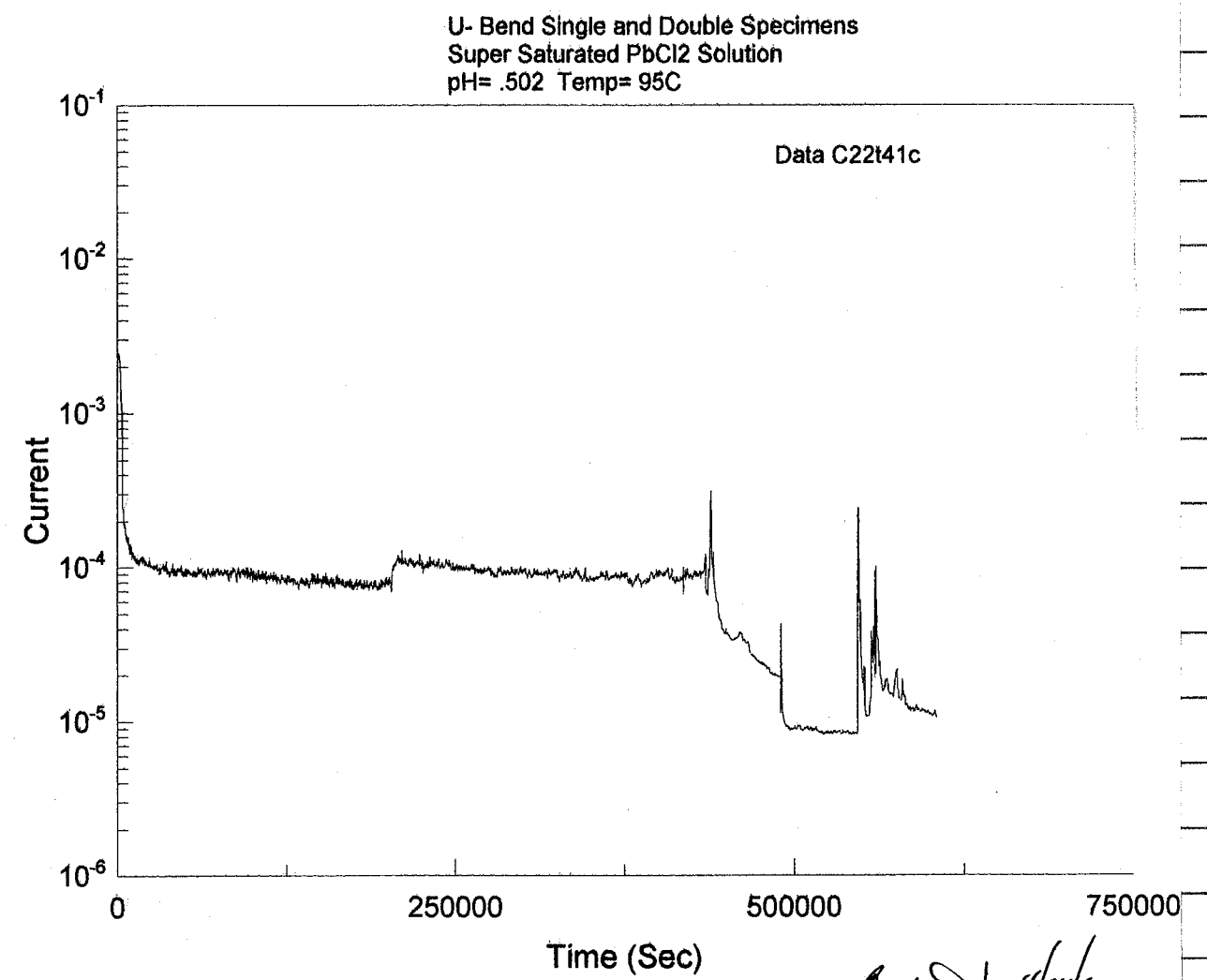
specimen Examination: Test C22+41b - Removed specimen from Test cell for Examination - Contact wires titanium from Double U-Bend Specimen Broke with little manipulation from my opinion we lost contact During Test - Under Magnification Surface Etching on Both Specimens Appears To have become Deeper But no cracking on Either Single or Double U-Bend Specimen After 336 hrs Total In  $PbCl_2$  Solution - will coat Ti wire with Microstop To Try To keep Contact with Specimens Restart Test Data C22+41c



B. K. Def 4/17/02

B. K. Def 4/17/02

specimen Examination: Test C22+41c - Removed specimens from Test cell for Examination - Contact wires looks Good - lost A Cooling Solution Pump Towards End of Test - Under Magnification Surface Etching Appears To be the same No cracking on Either Single or Double U-Bend Specimen After 504 hrs Total In  $PbCl_2$  Solution - will Restart Test AS = Data C22+41d

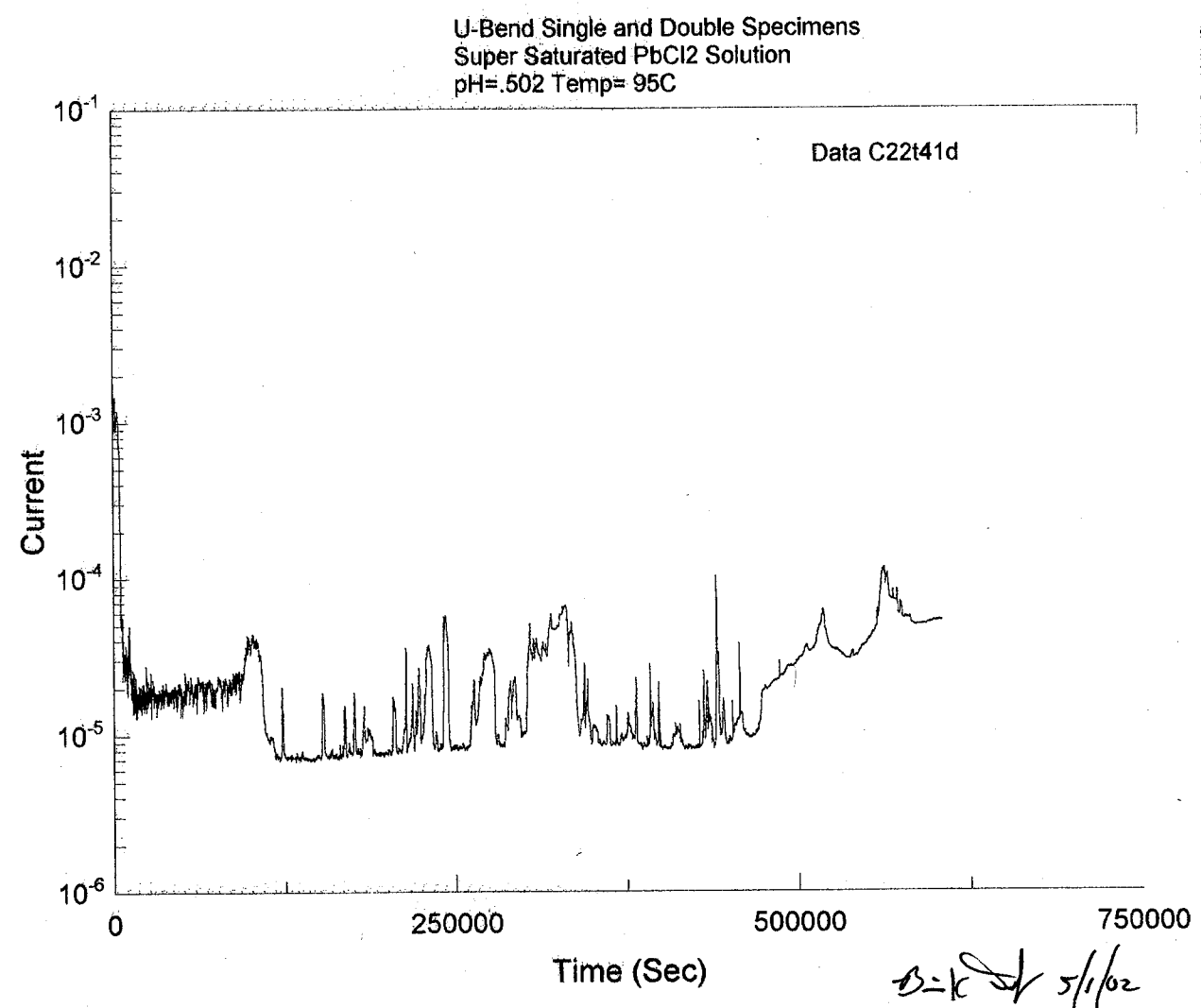


B. K. Def 4/24/02

B. K. Def 4/24/02

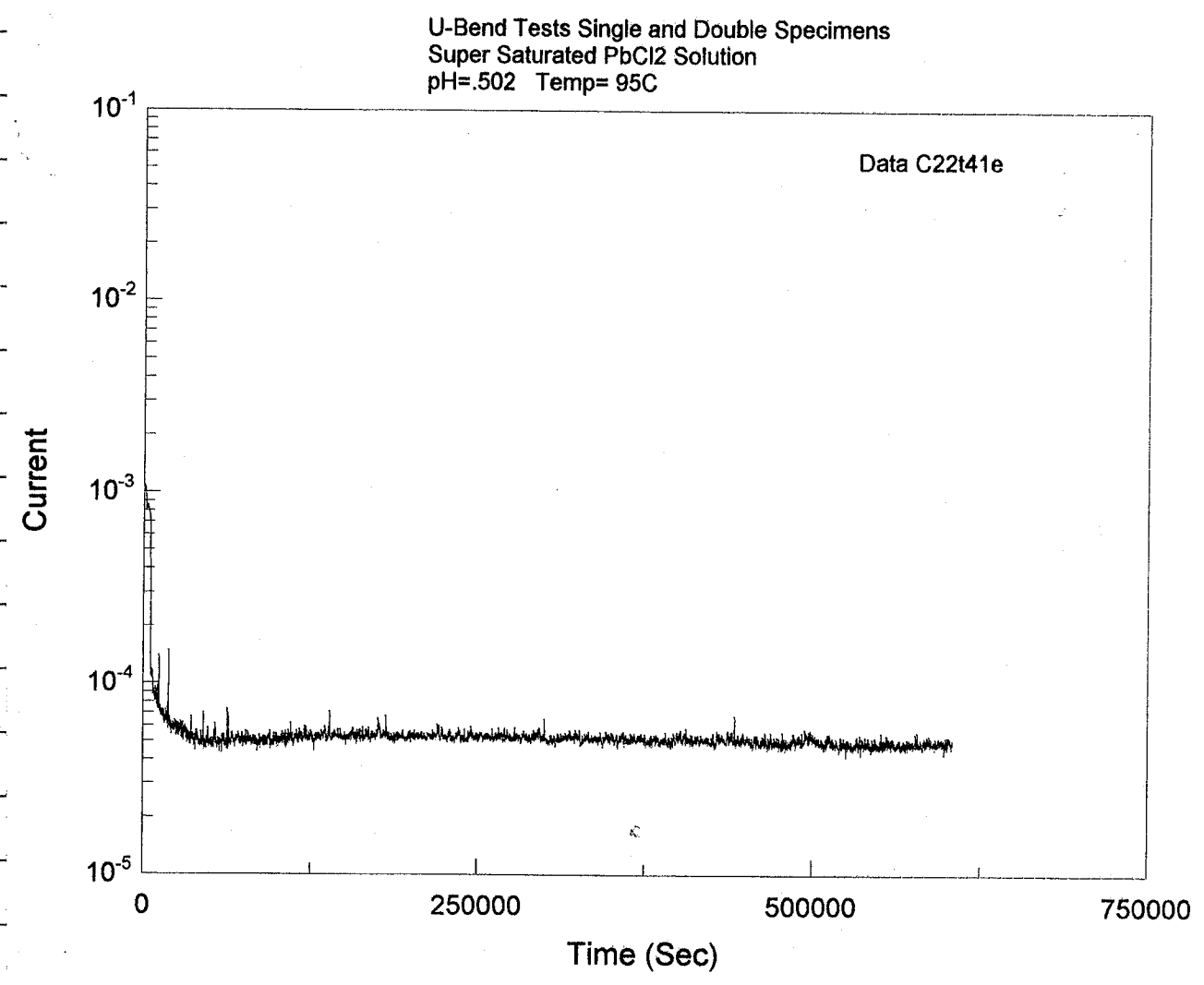


Specimen Examination: Test C22+41d - Removes Specimens from Test cell - Contact wires Great - Under magnification surface Etching Appears To Be the same As previous Test - No Cracking on Either Single or Double U-Bend specimen After 672 hrs Total In  $PbCl_2$  Solution will continue Testing  
Test Data C22+41c



Bik  
5/1/02

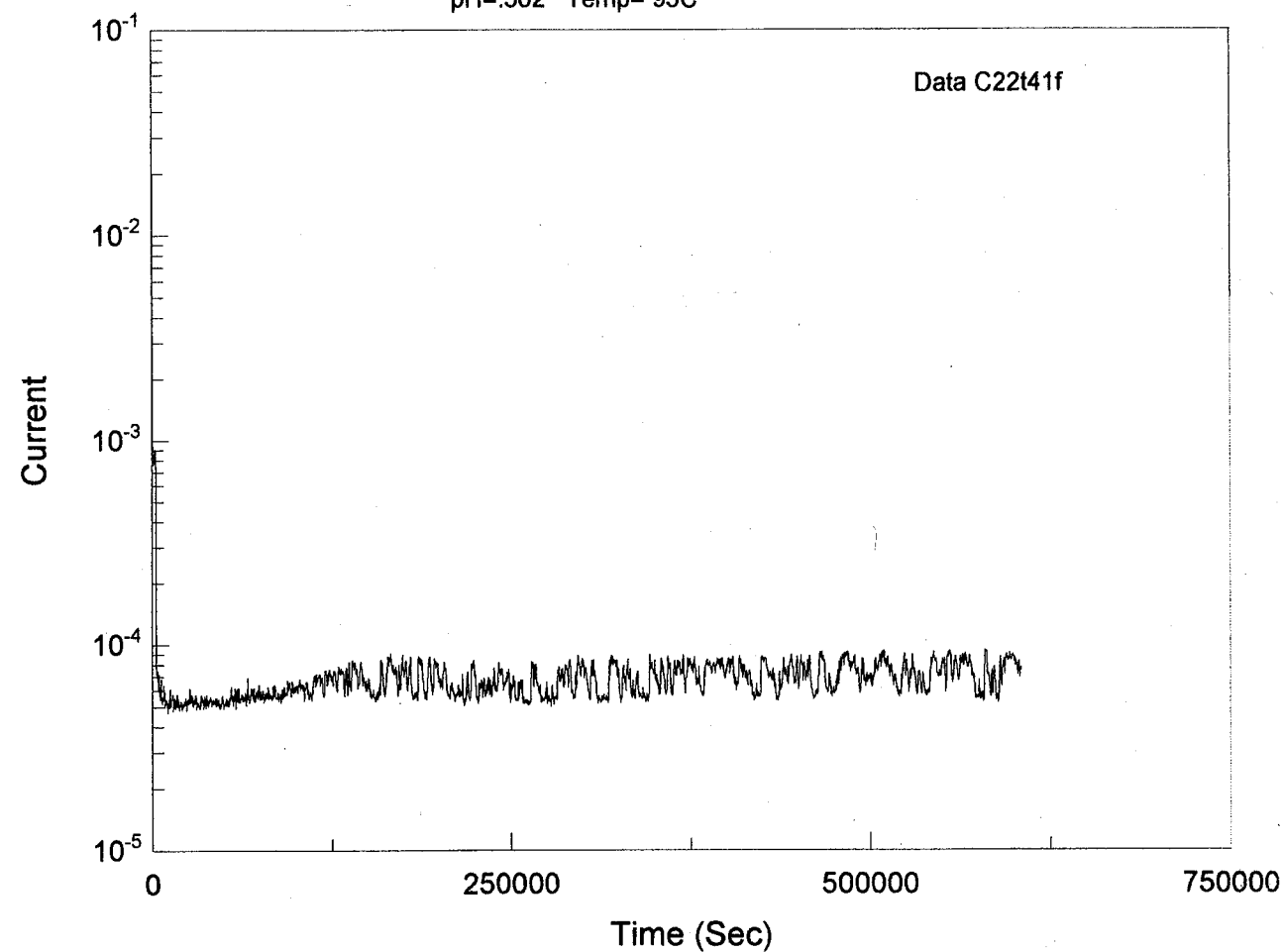
Specimen Examination: Test C22+41e - Removes Specimen from Test cell  
No Change Under Magnification Surface Etching Appears To Be the same As previous Test - No sign of Cracking on Either Single or Double U-Bend specimens After 840 hrs Total In  $PbCl_2$  Solution will continue Testing - New Test Data C22+41F



Bik  
5/9/02

Specimen Examination: Test C22+41F Removes Specimen from Test Cell  
 Test Completes with 972 hrs of testing in Super Saturated  
 $PbCl_2$  Solution on Both Single And Double U-Bend Specimen  
 Upon Examination of Specimen Under Magnification Notices A lot of  
 Surface Etching with grain Boundary Attack on All Specimens  
 Single And Double U-Bend Specimens Has staining on All Surfaces

U-Bend Tests Single and Double Specimens  
 Super Saturated  $PbCl_2$  Solution  
 pH=.502 Temp= 95C



B. K. D. J.  
 5/17/02

Potentiostatic Test Alloy C-22 U-Bend Specimen  
 $PbCl_2$  Solution @ -100mv

Objective: See pg #5

Specimen: Alloy C22 ASTM G-30 Single U-Bend Specimen #01

Dimensions 5"(L) x 3.975"(M) x 0.750"(W) x 0.125"(T) with 0.375 mounting Hole

Alloy C22 ASTM G-30 Double U-Bend Specimen #01

A= 5"(L) x 3.975"(M) x 0.750"(W) x 0.125"(T)

B= 5.395"(L) x 4.370"(M) x 0.750"(W) x 0.125"(T) with 0.375 mounting Hole x2

Start wt Single U-Bend: 58.20041g Sartorius Genius SN#12809099 cal 12/21/01

End wt Single U-Bend: 57.97741g

Start wt Double U-Bend: (A) 58.36225g (B) 63.76459g Sartorius Genius

End wt Double U-Bend: (A) 58.17556g (B) 63.53466g SN#12809099 cal 12/21/01

Solution:  $PbCl_2$  Super Saturated Solution from Testing C22 41

Start pH: .572 Fisher Accumet 950 meter SN#3340 cal 12/21/01

End pH: .592 pH probe #13-620-296 SN#110020Y

Potentiostat: Solartron 1287

Counter Electrode: PT Flay

Reference: Fisher 13-620-52 SN#057962<sup>SN 5/22/02</sup> 0199588

Temperature: 95°C Hg Thermometer SN#115749 cal 1/10/02

Solution Deaerates with 99.999%  $N_2$

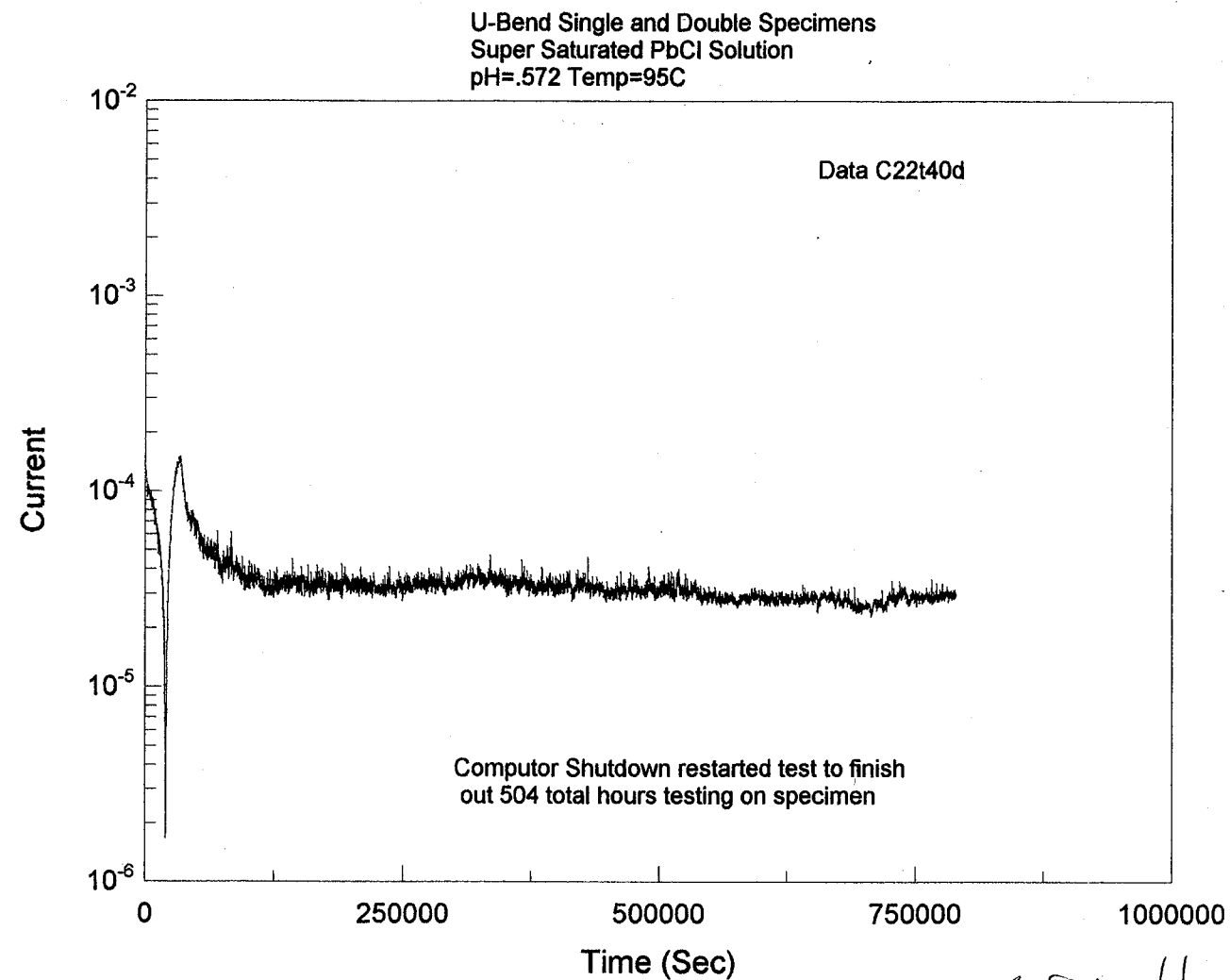
Econ = +117mv Keithley 617 SN#0579628 cal 3/10/02

Ept = +102mv

E Applied = -100mv

B. K. D. J.  
 5/22/02

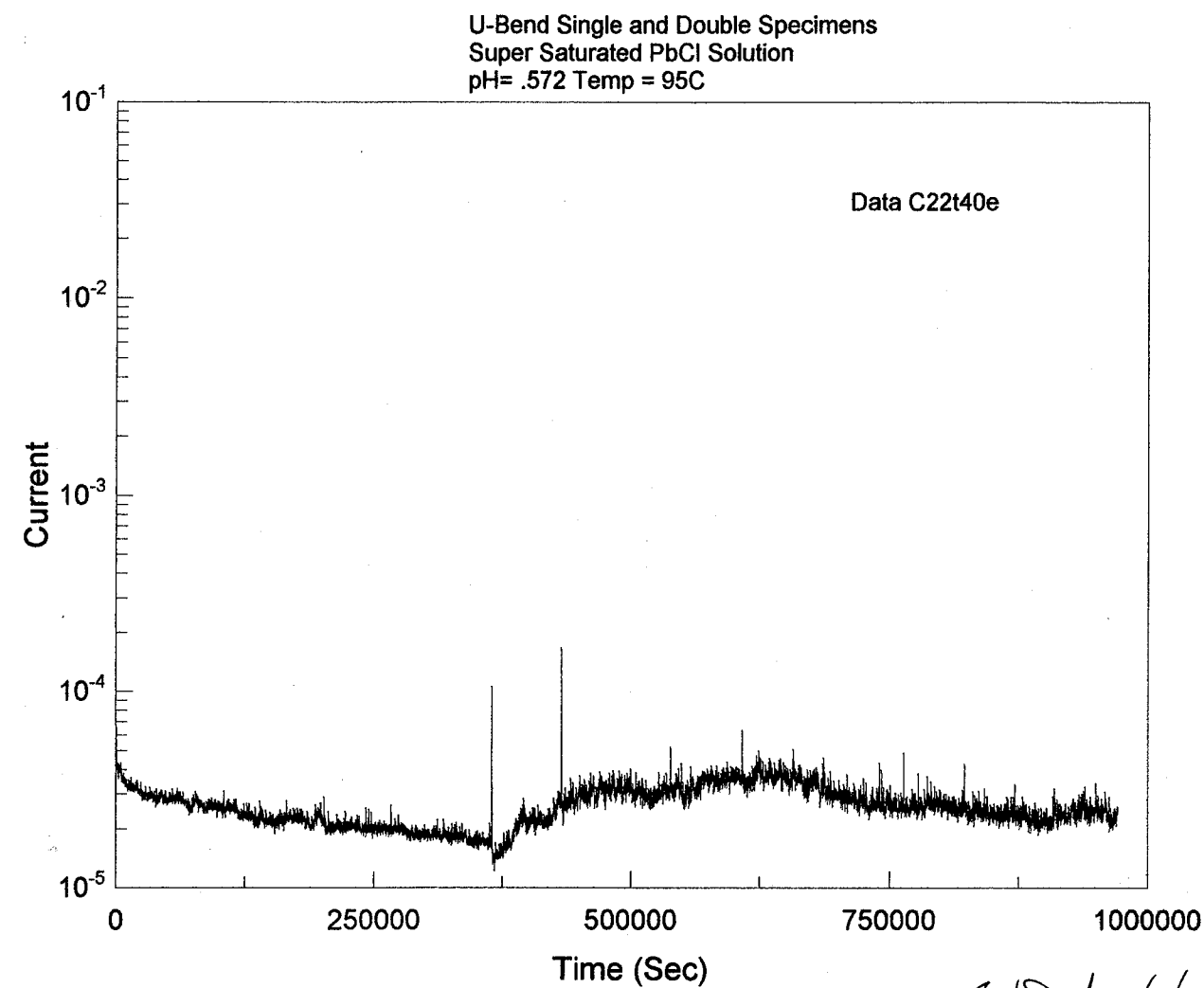
Test C22+40D : Computer shutdown, Don't break Test cell Apart  
 Restarted Test with same specimens And solution And same  
 Time Duration that we needed To finish Testing Procedure An Additional  
 270 hrs. The New Test Data C22+40e



B. P. D. 5/30/02

5/30/02

Test C22+40e : End of Testing with specimen #01 Total Test  
 Time Added To Specimen #01 Is 504 hrs - specimens Both Single  
 And Double U-Bends Have Etching that Is Deeper than Previous Testing  
 No Cracking on Either Specime



B. P. D. 6/11/02

6/11/02

U-Bero Solution Preparation

Solution: 2 Liters/2000mls prepared for U-Bero Testing

PbCl<sub>2</sub> Lot# L13K01 Deaerated with 99.999% N<sub>2</sub>

Total PbCl<sub>2</sub> Added To Solution 50.153g

6/11/02 : Starts Deaeration And stirring of 2L of DI water @ 3:30pm Heated To 95°C - kept At Temperature with stirring And Deaeration overnight (15 hrs.)

6/12/02 - @ 7:30 am Added 10.017g PbCl<sub>2</sub> Lot# L13K01 To Solution - Solution went cloudy - And Remained cloudy - kept Deaeration And stirring with Temperature @ 95°C for 2 hrs. Shut down Temp. Controller And let cell Cool overnight.

6/12/02 : Solution still cloudy  
Adjustment of pH

pH start = 4.576      Fisher Accumet 950 meter SN# 3340 cal 7/24/01

pH End = .509      pH Probe # 13-620-296 SN# 1100208

Added 94.5 mls of 6M HCl solution Lot# 002585 prepared 3/27/02

Starts To Reheat cell To 95°C @ 7:45 am solution still cloudy

Continue Deaeration And stirring of solution - solution went clear @ 8:30 am

Added 15.253g PbCl<sub>2</sub> Lot# L13K01 @ 8:40 am Temp: 65°C solution cloudy - then clear

Added 10.639g PbCl<sub>2</sub> Lot# L13K01 @ 9:40 am Temp 90°C - solution went cloudy

Added 5.036g PbCl<sub>2</sub> Lot# L13K01 @ 11:40 am Temp 95°C - solution cloudy with some small fine particles In bottom of cell

Added 5.206g PbCl<sub>2</sub> Lot# L13K01 @ 12:40 pm Temp 95°C - solution Remains cloudy - with small fine particles In bottom of cell

(continues on pg #139)

B.K. Duf 6/14/02

Added 4.002g PbCl<sub>2</sub> Lot# L13K01 @ 2:15pm Temp = 93°C  
solution Remains the same - cloudy with fine Particals

kept Deaeration And stirring going overnight @ 95°C

6/13/02 (7:15 am)

Solution slightly cloudy with fine Partical Drifts Around Bottom of cell - Break up material with glass Rod - solution

Returned To cloudy state with All material Broken Up. kept Deaeration And stirring going - Pulled Sample @ 9:00 am

for ICP Analysis with a 1:10 Dilution Ratio 20mls summates for Analysis - will continue with Deaeration And stirring @ 23°C

SOUTHWEST RESEARCH INSTITUTE  
PRELIMINARY SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SwRI  
Matrix: Liquid  
Work Order: 22646

Client: Division 20  
Date Received: 06/14/02  
Project No.: 20.01402.571

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.003
Lab Control	----	2.96
True Value	----	3.00
Recovery	----	98.7%
#1 PbCl2 Solution	207831	1470
Duplicate result	207831	1440
RPD	207831	2.06%
Spike result	207831	4500
Spike added	207831	3000
Recovery	207831	101.0%

Reporting Limit: 0.003 mg/L

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	205
True Value	----	200
Recovery	----	102%
#1 PbCl2 Solution	207831	1808
Duplicate result	207831	1767
RPD	207831	2.28%
Spike result	207831	2119
Spike added	207831	400
Recovery	207831	77.8%

Reporting Limit: 0.1 mg/L

B.K. Duf  
6/14/02



Cyclic Polarization of C-22 In  $\text{PbCl}_2$  Solution

Objective: See pg #5

Specimen: Alloy C22 Cylinder thermally Aged @  $870^\circ\text{C}$  for 5 min

Specimen Dimensions on pg #29 \* See bottom of page for thermally Age procedure

Start wt: 12.25591g Santarus Genius SN# 12809099 cal 6/4/02

End wt = 12.00648g

Solution: Super Saturated  $\text{PbCl}_2$  Lot# L13K01 See pg # 138-139

pH Start: .509 Fisher Accumet 950 meter SN# 3340 cal 7/24/01

pH End: Not Taken pH probe # 13-620-296 SN# 1100208

potentiostat = 1287 Solentum

Counter Electrode = Pt Flag

Reference = Fisher 13-620-52 SN# 0199588

Temperature =  $95^\circ\text{C}$  Hg Thermometer SN# 115749 cal 1/10/01

Ecorr: -203 mV Keithley 617 SN# 0579628 cal 5/10/01

Ept = -160 mV

Solution Deaerated with 99.999%  $\text{N}_2$ 

Specimen Examination = Pitting over Entire Surface - mla staining on Specimen

6/12/02

\* thermally Age C-22 Cylinders & U Bend Specimens  $870^\circ\text{C}$  for 5 min \*

over Linberg Model# 51333 - Omega Model HH22 microprocessor Thermometer

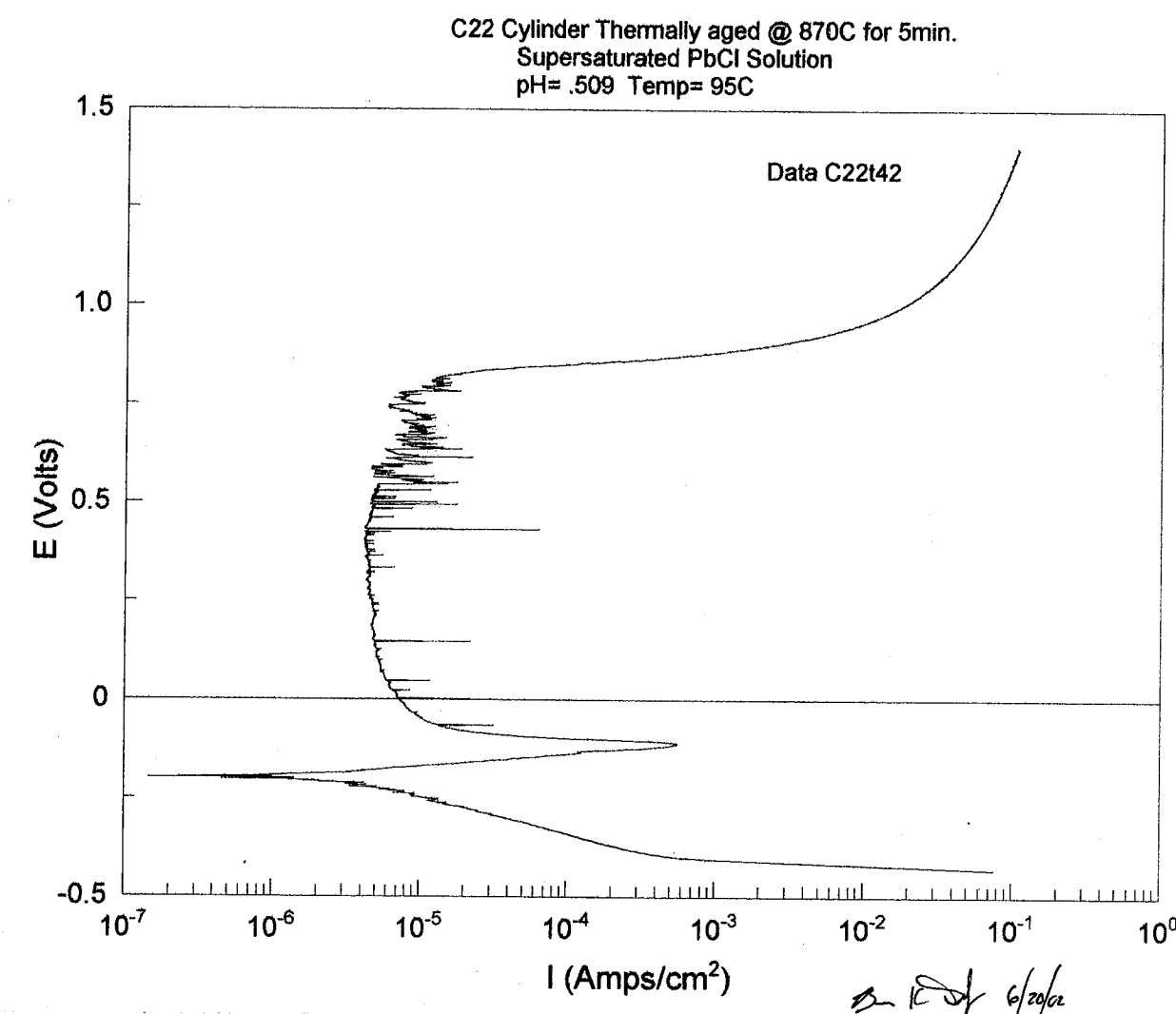
SN# T-94140 cal 3/22/02

thermocouple SN# 326 cal 1/11/02

over Set point @ 890 Meter Temp  $899.5^\circ\text{C}$  -  $903^\circ\text{C}$ 

Data C22t42

B. E. J. 6/24/02



B. E. J. 6/24/02

# Cyclic Polarization Using PT Flag In PbCl<sub>2</sub> Supersaturated Solution

objective: See pg #5

Specimen: PT Flag 9.72 cm<sup>2</sup> surface Area

Solution: Super Saturated PbCl<sub>2</sub> Lot # 413/K01 See pg # 138-139  
 start pH = .509 Fishen Accuret 950 meter SN# 3340 cal 7/24/01  
 End pH = Not Taken pH probe # 13-620-296 SN# 1100208

potentiostat: Solartec 1287

Counter Electrode: PT Flag

Reference Electrode: Fishen 13-620-52 SN# 0199588

Temperature: 95°C H<sub>2</sub> thermometer SN# 115749 cal 1/10/02

Deaeration: 99.999% N<sub>2</sub>

E<sub>corr</sub> = -263 mV Keithley 617 SN# 0579628 cal 5/10/02

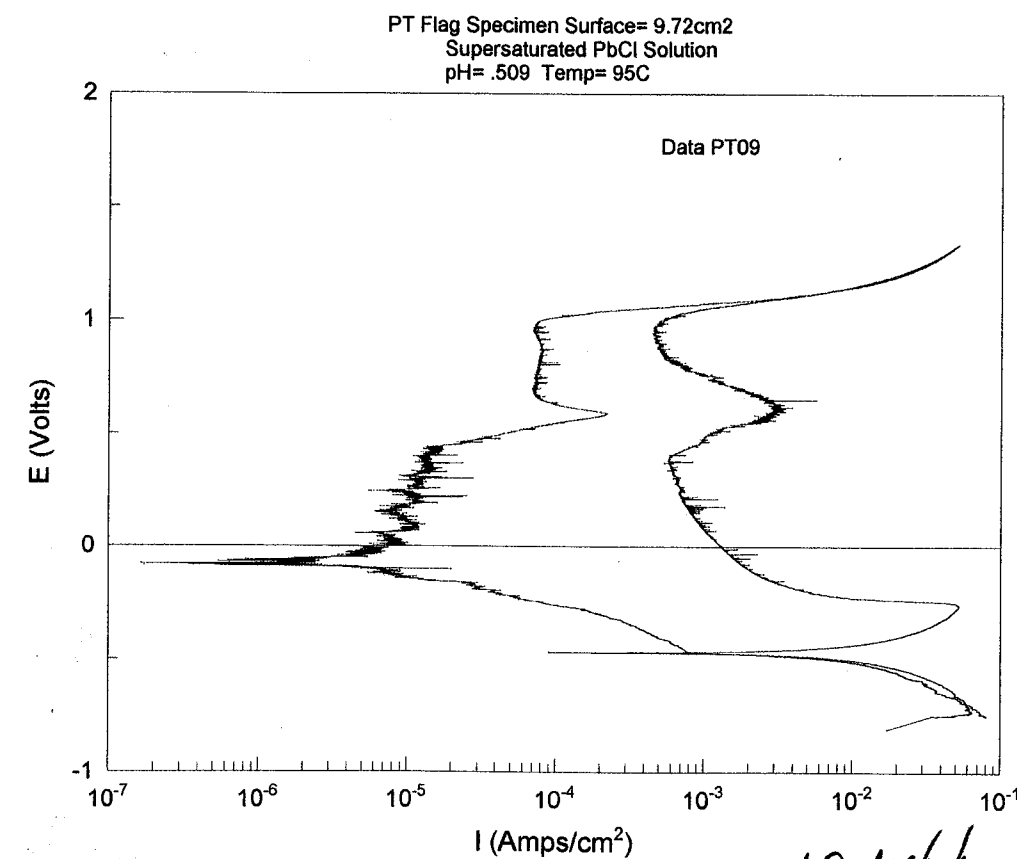
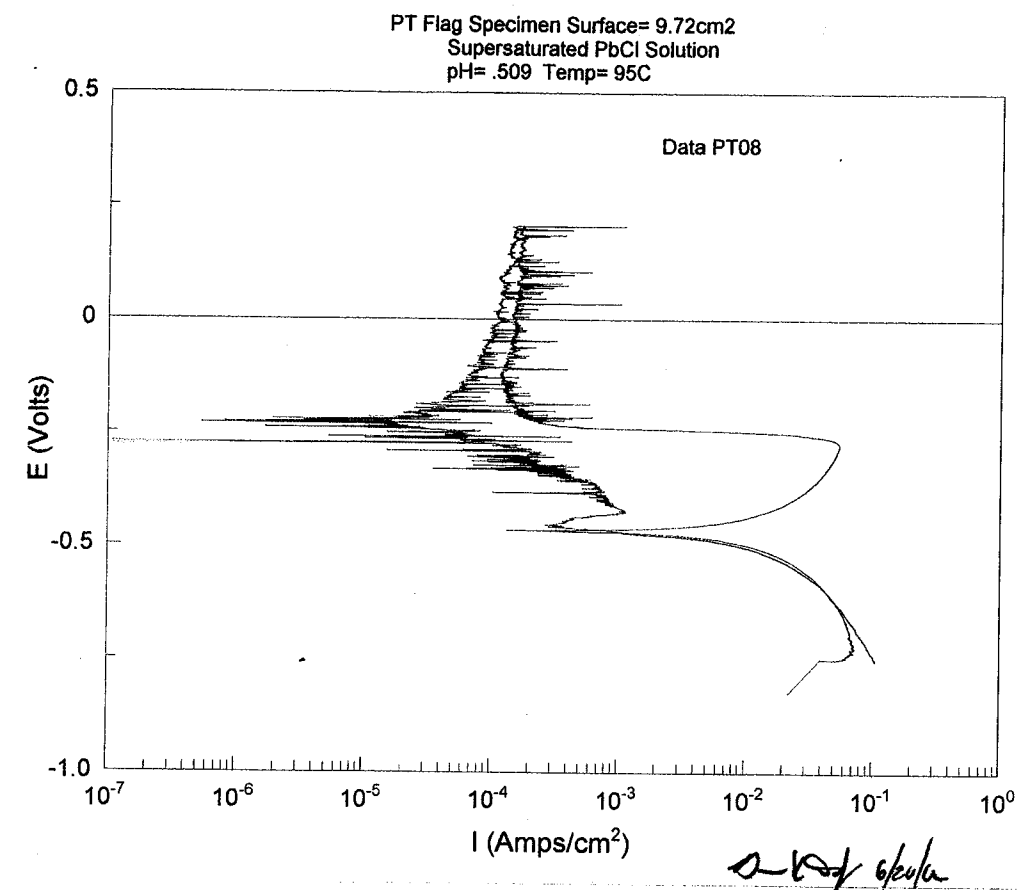
E<sub>pt</sub> = -466 mV

Solution Deaerates Before Each Test

Specimen Examination: A buildup of material from solution on both PT Flags  
 cleaned both surfaces in between tests

Data PT08 & PT09

6/24/02



6/20/02

# Cyclic Polarization of C-22 In PbCl<sub>2</sub> Solution

objective: see pg #5

specimen: Alloy C-22 cylinder - thermally Age @ 870°C for 5 min  
Specimen Dimension on pg #29 - see pg #140 for thermally Age procedure

start wt: 12.34753g Sartorius Genius SN# 12809099 cal 6/4/02

End wt: 12.34202g

solution: Supra Saturated PbCl<sub>2</sub> Lot # ~~613/01~~ <sup>614/01</sup> See pg #138-139

pH start: .509 Fisher Accumet 950 meter SN# 3340 cal 7/24/02

pH End: Not Taken pH probe # 13-620-296 SN# 1100208

potentiostat: Solartron 1287

Counter Electrode: Pt Flay

Reference: Fisher 13-620-52 SN# 0199588

Temperature: 95°C Mg Thermometer SN# 115749 cal 1/18/02

Ecorr: -258 mV Keithley 617 SN# 0579628 cal 5/10/02

Ept: -231 mV

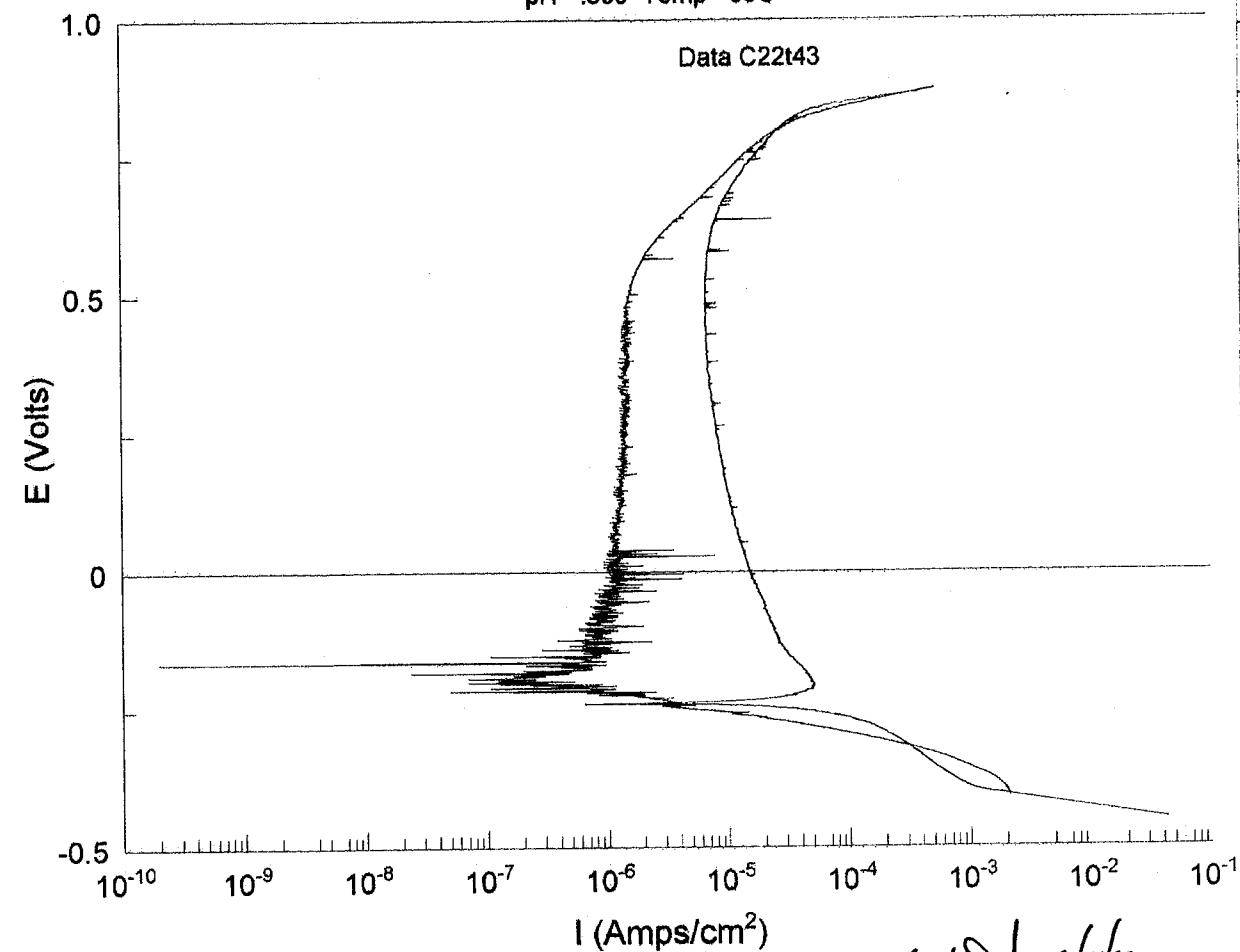
solution Degassed with 99.999% N<sub>2</sub>

Specimen Examination: Staining on surface of Specimen - Under Microscope  
Examination - some small pitting Notice on specimen

Data C22+45

B. J. D. J.  
6/24/02

C22 Cylinder thermally aged @ 870°C for 5 min.  
Supersaturated PbCl<sub>2</sub> Solution  
pH= .509 Temp= 95°C



B. J. D. J.  
6/21/02

B. J. D. J.  
6/21/02

# Cyclic Polarization of C-22 In $PbCl_2$ Solution

object: See pg #5

specimen: Alloy C22 Cylinder - Specimen Dimensions on pg #29

Start wt: 12.51707 g      Sartorius Genius SN# 12509099 cal 6/4/02

End wt: 12.51601 g

Solution: Super Saturated  $PbCl_2$  Lot# 413 K01 See pg # 138-139

pH start = .509      Fisher Accumet 550 meter SN# 3340 cal 7/24/01

pH End = Not Taken      pH probe # 13-620-296 SN# 1100202

potentiostat = 1287 Solution

Counter Electrode: Pt Flay

Reference: Fisher 13-620-52 SN# 0199588

Temperature: 95°C      Hg thermometer SN# 115745 cal 1/10/02

E<sub>com</sub> = -241 mV      Keithley 617 SN# 0579628 cal 5/10/02

E<sub>pt</sub> = -251 mV

Solution Degassed with 99.999%  $N_2$

Specimen Examination: Staining on specimen at solution level - Under microscopic

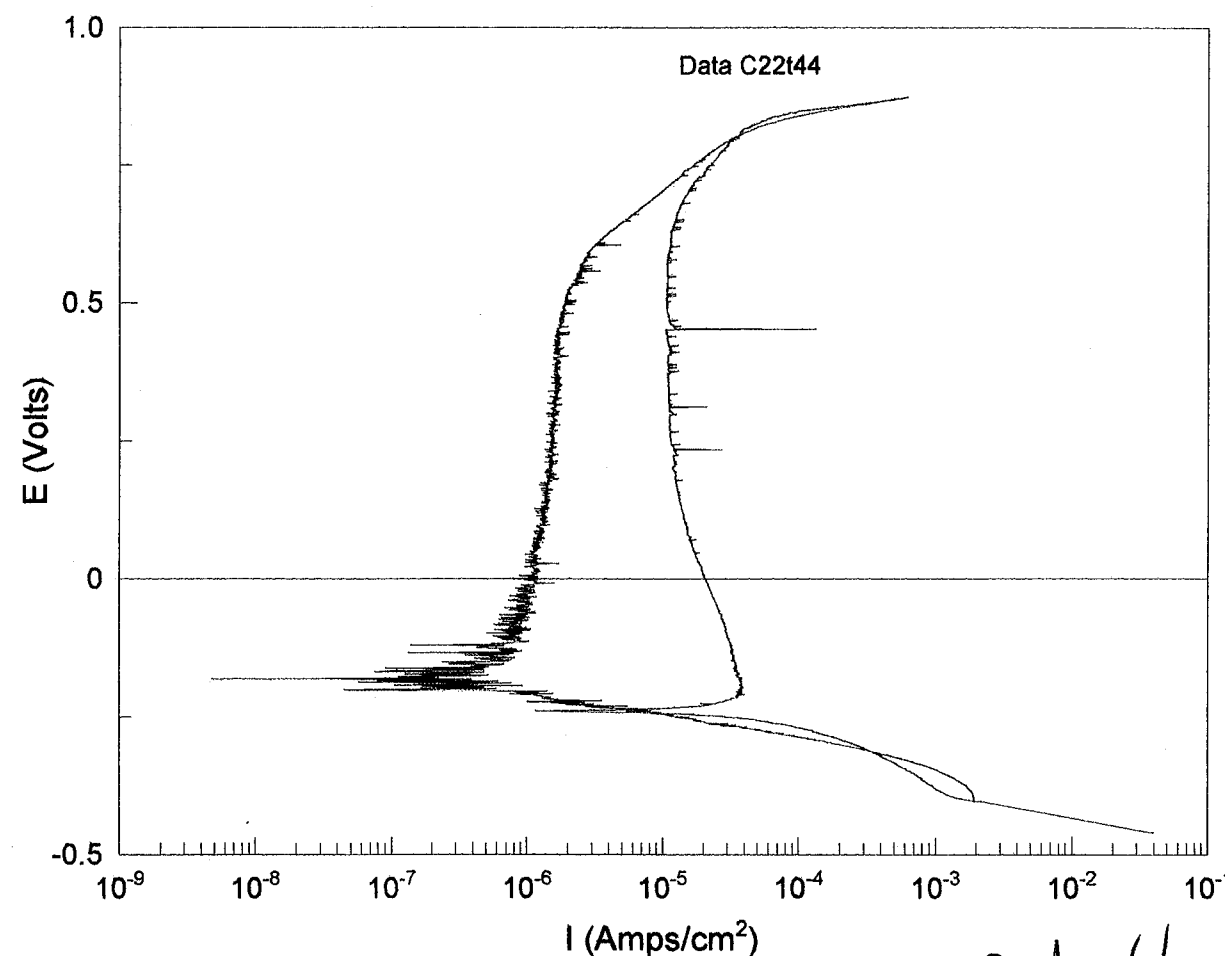
Examination: some small pitting noticed

\* Note Specimen Repolished for Further Testing

Data C22+44

B. P. D. / 6/21/02

C22 Cylinder      Supersaturated  $PbCl_2$  Solution  
pH = .509      Temp = 95C



B. P. D. / 6/21/02

B. P. D. / 6/21/02



After Running Test C22+42 - PTO8 - PTO9  
C22+43 And C22+44. Pulled 10mls of  
Super Saturates PbCl<sub>2</sub> - (See pg #138 for Solution And Previous  
ICP Results) ICP Analysis of Solution.

1:10 Dilution Ratio on Solution

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

Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SwRI      Date Received: 06/21/02  
Matrix: Liquid      Project No.: 20.01402.571  
Work Order: 22696

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.005
Lab Control	----	548
True Value	----	500
Recovery	----	110%
#1 PbCl <sub>2</sub> Solution	208240	1190
Duplicate result	208240	1214
RPD	208240	2.00%
Spike result	208240	1750
Spike added	208240	500
Recovery	208240	112%

Reporting Limit: 0.005 mg/L

B. R. 6/25/02

B. R. 6/25/02

ICP Chlorine Results from Solution on P. #148  
1:10 Dilution Ratio

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

Lab Name: Southwest Research Institute      Client: Division 20  
Lab Code: SwRI      Date Received: 06/21/02  
Matrix: Liquid      Project No.: 20.01402.571  
Work Order: 22696

Sample ID	Lab System ID	Chloride Results (mg/L)
Prep Blank	----	<0.1
Lab Control	----	205
True Value	----	200
Recovery	----	103%
#1 PbCl <sub>2</sub> Solution	208240	1592
Duplicate result	208240	1612
RPD	208240	1.25%
Spike result	208240	1798
Spike added	208240	200
Recovery	208240	103%

Reporting Limit: 0.1 mg/L

B. R. 7/3/02

B. R. 7/3/02

Potentiostatic Test Alloy C-22 U Bend Specimen  
PbCl<sub>2</sub> Solution @ -100 mV

objective: See pg #5

Specimen: Alloy C22 Astm G-30 Double U-Bend Specimen #03

Thermally Aged 5 min @ 870°C + Double U-Bend Specimen #05 <sup>As Rec'd</sup>

Dimensions 5"(L) x 3.975"(m) x 0.125"(T) = A plus x 0.750"(w)

5.395"(L) x 4.370"(m) x 0.750"(w) x 0.125"(T) = B

with 0.375" mounting Hole x 2

Start wt #03 = A.) 59.99193g B.) 64.32725g Sartorius Genius SN# 12809099

End wt #03 = A.) 59.63472g B.) 63.76857g cal 6/4/02

Start wt #05 A.) 60.28828g B.) 64.64974g Sartorius Genius SN# 12809099

End wt #05 A.) 60.04064g B.) 64.28992g cal 6/4/02

Solution: PbCl<sub>2</sub> Super Saturated - Solution Preparation pg #138

Start pH = .509 Fisher Accumet 950 meter SN# 3340 cal 7/24/02

End pH = .635 pH probe 13-620-296 SN# 1100208

Potentiostat: Solartron 1287

Counter Electrode: Pt Flay

Reference: Fisher 13-620-52 SN# 049588

Temperature: 95°C H<sub>2</sub> Thermometer SN# 115749 cal 1/10/02

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

E<sub>corr</sub> = -245 mV Keithley 617 SN# 0579628 cal 7/10/02

E<sub>pt</sub> = -237 mV

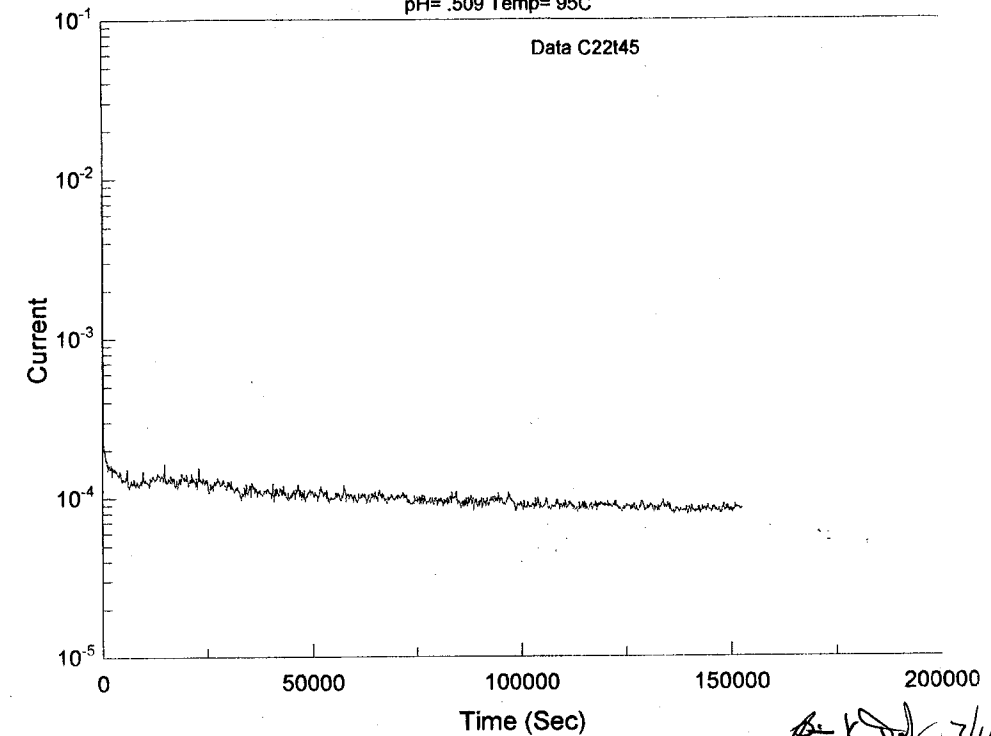
E<sub>Appl</sub> = -100 mV

Test C22+45

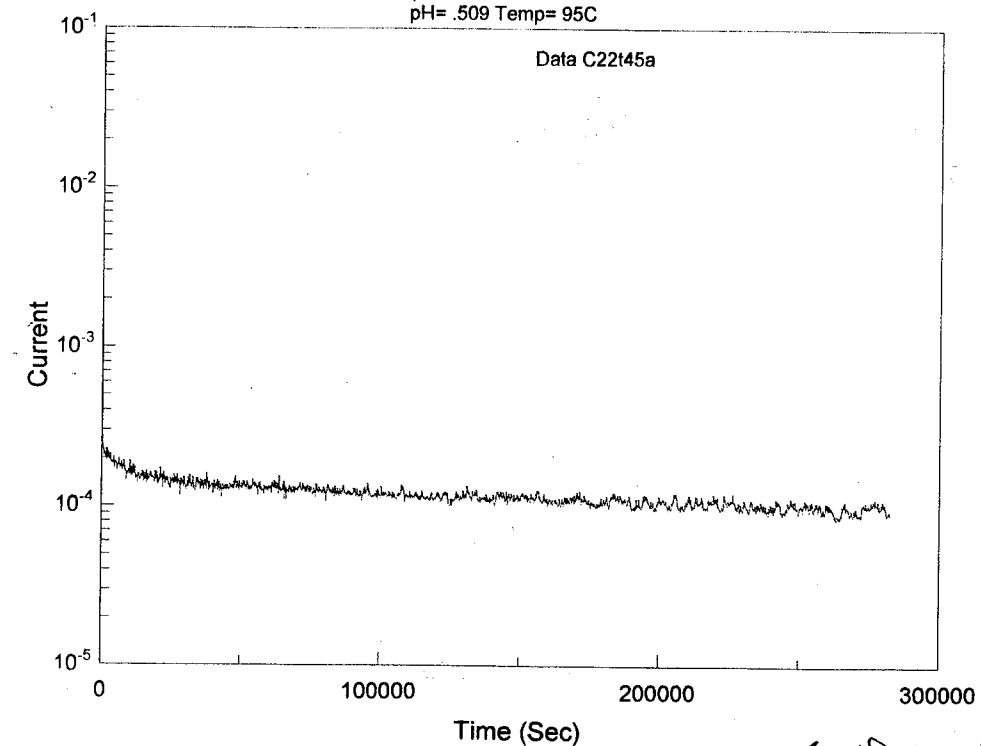
B. K. D. J.  
6/26/02

No Specimen Examination - other than Visual thru cell

U-Bend Tests both specimens Double U-Bend  
#03 Thermally aged 5 min @ 870C  
Super Saturated PbCl<sub>2</sub> Solution  
pH = .509 Temp = 95C



U-Bend Tests both specimens Double U-Bend  
#03 Thermally aged 5 min @ 870C  
Super Saturated PbCl<sub>2</sub> Solution  
pH = .509 Temp = 95C

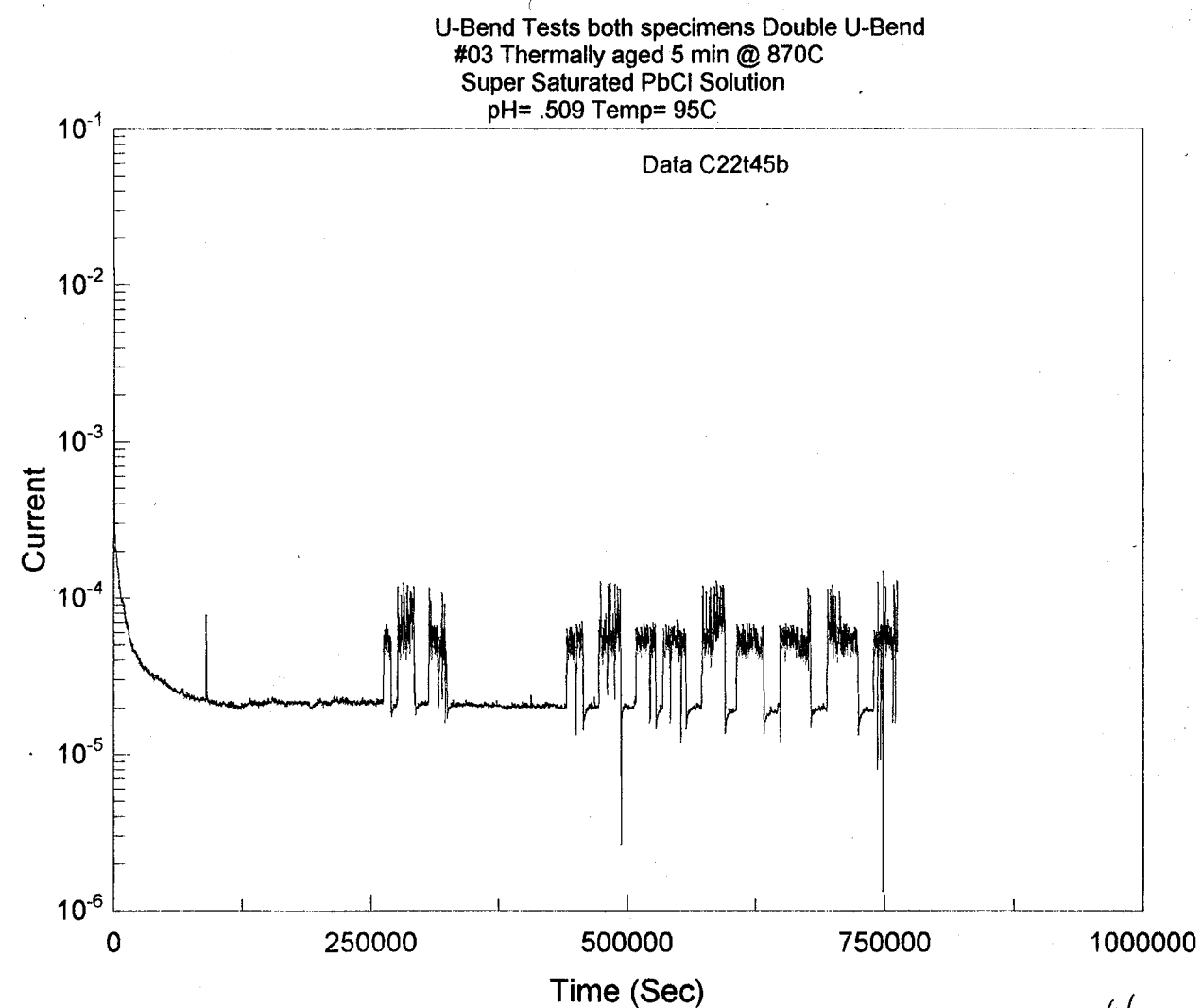


Note: Bad Electrical storms computer shutdowns Replaced

UPS for further testing

B. K. D. J.  
7/16/02

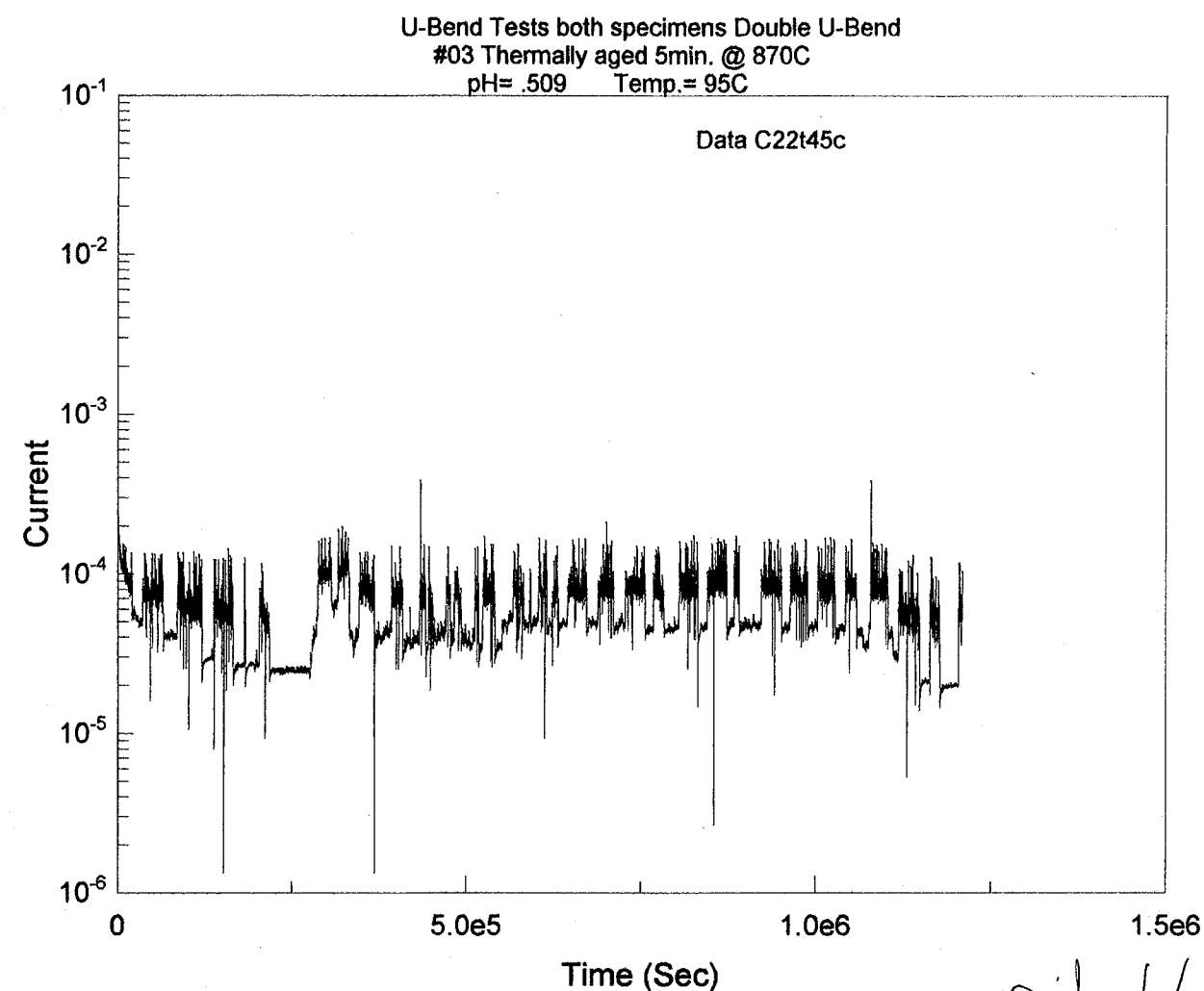
Test: C22+45b. Removes Specimens from Test Cell. Both Specimens look Great. 335 hrs of Testing In Super Saturated  $PbCl_2$  Solution. No Cracking or Surface Etching Just mild Staining will Restart Test. New Test: C22+45c for 336 hrs.



B. J. 7/10/02

B. J. 7/10/02

Test: C22+45c. Removes Specimens from Test Cell. Both Specimens show some Surface Etching. In Vapor phase Area of Specimen Around Crevice Bolt. 671 hrs Total Test Time In Super Saturated  $PbCl_2$  Solution. No Cracking Noticed on Either Specimen. Mild Etching. Mild Staining will Restart Test. New Test C22+45d for 336



B. J. 7/25/02

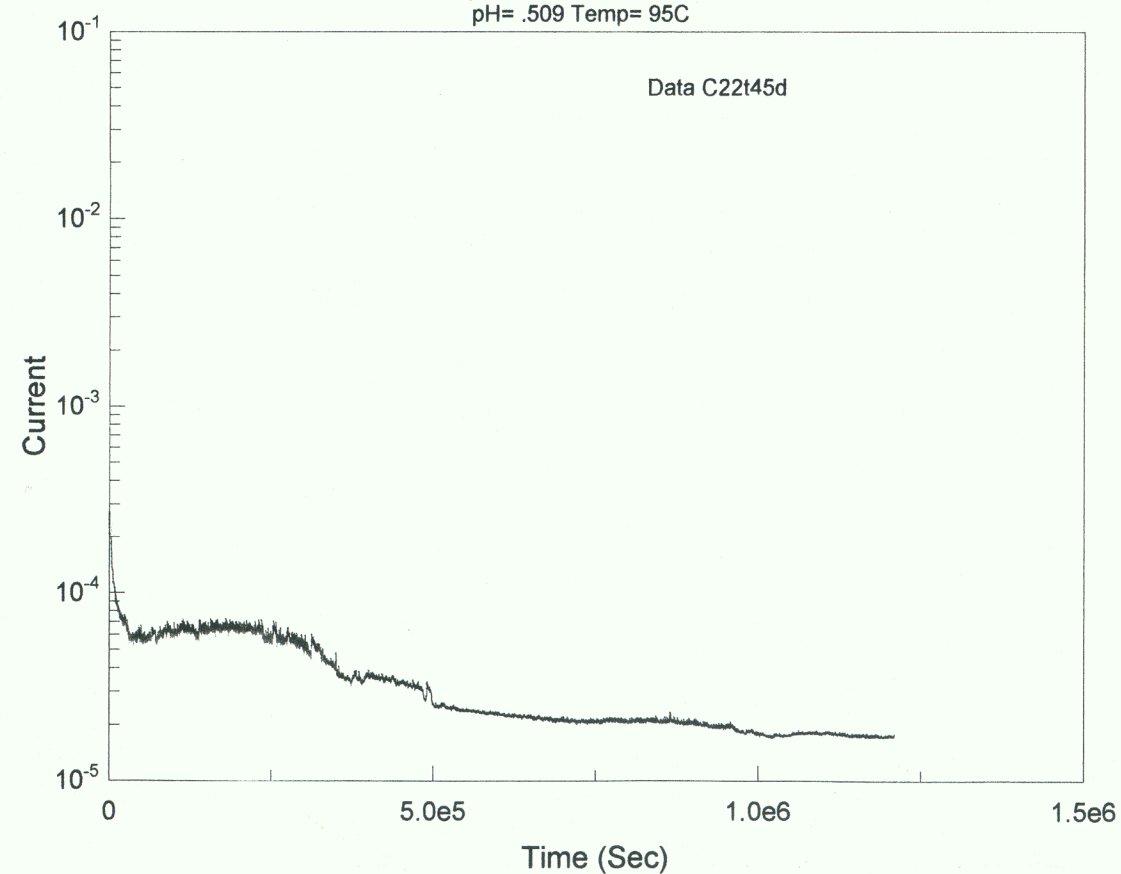
B. J. 7/25/02



Test C22+450 End of Testing on Specimen #03  
 Thermally Aged at 870°C for 5 min. And Specimen #05  
 Total <sup>800</sup>822/a Total Test Time 1007 hours on Both Specimens  
 Specimen Examination shows surface Etching on Both Specimens  
 In Vapor phase of Test cell. Staining on Both Specimens Mild  
 No cracking on either Specimen This is the end Testing of  
 Test 45 In super Saturated  $\text{PbCl}_2$  solution

\* Note this Test was run with A New UPS for the Computer  
 And Solution 1287. Cut Noise level Considerably on Testing  
 Compare All Tests of C22+45 - C22+45d

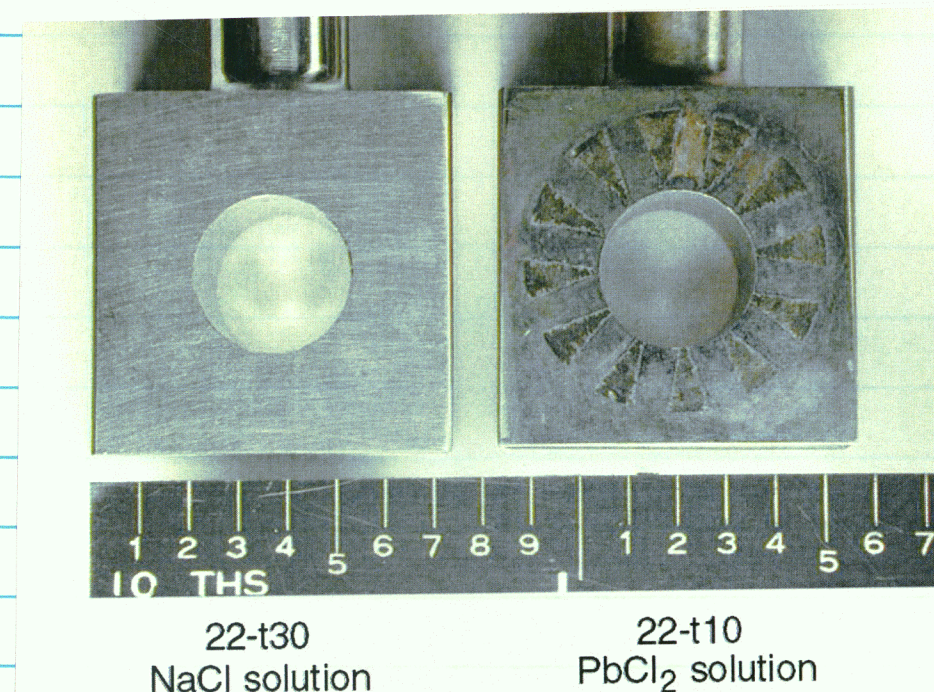
U-Bend Tests both specimens Double U-Bend  
 #03 Thermally aged 5 min @ 870C  
 Super Saturated  $\text{PbCl}_2$  solution  
 pH= .509 Temp= 95C



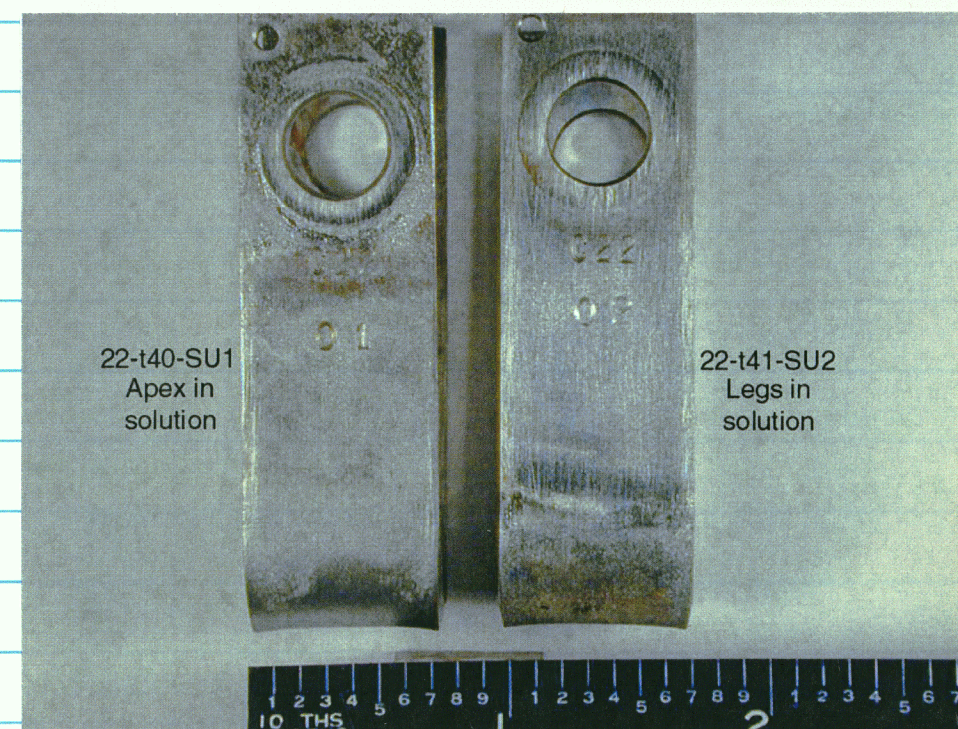
Bi R  
 8/23/02

# Photographs of Alloy 22 Specimens

## 1. crevice specimens 22-t10 and 22-t30



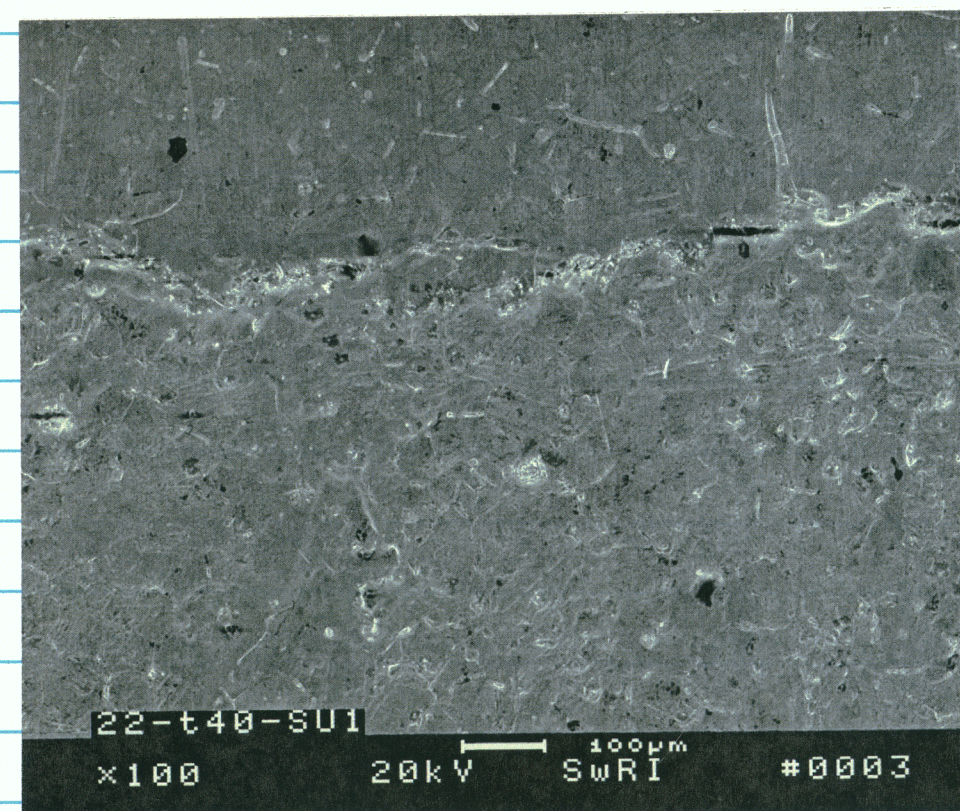
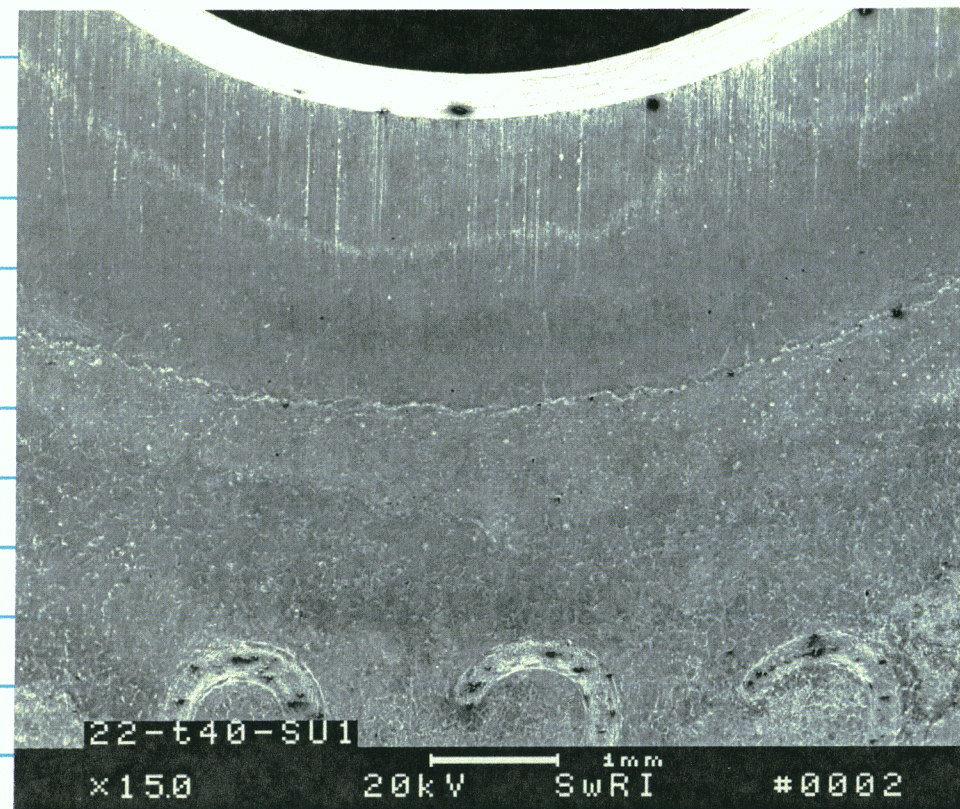
## 2. Single U-bend specimens 22-t40-SU1 and 22-t41-SU2



Yi Pan  
 8/26/02

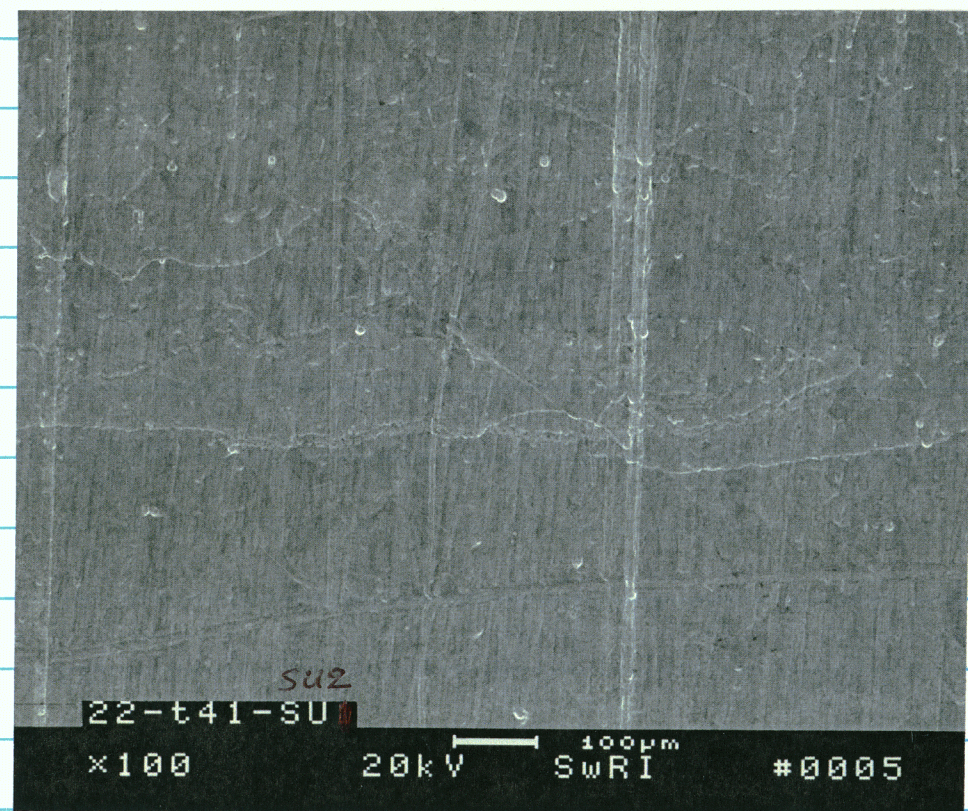
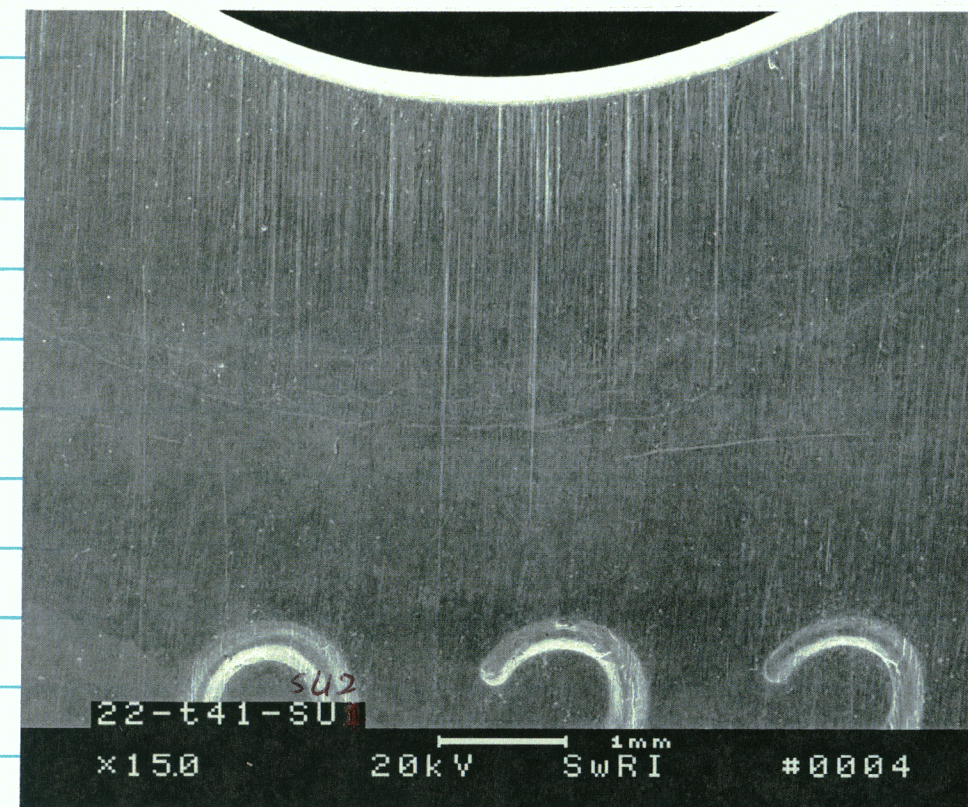


1. Specimen 22-t40-SU1 showing surface attack on the leg above the vapor/solution interface



*Yij Pan*  
8/26/02

2. Specimen 22-t41-SU2 showing minor attack on the leg immersed in the solution



*Yij Pan*  
8/26/02  
Copies sent to QA 9/11/02 *Bi-Kay*



New U-Bend Specimens for Testing

Darrell S. Dunn SwRI-CNWR Phone: (210) 522-6090 Fax: (210) 522-5184 e-mail: ddunn@swri.org	U-bend specimen CNWRA 20.06002.01.081.003 All Dimensions $\pm 0.010"$ unless otherwise specified  Page 1 of 2	To be completed at time of order: Material: <u>Welded Alloy 22 NG-GTAW</u> <u>(58Ni-22Cr-13.5Mo-3W-3Fe)</u> Heat: <u>059902LL2 &amp; XX2048BG</u> Specimen Orientation: <u>As shown</u> Other: <u>Framatome D62X plate</u>
--	--	---

MATERIAL INFORMATION  
NOTEBOOK 505  
PAGES 2-25

0.75"  
0.50"  
Weld  
2.50"  
0.375" dia  
0.50"  
0.125  
+/- 0.005"  
5.00"

*Darrell S. Dunn* 2/12/03  
Initiated by: D. Dunn Date

*V. Jain* 2/12/03  
Reviewed by V. Jain Date

*B. Mabrito* 2/12/2003  
QA Approval B. Mabrito Date

Continued on pg #159

*B. Mabrito*  
2/26/03

Darrell S. Dunn SwRI-CNWR Phone: (210) 522-6090 Fax: (210) 522-5184 e-mail: ddunn@swri.org	Location of U-bend specimens CNWRA Drawing 20-06002-01-081-003  Page 2 of 2	To be completed at time of order: Material: <u>Welded Alloy 22 NG-GTAW</u> <u>(58Ni-22Cr-13.5Mo-3W-3Fe)</u> Heat: <u>059902LL2 &amp; XX2048BG</u> Specimen Orientation: <u>As shown</u> Other: <u>Framatome D62X plate</u>
--	--	---

Mill 0.020" from original surface  
Top and bottom surfaces

Specimen orientation:

7.87"  
1.50"

*Darrell S. Dunn* 2/12/2003  
Initiated by: D. Dunn Date

*V. Jain* 2/12/03  
Reviewed by V. Jain Date

*B. Mabrito* 2/12/2003  
QA Approval B. Mabrito Date

*B. Mabrito*

## U-Benz Solution Preparation

Solution: 2 Liters / 2000mls prepared for U-Benz Testing

$\text{PbCl}_2$  Lot# L13K01 Degassed with 99.999%  $\text{N}_2$

Total  $\text{PbCl}_2$  Added To Solution 50.462g

2/25/03: Started To heat Solution of DI water @ 7:30 A.M

\*Note: solution was degassed with  $\text{N}_2$  over the weekend At Room Temp

Also solution was stirred At the same Time.

Reaches Temperature of  $95^\circ\text{C}$  @ 9:20 A.M

Added 10.174g  $\text{PbCl}_2$  Lot# L13K01 @ 10:05 A.M

Solution went cloudy Continued To stir And Degassate

shut Down Heater @ 3:00 pm Solution cloudy

2/26/03 Cell cooled overnight solution cloudy still

Adjusted pH To 0.506

Starting pH 4.578

Used 6M HCl Solution Lot# 023844

Added 76.5 mls of 6M HCl for pH adjustment

Started To Reheat @ 9:26 A.M

Solution still Has some small Fine particles floating

In the Solution But went clearer when HCl was added

Solution went clear around  $60^\circ\text{C}$  will begin To Add

More  $\text{PbCl}_2$

See pg #161

B. K. J.  
2/27/03

2/26/03

9:55 Added 15.034g  $\text{PbCl}_2$  Lot# L13K01 @  $58.3^\circ\text{C}$

Solution went Clear At  $72.6^\circ\text{C}$

10:44 Added 10.019g  $\text{PbCl}_2$  Lot# L13K01 @  $80.8^\circ\text{C}$

Solution went cloudy then cleared up

some small Fine particles starting To form In Bottom of cell

11:31 Added 5.110g  $\text{PbCl}_2$  Lot# L13K01 @  $90.9^\circ\text{C}$

Solution same As previous cloudy with particles

\* Continued stirring And Degassing During Solution Mixing

12:59 Added 5.034g  $\text{PbCl}_2$  Lot# L13K01 @  $95^\circ\text{C}$

Solution same As previous

2:01 Added 5.091g  $\text{PbCl}_2$  Lot# L13K01 @  $95^\circ\text{C}$

Solution same As previous - cloudy with small particles At Bottom of cell

Total  $\text{PbCl}_2$  Lot# L13K01 Added To Cell = 50.462g

B. K. J.  
2/27/03

Cyclic Polarization of C-22 Cylinder In  $PbCl_2$ 

Objective: See pg #5

Specimen: Alloy C22 Cylinder Specimen Dimensions on pg #29  
600 Grit FinishStart wt = 12.5064g Santaricus Genius SN# 12509099 cal 11/15/02  
End wt = 12.50913g Due 5/15/03

Solution: See preparation on pg #160-161

Start pH: 5.07 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 Due 8/7/03

End pH: Not Taken pH probe # 13-620-296 SN# 2291257 P6

Potentiostat: Solartron 1287

Counter Electrode: Pt Flag

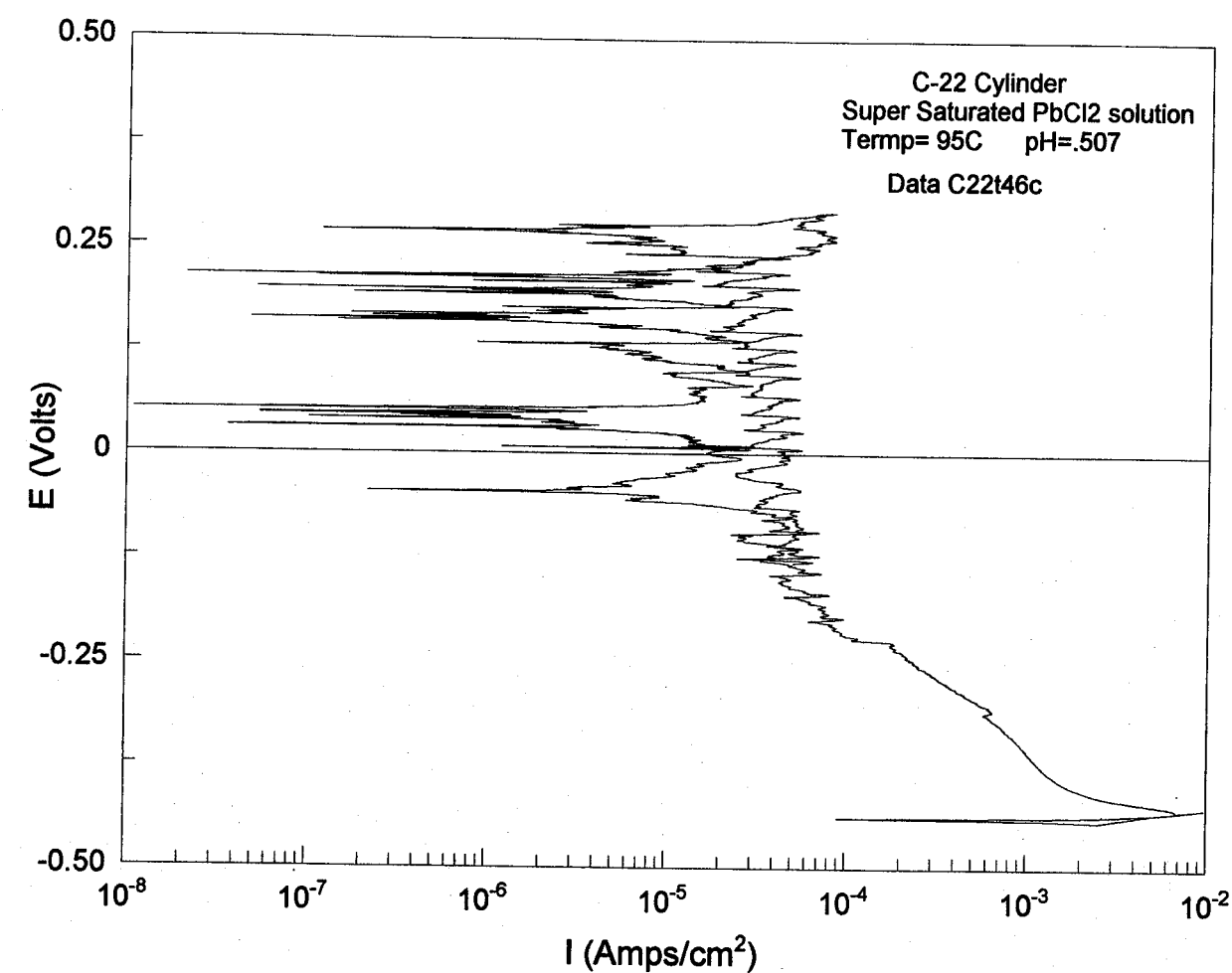
Reference: Fisher 13-620-52 SN# 0199588

Temperature: 95°C Hg Thermometer SN# 096-649 cal 1/10/03  
Due 7/10/03Solution Deaerates with 99.999%  $N_2$ 

Ecorr = +301 Keithley 617 SN# 0579628 cal 9/22/02

Ept = +145 Due 9/22/03

Specimen Examination: No Signs of Any Pitting or Corrosion

B. E. J.  
3/4/03B. E. J.  
3/4/03



# Cyclic Polarization of C-22 Crevice Specimen

Objective: See pg #5

Specimen: Alloy C-22 Crevice Specimen for Dimensions See pg #6

polished To A 600 Grit Finish - with 2 PTFE Crevice Washers

Attached At 50 In-Oz Using Proto 6104 SN# 139672 cal 8/28/02 due 2/28/03

Start wt: 40.18553g Sartorius Genius SN# 12809099 cal 11/15/02

End wt: 40.17851g due 5/15/03

Solution: See preparation on pg # 160-161

Start pH: .507 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/7/03

End pH: Not Taken pH probe # 13-620-296 SN# 2291257 P6

Potentiostat: Solartron 1287

Counter Electrode: PTFE

Reference: Fisher 13-620-52 SN# 0199588

Temperature: 95°C Hg Thermometer SN# C96-649 cal 1/10/03 due 7/10/03

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

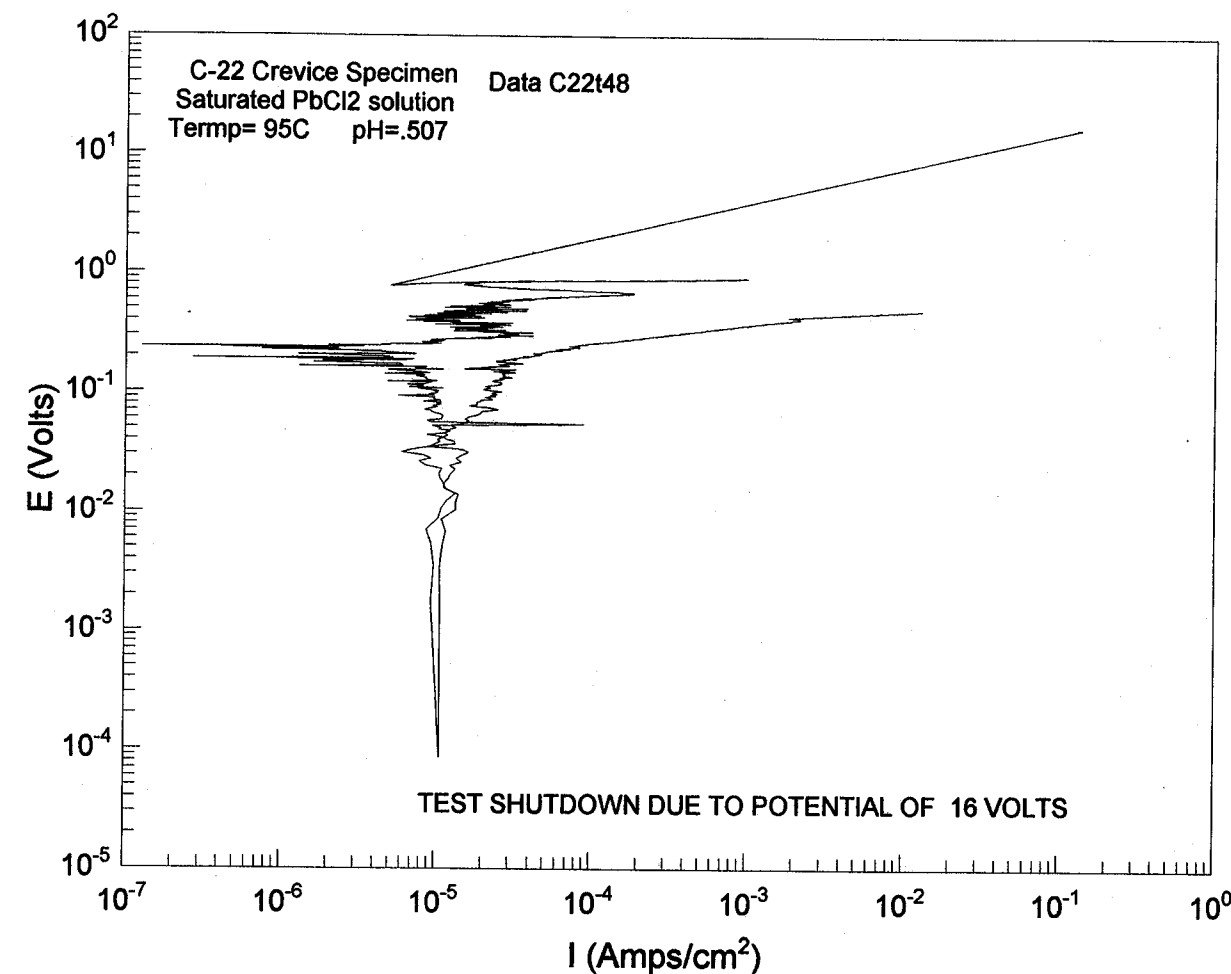
Ecorr = +319 mV Keithley 617 SN# 0579628 cal 9/22/02

Ept = +534 mV due 9/23/03

Specimen Examination: No Sign of Crevice Corrosion on Specimen

or staining - Repolish for Next Test Data C22t48

B. F. J.  
3/4/02



\* Generated Chlorine Gas Test shutdown

B. F. J.  
3/4/02

## U-Beno Solution Preparation

Solution: 2 Liters/2000mls prepared for U-Beno Testing  
 $\text{PbCl}_2$  Lot # L13 K01 Deaerated with 99.999%  $\text{N}_2$  + Stirrer  
 2 Batches Made #1 Batch Total  $\text{PbCl}_2 = 50.228\text{g}$   
 #2 Batch Total  $\text{PbCl}_2 = 50.035\text{g}$

#1

#2

2/28/03 - Deaerates 2 Liters  
 DI over the weekend  
 with 99.999%  $\text{N}_2$  while stirring

2/28/03 - Deaerates 2 Liters DI  
 over the weekend with  
 99.999%  $\text{N}_2$  while stirring

3/3/03 - Started To heat cell to  $95^\circ\text{C}$   
 @ 8:30 A.M. - still Deaerating + stirring

3/3/03 started To heat cell To  
 $95^\circ\text{C}$  @ 8:30 AM still  
 Deaerating And stirring

At 12:00pm Added 10.339g  $\text{PbCl}_2$   
 Kept Up To Temp And Deaeration  
 And stirring overnight

At 12:00pm Added 10.176g  $\text{PbCl}_2$   
 Kept Up To Temp And Deaeration  
 And stirring overnight

3/4/03 shut down Temp Controller  
 To let cell Cool 9:00 A.M.  
 Kept stirring And Deaeration going  
 over night

3/4/03 shut down Temp  
 Controller To let cell Cool 9:00 A.M.  
 Kept stirring And Deaeration  
 going overnight

3/5/03 - Adjusted pH  
 start pH = 4.839

3/5/03 Adjusted pH  
 start pH = 4.579

Adjusted To .501 with 86 mls  
 of 6M HCl Lot # 023844

Adjusted To .497 with  
 81 mls 6M HCl Lot # 023844

(Continued on pg # 167)

Bit D 3/10/03

#1

#2

3/5/03

Started To Reheat cells To  $95^\circ\text{C}$   
 while Deaerating + stirring  
 @ 8:40 A.M.

Solution went Clear @  $55.3^\circ\text{C}$

Added 15.294g  $\text{PbCl}_2$  @ 10:00am

Temp was  $85.6^\circ\text{C}$  - Solution Clear

3/5/03

Started To Reheat cells To  $95^\circ\text{C}$   
 while Deaerating + stirring  
 @ 8:40 A.M.

Solution went Clear @  $50.1^\circ\text{C}$

Added 15.285g  $\text{PbCl}_2$  @ 10:00am

Temp  $91.2^\circ\text{C}$  - Solution Clear

Added 10.206g  $\text{PbCl}_2$  @ 1:00pm  
 Solution Cloudy

Added 10.304g  $\text{PbCl}_2$  @ 1:00pm  
 Solution Cloudy

Added 5.016g  $\text{PbCl}_2$  @ 2:00pm  
 Solution cloudy

Added 5.017g  $\text{PbCl}_2$  @ 2:00pm  
 solution cloudy

Added 5.322g  $\text{PbCl}_2$  @ 3:00pm  
 solution cloudy with some fine particles  
 stirring on Bottom of cell

Added 5.214g  $\text{PbCl}_2$  @ 3:00pm  
 solution cloudy with fine particles  
 stirring on Bottom of cell

Added 4.057g  $\text{PbCl}_2$  @ 3:45pm  
 Solution cloudy with Solos stirring  
 In bottom of cell

Added 4.039g  $\text{PbCl}_2$  @ 3:45pm  
 Solution cloudy with Solos  
 stirring In bottom of cell

Dropped Temp To  $75^\circ\text{C}$  overnight  
 stirring And Deaeration continues

Dropped Temp To  $75^\circ\text{C}$  overnight  
 stirring And Deaeration continues

3/6/03 Reheats To  $95^\circ\text{C}$  Both cells - made sure All Solution were stirring Good  
 Pulled ICP Analysis sample @ 9:30 for each cell

Bit D 3/10/03

ICP Analysis for Test Solution Preparation  
From pg # 166 + #167

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 03/06/03

Matrix: Liquid

Project No.: 20.06002.01.081

Work Order: 23945

Sample ID	Lab System ID	Lead Results (mg/L)	Chloride Results (mg/L)
Prep Blank	----	<0.003	<0.1
Lab Control	----	0.497	208
True Value	----	0.500	200
Recovery	----	99.4%	104%
Solution # 1	221526	1620	2099
Duplicate result	221526	1620	2101
RPD	221526	0.00%	0.10%
Spike result	221526	2080	2472
Spike added	221526	500	400
Recovery	221526	92.0%	93.3%
Solution # 2	221527	1110	1398

Reporting Limit:                      0.003 mg/L                      0.1 mg/L

*B. E. D.* 3/19/03

ICP Analysis for Test Solution Preparation  
From pg # 160 + #161

**SOUTHWEST RESEARCH INSTITUTE**  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Client: Division 20

Lab Code: SwRI

Date Received: 02/28/03

Matrix: Liquid

Project No.: 20.06002.01.081

Work Order: 23905

Sample ID	Lab System ID	Lead Results (mg/L)	Chloride Results (mg/L)
Prep Blank	----	<0.003	<0.01
Lab Control	----	0.445	204
True Value	----	0.500	200
Recovery	----	89.0%	102%
#1	221084	887	1207
Duplicate result	221084	876	1206
RPD	221084	1.25%	0.08%
Spike result	221084	1350	1397
Spike added	221084	500	200
Recovery	221084	92.6%	95.0%

Reporting Limit:                      0.003 mg/L                      0.01 mg/L

*B. E. D.* 3/19/03

Cyclic Polarization of C-22 In  $PbCl_2$ 

Objective: See pg #5

Specimen: Alloy C-22 Crevice Specimen for Dimensions See pg #6

polished To A 600 Grit Finish - with 2 PTFE crevice washers

Attached At 50 In. Oz Using Photo 6104 SN# 139072 cal 8/06/03 due 9/06/03

Start wt: 40.16311 g Sartorius Genius SN# 12809099 cal 11/15/02

End wt = 40.16278 g due 5/15/03

Solution: See preparation on pg #166-167, #1 solution, Imp Analysis pg #168

Start pH: .501 Fisher Accum 950 meter SN# 33410 cal 8/7/02 due 8/7/03

End pH: .634 pH probe #13-620-296 SN# 2291257 Pb

potentiostat: Solartron 1287

Counter/Electronics: PT Fly

Reference: Fisher 13-620-52 SN# 0199568

Temperature: 95°C Hg thermometer SN# C96-649 cal 1/10/03 due 7/10/03

Solution Denotes with 99.999%  $N_2$ 

Ecorr = -156 mV Keithley 614 cal = 5/26/02

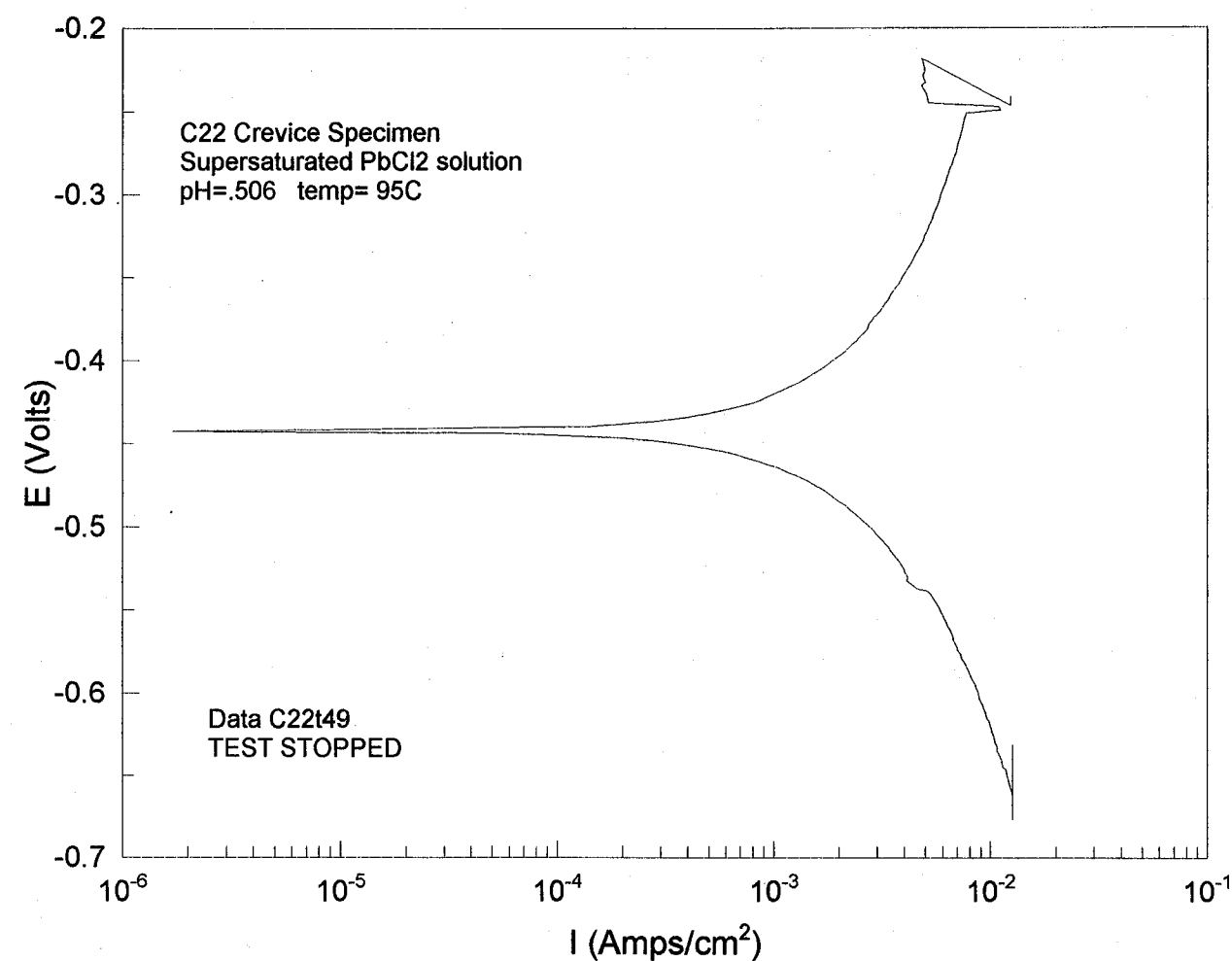
Ept = +350 mV SN# 0704934 Due: ~~5/26/02~~ <sup>6/10/02</sup> 5/26/03

Specimen Examination: No Crevice Corrosion - Test stopped - this specimen was

from prior test #48 - will not Repolish will use New Specimen for Next Test

Data C2249

3/19/03

B. J. D.  
3/19/03



## Cyclic Polarization of C-22 Crevice Specimen

Objective: See pg #5

Specimen: Alloy C-22 Crevice Specimen For Dimensions See pg #6

polished To A 600 Grit Finish - with 2 PTFE Crevice Washers Attached At  
SO In-Oz Using Photo 6104 SN#139072 cal 3/6/03 due 9/6/03Start wt: <sup>24/5/03</sup>~~40.822~~ 40.4815g Satorious Genius SN#12809099 cal 11/15/02 due 5/15/03End wt: <sup>24/5/03</sup>~~40.836~~ 40.4003g

Solution: See preparation on pg #166-167 - #1 Solution - Icp Analysis pg #168

Readjusted pH down Again with 23ul of 6M HCl lot# 023844 start .634

pH start = .521 Fisher Accuret 950 meter SN#3340 cal 8/7/02 due 8/7/03

pH End = .536 pH probe #13-620-296 SN#2291257 Pb

potentiostat: Solartron 1287

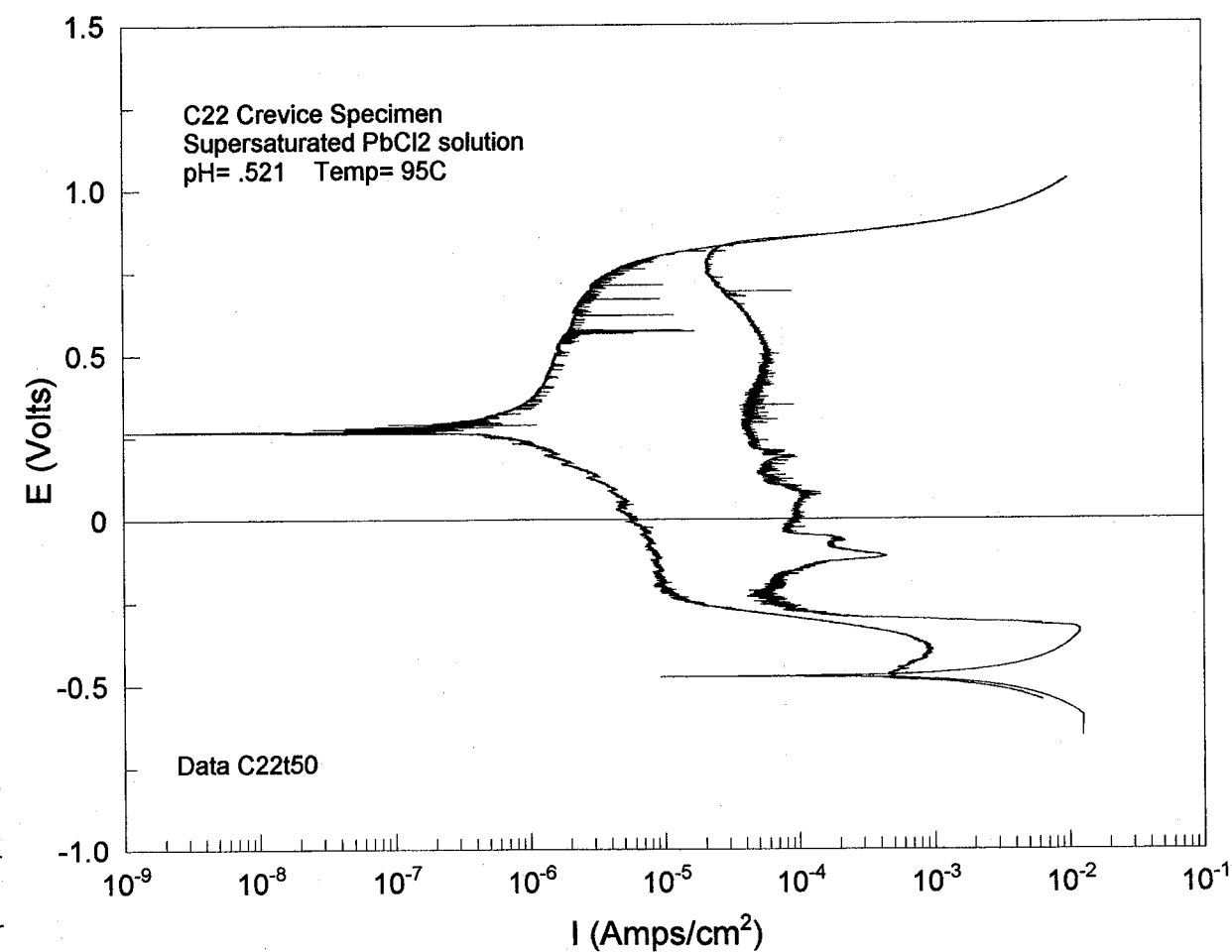
Counter Electrode: Pt Flg

Reference: Fisher 13-620-52 SN#0199568

Temperature: 95°C H<sub>2</sub> thermometer SN#C96-649 cal 1/10/03 due 7/10/03Solution Deaerated with 99.999% N<sub>2</sub>

Ecorr = -220 mV Keithley 614 SN#0704934 cal 5/26/02 due 5/26/03

Ept -123 mV

Specimen Examination: Crevice Corrosion on 24/24 feet of Crevice Washer  
mild staining on All SurfacesData C22t50  
B-K 3/19/03

B-K 3/19/03

## Cyclic Polarization of C-22 Crevice Specimen

objective: See pg #5

Specimen: Alloy C-22 Crevice Specimen for Dimensions See pg #6  
 polished To A 600 Grit Finish - with 2 PTFE Crevice Washers  
 Attached At 50 In-Oz Using Pauto 6104 SW#139072 Cal 3/6/03 due 8/6/03

Start wt: 40.60224g Santarious Genius SW#12809099 Cal 11/10/02  
 End wt: 40.59552g due 5/15/03

Solution: See preparation on pg #166-167 - #2 Solution - Top Analysis pg #168  
 Start pH: .497 Fisher Accumet 950 meter SW#3340 Cal 8/7/02 due 8/7/03  
 End pH: Not taken pH probe #13-620-296 SW#2291257 P6

potentiostat: Solartron 1287

Counter Electrode: Pt Fly

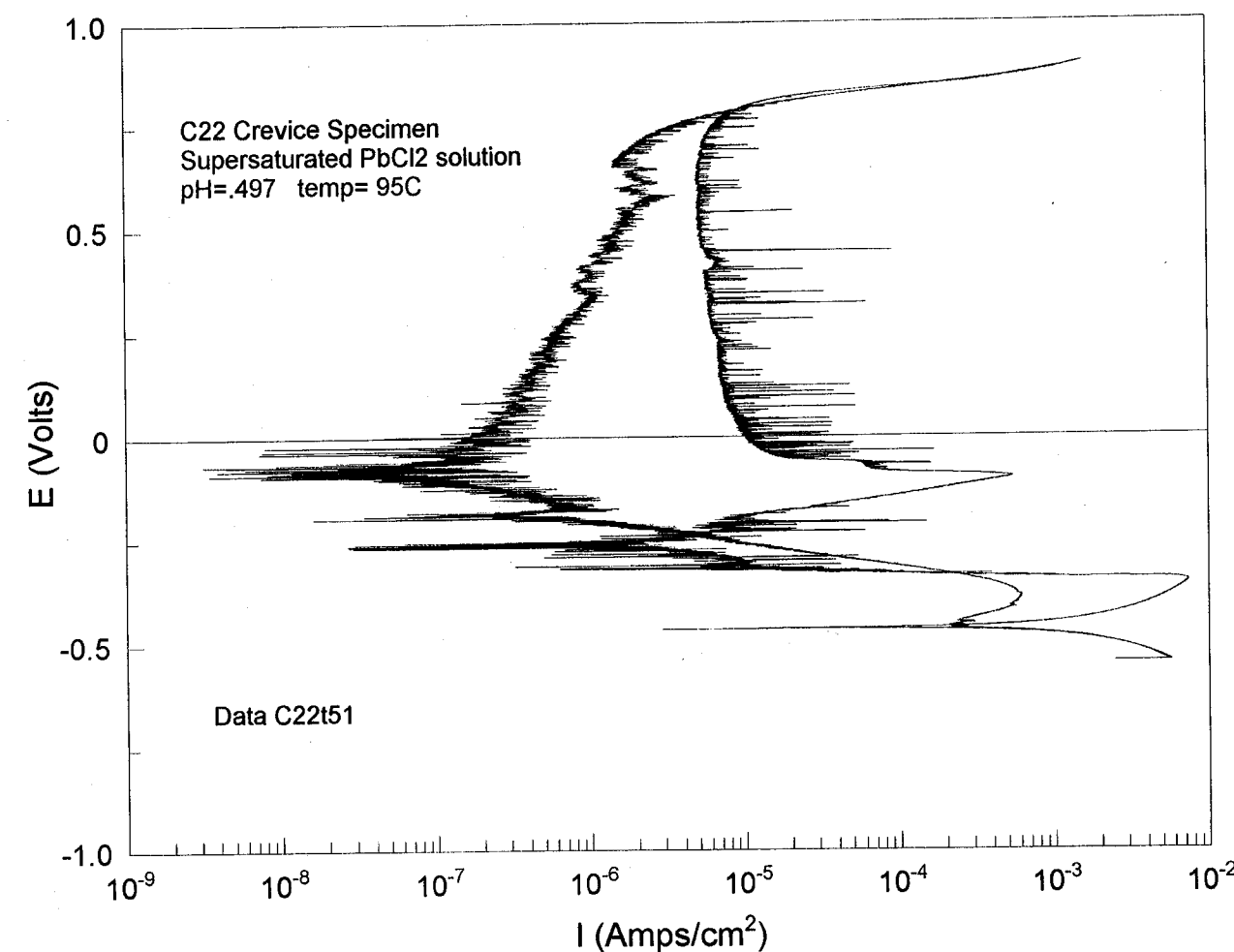
Reference: Fisher 13-620-52 SW#0199588

Temperature: 95°C Hg Thermometer SW#C96-649 Cal 1/10/03 due 7/10/03  
 Solution Deaerated with 99.999%  $N_2$

Ecorr: -189mV Keithley 614 SW#0704934 Cal 5/26/02 due 5/26/03  
 Ept: -465mV

Specimen Examination: No Crevice Corrosion - Transpassive Dissolution And  
 staining on Specimen - Very mild staining

Data C22t51  
 Bill [Signature] 3/19/03



Bill [Signature]  
 3/19/03

Cyclic Polarization of C-22 Crevice Specimen

Objective: See pg #5

Specimen: DOE Alloy NO6022 - Allegheny Ludlum Heat 059902142 Inco Alloy 622  
heat #204886 filler - Specimen Contains weld Material See NB# 505 pg# 2-25  
polishes to A 600 Grit Finish with 2 PTFE Crevice washers Attached At 50 In. O2  
Using Proto 6104 SN# 139072 cal 5/6/03 due 9/6/03

Start wt: 34.15822g Santarimus Genius SN# 12809099 cal 11/15/02  
End wt: 34.14988g due 5/15/03

Solution: See preparation on pg# 160-161 - #2 solution - ICP Analysis pg# 168  
pH Start = .497 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 due 8/7/03  
pH End Not Taken pH probe # 13-620-296 SN# 2291257 PL

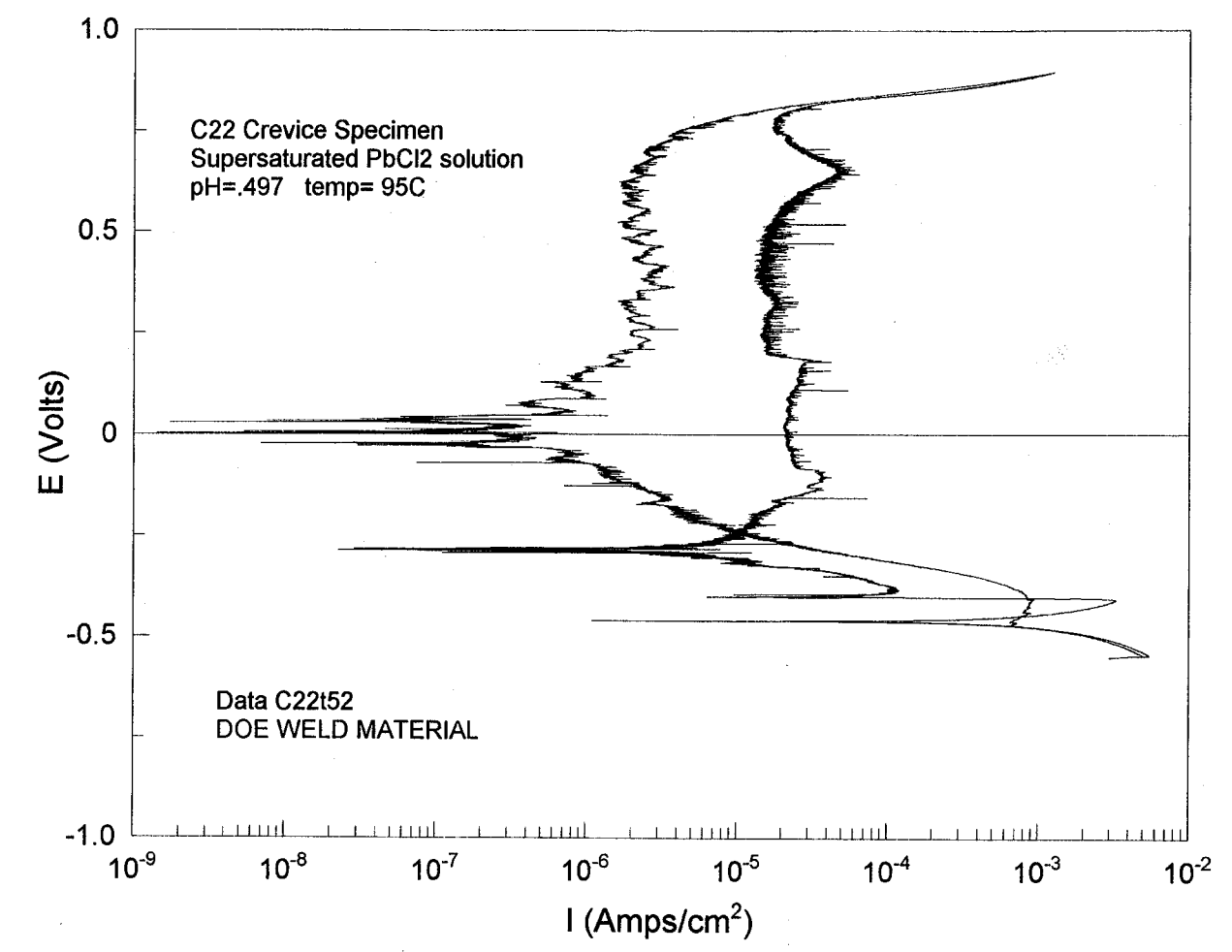
Potentiostat: Solartron 1287  
Counter Electrode: PT Flag  
Reference: Fisher 13-620-52 SN# 0199588  
Temperature: 95°C H<sub>2</sub> thermometer SN# 096-649 cal 1/10/03 due 7/10/03

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

E<sub>corr</sub> = -186 Keithley 614 SN# 0704934 cal 5/26/02  
E<sub>pt</sub> = -132 due 5/26/03

Specimen Examination: Transpassive Dissolution And Staining on Specimen

\* Repolishes specimen for further testing  
Data C22t52  
B. K. Duf 3/24/03



Copies sent to QA Records  
J. Pa  
4/2/03

B. K. Duf  
3/20/03

New Solution Preparation for PbCl<sub>2</sub>

Solution: 5 Liters / 5000 mls prepared for Testing  
PbCl<sub>2</sub> Lot # L13K01 Super Saturated Solution  
Deaerated with 99.999% N<sub>2</sub> while stirring Total PbCl<sub>2</sub> Added = 125.132g

3/28/03 : started heating 5 Liters DI while stirring and Deaerating  
At 95°C over weekend (48 hrs)

3/31/03 : Added 25.059g PbCl<sub>2</sub> Lot # L13K01 @ 8:00 a.m.  
Kept stirring and Deaerating overnight (24 hrs)

4/1/03 : Shut off heater controller @ 7:30 a.m.  
@ 2:05pm Solution @ Room Temp  
pH Adjustment  
pH Start = 4.487  
pH Adjusted To: .498  
used 137 mls 6M HCl Lot # 023844 prepared 4/1/03

started to Reheat solution To 95°C @ 2:21 pm while stirring  
and Deaerating Solution: Solution went clear @ 67.4°C  
Kept @ 95°C overnight while Deaeration + stirring To In progress

4/2/03 PbCl<sub>2</sub> Added Lot # L13K01:  
Added 37.57g PbCl<sub>2</sub> @ 8:00 AM - Solution cloudy To Clear  
Added 25.013g PbCl<sub>2</sub> @ 9:58 AM - Solution cloudy with fine particles  
Added 15.091g PbCl<sub>2</sub> @ 1:30 pm - Solution cloudy with fine particles  
still floating through solution  
(Continued on p#179)

B. K. D. 4/4/03

4/2/03 Added 11.087g PbCl<sub>2</sub> @ 2:30 pm - Solution Same  
Added 11.312g PbCl<sub>2</sub> @ 3:40 pm. Solution Same

4/3/03 Kept solution @ 95°C stirring and Deaeration going  
fine particles drifts on bottom of cell - Broke  
up with stirrer @ 7:30 a.m.  
Pulled 2 samples for ICP Analysis 1:10 Dilution  
@ 9:30 a.m.

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute Client: Division 20  
Lab Code: SwRI Date Received: 04/03/03  
Matrix: Water Project No.: 20.06002.01.081  
SPR: 24075 TO: 030403-8

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.003
Lab Control	----	0.495
True Value	----	0.500
Recovery	----	99.0%
#1 PbCl <sub>2</sub> Soln	223134	1600
Duplicate result	223134	1630
RPD	223134	1.86%
Spike result	223134	2100
Spike added	223134	500
Recovery	223134	100%
#2 PbCl <sub>2</sub> Soln	223135	1520

chloride 77p 10/6/03  
Results (mg/L)  
<0.1  
201  
200  
101 %  
1399  
1387  
0.86 %  
1718  
400  
79.8 %  
1292  
0.1 mg/L

Reporting Limit: 0.003 mg/L

B. K. D. 4/4/03



# Cyclic Polarization of C-22 Specimen

objective: See pg #5

Specimen: DOE Alloy NO6022 - Allegheny Ludlum Heat 05950242 Inco Alloy 622

Heat XX204886 Filler - Specimen Contains Weld Material - See NG#505 pg# 2-25

polished to # 600 Grit Finish with 2 PTFE Crevice Washers Attached At 50 In-C

Using Photo 6104 SN# 139072 cal 3/6/03 due 9/6/03

start wt = 34.09840g Sartorius Genius SN# 12809099 cal 11/15/02 due 5/15/03

End wt = 34.05289g

Solution: See preparation on pg #175-179 - ICP Analysis pg# 179 - Used 1200ml/s

pH start = .498 Fisher Accumet 950 meter SN# 3340 cal 5/7/02 due 8/7/03

pH End .693 pH probe #13-620-296 SN# 2291257 PL

potentiostat = Solatron 1287

Counter Electrode: RT Plg

Reference: Fisher 13-620-52 SN# 099588

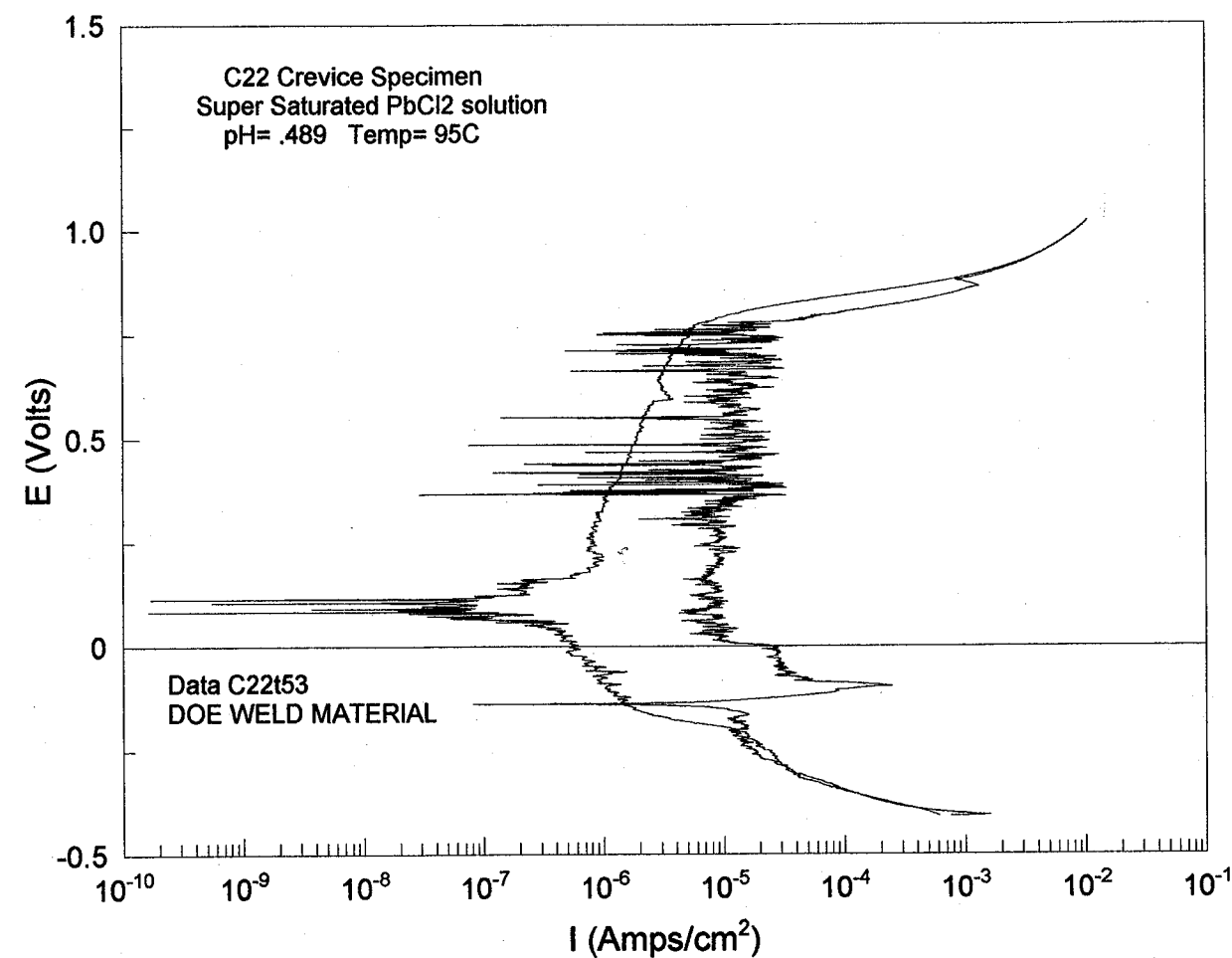
Temperature: 95°C Hg Thermometer SN# C56-649 cal 1/10/03 due 7/10/03

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

E<sub>scan</sub> -100mV Keithley 617 SN# 537418 cal 4/2/03 due 10/2/03

E<sub>pt</sub> +400mV

Specimen Examination: More Surface Etching on Specimen 1/4 feet No Crevice  
Corrosion. weld surface Etching with staining  
Data C22+S3 B-FSA 4/7/03



B-FSA 4/9/03

## Cyclic Polarization of C-22 Specimen

Objective: See pg #5

Specimen: DOE Alloy N06022 - Allegheny Ludlum Heat 059902LL2  
 Inco Alloy 622 Heat XX204806 filler - Specimen Contains weld material see N6#505 pg #2-25  
 polished to A 600 grit finish with 2 PTFE Crevice Washers Attached At 5021-02  
 Using Proto 6104 SN#139072 cal 3/6/03 due 9/6/03

Start wt: 34.14064g Sartorius Genius SN#12809089 cal 11/15/02  
 End wt: 34.13875g due 5/15/03

Solution: See preparation on pg # <sup>8/10/03</sup> 178-179 ICP Analysis pg #179  
 pH Start: .498 Fisher Accumet 950 meter SN#3340 cal 8/7/02 due 8/7/03  
 pH End: .788 pH probe #13-620-296 SN#2291257 PL

Potentiostat: Solartron 1287

Counter Electrode: Pt Flag

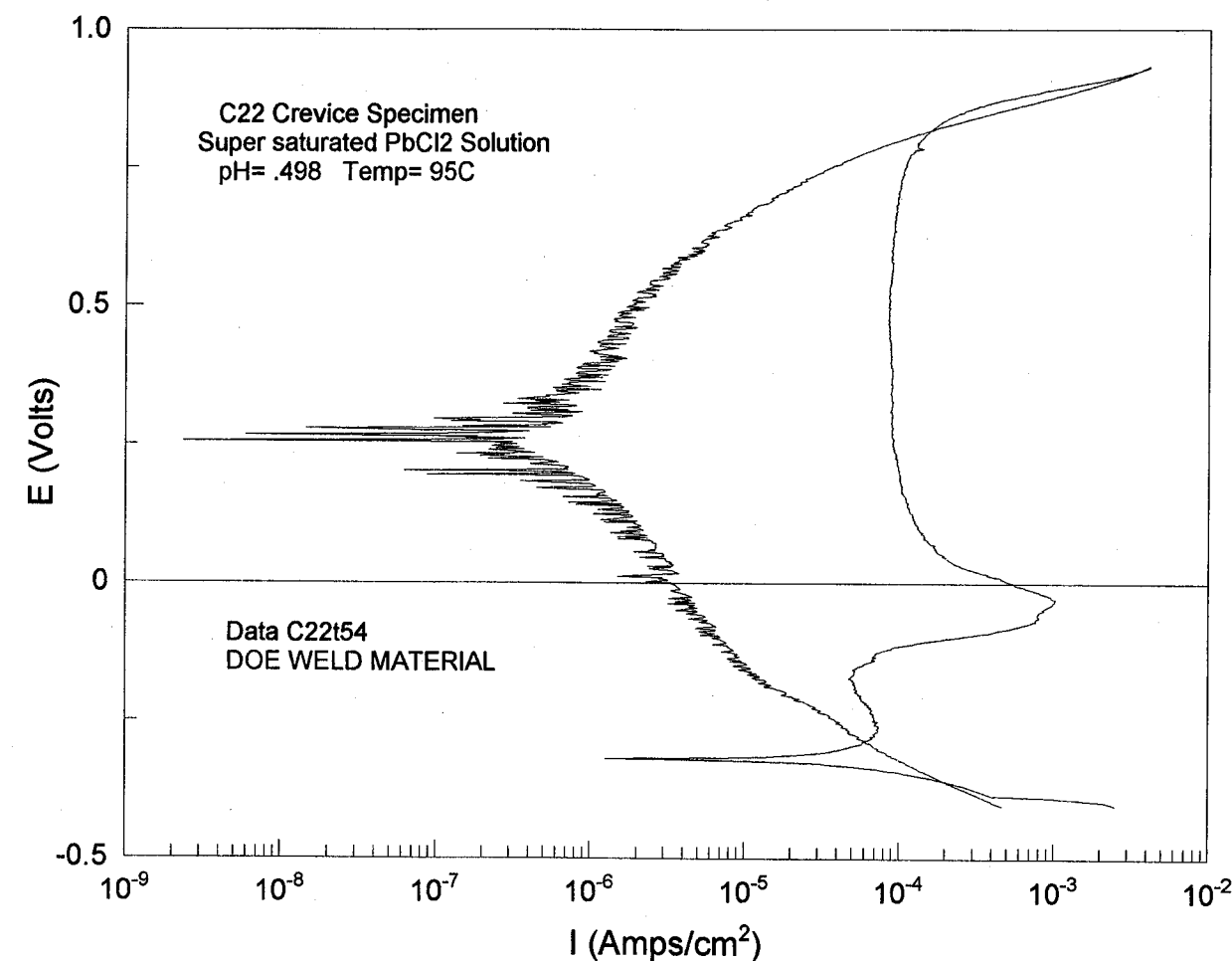
Reference: Fisher 13-620-52 SN#0199581

Temperature: 95°C H<sub>2</sub> Thermometer SN#C96-649 cal 1/10/03 due 7/10/03Solution Deaerated with 99.999% N<sub>2</sub>E<sub>corr</sub> = -149 mV Keithley 614 SN#0704934 cal 5/26/02E<sub>pt</sub> = +395 mV due 5/26/02

Specimen Examination: No crevice corrosion 24 feet of crevice washer

Very mild surface staining

Data C22154  
 B. R. J. 4/10/03



(Scan Rate 16.67)

B. R. J.

Potentiostatic Test Alloy C-22 U-Bend Specimens  
Double U-Bend Specimens X 2 @ 0 potential And -100

objective: See pg #5

Specimen: Alloy C-22 ASTM G-30 for outer specimen: DOE web Alloy 05990242

Plate # D 62X - filler XY 204886 Inner specimen (No #505 pg #2-25)

Specimen A = DOE C-22 Dimensions 5"(L) X 3.975"(m) X 0.750"(w) X 0.125"(T)

B = C-22 Alloy Dimensions 5.395"(L) X 4.370"(m) X 0.750"(w) X 0.125"(T) with 0.375" mounting Hole X 2

Specimen #1 Start wt = (A) 62.3640g (B) 64.5070g Sartorius Genius SN#12809099

B = #07 End wt = (A) 61.8223g (B) 63.5480g cal 11/15/02 due 5/15/03  
A = DOE T28

Specimen #2 Start wt = (A) 62.9255g (B) 64.8028g Sartorius Genius SN#12809099

A = DOE 03 End wt = (A) 62.7749g (B) 64.7126g cal 11/15/02 due 5/15/03

B = C22 #9

Solution: PbCl<sub>2</sub> Super Saturated See pg #178-179

pH start = 0.498 Fisher Accumet 950 meter SN#3340 cal 8/1/02 due 8/7/03

pH End = 0.674 pH probe #13-620-296 SN#2291257 P6

Potentiostat = Solartron 1480 multichannel

Counter Electrode = PT Flay X2

Reference: Fisher 13-620-52 SN#0199588

Temperature: 95°C Hg Thermometer SN#096-649 cal 1/10/03 due 7/10/03

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

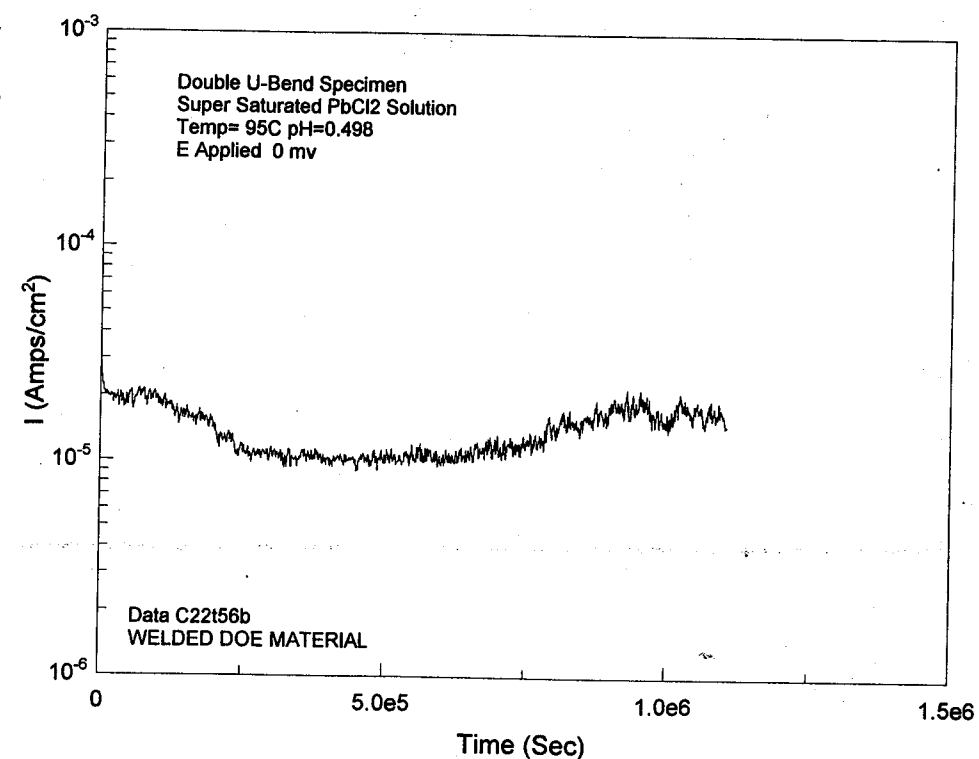
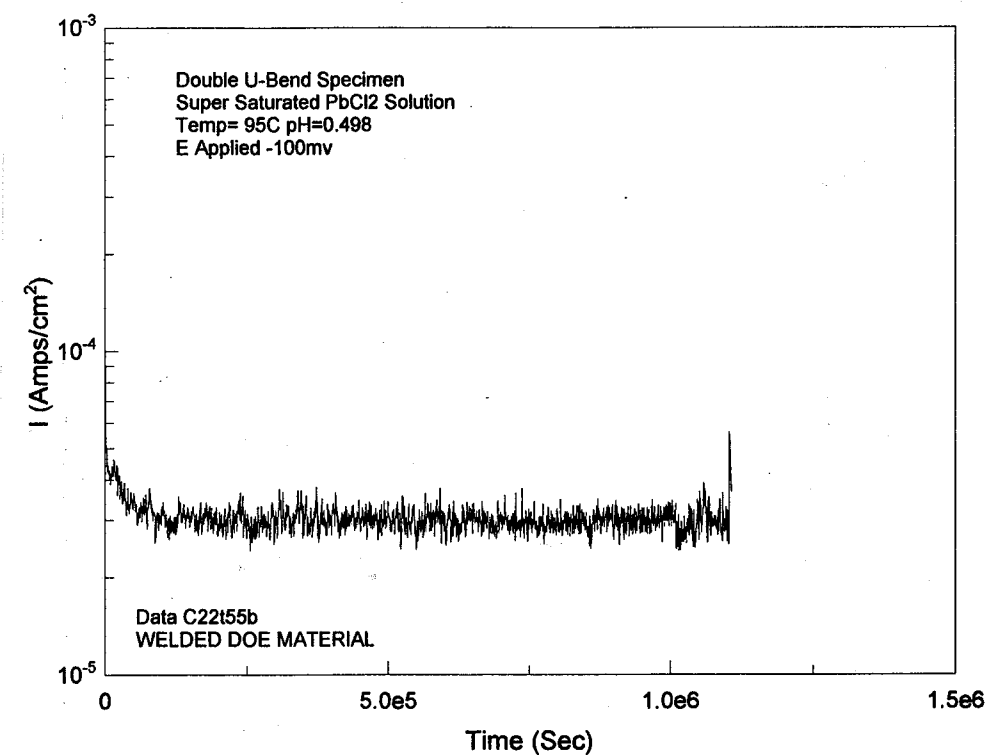
#1 Specimen #2 Specimen

E<sub>app</sub> = -199mV E<sub>app</sub> = -189mV Keithley 614 SN#0704934

E<sub>p</sub>T = +297mV E<sub>p</sub>T = +300mV cal 8/26/02 due 5/26/03

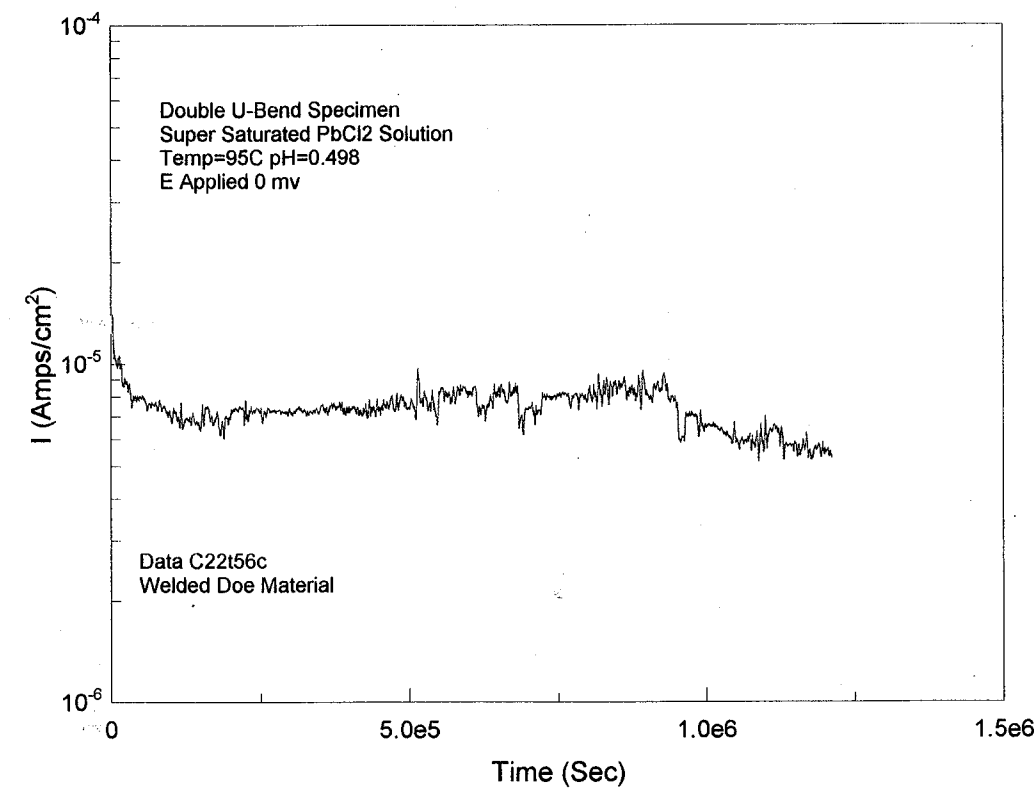
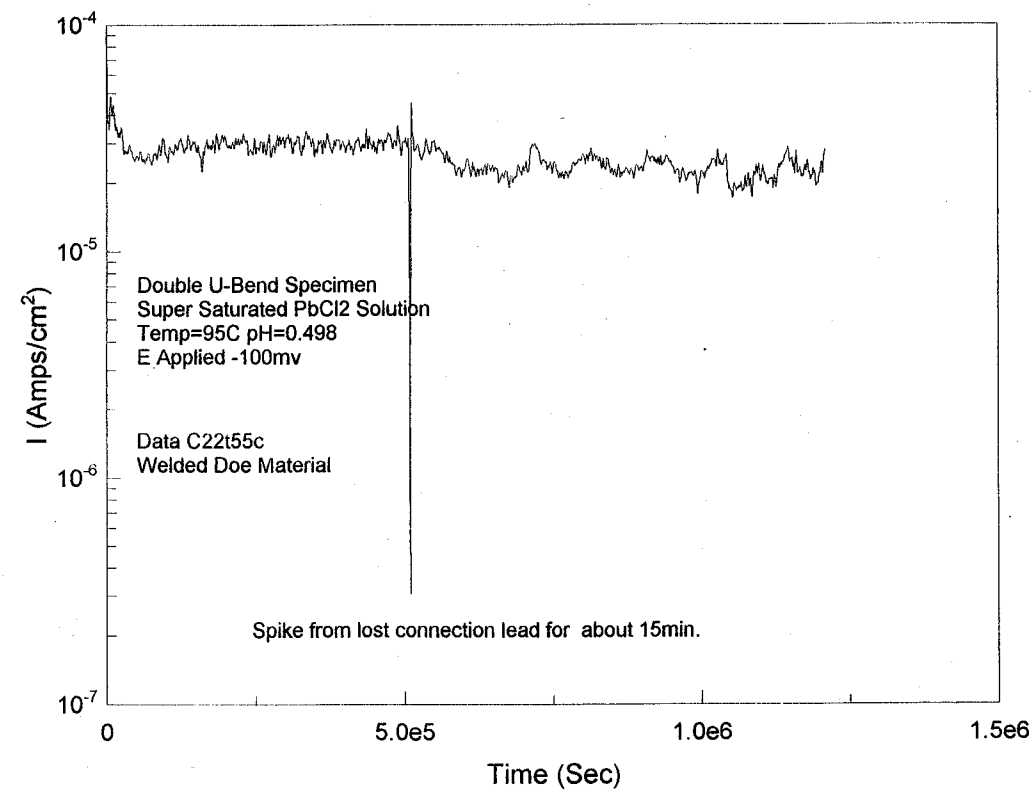
E Applies = -100mV E Applies = 0 mV B. E. J. 4/12/03

Specimen Examination: 5/1/03. Visual Examination All Specimens Have slight staining on surface And mild Bulup of Material - Solution Has Green tint - No Sign of Cracking on Either Specimen DOE or the Outer C-22 Alloy Specimen - will Continue Testig New Data File C22+55c - C22+56c



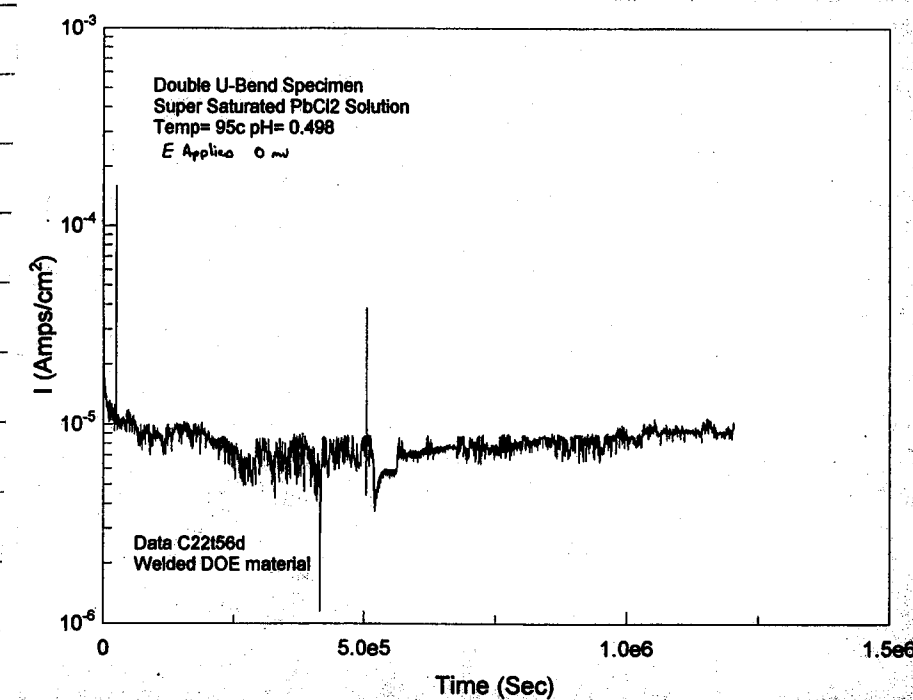
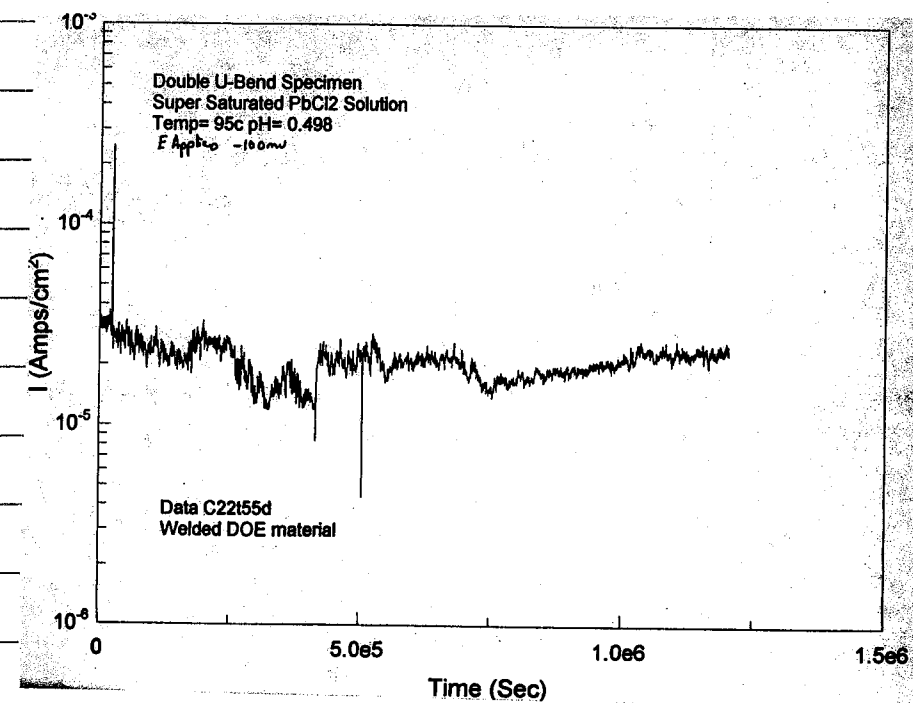
B. E. J.  
6/6/03

Specimen Examination: 5/15/03 Visual Inspection of Specimens Same As Previous  
staining slight surface Etching - No sign of Cracking on Either Specimen - Solution  
still has Green tint - will continue Testing File Name C22t55d - C22t56d



B. P. J.  
6/4/03

Specimen Examination: 5/28/03 Test End @ 10:50am  
Visual Examination After Cleaning Surface Staining And surface Etching on Specimens  
#1 Specimen Has A Apparent Depth To the Etching - More so than Specimen #2  
Also Appears To Have slight cracks By Bolt Hole - Both Specimens will Have  
Microscopic Examination



B. P. J.  
6/9/03



Solution Preparation for PbCl<sub>2</sub>

Solution: 5 Liters / 5000 ml prepared For Testing  
PbCl<sub>2</sub> Lot# L13K01 Super Saturated Solution  
Deaerated with 99.999% N<sub>2</sub> while stirring Total PbCl<sub>2</sub> Added = 125.230g

5/30/03: started heating 5 Liters DI while stirring And Deaerating  
@ 95°C over weekend (48 hrs)

6/2/03: started Adding PbCl<sub>2</sub> Lot# L13K01 @ 8:00 AM - Added 25.085g  
Kept stirring And Deaerating overnight (24 hrs)

6/3/03: shut off heater controller @ 7:00 AM To Cool To Room Temp  
@ 3:30 PM pH Adjustment  
pH Start = 4.417  
pH Adjusted To = .547

Used 6M HCl Lot# G23844 prepared 4/1/03 used 162mls

6/4/03  
started To Reheat @ 7:00 A.M while stirring And Deaerating  
solution went clear @ 62.7°C

Added 37.62g PbCl<sub>2</sub> Lot# L13K01 @ 7:40 - solution went cloudy  
Added 25.011g PbCl<sub>2</sub> Lot# L13K01 @ 8:40 - solution cloudy with particles  
Added 15.865g PbCl<sub>2</sub> Lot# L13K01 @ 10:20 - solution cloudy with particles  
Added 11.223g PbCl<sub>2</sub> Lot# L13K01 @ 2:30 1:10 - solution cloudy with particles  
Added 11.026g PbCl<sub>2</sub> Lot# L13K01 @ 2:40 - solution same  
Kept stirring And Deaeration continued 95°C Temp  
Pulled 2 samples for ICP Analysis @ 1:10 Dilution @ 3:45  
B-K 6/20/03

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute  
Lab Code: SWRI  
Matrix: Liquid  
SRR: 24405

Client: Division 20  
Date Received: 06/04/03  
Project No.: 20.06002.01.081  
TO: 030604-8

Sample ID	Lab System ID	Lead Results (mg/L)	Chloride Results (mg/L)
Prep Blank	----	<0.003	<0.1
Lab Control	----	0.495	212
True Value	----	0.500	200
Recovery	----	99.0%	106%
#1 1:10 Dilution PbCl <sub>2</sub> Solution	227265	1200	1170
Duplicate result	227265	1150	1171
RPD	227265	4.26%	0.09%
Spike result	227265	1700	1548
Spike added	227265	500	400
Recovery	227265	100%	94.5%
#2 1:10 Dilution PbCl <sub>2</sub> Solution	227266	1200	1442

Reporting Limit: 0.003 mg/L 0.1 mg/L

B-K 6/20/03

# Potentiostatic Test Alloy C-22 U-Bend Specimens

Double U-Bend Specimens X2 @ 0.05 And -0.05 Applied Potential

Objective: See pg #5

Specimen: Alloy C-22 ASTM G-30 for outer Specimen DOE web Alloy 059902442

Plate # 062X - filler XX 204886 Inner Specimen (NB#505 pg #2-25)

Specimen A = DOE C-22 Dimensions 5"(L) x 3.95"(m) x 0.750"(w) x 0.125"(T)

B: C-22 Alloy Dimensions 5.395"(L) x 4.370"(m) x 0.750"(w) x 0.125"(T) w/ 0.375" mounting Hole X2

Specimen #1 start wt = (A) 62.53136g (B) 64.82030g Sartorius Genius SN#12809099

A: T1A End wt = (A) 62.15083g (B) 63.83064g cal 5/15/03 due 11/15/03  
B: #11 64.24388g

Specimen #2 start wt = (A) 62.30656g (B) 64.53884g Sartorius Genius SN#12809099

A: T2A End wt = (A) 61.79646g (B) 63.83064g cal 5/15/03 due 11/15/03  
B: #10

Solution: PbCl<sub>2</sub> Super Saturates See pg #188-189

pH Start: .547 Fisher Accumet 950 meter SN#3340 cal 8/7/02 due 8/7/03

pH End: .588 pH probe #13-620-296 SN#2291257 PL

Potentiostat: Solartron 1480 Multichannel

Counter Electrode = PT Flay X2

Temperature = 95°C Hg Thermometer SN#C96-649 cal 1/10/03 due 7/10/03

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

#1 Specimen #2 Specimen

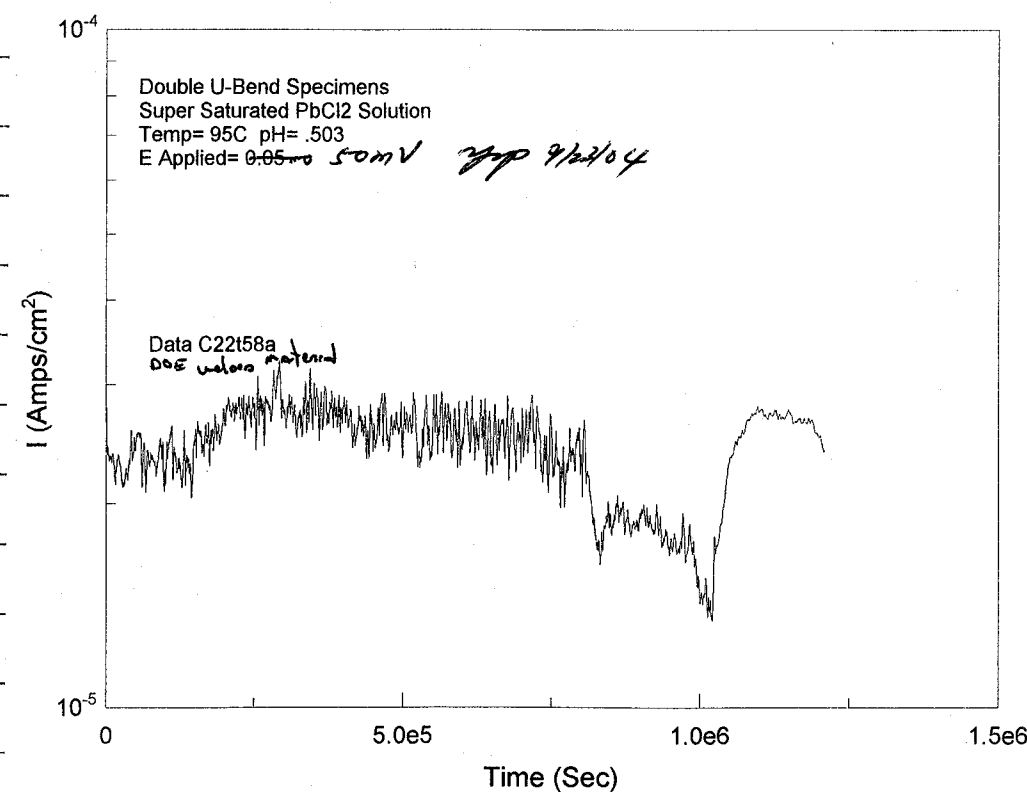
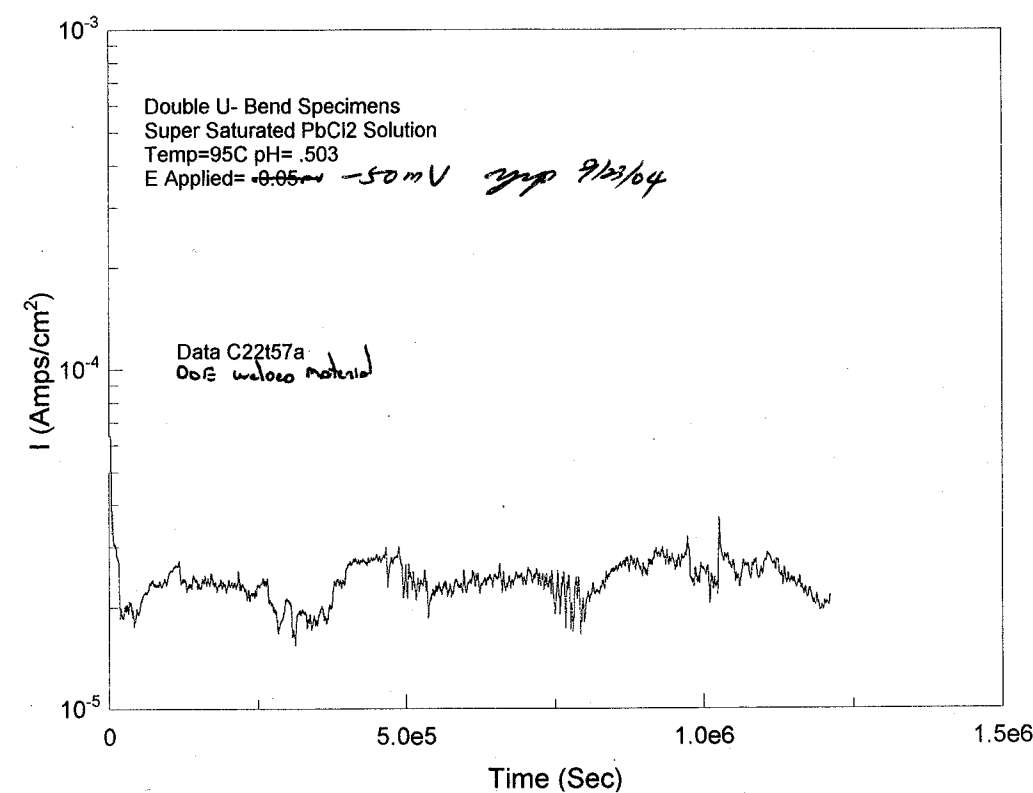
E<sub>corr</sub> = -222mV E<sub>corr</sub> = -217mV Keithley 617 SN#537918  
due 6/20/03

E<sub>pt</sub> = +242mV E<sub>pt</sub> = +190mV cal 4/2/03 due 10/2/03

E Applies 0.05mV E Applies = -0.05mV

B. E. D. J.  
6/20/03

Specimen Examination: 6/25/03 - Visual Examination - All Specimen show mild surface staining solution has developed a slight green tint - mild surface buildup of material on both specimens. No sign of cracking or etching on either. Specimen will continue test. data Filr C22+57b - C22+58b

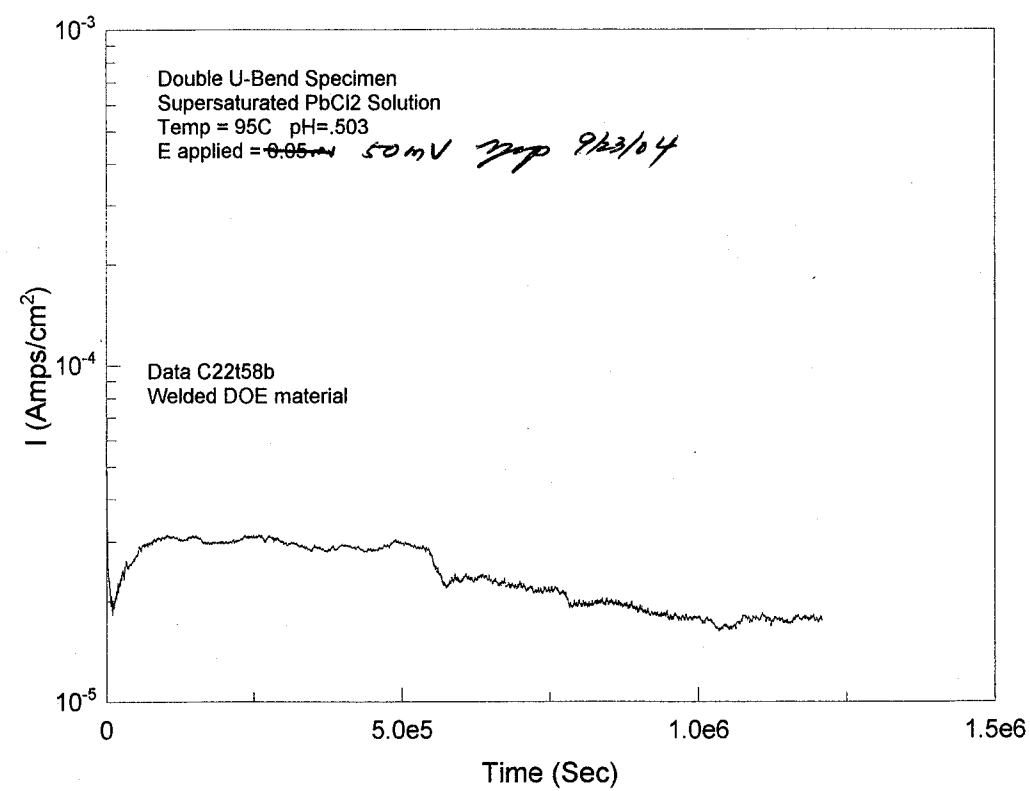
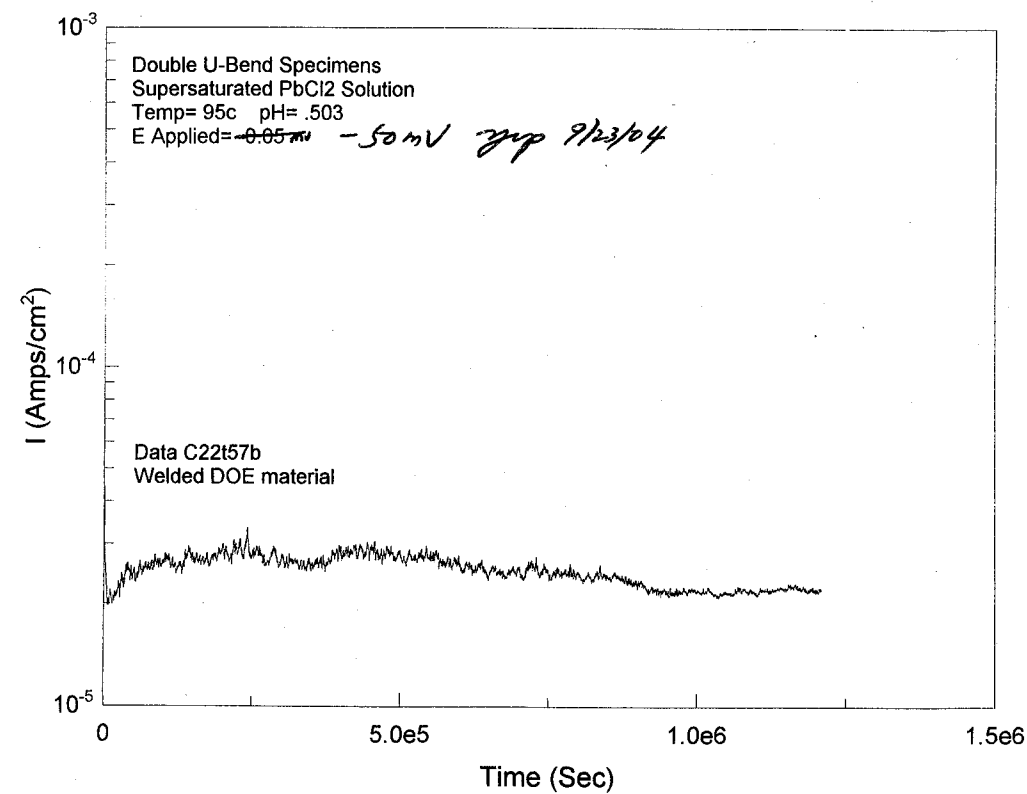


Both Have  
DOE web material

B. E. D. J.  
6/25/03

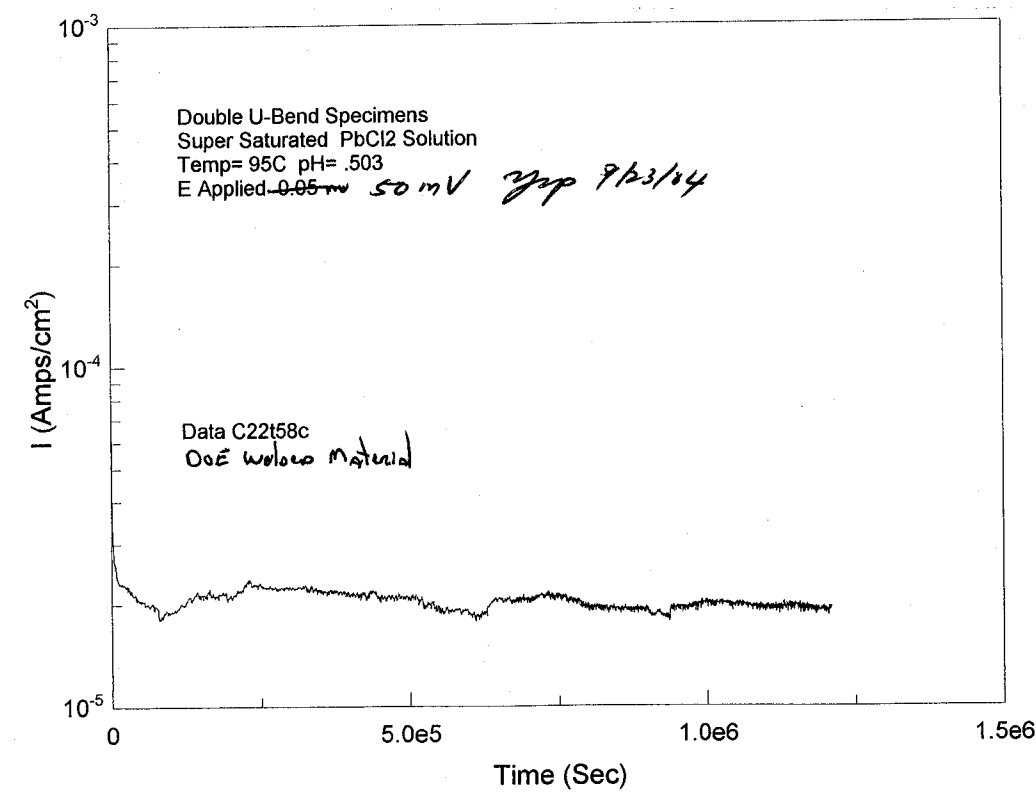
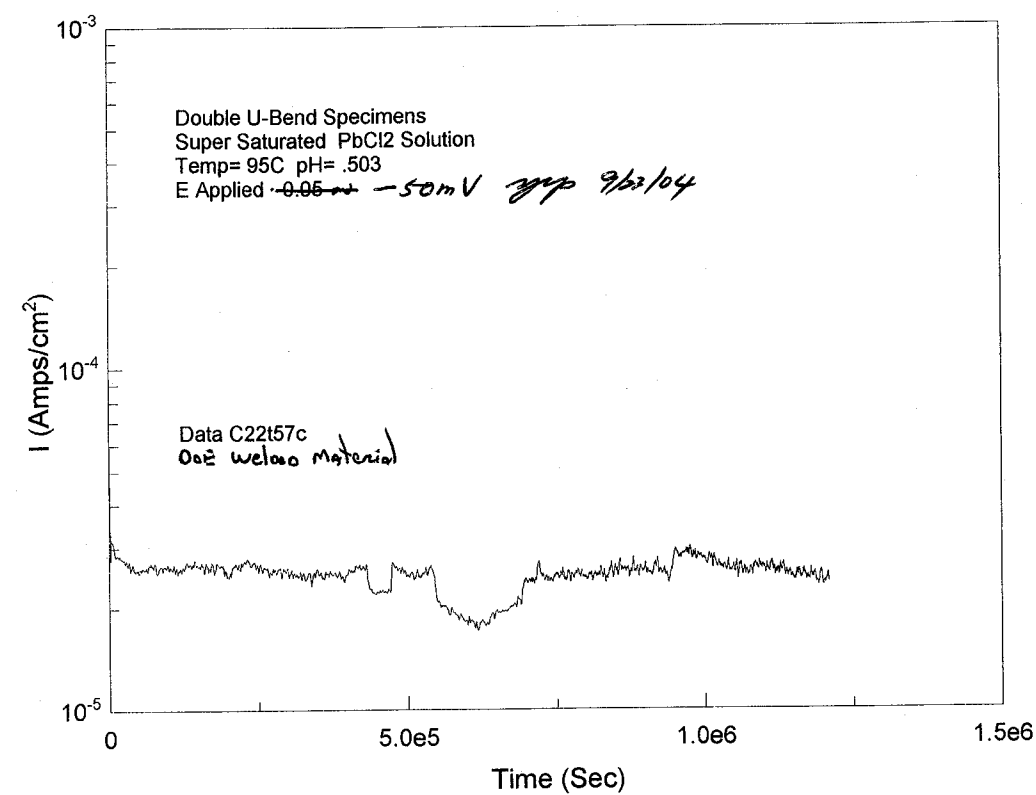
7/10/03: Visual Exam - Both specimens Have slight buildup of material on their surfaces - Mild staining on All Surfaces - No sign of Etching or cracking on either Specimen will continue Testing

Data File C22t57c - C22t58c



*Diary 7/10/03*

7/25/03: Test Enden - Both Have Build up of Material on Surface Stain on All Surfaces And Between Specimens - No sign of cracking



*Diary 7/25/03*

3000 mls PbCl<sub>2</sub> Solution left from last batch - 95°C  
Reheated Test Solution from pg # 188 - 189 Plus Deaerated with N<sub>2</sub>  
Pulled 2 samples At A 1:10 Dilution for  
ICP Analysis Sample #1 Pulled from 3 Inches Into Solution  
Sample #2 Pulled 3 Inches from bottom of Cell  
After Solution was Deaerated And Held @ 95°C for 15 hours

FORM FOR REQUESTING WORK FROM OTHER DIVISIONS

A. TO BE COMPLETED BY DIVISION 20 PERSONNEL

Requester: Y. Ming Pan Request Date: 7/25/03  
Project No.: 20.06002.01.081 Phone No.: X 6640  
Description of Work Requested:  
ICP Analysis of PbCl<sub>2</sub> Solution for  
Lead And Chloride Concentrations

☐ 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
#1 PbCl <sub>2</sub> Solution	
#2 PbCl <sub>2</sub> Solution	

B. TO BE COMPLETED BY DIVISION PERFORMING WORK<sup>1</sup>

☐ 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): \_\_\_\_\_

<sup>1</sup> 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.

B. P. J.  
7/29/03

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

SRR: 24714

Client: Division 20

Date Received: 07/28/03

Project No.: 20.06002.01.081

TO: 030728-4

Sample ID	Lab System ID	Lead Results (mg/L)
Prep Blank	----	<0.003
Lab Control	----	0.501
True Value	----	0.500
Recovery	----	100%
#1 PbCl <sub>2</sub> Solution	231141	1220
Duplicate result	231141	1230
RPD	231141	0.82%
Spike result	231141	1710
Spike added	231141	500
Recovery	231141	98.0%
#2 PbCl <sub>2</sub> Solution	231142	1380

Reporting Limit: 0.003 mg/L

SOUTHWEST RESEARCH INSTITUTE  
SAMPLE ANALYSIS DATA SHEET

Lab Name: Southwest Research Institute

Lab Code: SwRI

Matrix: Water

SRR: 24714

Client: Division 20

Date Received: 07/28/03

Project No.: 20.06002.01.081

TO: 030728-4

Sample ID	Lab System ID	Lead Results (mg/L)	Chloride Results (mg/L)
Prep Blank	----	<0.003	<0.1
Lab Control	----	0.501	202
True Value	----	0.500	200
Recovery	----	100%	101%
#1 PbCl <sub>2</sub> Solution	231141	1220	1564
Duplicate result	231141	1230	1547
RPD	231141	0.82%	1.09%
Spike result	231141	1710	1921
Spike added	231141	500	400
Recovery	231141	98.0%	89.3%
#2 PbCl <sub>2</sub> Solution	231142	1380	1652

Reporting Limit: 0.003 mg/L 0.1 mg/L

B. P. J.  
7/29/03



# Thermally Ages Procedure And Solution Annealed Procedure For Double U-Bend Specimens

First Solution Annealed All Specimens

Specimens DOE web Alloy 059902442 plate # D62X filler XX 2048 B6  
Specimen Markers #B3B (Inner Specimen) And #T3B (Inner Specimen)  
C-22 Alloy #12 (Outer Specimen) And #13 (Outer Specimen)

Oven set point  $1130^{\circ}\text{C}$  Thermocouple Reading 1127.2  
for 12 mins Total Time In Oven then Quenched

Oven: Linberg SN# 909172 model #57333

Temperature Measurement: Omega Microprocessor thermometer model #4H22 SN# J-94140  
cal 5/8/03 Due 11/8/03  
Thermocouple #332 cal 7/21/03 Due 1/21/04

Then Specimen #12 C-22 Alloy + DOE #B3B web C-22

Thermally Aged  $870^{\circ}\text{C}$  for 30 min

Used Same Oven And Thermal couple but oven set point was  $875^{\circ}\text{C}$   
And Thermal couple Reading was  $872.6^{\circ}\text{C}$

B. F. J.  
8/2/03

Potentiostatic Test Alloy C-22 U-Bend Specimens  
Double U-Bend Specimens X2 @  $-100\text{mV}$  potential

objective: See pg #5

Specimen: Alloy C-22 ASTM G-30 for Outer Specimen: DOE web Alloy 059902442  
Plate #D62X filler XX 2048 B6 Inner Specimen (NB#505 pg #2-25)

Specimen A: DOE C-22 Dimensions  $5^{\prime\prime}(\text{L}) \times 3.975^{\prime\prime}(\text{m}) \times 0.750^{\prime\prime}(\text{w}) \times 0.125^{\prime\prime}(\text{T})$

B: C-22 Alloy Dimensions  $5.395^{\prime\prime}(\text{L}) \times 4.370^{\prime\prime}(\text{m}) \times 0.750^{\prime\prime}(\text{w}) \times 0.125^{\prime\prime}(\text{T}) \text{ w/} 0.375^{\prime\prime}$

mounting Holes In Both Specimens

All Specimens Solution Annealed And Specimen #12 C-22 #B3B DOE Thermally Aged  
See pg #196 for Details

Specimen #1

A: #12 C-22 Start wt: (A) 63.63457g (B) 61.55754g Sartorius Genius SN# 1280909

B: #B3B DOE End wt: (A) 62.54610g (B) 60.97030g cal 5/15/03 Due 11/15/03

Specimen #2

A: #13 C-22 Start wt: (A) 62.34303g (B) 61.76262g Sartorius Genius SN# 12809099

B: #T3B DOE End wt: (A) 61.52301g (B) 61.06229g cal 5/15/03 Due 11/15/03

Solution:  $\text{PbCl}_2$  Super Saturated See pg #188-189 Rechecked pg #194-195

pH Start: 0.503 Fisher Accumet 950 meter SN# 3340 cal 8/7/02 Due 8/7/02

pH End: 0.458 pH probe #13-620-296 SN# 2291257 PL

potentiostat = Solatron 1480 Multichannel

Counter Electrode: PT Flay X2

Reference: Fisher 13-620-52 SN# 0199588

Temperature:  $95^{\circ}\text{C}$  Hg Thermometer SN# C96-649 cal 7/15/03 Due 1/15/04

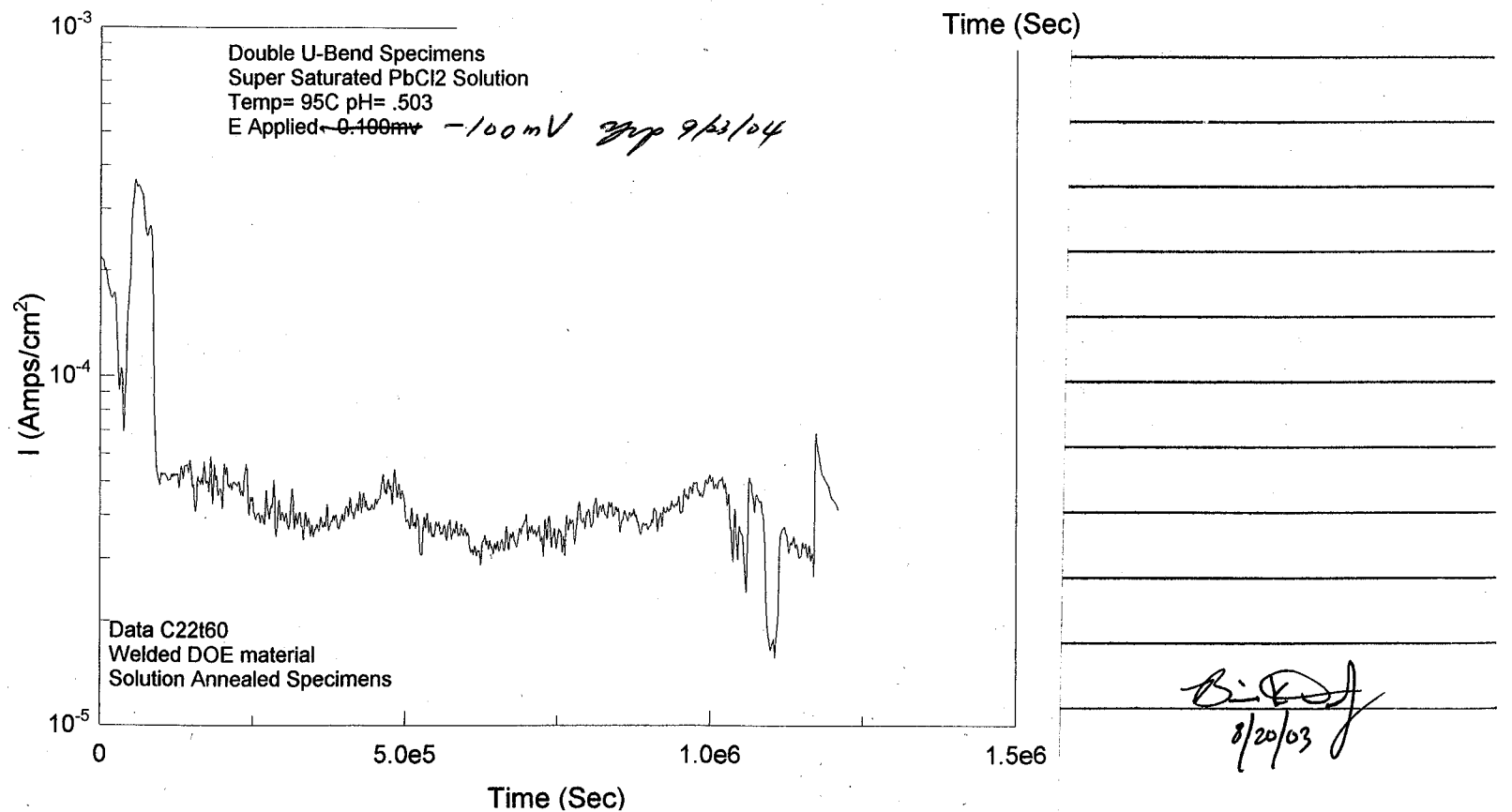
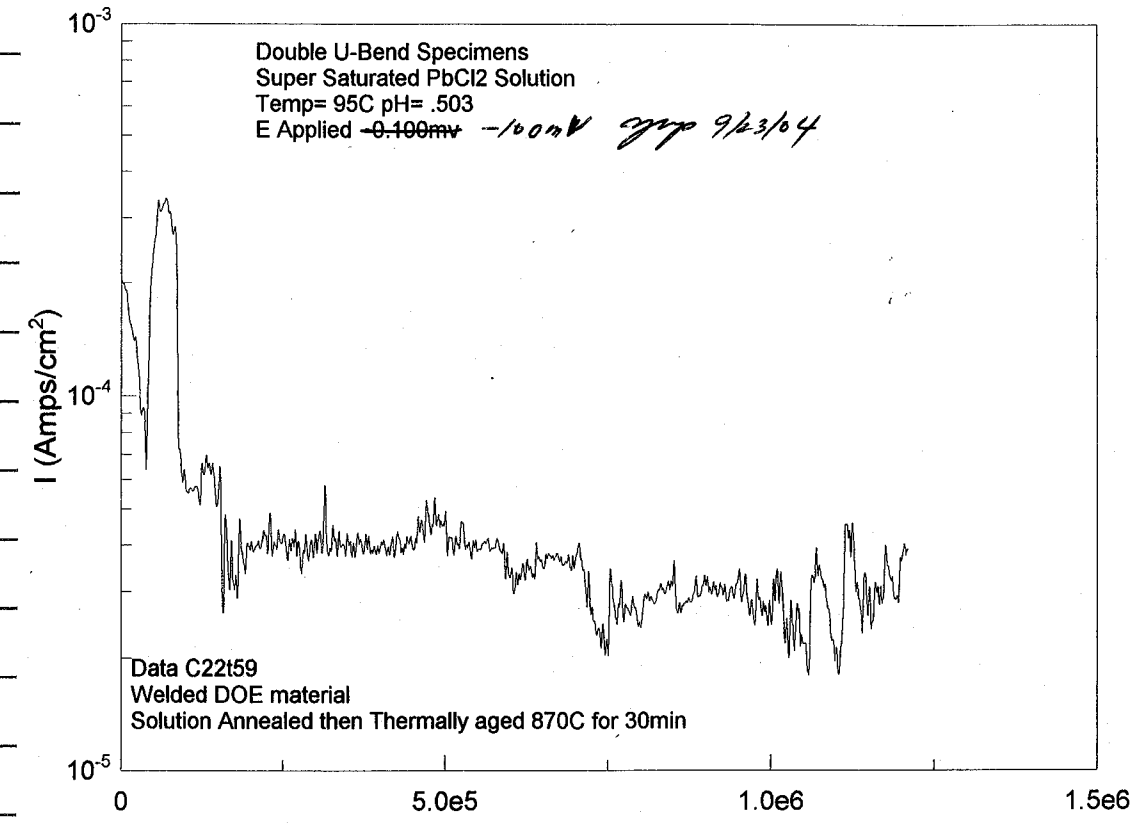
#1 Ecorr =  $-153\text{mV}$  #2 Specimen Ecorr =  $-163\text{mV}$  Keithley 617 SN#

E<sub>pt</sub> =  $+263\text{mV}$  E<sub>pt</sub> =  $+261\text{mV}$  cal: Due:

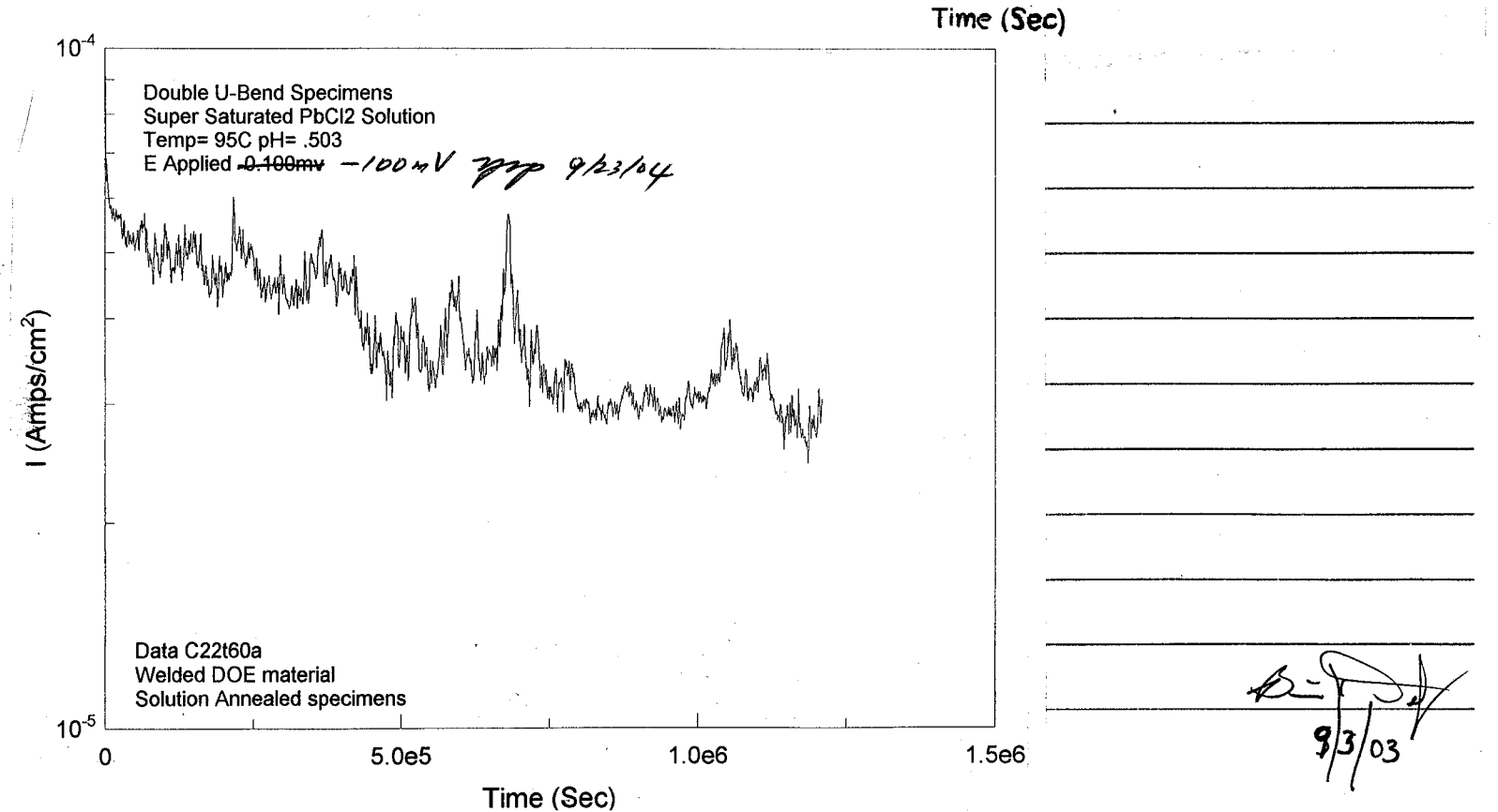
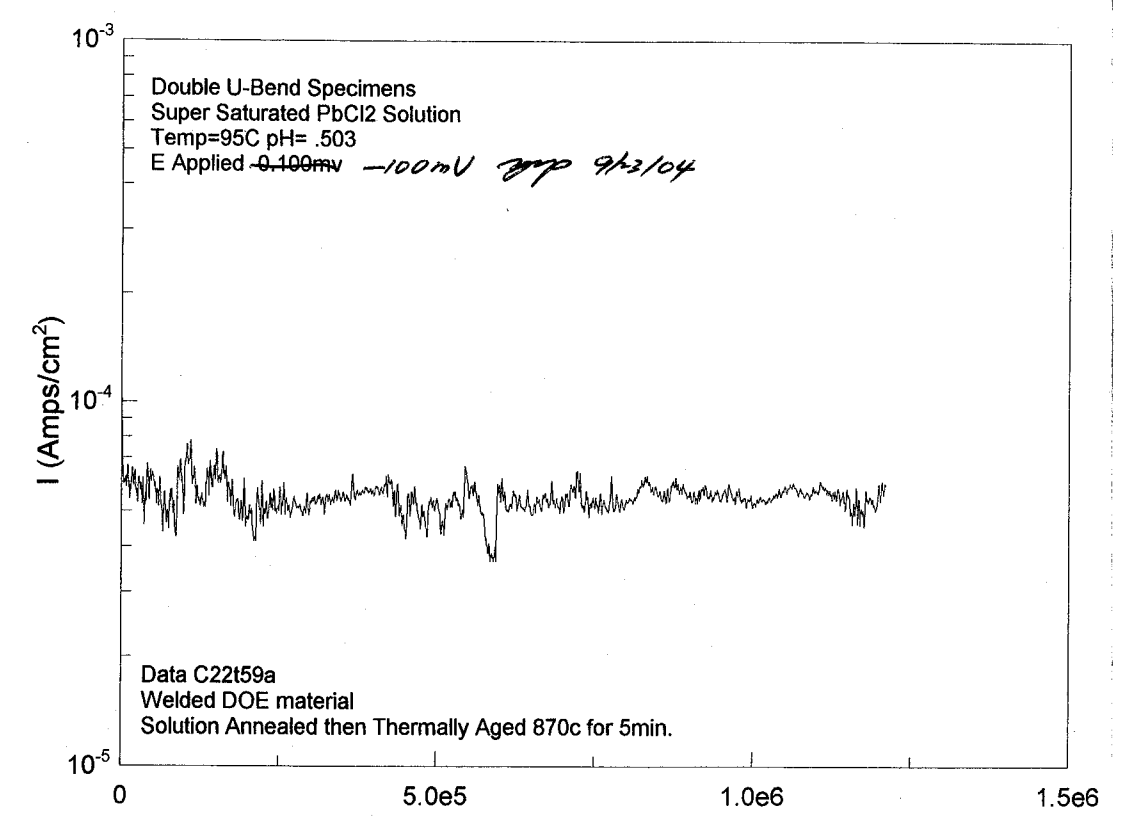
E Applied =  $-100\text{mV}$  E Applied =  $-100\text{mV}$

B. F. J.  
8/7/03

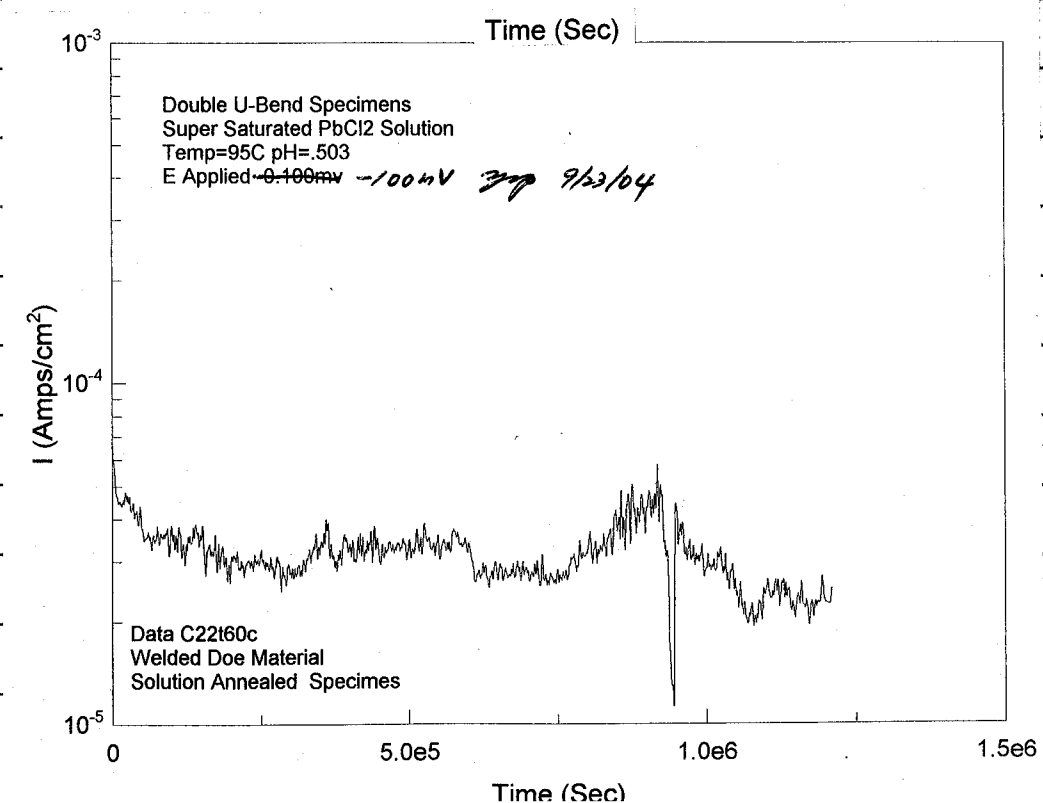
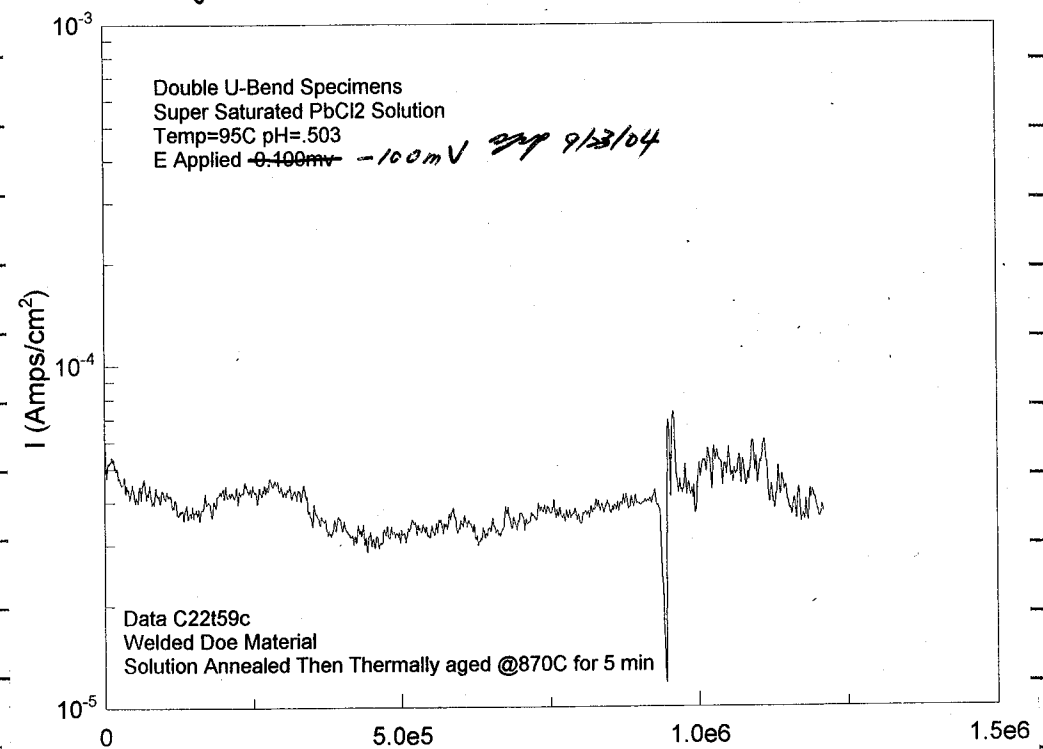
8/20/03 Specimen Examination: Both specimens have slight build up of material  
At solution Vapor phase - No sign of Etching or Cracking - Both have mild  
staining on surface. Specimen #2 cable broke specimen remains attached/connected  
See Graph - will Restart Tests Data Files C22+59a And C22+60A



9/8/03: Specimen Examination: Both specimens have slight build up of material  
At solution Vapor phase - Showing No sign of Etching or Cracking on Either specimen  
Both have mild staining - will keep on + continue with Test New Data File  
C22+59b And C22+60b



C22+60b 8/21/03  
 \*Note Test C22+59b And C22+60b were shut down since Test specimen Holder Beaker will Restart with New Test Data File C22+59c And C22+60c  
 Specimen Examination: Both Specimens show signs of Etching on All Surfaces Especially Tabetween Specimens - Staining on Both Specimens. No Cracking on Either Specimen  
 \* Continue Testing In Notebook # 602



Copies Sent  
 TO QA 10/3/03  
 B. H. J.  
 9/23/03

SN# 465  
 Stress Corrosion Cracking and Hydrogen Embrittlement of Container and Drip Shield Materials, CNWRA 2003-02, October 2002

Y.-M. Pan, D.S. Dunn, L. Yang, and G.A. Cragnolino, Corrosion and Stress Corrosion Cracking of Alloy 22 in Lead-Containing Solutions, *Mat. Res. Soc. Symp. Proc.* Vol. **757**, 743-750, 2003

A.A. Csontos, Y.-M. Pan, D.S. Dunn, L. Yang, and G.A. Cragnolino, The Effect of Environmental Chemistry on the Pb Assisted Stress Corrosion Cracking Susceptibility of Mill-Annealed Alloy 22 and GTAW Weldments, *Mat. Res. Soc. Symp. Proc.* Vol. **824**, 39-44, 2004

A.A. Csontos, Y.-M. Pan, D.S. Dunn, L. Yang, and G.A. Cragnolino, The Effect of Potential and Aging on the Pb-Assisted Stress Corrosion Cracking Susceptibility of Alloy 22 Gas Tungsten Arc-Welded Weldments, *Metall. Mater. Trans.*, Vol. **36A**, 1169-77, 2005

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]* 12/2/2005

**ADDITIONAL INFORMATION FOR SCIENTIFIC NOTEBOOK NO. 465**

<b>Document Date:</b>	05/24/2001
<b>Availability:</b>	Southwest Research Institute® Center for Nuclear Waste Regulatory Analyses 6220 Culebra Road San Antonio, Texas 78228
<b>Contact:</b>	Southwest Research Institute® Center for Nuclear Waste Regulatory Analyses 6220 Culebra Road San Antonio, TX 78228-5166 Attn.: Director of Administration 210.522.5054
<b>Data Sensitivity:</b>	<input checked="" type="checkbox"/> "Non-Sensitive" <input type="checkbox"/> Sensitive <input type="checkbox"/> "Non-Sensitive - Copyright" <input type="checkbox"/> Sensitive - Copyright
<b>Date Generated:</b>	09/17/2004
<b>Operating System:</b> (including version number)	Windows
<b>Application Used:</b> (including version number)	Unknown
<b>Media Type:</b> (CDs, 3 1/2, 5 1/4 disks, etc.)	1 - CD
<b>File Types:</b> (.exe, .bat, .zip, etc.)	.cor
<b>Remarks:</b> (computer runs, etc.)	Media contains: data files.