CNWRA-012

CORROSION

SOUTHWEST RESEARCH INSTITUTE

20-3606

PRELIMINARY EXPERIMENTS AND PROCEDURE DEVELOPMENT FOR NRC POLARIZATION TESTS

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JWPE NOTEBOOK - 012

5/89 - 3/90

DATA DISKETTE INSIDE

IWPE - 1

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TITLE PRELIMINARY EXPERIMENTS & PROCEDURE DEVELOPMENT

PROJECT NO. 20-3606

BOOK NO. 1

Preparation of J-13 simulated water from stock Solutions A & B.

Preparation of Stock Solution A:

Approx 950 ml of DI water was added to a 1L volumetric flask. To this was added 1.377 gr $CaSO_4 \cdot 2H_2O$ and stirred until dissolved. Then the following were added: 0.944 gr $Ca(NO_3)_2 \cdot 4H_2O$
0.119 gr KCl
0.569 gr $MgCl_2 \cdot 6H_2O$
and stirred into solution. The solution was filtered through a Whatman #1 filter and then diluted to volume with DI water.

Preparation of Stock Solution B:

Approx 950 ml of DI water was added to a 1L volumetric flask. To this was added 6.720 gr $NaHCO_3$ and 0.232 gr KF. These were stirred into dissolution and then diluted to volume with DI water.

To prepare simulated J-13 water 250 ml of Stock Soln A and Stock Soln B are pipetted into 925 ml of DI water in a 1L volumetric flask. To this is added 0.0834 gr silicic acid and stirred. The pH is checked and adjusted to 7 ± 0.2 with 0.1N HCl or 0.1N NaOH as needed. Then the solution is diluted to volume with DI water.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 5/1/89

DISCLOSED TO AND UNDERSTOOD BY N Sridhar DATE 3/14/90 WITNESS DATE

SWR 01 TP 304L

Stock Soln A-1 and B-1 prepared 3/15/89

① Simulated J-13 prepared from A-1 and B-1 as described in procedure. Initial (pre-test) pH of this solution was 7.12 at RT. Post test pH 9.18

② 900 ml. of the J-13 solution was added to the corrosion cell where it was heated to 95°C. A constant stream of 99.990% Argon was sparged through the cell to eliminate oxygen.

③ A cylindrical specimen of approx 10 cm² area of 304L SS was prepared by hand polishing first with 220 grit followed by a finishing polish with 600 grit. Wet go polishing was used in this process. (*)

④ ~~Argon was sparged for 1 hour~~ The redox potential of the Pt electrode was monitored until it reached a steady state. After one hour the potential was +106 mV vs SCE.

⑤ After one hour of solution conditioning, the specimen was inserted such that the crevice and at the TFE washer was above the level of the liquid. The specimen was allowed to remain in solution for 15 minutes prior to activating the polarization experiment in order to get a "feel" for the equilibrium potential which was -360 mV vs SCE

⑥ A cyclic polarization scan was performed and stored as SWR01. The Pt redox data is SWR01

⑦ The specimen area was determined after the experiment by calculating the immersed portion. 0.246" ϕ and 1.775" length. Area (immersed) = 9.157 cm²

(*) Specimen degreased in acetone (15 min ultrasonic), rinsed w/DI water, then methanol and dried under hot air.

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SWR 02 TP 304L

① J-13 water prepared from stock A-1 and B-1
~~final~~ pH was 6.97 after adjustment.

② 304L specimen polished (wet) with 220 then 600 grit paper. It was degreased in acetone (15 min ultrasonic) rinsed with DI water, then methanol and dried under hot air.

③ 900 ml. of the J-13 water were added to the corrosion cell and heated to 95°C. At the same time, de-aeration with high purity argon was begun. Redox potentials of a Pt electrode were monitored for one hour (SWRI02 and SWRI02-) The redox potential after one hour was +113 mV vs SCE.

④ The specimen was inserted and a hold of 15 minutes was done prior to starting the experiment. The equilibrium potential at this time was -525 mV vs SCE.

⑤ Approx. 15 minutes into the program, it was noticed that the service area at the TFE washer was immersed. The run was stopped, the specimen repositioned, and the experiment restarted from the beginning of the program.

⑥ The data was stored as SWR 02. Pt redox data is SWRI 02 and SWRI 02-.

⑦ Area of specimen that was immersed was calculated from the following measurements 0.246" ϕ 1.751" length Area = 9.037 cm^2

This specimen (as SWR01) showed multicolored staining as well as pitting. A high leakage rate from the bridge of saturated KCl was observed. Both solutions had a distinct yellowish color after the scan. It is suspected that these experiments are "chloride contaminated" and not representative of 304L SS in "normal" J-13 water.

Post test pH 8.90

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SWR 03 This test will be run with J-13 water in the bridge, which will be electrically connected back to the reference jack at the electrometer thru a 0.1 mfd capacitor. This was suggested by EGG/PARC applications personal to attempt to correct impedance effects in the low conductive water in the bridge.

(1) A liter of J-13 water was prepared from Stock Soln. A-1 and B-1 The adjusted pH was 6.88. Post test was 9.14

(2) A 304L specimen was wet polished with 220 and then with 600 grit paper. It was then degreased in acetone (15 min. ultrasonic), rinsed in DI water, then methanol and dried in hot air

(3) An Ecor vs Time on Pt was run for one hour. At about 45-50 min the specimen was inserted into the test cell but not immersed. E vs T SWRT03

(4) At one hour the specimen was immersed (crevice above liquid) and a 1 hour Ecor vs. Time (SWRT S3) was done. Ecor at the end of 1 hour was -433 vs SCE.

(5) The cyclic polarization scan was then performed, starting at the Ecor potential. This is saved as SWR 03

(6) The immersed specimen dimensions were 0.246" ϕ 1.775" l for an area of 9.157 cm².

(7) The entire scan ran relatively noise free and without the impedance problems experienced on earlier experiments.

The specimen showed staining and evidence of pits. Two potentiostatic experiments were run to determine whether pits would continue to grow if a potential in the proper range was applied. This is stored as SWRT 03A and 03B.

Comparison of the SCE used vs another (new) one showed a difference of 8 mV.

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Run of ASTM G-5 Standard curve (SWRJ G5A)

① A 1.0 N H_2SO_4 solution was prepared by diluting 27.2 mL of 98% reagent grade H_2SO_4 to a volume of 1 liter in a volumetric flask.

② The 430SS specimen used was the standard material purchased from ASTM. The specimen had been used before, but was repolished to a 600 grit finish according to ASTM G-5.

③ The cell was heated and de-aerated at least 30 min before insertion of the specimen. 99.990% Argon was used. The specimen was immersed for one hour prior to scan initiation.

④ The platinum potential at 50 minutes was −209 mV vs SCE
The specimen potential at 55 minutes was −513 mV vs SCE
The specimen dimensions & area were:
0.367" ϕ 0.365" l Area = 3.398 cm^2

⑤ The run was aborted shortly after passing thru the first large knee, and it appeared impedance effects were coming into play. A loose connection was noticed on the capacitor line. Also, some moisture detected in spec. holder (leak).

⑥ The specimen was repolished and re-mounted and test was initiated in the same solution (including one hour hold). The restart Pt voltage was −208 mV vs SCE; Spec at 55 min E = −515

⑦ The run was aborted when the instrument went into overload at the same areas as previous experiments

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SIGNATURE Walter J. Machowski DATE 5/4/89

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Run of ASTM G-5 Standard curve (SWRJ G5A) DUPLICATE

(1) A 1.0 N H_2SO_4 solution was prepared by diluting 27.2 mL of 98% reagent grade H_2SO_4 to a volume of 1 liter in a volumetric flask.

(2) The 430SS specimen used was the standard material purchased from ASTM. The specimen had been used before, but was repolished to a 600 grit finish according to ASTM G-5.

(3) The cell was heated and de-aerated at least 30 min before insertion of the specimen. 99.990% Argon was used. The specimen was immersed for one hour prior to scan initiation.

(4) The platinum potential at 50 minutes was −209 mV vs SCE
The specimen potential at 55 minutes was −513 mV vs SCE
The specimen dimensions & area were:
0.367" φ 0.365" l Area = 3.398 cm^2

(5) The run was aborted shortly after passing thru the first large knee, and it appeared impedance effects were coming into play. A loose connection was noticed on the capacitor line. Also, some moisture detected in spec. holder (leak).

(6) The specimen was repolished and re-mounted and test was initiated in the same solution (including one hour hold). The restart Pt voltage was −208 mV vs SCE; Spect at 55 min E = −515

(7) The run was aborted when the instrument went into overload at the same areas as previous experiments

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SIGNATURE Walter J. Machowski DATE 5/4/89

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SwRI 04

① A J-13 working solution was prepared from Stock Soln A-1 and B-1. Adjusted pH 7.18.

② A 316L specimen was prepared by ~~good~~ polishing w/320 and the 600 grit finish.

③ Some problems were encountered in starting-up the test. Finally, the specimen was immersed for one hour prior to the start of a cyclic polarization scan. The redox potential and specimen potential were not manually read at 50 and 55 minutes.

④ The scan was run to completion. The immersed portion of the specimen was 0.246" Ø and 1.795" L. Area = 9.242 cm²

⑤ There is a "blip" in the scan which may have been caused by a bubble in the bridge

⑥ The electrometer was grounded to the fume hood for this run.

⑦ E_{corr} was −512 mV E_{pit} = +417 mV E_{prot} = +129 mV
all vs SCE

The post test solution pH was 8.33.
The post test solution pH was 8.33.

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SWR05

① J-13 water was prepared from stock A-1 and ~~B-2~~ B-1 conc. The adjusted pH was 7.03.

② A 316L specimen was wet polished with 220 and then 600 grit finish. It was then rinsed in acetone (ultrasonic) washed with DI water, rinsed w/methanol and hot air dried.

③ Approx 900 ml. of the J-13 water was put in the test cell and heated to 95°C while undergoing de-aeration with 99.999% Argon. The specimen was held above the liquid while de-aeration was being done. The specimen was immersed in the liquid after 30 minutes of de-aeration. No redox potentials ~~of~~ on Pt were taken.

④ The scan was started at E_{corr} instead of some potential cathodic to E_{corr} by an entry error in the program. E_{corr} was −736 mV vs SCE at test start.

⑤ A faradaic cage of aluminum screen wire was placed completely around the cell and grounded thru the electrometer.

⑥ Reading values off of edit plot: E_{corr} = −730 mV E_{pit} = +464 mV

E_{prot} = +131 mV all vs SCE

The immersed portion of the specimen was 0.248" ϕ and 1.763" l Area = 9.173 cm²

The post-test solution pH was 9.53

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SIGNATURE Walter J. Machowski DATE 5/10/89

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SWR 06

① J-13 water was prepared from Stock Soln's A-1 and B-1. The adjusted pH was 9.13 Note: all pH adjustment to date have been with 0.1N HCl since all prepared J-13 waters have had a pH in XS of 7.20.

② A 316L specimen (one with indentation in bottom) was filed down until the indentation was gone. Then it was wet polished w/220 and finally 600 grit paper. Degreased in acetone (ultrasonic), rinsed w/DI water, rinsed in MeOH and hot air dried.

③ Approx 900 ml of J-13 were added to the test cell and heated to 95°C ±1° and de-aerated w/argon. The spec was held above the liquid during the 30-35 min de-aeration step. It was then immersed so that the wash interface was above the liquid level (as were previous tests)

④ Specimen was immersed 1 hour before a cyclic polarization scan was performed starting at +200 mV vs E_{corr}. Potential at 55 min E_{55} = -720 vs SCE

The run was run to completion. The area of the immersed portion of the specimen was measured 0.246" ϕ 1.784" l Area = 9.202 cm²

The values read off the edit plot were:

E_{corr} = -823 mV; E_{pit} = +511 mV; E_{prot} = +110 mV vs SCE

The post-test solution pH was 8.90

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SIGNATURE Walter J. Mochowski DATE 5/10/89

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SWR 07

① J-13 water was prepared from Stock Sol'n A-1 and B-1. The adjusted pH was 6.83. Post-test pH was 9.10

② A 304L SS specimen was prepared (indentation on bottom was filed off) by wet polishing with 220 and 600 grit paper. It was rinsed in acetone (ultrasonic), washed w/DI water, rinsed with MeOH and hot air dried.

③ Approx 900 ml of J-13 water were added to the test cell which was heated to 95°C and de-aerated with Argon. The spec was held out of the liquid until more than 30 minutes of de-aeration were accomplished then inserted such the crevice at the washer interface was above the liquid. E_{corr} at 55 min was −452 mV vs SCE

④ After the test the immersed area of the specimen was calculated.

0.247" ϕ 1.812" l Area = 9.380 cm^2

⑤ The parameters on the curve were

E_{corr} = −622 mV; E_{pit} = +107 mV; E_{prot} = +451 mV vs SCE

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SIGNATURE Walter J. Machowski DATE 11 May 89

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SWRI 08

① A liter of simulated J-13 water was prepared from stock solns A-1 and B-1. The adjusted soln pH was 7.06

② A 304L SS specimen was prepared (indentation on bottom) by filing off the bottom indentation and wet polishing with 320 grit and finishing with 600 grit. It was rinsed in acetone (ultrasonic - 15 min), washed w/ DI water, rinsed with MeOH and hot air dried.

③ Approx 900 mL of the test solution was added to the corrosion cell, heated to 95°C while de-aerating w/ Argon. The specimen was held above the liquid until 30 min. of de-aeration had passed. It was then immersed such that the washer interface was above the liquid level.

E at 55 min = −520 mV vs SCE

④ When the anodic scan was at approx +1300 mV and 5000 μA, it was accidentally aborted instead of manually reversed. Quickly a second experiment (SWRI 08A) was instituted to do a potentiodynamic experiment from +1300 mV on down. This plot could be overlayed on the other.

⑤ The immersed portion of the specimen was measured, and its area calculated. 3/14/90

0.246" ϕ ~~[illegible]~~ 1.796" l

Area = 9.261 cm²

The parameters of the curve were E_{corr} = −551 mV

E_{pot} = +128 E_{pit} = +514 mV all vs SCE

The post-test pH was 7.55. The specimen showed very little staining and only very few pits compared to most of the other tests.

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SIGNATURE Walter J. Machowski DATE 12 May 89

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WSM 3/20/90

SWRI 09

① One liter of simulated J-13 water was prepared from stock solutions A-1 and ~~A-2~~ B-1. The adjusted solution pH was 6.97. Post test pH was 9.22.

② A 304L SS specimen (with small indentation @ bottom) was prepared by filing off the indentation & then wet polishing with 220 and finally 600 grit paper. It was rinsed in acetone (ultrasonic - 10 min), washed w/DI water, rinsed w/MeOH and hot air dried.

③ Approx 900 ml of test solution was added to the corrosion cell, heated to 95°C and de-aerated w/argon. The specimen was held above the liquid for a minimum 30 min. before immersion. Immersion was with the washer interface above the liquid. It was immersed 1 hour before the cyclic polarization scan was started. E_{55min} = -746 mV vs SCE

④ The immersed portion of the specimen was measured.
0.246" ϕ 1.754" l Area = 9.052 cm²

The parameters from the curve were:

E_{corr} = -814; E_{prot} = +99; E_{pit} = +476 mV vs SCE

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SIGNATURE Walter J. Machowski DATE 15 May 89

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SWRI 10

① One liter of J-13 simulated water was prepared from stock solutions A-1 and B-1. The adjusted solution pH was 6.91. Post test pH was 8.11.

② A 304L SS specimen (with indentation) was prepared by filing off the indentation and then wet polishing with 220 and finally 600 grit paper. It was degreased in acetone (ultrasonic - 10 min), rinsed in DI water, rinsed in MeOH and hot air dried.

③ Approx. 900 ml. of the solution was transferred to the test cell and heated to 90°C. There was no de-aeration of the solution, it was run w/out any gas being ~~purged~~ sparged thru it.

④ The specimen was held in solution one hour prior to starting a polarization scan @ 1 mV/sec.

The immersed portion was measured:

0.247" ϕ 1.772" l Area = 9.180 cm²

The parameters off the curve were:

E_{corr} = −208; E_{pit} = +417; E_{prot} = +221 mV vs SCE

The specimen was moderately stained + pitted.

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SIGNATURE Walter J. Machowski DATE 16 May 89

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SWRI 11

① One liter of simulated J-13 water was prepared from Stock Solutions A-1 and B-1. The adjusted pH was 6.89

② A 316L specimen (with indentation) was filed down until the indentation was gone and then wet polished with 220 and finally 600 grit paper. It was then degreased in acetone (ultrasonic - 10 min), washed with DI water, then rinsed with MeOH and hot air dried.

③ Approx. 900 ml. of the test solution was put in the corrosion cell and heated to 90°C. No de-aeration or sparging of any gas was performed.

④ The specimen was held in solution for one hour prior to starting a cyclic polarization scan at 1 mV/sec.

⑤ The immersed portion of the specimen was measured and the area calculated.

0.247" ϕ 1.834" l Area = 9.491 cm²

The parameters obtained from the curve were:

E_{corr} = −158 mV; E_{pot} = +267 mV; and E_{pt} = +423 mV

all vs SCE

The final sol'n pH was 7.19

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SIGNATURE Walter J. Machowski DATE 16 May 89

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SWRI 12

① One liter of sim J-13 water was prepared from Stock Solutions A-1 and B-1. For this test, the chloride content was increased to 1000 ppm. This was accomplished by adding 1.637 gr. of reagent grade sodium chloride to the solution makeup process. The adjusted pH was 7.17. The final pH was 8.97.

② A 304L (with indentation) was prepared as previously described in experiment SWRI 10.

③ Approx. 900 of the test solution was heated to 95°C while de-aerating w/Argon. After 30 minutes of de-aeration the specimen was immersed (washer interface above liquid) and held for one hour prior to starting a cyclic polarization scan.

④ After returning 2½ hours later, it was noticed that a lot of "noise" was present in the data. There had been a heavy rain shower, but no electrical activity. It was also observed that the specimen holder may have leaned slightly causing the specimen to contact the reference electrode salt bridge. This is the most likely cause of the "noisy" curve.

⑤ The dimensions of the immersed portion were measured.
0.248" ϕ 1.871" l Area = 9.716 cm²

The parameters from the curve are:
E_{cor} = −693 mV; E_{pit} = −129 mV; and E_{prot} = −48 mV
all vs SCE

These values are suspect due to problem mentioned.

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SIGNATURE Walter J. Machowski DATE 18 May 89

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WDM 3/20/90

SWRI ~~12~~ 13

WDM 3/20/90

① One liter of simulated J-13 water was prepared from Stock Sol'ns A-1 and B-1. ~~The adjusted pH was~~ To this was added 1.637 grams of AR grade NaCl to adjust the chloride content to 1000 ppm. The adjusted pH was 6.88. The post test pH was 9.11.

WDM 3/20/90

② A 316L SS specimen was prepared as in SWRI 10.

③ Approx 900 ml of the test solution, in the cell, was heated to 95°C while sparging argon. After 30 min. of de-aeration, the specimen was immersed (washer interface above the liquid). After 1 hour of immersion, a cyclic polarization scan was started. $E_{SS} = -721$ mV.

The immersed portion of the specimen measured:
0.247" ϕ 1.870" l Area = 9.671 cm²

The parameters from the curve were:

$E_{corr} = -800$ mV; $E_{pit} = +21$ mV; $E_{prot} = -58$ mV vs SCE

Comparison of the SCE with another "fresh" electrode showed a difference of 2 mV.

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SIGNATURE Walter J. Machowski DATE 18 May 89

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SWRI 14

① One liter of J-13 simulated water was prepared from Stock Soln's A-1 and B-1. The adjusted pH was 7.18.⊛ The final pH was 9.20.

② A Hast C-22 specimen was prepared by wet polishing with 220 grit and finally with 600 grit paper. It was degreased in acetone (ultrasonic 10 min), rinsed with DI water, rinsed w/ MeOH and hot air dried.

③ Approx 900 ml of the test solution was heated in the corrosion cell while de-aerating w/ argon. The specimen was held above the liquid until 30 minutes had passed. It was then immersed for one hour prior to starting a cyclic polarization scan. E_{ss} not taken.

The immersed portion of the specimen was measured
0.247" Ø 1.770" ℓ Area = 9.170 cm^2

The specimen was stained w/ a yellow-orange color and may have had some pitting starting.

⊛ All adjustments to water pH from SWRI 1 to SWRI 14 have been with 0.1N HCl to lower the pH into the proper range.

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SIGNATURE		DATE
Walter J. Machowski		19 May 89
DISCLOSED TO AND UNDERSTOOD BY	DATE	WITNESS	DATE
N. Sridhar	2/14/90		

Preparation of Stock Solutions A-2 and B-2.

The two stock solutions were pepared according to the procedures described previously. Stock Soln A was left over the weekend before filtering out any remaining particles and diluting to volume w/DI water.

This solution is dated 5/19/89.

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SIGNATURE		DATE		DATE
Walter J. Machowski		5/19/89		
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N. Sridhar	3/14/90			

① One liter of simulated J-13 water was prepared from stock solutions A-2 and B-2. The pH of the solution before pH adjustment was 7.68. It was adjusted to a pH of 7.18 by addition of 3.0 ml of 0.1N HCl.

② A 304L SS specimen which had been previously used (304L-005) was prepared by wet polishing with ~~240~~ 320 and finally 600 grit paper. It was then degreased in acetone (ultrasonic - 10 min), washed w/DI water, rinsed with methanol and hot air dried.

③ 900 ml of the prepared water was placed in the corrosion flask and heated to 95°C. No de-aeration or sparging of any gas was used. One part of the flask was left open to allow natural aeration of the test solution.

④ Since no de-aeration was being done, the specimen was partially immersed (to avoid crevice effects) in the solution after it reached 95°C. The specimen was immersed for one hour prior to initiating polarization. The platinum potential after 50 minutes was +130 mV vs SCE. The specimen potential at one second before polarization was -144 mV vs SCE.

⑤ The cell was set up w/a 100 nF capacitor on the reference bridge; a 10 μF capacitor between the working electrode + ground to the electrometer. No Faraday cage.

⑥ The immersed portion of the specimen was measured and its area calculated.

0.247" Ø 1.741" l Area = 9.025 cm²

Parameters were E_{corr} = -136 mV; E_{pit} = +809 mV; E_{prot} = +768 mV vs SCE
off the curve

Post-test pH 9.12 SCE vs another SCE Δ 002 mV

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Mackowski DATE 6/9/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

TITLE

PROJECT NO. 20-3606

BOOK NO. 1

SWRI 16

① One liter of simulated J-13 water was prepared from Stock Solutions A-2 and B-2. The pH prior to adjustment was 7.76. 4.0 ml of 0.1 HCl were added to adjust the pH to 6.99. Final solution pH was ~~8.77~~ 8.77

② A 316L specimen which been previously used (316L-002) was prepared by wet polishing with ~~240~~ 220 and finally 600 grit paper. It was then degreased in acetone (ultrasonic - 10 min) rinsed in DI H_2O, rinsed with MeOH and hot air dried.

③ 900 ml of the solution was placed in the test cell and heated to 95°C. No de-aeration or sparging of any gas was used. One port of flask left open to allow natural aeration.

④ The specimen was immersed (partially to leave washer interface above liquid) for one hour prior to polarizing. The ref bridge appeared to be plugged (potentials too positive) and the SCE was positioned directly in the solution. E of Pt at 50 min was +98 mV. E spec at start was −118 mV.

✓ volume lost

⑤ The cell had a 10 μF capacitor between WKG-E and ground. No Faraday cage was used.

⑥ Upon returning from lunch, it appeared that something may have gone awry w/ the temp control. Only 1/3 the specimen was immersed and ~~about~~ the liquid level had dropped 3/4" to 1" due to evaporation. Also, the solution had taken on a distinct yellow color.

⑦ The run was completed and cataloged.

$E_{corr} = -120$ $E_{pit} = +537$ and $E_{pwt} = +32$ mV vs SCE

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 6/13/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 17

① One liter of simulated J-13 water was prepared from stock solutions A-2 and B-2. The pre-adjustment pH was 7.60. 4 ml of 0.1 HCl were added, and the final adjusted pH was 7.02. Final pH was ~~8.45~~. 8.63

② A 316L specimen which had been previously used (SWR 05) was repolished. Wet polishing w/ 220 grit until all pits were gone. A final polish w/ 600 grit was then done. It was degreased in acetone (ultrasonic - 10 min), rinsed with DI water, rinsed w/ MeOH and hot air dried.

③ 900 ml of the solution were transferred to the test cell and heated to 95°C. No de-aeration or sparging of any gas was used. The fitting for sparging was put in place rather than leaving a port open. This allowed the cell to "breathe", yet minimized evaporation of the test solution.

④ The specimen was immersed leaving the washer interface above the liquid level for one hour prior to starting polarization. The ref bridge had a .1 MFD capacitor to the electrometer, and a 10 μFD capacitor was used between the working electrode and the electrometer ground. No Faraday screen was used.

⑤ The Platinum potential after 50 minutes was +146 mV and the specimen potential one second before startup was −213 mV vs SCE.

⑥ The immersed portion of the specimen was measured:
0.232" ϕ 1.816" l Area = 8.812 cm²

⑦ The parameters off the curve were:
E_{corr} = −212 mV E_{pit} = +497 mV; E_{prot} = +229 mV vs SCE

⑧ The SCE was compared with another at test end and the difference was 4 mV.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 6/14/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 18

① One liter of simulated J-13 water was prepared from stock solutions A-2 and B-2. The pre-adjustment pH was 7.67. 4 ml of 0.1N HCl were added, and the adjusted pH was 7.04

② An Incoloy 825 specimen was prepared by wet polishing with 320 grit paper and finally with 600 grit paper. It was then degreased in acetone (ultrasonic - 10 min), rinsed w/DI water, rinsed w/MeOH and hot air dried.

③ 900 ml of the water was transferred to the test cell and heated to 95°C. No de-aeration or sparging of any kind was performed. The sparging fitting was used as in SwRI 17.

④ The test specimen was partially immersed in the water, leaving the washer interface above the liquid, for one hour prior to starting polarization. The reference bridge had a .1 mfd capacitor ground to the ref. plug on the electrometer. A 10 mfd capacitor was placed between the working electrode and ground to the electrometer.

⑤ The platinum potential after 50 minutes was +206 mV and the specimen potential (one second before startup) was -179 mV vs SCE.

⑥ The parameters of the curve were E_{corr} = -180 mV and the break point was +833 mV vs SCE.

⑦ The immersed part of the specimen was measured 0.248" ϕ 1.826" l Area = 9.158 cm²

Comparison of the test SCE against another showed a Δ 5 mV.

Final (post-test) pH was ~~8.29~~ 8.42 3/14/90

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE		DATE
Walter J. Machowski		6/15/89
DISCLOSED TO AND UNDERSTOOD BY	DATE	WITNESS	DATE
N. Sridhar	3/14/90		

SwRI 19

① One liter of J-13 simulated water was prepared from stock solutions A-2 and B-2. The pre-adjustment pH was 7.62. The pH of this solution was adjusted by bubbing CO_2 gas through the solution. This caused the pH to drop to 6.60. Approximately 4-5 ml. of 0.1N NaOH were added to bring the pH up to 7.07.

② A Hastelloy C-22 specimen was prepared by wet polishing with 220 grit and then finally with 600 grit paper. It was degreased with acetone (ultrasonic - 10 min), rinsed with DI water, rinsed w/ MeOH and hot air dried.

③ 900 ml. of the water was transferred to the corrosion cell and heated to 95°C. No deaeration or sparging of any kind was done. The sparging tube was in place as in SwRI 18.

④ The test specimen was partially immersed in the water to keep the washer interface above the liquid level. The spec. was immersed one hour prior to start of polarization. A 10 mFD capacitor was in place between the WKE and ground. A 0.1 mFD capacitor was on the reference bridge to the electrometer.

⑤ The Pt potential at 50 min was +169 mV vs SCE
The spec potential at startup was −200 mV vs SCE

⑥ The immersed portion of the specimen was measured and the area calculated.
0.248" ϕ 1.871" l Area = 9.716 cm²

Comparison of the SCE with another, showed a potential difference of 8 mV.

⑦ The hysteresis loop "knee" was at +769 mV and intersected on the cathodic scan at +580 mV.
The post-test pH was 8.18

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 6/16/89

DISCLOSED TO AND UNDERSTOOD BY N. Srinivas DATE 3/14/90 WITNESS DATE

SwRI 20

① One liter of simulated J-13 water was prepared from Stock Solutions A-2 and B-2. The pre-adjustment pH was 7.82; this was adjusted by bubbling Cl_2 gas through the solution. The final solution pH was 7.06 after adjustment with 3 ml of 0.1 N NaOH.

② An Incoloy 825 specimen was prepared by wet polishing with 220 grit and finally 600 grit paper. It was then degreased in acetone (ultrasonic-10 min), rinsed with DI water, rinsed w/MeOH and hot air dried.

③ 900 ml. of the water was transferred to the corrosion cell and heated to 95°C. No de-aeration or sparging of any kind was performed. The sparging tube was in place.

④ The test specimen was partially immersed in the water, keeping the crevice interface above the liquid level. The specimen was immersed for one hour prior to start of polarization. A 10 mfd capacitor was between the WKE and CRD. A 0.1 mfd capacitor was between the reference bridge and the electrometer connection.

⑤ The Pt potential after 50 min. was +138 mV vs SCE
The spec potential at test start was −230 mV vs SCE.

⑥ The immersed portion of the specimen was measured and the area calculated.
0.250" Ø 1.843" l Area = 9.655 cm^2

Reference electrode comparison showed a difference of 6 mV.

The post test pH was 9.31

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 6/27/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 21

① One liter of simulated J-13 water was prepared from Stock Solutions A-2 and B-2. For this test, a chloride ion concentration of 1000 ppm was needed so 1.657 grams of AR Grade NaCl were added to the preparation. The pre-adjustment pH was 7.72 and it was reduced to 7.15 by bubbling gaseous CO_2

② An Incoloy 825 specimen was prepared by wet polishing with 220 grit and then finally 600 grit paper. It was then degreased in acetone (ultrasonic - 10 min), washed w/ DI water, rinsed w/ MeOH and hot air dried.

③ 900 ml. of the solution was placed in the corrosion flask and heated to 95°C. No de-aeration or gas sparging was in effect. Sparging tube was in place.

④ The test specimen was partially immersed (keeping washer interface above the liquid) for one hour prior to starting polarization. A 10 mfd capacitor was between the WRE and CRD, and a 0.1 mfd capacitor was between the bridge and the electrometer.

⑤ The Pt potential after 50 min was +140 mV vs SCE and the test specimen at 1 startup was -257 mV vs. SCE.

Shortly before test end, a power "glitch" knocked the experiment off line and aborted it. Most of the data had already been obtained. The solution was quite yellowish with some fine precipitate evident.

Comparison of reference electrodes showed a difference of ~~N~~ 5 mV.

The post test pH was 8.90

Immersed portion 0.248" ϕ 1.825" l A = 9.470 cm²

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 6/28/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SWRI 22

① One liter of simulated J-13 water was prepared from stock solutions A-2 and B-2. The pre-adjustment pH was 7.88. It was brought down with gaseous CO_2 to 7.09. The chloride ion was measured with a specific ion electrode and measured 6 ppm vs lab prepared standards.

② A Hast C-22 specimen was prepared by wet polishing with 220 grit and finally with 600 grit paper. Examination under low power optical microscope showed a smooth, pit free finish. It was then cleaned in detergent (ultrasonic), rinsed w/ DI water, rinsed with MeOH and hot air dried.

③ 900 ml of the solution was transferred to the test cell and heated to 95°C. No de-aeration of ~~argon~~ or sparging of any kind was done. The sparging tube was in place.

④ The test specimen was partially immersed (keeping the washer interface above the liquid level) for one hour prior to initiating polarization. A 10 mFD capacitor was between the WE and CRD, and a 0.1 mFD capacitor was between the salt bridge and the electrometer.

⑤ The Pt potential after 55 min. was +213 mV vs SCE
The spec potential at startup was −191 mV vs SCE

⑥ The immersed portion of the specimen was measured
0.246" Φ 1.804" l Area = 9.301 cm²

Parameters from curve: E_{corr} = −192 E_{pit} = +761 E_{prot} = +513

Specimen had many small pits. Water only slightly off color (yellowish)
SCE compared to another showed Δ mV = 6 mV
After SCE cooled overnight Δ mV = 1 mV
Post test pH was 8.65

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 7/11/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

TITLE PLAT

PROJECT NO. 20-3606

BOOK NO. 1

This test was a potentiodynamic scan in simulated J-13 water using a platinum wire as the working electrode. This scan would demonstrate where the oxygen potential would occur.

① One liter of simulated J-13 water was prepared from Stock Solutions A-2 and B-2. The pre-adjustment pH was 7.98 and was brought down to 6.90 with gaseous CO_2. The Cl^- was measured w/a ISE at 6.5 ppm Cl^-.

② 900 ml. of the solution was transferred to the test cell and heated to 95°C. No de-aeration or gas sparging was done. The sparging tube was in place.

③ A piece of platinum wire 0.038"Φ, 2.254"l, area = 1.743 cm² was immersed in the solution prior to starting polarization. A one hour delay was used, as in other experiments.

The potential of the Pt counter electrode at 50 minutes was +225 mV vs SCE. The platinum wire potential at startup was +235 mV vs SCE.

A cyclic scan was run.

Solution pH at the end of the test was 8.49

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE		DATE
Walter J. Machowski		7/12/89
DISCLOSED TO AND UNDERSTOOD BY	DATE	WITNESS	DATE
N. Sridhar	3/14/90		

SwRI 53

① One liter of simulated J-13 water was prepared from Stock Solution A-2 and B-2. The pre-adjustment pH was 7.90. This was brought down to 6.93 with gaseous CO_2. About 5-6 ml 0.1N HCl were added because the solution was acidified to much to 6.65. The Cl^- was measured to be 7.5 ppm.

② An Incoloy 825 specimen was prepared by wet polishing with 220 and finally 600 grit paper. It was then degreased and cleaned with acetone, DI water, and MeOH and hot air dried. An O-ring was placed at the ~~[illegible]~~ longitudinal center to form an artificial crevice. WDM 3/21/90

③ 900 ml. of the solution was placed in the corrosion cell and heated to 95°C. No de-aeration or gas sparging was in effect. The sparging tube was in place.

④ The test specimen was partially immersed (keeping the washer interface above the liquid level) for one hour prior to initiating polarization. A 10 mfd capacitor was between WKC E and CR D; a 0.1 mfd capacitor was between the bridge and the electrometer.

⑤ The Pt potential after 50 min was +173 mV vs SCE and the specimen at startup was -231 mV vs SCE. The immersed portion of the specimen was measured 0.246" ϕ 1.831" l Area = 9.367 cm²

A no pitting results was obtained.

Comparison of reference electrode to another 5 mV = 1 mV.

The post-test pH was 8.60.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Nachoraski DATE 7/14/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 24

① One liter of simulated T-13 water was prepared from stock solutions A-2 and B-2. To this, 1.637 grams of NaCl were added to adjust the Cl^- concentration to 1000 ppm. The pre-adjustment pH was 7.78 and it was adjusted w/ gaseous CO_2. About 3ml of 0.1N HNO3 were added to get a final adjustment of 6.92. The Cl^- was measured to be 1020 ppm.

② An Incoloy 825 specimen was prepared by wet polishing w/ 220 and finally 600 grit paper. It was then degreased and washed with acetone, DI water, MeOH and hot air dried. An O-ring was placed on the longitudinal center to form an artificial crevice

③ 900 ml of the test solution was placed in the test cell and heated to 95°C. No de-aeration or gas sparging was done.

④ The test specimen was immersed (leaving the washer interface above the liquid) for one hour prior to initiating polarization. A 10 mFd capacitor was between WKE & and RRD. And a 0.1 mFd capacitor was between the bridge and the electrometer.

⑤ The Pt potential at 50 minutes was +134 mV vs SCE
The specimen potential at startup was −261 mV vs SCE
The immersed portion of the specimen was measured.
0.246" ⌀ 1.869" l Area = 9.625 cm^2

The solution had taken a definite yellowish color by the time the reverse scan had started.

Post-test pH was 8.46

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Nachowski DATE 7/17/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 25

① One liter of simulated J-13 water was prepared from Stock Solutions A-2 and B-2. To this, an additional 1.637 grams of NaCl was added to adjust Cl^- concentration to 1000 ppm. Measured Cl^- concentration was 1000 ppm. The pre-adjustment pH was 7.93 and it was lowered w/ gaseous CO_2 to 6.86. About 3 ml of 0.1 N NaOH were added for final adjustment

② A 304L SS specimen (the first from Metal Samples) was prepared by wet polishing with 220 and finally 600 grit paper. It was degreased and washed with acetone, rinsed w/ DI water, MeOH and dried.

③ 900 ml. of the test solution was placed in the flask and heated to 95°C. No de-aeration or gas sparging was done. The sparging tube was in place.

④ The test specimen was partially immersed (leaving the washer interface above the liquid) for one hour prior to initiating polarization. A 10 mfd capacitor was between WKO-E and CRD, and a 0.1 mfd capacitor was between the bridge and the electrometer.

⑤ The Pt potential after 50 minutes was +168 mV vs SCE
Specimen potential at startup was −265 mV vs SCE

The immersed portion of the specimen was measured.
0.246" ϕ 1.796" l Area = 9.261 cm^2

Solution was a cloudy brownish-yellow. Specimen had much multi-colored staining.

Comparison of SCE with another showed 5 mV = 4 mV

Post-test pH was 8.59

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/18/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SwRI 26

① One liter of simulated J-13 water was prepared from Stock solutions A-2 and B-2. The pre-adjustment pH was 7.94, and this was lowered to 6.92 with gaseous CO_2. The chloride ion was measured at 6 ppm.

② A 304L SS specimen (from Metal Samples) was prepared by wet polishing with 220 and finally 600 grit paper. It was then degreased in acetone, rinsed w/DI water, rinsed w/MeOH and dried. An elastomeric O-ring was placed on the longitudinal center to form an artificial crevice.

③ 900 ml. of the test solution were placed in the test cell and heated to 95°C. No de-aeration or gas sparging was done. Sparging tube was in place.

④ The test specimen was partially immersed (leaving the washer interface above the liquid) for one hour prior to initiating polarization. A 10 μfd capacitor was between WKR-E and CRD, and a 0.1 mfd capacitor was between the bridge and the electrometer.

⑤ The Pt potential after 50 min. was +164 mV vs SCE
The specimen potential at start-up was −328 mV vs SCE.

The immersed portion of the specimen was measured:
0.245" ϕ 1.778" l Area = 9.133 cm²

The solution remained water clear.
Post test pH was 8.65

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 7/19/89

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 8/14/90 WITNESS DATE

Preparation of Stock Solution A-3/B-3

Two stock solutions were prepared according to the general procedures described on page #1. Two major differences in the preparation are:

① "Nanopure" water (17-18 Mohm) was used instead of 200 Kohm de-ionized water.

② 0.376 grams of $KF \cdot 2H_2O$ were added to account for the waters of hydration.

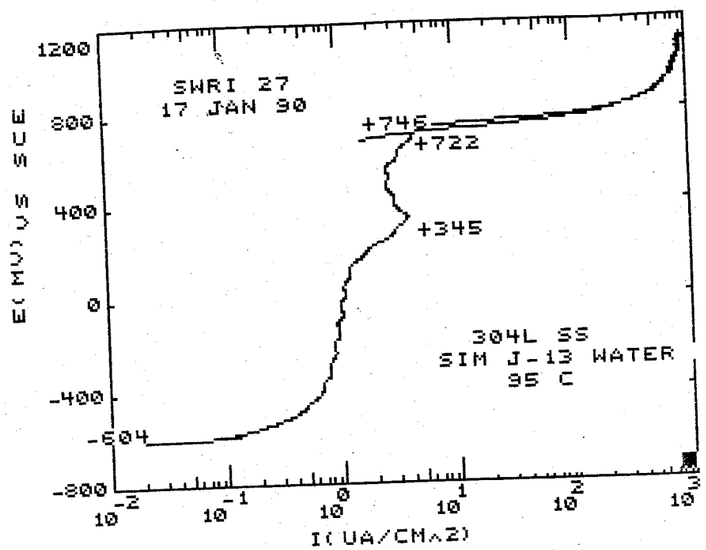
No filtration of any particles was performed.

The solutions are dated 1/12/90.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/12/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI27
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	~~5000~~ manually reversed at 1109 mV and 9250 μA (925 μA/cm²)
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-602
MV/PT	4
DATA MAX	951
DATA MIN	-20.48
ABS MIN	.02
ABS MAX	951

LEGEND

304L SS IN SIM J-13 WATER AT 95C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 6.92

17 JAN 1990

① One liter of simulated J-13 water was prepared from Stock Solution A-3 and B-3. No silicic acid was added to this solution. The pre-adjustment pH was 7.93, ~~and~~ and this was lowered to 6.92 with gaseous CO_2. The chloride ion concentration was measured to be 7.1 ppm. High ~~of~~ purity water (17-18 Mohm) was used in the preparation.

② A 304L specimen (Metal Samples) was prepared by passivating in 20% HNO_3 overnight (min. 16 hrs). It was then polished with 220 and then 600 grit paper - except the top surface under the gasket to minimize crevice effects. It was then degreased in acetone rinsed w/ DI water, rinsed w/ Et OH and dried.

③ 900 ml. of the test solution was placed in the test cell and heated to 95°C and de-aerated w/ argon for one hour prior to immersion of the specimen. This procedure is in general accordance w/ ASTM G-61.

④ The test specimen was fully immersed in the test solution, and remained immersed for one hour prior to initiation of polarization. The PAR 173/276 was used without any external capacitors.

⑤ The Pt potential after 50 min was −137 mV vs SCE
The specimen potential at start-up was −603 mV vs SCE

⑥ The scan was manually reversed at 1109 mV / 9250 μA since the transpassive break was finished and current was not increasing much with increased potential. There was no point in letting the pre-programmed point of 5×10^{-3} A/cm² be reached.

⑦ The solution was water clear at the end of the test. Post-test pH was 8.71.

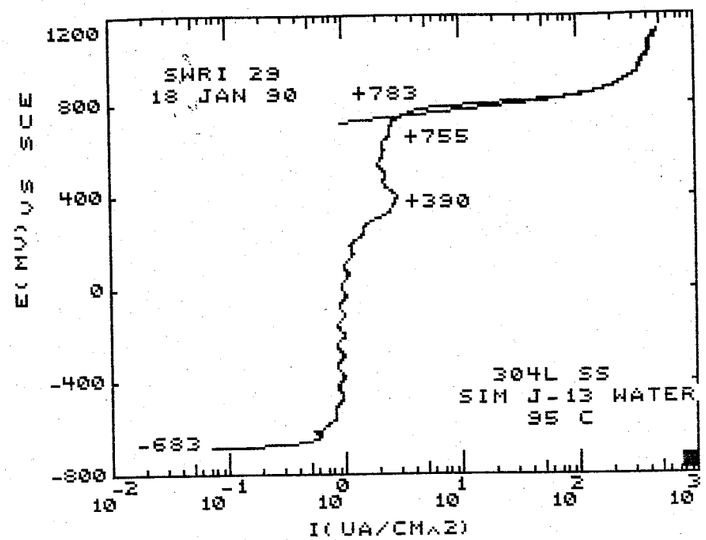
Comparison of the SCE with another showed ΔmV = 2.

This specimen was fabricated to have a nominal surface area of 10 cm² when totally immersed.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/17/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI29
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	~~800~~ manually reversed at 1120mV and ~460μA/cm²
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-683
MV/PT	4
DATA MAX	484
DATA MIN	.072
ABS MIN	.072
ABS MAX	484

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
DE-AERATION W/ ARGON ACC. TO G-61
INITIAL PH 6.99

18 JAN 1990

① One liter of simulated J-13 water was prepared from stock solution A-3/B-3 using high purity water (17-18 MΩ). No silicic acid was added to this solution. The pre-adjustment pH was 8.12 and it was lowered to 6.99 with gaseous CO_2. Chloride ion concentration was measured to be 7.0 ppm.

② A 304L specimen was prepared by passivating it in a 20% HNO_3 solution overnight (min. 16 hrs). It was then polished with 220 grit paper followed by 600 grit paper - except top surface which was left passivated. It was then degreased in acetone (rinse), cleaned w/detergent (ultrasonic), rinsed w/DI water, rinsed w/ETOH and dried.

③ 900 ml. of the solution was transferred to the test cell and heated to 95°C, and de-aerated w/argon for one hour prior to specimen immersion.

④ After one hour of de-aeration, the specimen was fully immersed in the test solution. A one hour delay was used prior to start of polarization. The PAR 173/276 was used without external capacitors.

⑤ The Pt potential after 50 min. was -192 mV vs SCE
The specimen potential at start-up was -683 mV vs SCE

⑥ The scan was manually reversed at 1120 mV / 460 μA since the transpassive break was complete.

⑦ Solution was water clear at the end of the test.
Post-test pH was 8.30

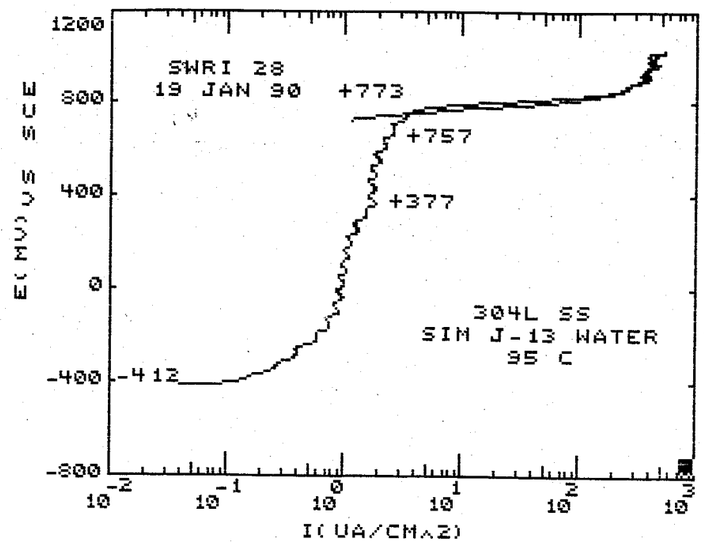
Comparison of SCE w/another showed ΔmV = 4

Specimen was 10 cm².

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Mackowski DATE 1/18/90

DISCLOSED TO AND UNDERSTOOD BY N. Nicholas DATE 3/14/90 WITNESS DATE



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI28
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	500
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-412
MV/PT	4
DATA MAX	529
DATA MIN	.0408
ABS MIN	.0408
ABS MAX	529

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 7.02

19 JAN 1990

① One liter of simulated J-13 water was prepared from stock solutions A-3/B-3 using hi-purity (17-18 Mohm) water. No silicic acid was added to the solution. The pre-adjustment pH was 8.08, and this was lowered to 7.02 w/ gaseous CO_2. Chloride ion concentration was found to be 6.3 ppm.

② A 304L specimen (Metal Samples) was passivated overnight (min 16 hrs) in 20% HNO_3. It was then polished w/ 220 followed by 600 grit finish. Polishing was done in a stirring motor rather than by hand as in previous tests. The threaded end was left unpolished. It was degreased in acetone (rinse), detergent (ultrasonic), rinsed w/ DI water, then EtOH and dried.

③ 900 ml of the solution was transferred to the test cell and heated to 95°C while de-aerating with argon. De-aeration was conducted for one hour before the specimen was immersed.

④ The specimen was then fully immersed in the test solution and was at open circuit for one hour before polarization began. The PAR 173/276 was used w/out external capacitors.

⑤ The Pt potential after 50 min was +35 mV vs SCE
The specimen potential at start-up was −413 mV vs SCE.

The Pt potential was considerably different from the prior two tests. Inspection of the experimental set-up revealed no abnormalities & the test was allowed to continue.

⑥ The solution was water clear at the end of the test.

Post-test pH was 8.23

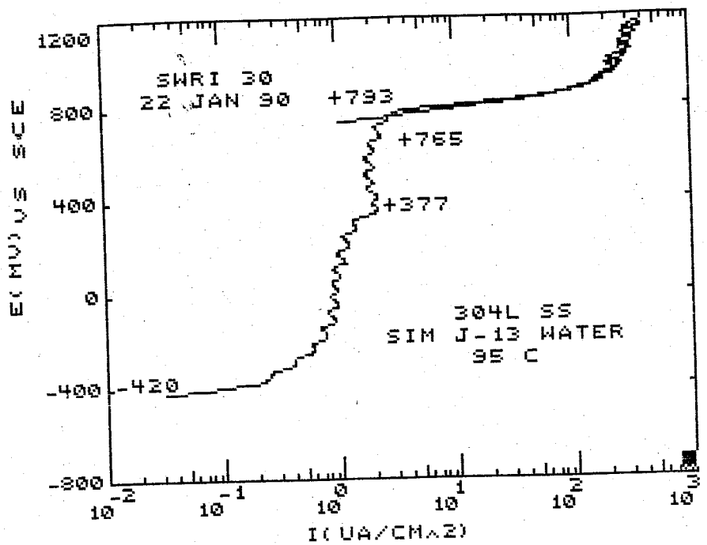
Comparison of the SCE with another showed ΔmV = 5
Specimen area was 10.0 cm²

NOTE: Thermo-Watch® temperature regulator put into use w/ this experiment & used on subsequent tests.
Scan was manually reversed.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/19/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI30
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	~~500~~ manually reversed at 1165 mV and 3760 μA
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-420
MV/PT	4
DATA MAX	394
DATA MIN	-20.48
ABS MIN	.032
ABS MAX	394

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 6.98

22 JAN 1990

TITLE SwRI 30

PROJECT NO. 20-3704

BOOK NO. 1

① One liter of simulated J-13 water was prepared from Stock Solutions A-3/B-3 using high purity (17-18 MΩ) water. No silicic acid was added to the solution. The pre-adjustment pH was 8.03 and this was lowered to 6.98 with gaseous CO_2. Chloride ion concentration was 6.8 ppm.

② A 304L specimen was passivated in 20% HNO_3 for ~65 hours (over the weekend). It was then polished by mounting in a motor drive and spinning. A 220 grit, followed by a 600 grit polish was performed. It was then degreased in acetone (rinse), detergent (ultrasonic), DI water rinse, EtOH rinse and dried.

③ 900 ml. of the solution was transferred to the test cell and heated to 95°C while de-aerating w/argon. Solution was de-aerated for one hour prior to specimen immersion.

④ The specimen was fully immersed in the test solution, and remained at open circuit potential for one hour prior to the start of polarization. The PAR 173/276 was used w/out capacitors.

⑤ The Pt potential after 50 minutes was +39 mV vs SCE.
The specimen potential at start-up was −420 mV vs SCE.

⑥ The solution was water clear at the end of the test.

Post test pH was 8.20

Comparison of the SCE with another showed ΔMV = 3.

Specimen area was 10 cm².

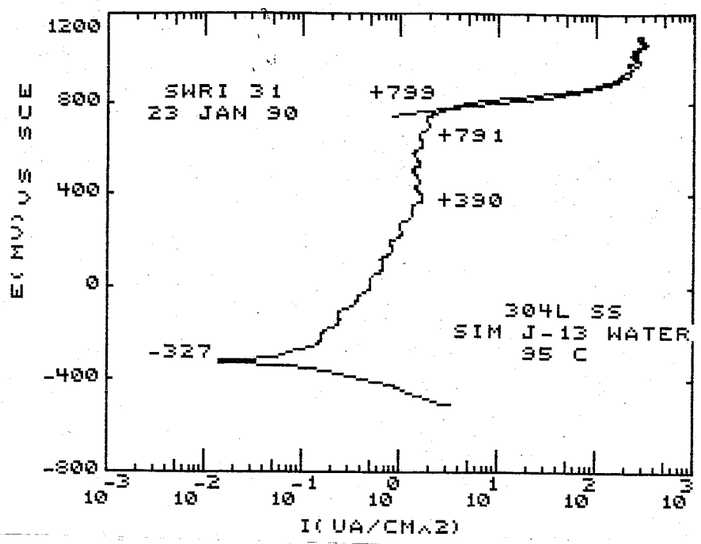
NOTE: Scan was manually reversed

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/22/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

SWRI31
CYCLIC POLN



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI31
INITIAL E(MV)	-200 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	~~500~~ manually reversed at 1102 mV and 2870 μA
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	1800 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-307
MV/PT	4
DATA MAX	326
DATA MIN	-3.28
ABS MIN	0
ABS MAX	326

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
START SCAN IN CATHODIC REGION
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 6.92 / FINAL 8.22

23 JAN 1990

① One liter of simulated J-13 water was prepared from stock solutions A-3 / B-3 using high purity (17-18 MΩ) water. No silicic acid was added. The pre-adjustment pH was 8.12 and this was lowered to 6.95 with gaseous CO_2. Chloride ion concentration was 6.8 ppm.

② A 304L specimen was passivated in 20% HNO_3 overnight (min. 16 hours). It was then polished on a motor drive with 220 followed by 600 grit polishing paper. It was then degreased in acetone (rinse), detergent (ultrasonic) rinsed w/DI water, rinsed w/EtOH and dried.

③ 900 ml. of the test solution was transferred to the test cell and heated to 95°C while de-aerating with argon. The solution was de-aerated for one hour prior to immersion of the specimen.

④ After one hour of de-aeration, the specimen was fully immersed and remained at open circuit potential prior to the start of polarization. During this equilibration period, power was inadvertently turned off so that the computer quit the program. It was restarted with the program time modified so that a one hour wait would still be obtained prior to start of polarization. Started in the cathodic region.

⑤ The Pt potential at 50 min. was +121 mV vs SCE
The specimen potential at start-up was -308 mV vs SCE

The scan was manually reversed.

The solution was water clear at the test's end.
Final pH was 8.22.

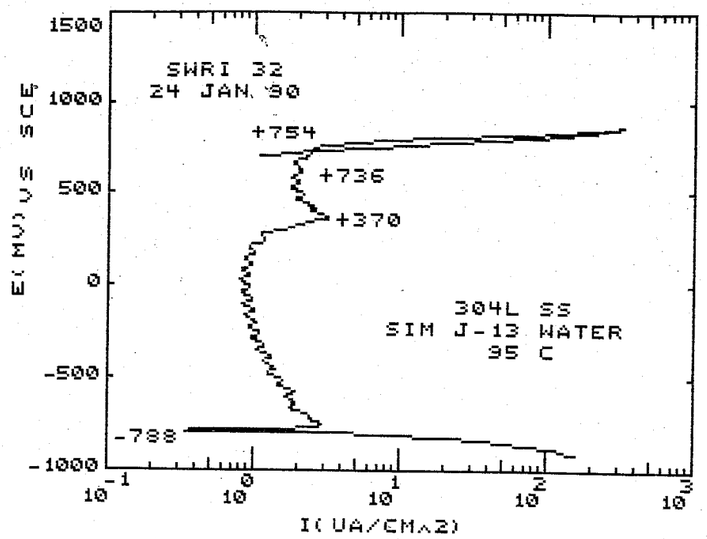
Comparison of the SCE w/another showed ΔmV = 1

Specimen area was 10 cm².

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE	DATE	WITNESS	DATE
Walter J. Machowski	1/23/90		
DISCLOSED TO AND UNDERSTOOD BY N. Nichols	DATE 3/14/90		

SWRI32
CYCLIC POLN



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI32
INITIAL E(MV)	-200 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	300
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-708
MV/PT	4
DATA MAX	333
DATA MIN	-204.8
ABS MIN	.344
ABS MAX	333

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
START SCAN IN CATHODIC REGION
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 6.92

24 JAN 1990

① One liter of simulated J-13 water was prepared using Stock Solutions A-3/B-3 and high purity (17-18 MΩ) water. No silicic acid was added. The pre-adjustment pH was 8.12 and this was lowered to 7.06 with gaseous CO_2. Chloride ion concentration was 6.8 ppm.

② A 304L specimen was passivated in 20% HNO_3 for a minimum of 16 hours. It was then polished on a motor drive with 220 grit followed by a 600 grit paper. It was then degreased in acetone (rinse), detergent (ultrasonic), rinsed in DI water, rinsed with ETOH and dried.

③ 900 ml of the test solution was transferred to the test cell and heated to 95°C while de-aerating w/argon. De-aeration was conducted for one hour prior to specimen immersion. A flask with water was used as a water trap for the exiting argon. A higher gas flow was used on this test compared with previous tests. This was determined by visually watching the gas bubbles through the solution.

④ After one hour of de-aeration, the specimen was fully immersed in the solution, and remained at open circuit for one hour prior to initiating polarization. Polarization was started in the cathodic region.

⑤ The Pt potential after 50 minutes was −352 mV vs SCE
The specimen potential at start-up was −708 mV vs SCE
Final pH was 8.73 ⊛

Comparison of SCE w/another showed ΔmV = 2
Specimen area was 10 cm².

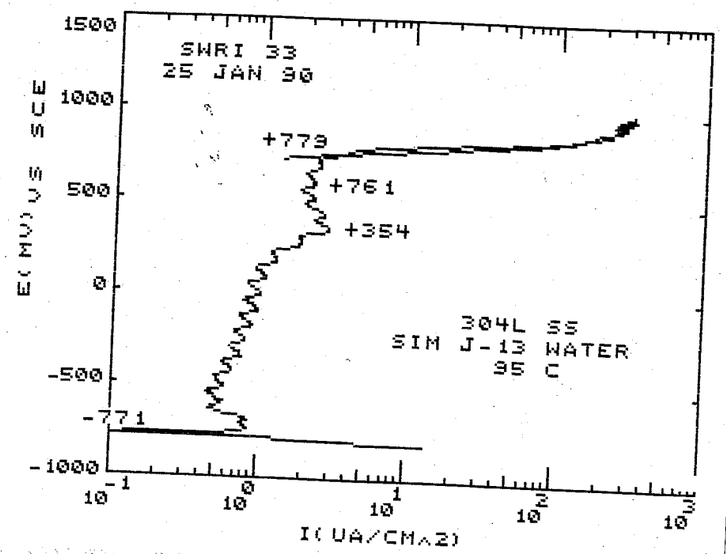
⊛ NOTE: The pH electrode was behaving erratically – it will be replaced w/a new one.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/24/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 2/14/90 WITNESS DATE

SWRI33
CYCLIC POLN



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI33
INITIAL E(MV)	-200 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	300
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-611
MV/PT	4
DATA MAX	324
DATA MIN	-13.4
ABS MIN	.102
ABS MAX	324

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
START SCAN IN CATHODIC REGION
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 7.12

25 JAN 1990

TITLE SwRI 33

PROJECT NO. 20-3704

BOOK NO. 1

① One liter of simulated J-13 water was prepared from stock solutions A-3/B-3 using high purity (17-18 MΩ) water. No silicic acid was added. The pre-adjustment pH was 8.21 and this was lowered to 7.12 w/gaseous CO_2. The chloride was not determined.

② A 304L specimen was passivated in 20% HNO_3 overnight (minimum 16 hrs). It was then polished on a motor drive with 220, followed by 600 grit paper. It was then degreased in acetone (rinse), detergent (ultrasonic), rinsed w/DI water, rinsed with EtOH and dried.

③ 900 ml. of the test solution was transferred to the test cell and heated to 95°C, while de-aerating with argon (water trap). De-aeration continued for one hour before the test specimen was immersed in the solution.

④ After one hour, the specimen was fully immersed and kept at open circuit potential for one hour prior to initiating polarization. Polarization was started in the cathodic region.

⑤ The Pt potential after 50 min was −217 vs SCE.
Specimen potential at start up was −611 vs SCE.

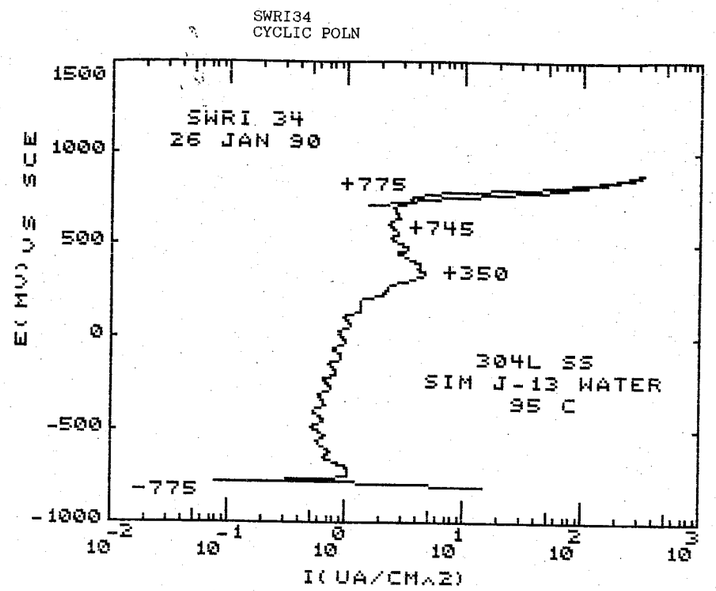
Post test pH was 9.65.

Comparison of SCE w/another showed ΔmV = 1
Solution remained water clear
Specimen area was 10 cm².

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 1/25/90

DISCLOSED TO AND UNDERSTOOD BY M Sridhar DATE 3/14/90 WITNESS DATE



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	SWRI34
INITIAL E(MV)	-200 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	100 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	300
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-615
MV/PT	4
DATA MAX	338
DATA MIN	-14.5
ABS MIN	.078
ABS MAX	338

LEGEND

304L SS IN SIM J-13 WATER AT 95 C
PH ADJUSTED W/CO2:NO SILICIC ACID
RUN ON PARC 173/276
START SCAN IN CATHODIC REGION
DE-AERATION W/ARGON ACC. TO G-61
INITIAL PH 6.93

26 JAN 1990

① One liter of simulated J-13 water was prepared from stock solutions A-3/B-3 using high purity (17-18 MΩ) water. No silicic acid was added. The pre-adjustment pH was 8.41 and this was lowered to 6.93 with gaseous CO_2. Chloride concentration was 6.5 ppm.

② A 304L specimen was prepared by passivating in 20% HNO_3 overnight (minimum 16 hr). It was then polished on a motor drive with 220 grit followed by 600 grit paper. It was then degreased in acetone (wipe), detergent (ultrasonic), rinsed w/ DI water, rinsed w/ EtOH and dried.

③ 900 ml of the test solution was transferred to the test cell and heated to 95°C while de-aerating with argon (water trap). De-aeration continued for one hour prior to specimen immersion.

④ After one hour, the specimen was fully immersed and remained at open circuit potential for one hour prior to the start of polarization. Polarization was started in the cathodic region.

⑤ The Pt potential after 50 min. was -300 mV vs SCE
The specimen potential at start-up was -614 mV vs. SCE
Post test pH was 9.68.

Comparison of SCE w/another showed ΔmV = 5

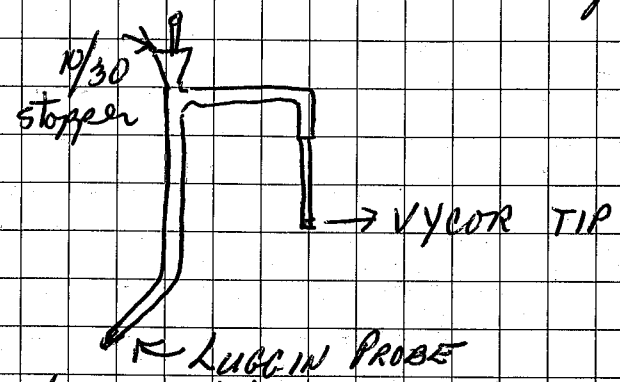
Specimen area was 10 cm²
Solution remained water clear.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

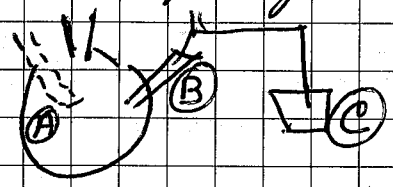
SIGNATURE Walter J. Machowski DATE 1/29/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 1/14/90 WITNESS DATE

① In order to place the reference electrode in another container to avoid it being placed in hot solution as happens with the PARC bridge. A salt bridge was constructed to connect the PARC bridge with a beaker in which solution and the reference electrode would reside. The bridge looked like:



When filled completely, and stoppered minimal leakage occurred.



Three points at which the reference was placed were checked to measure what variations in potential might be observed. (A) was the reference directly in the test solution (B) was reference at the PARC bridge (C) was the reference at an extend pt. w/ new bridge. An arbitrarily picked 304L specimen (already used) was the specimen. Solutions were emheated and not de-aerated.

② Using simulated J-13 water in cell, both bridges, and beaker, the PARC 173 was used to measure potential

at (A) +203 mV vs SCE

(B) +249 mV " SCE

(C) +245 → +280 mV vs SCE (E varied sinusoidally)

Measurements at the same points w/ 602 Keithley electrometer

(A) +203 mV vs SCE

(B) +249 " "

(C) +260 " "

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/1/90

DISCLOSED TO AND UNDERSTOOD BY N. Nicholas DATE 3/14/90 WITNESS DATE

(3) A 3.5% solution of NaCl and water was prepared, and the cell, bridges, etc were filled with this solution. After 30 minutes of equilibrating to let the vycor tips wet out, the potentials at (A), (B), and (C) were again measured.

with PARC 173:	(A)	+245 mV	with Keithley	(A)	+245 mV
	(B)	+244 "		(B)	+244 "
	(C)	+245 "		(C)	+246 "

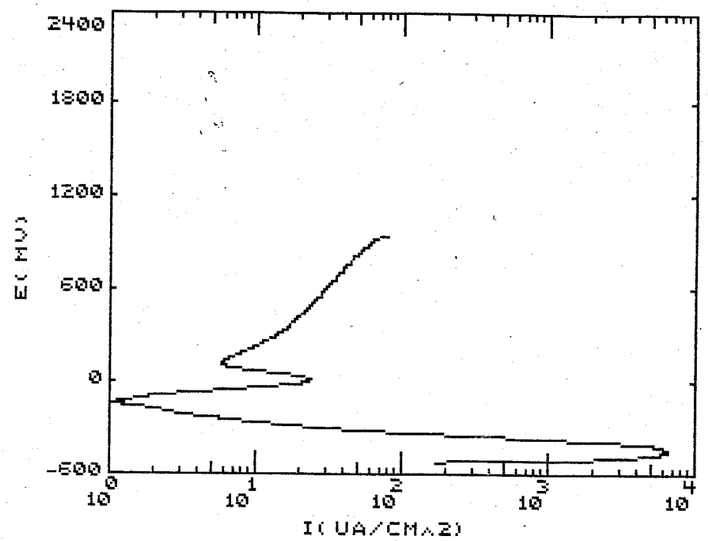
The higher conductivity solution gave consistent readings at the various sampling points.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/1/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

G5-757
POTENTIODYNAMIC



Test aborted – specimen holder leaked

RUN PARAMETERS

TECHNIQUE	POTENTIODYNAMIC
ORIGINAL NAME	G5-757
INITIAL E(MV)	0 VS E
FINAL E(MV)	1600 VS R
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	3.562
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-521
MV/PT	2
DATA MAX	6457.046
DATA MIN	1.055587
ABS MIN	1.055587
ABS MAX	6457.046

LEGEND

G5 ON PARC 273/AFTER REPAIR
DE-AERATE W/ARGON
USE RE-POLISHED 430 SS SPECIMEN

13 FEB 1990

① A 1.0N solution of H_2SO_4 was prepared according to ASTM G5 using high purity water.

② A 430SS specimen was also prepared by polishing w/220 grit and finishing with 600 grit.

③ De-aeration with high purity argon was done.

The Pt potential 10 min prior to polarization was -212 mV vs SCE

The specimen was immersed for one hour prior to polarization, it's potential at start-up was -521 mV vs SCE

④ This was the first test w/ PARC 273 (S# 41108) after its return from repairs. It appeared to be operating properly.

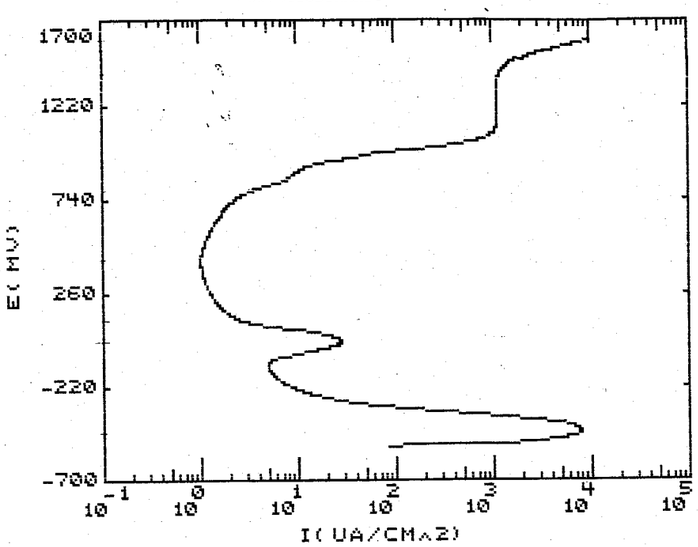
⑤ Noticed that curve was not matching the standard G5 curve. Further examination revealed a leak into the working specimen holder. The test was aborted at this time.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/13/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 2/14/90 WITNESS DATE

G5-857
POTENTIODYNAMIC



RUN PARAMETERS

TECHNIQUE	POTENTIODYNAMIC
ORIGINAL NAME	G5-857
INITIAL E(MV)	0 VS E
FINAL E(MV)	1600 VS R
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	4.195
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-524
MV/PT	2
DATA MAX	9392.133
DATA MIN	1.020262
ABS MIN	1.020262
ABS MAX	9392.133

LEGEND

RUN ON PARC 273/AFTER REPAIR
DE-AERATE W/ARGON

14 FEB 1990

① A 1.0 N sulfuric acid solution was prepared according to G5 using high purity (17-18 MΩ) for dilution.

② A 430 SS was prepared by polishing w/ 220 grit and finished with 600 grit. Polishing was done on a motor drive.

③ de-aeration was performed using high purity argon

The specimen was 1.24 cm × 0.91 cm ϕ ∴ Area = 4.195 cm²

The Pt potential 10 min before polarization was -208 mV vs SCE

The specimen potential at start up was -524 mV vs SCE

④ Everything seemed to operate properly, and a good G5 curve was obtained.

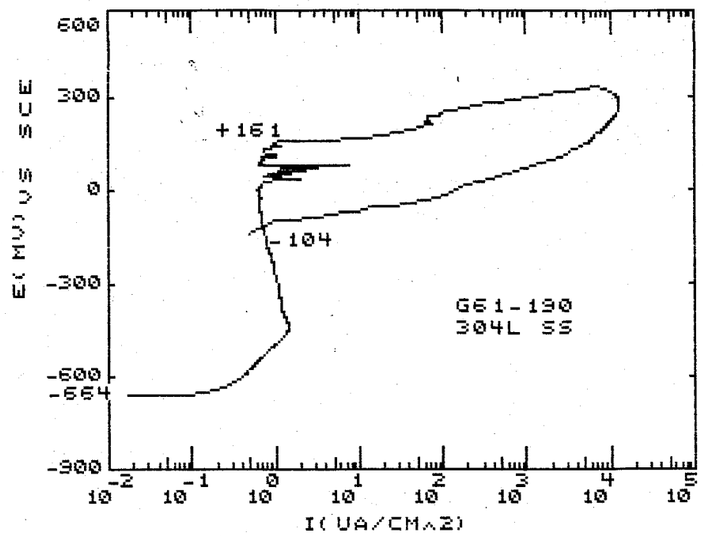
SCE reference used was #2

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/14/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

G61-190
CYCLIC POLN



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	G61-190
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	-1 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	5000
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	~~PASS~~ 1 HOUR IMMERSION

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-663
MV/PT	4
DATA MAX	12790
DATA MIN	.0174
ABS MIN	.0174
ABS MAX	12790

LEGEND

RUN ON PARC/273 AFTER REPAIR
DE-AERATION W/ARGON
CYLINDRICAL SPEC/10 SQ CM AREA

15 FEB 1990

TITLE G61-190

PROJECT NO. 20-3704-042

BOOK NO. 1

① A 3.56% w/w solution of NaCl was prepared using reagent grade salt and high purity (17-18 MΩ) water.

② A 10 cm² cylindrical specimen was polished with 240 grit and finished with 600 grit paper.

③ De-aeration was done according to C61 using argon. Specimen was immersed for one hour prior to start of polarization.

④ The Platinum potential 10 min prior to start: +68 mV vs SCE
Specimen potential at start − 663 mV vs SCE

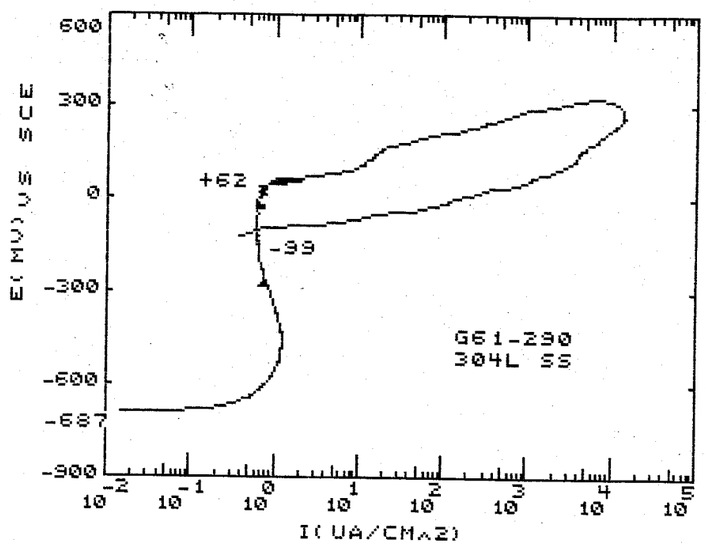
⑤ SCE reference used was # 2
Comparison with "calibrated" SWV = 2

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/15/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 2/14/90 WITNESS DATE

G61-290
CYCLIC POLN



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	G61-290
INITIAL E(MV)	0 VS E
VERTEX E(MV)	100 VS E
FINAL E(MV)	-1 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	5000
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA(CMS^2)	10
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-686
MV/PT	4
DATA MAX	13820
DATA MIN	.0145
ABS MIN	.0145
ABS MAX	13820

LEGEND

RUN ON PARC 273/AFTER REPAIR
DE-AERATION W/ARGON
CYLINDRICAL SPEC/10 SQ CM AREA

16 FEB 1990

TITLE G61-290

PROJECT NO. 20-3704-042

BOOK NO. 1

① A 3.65% w/w solution of NaCl was prepared with reagent grade salt and high purity (17-18 MΩ) water.

② A 10 cm² cylindrical specimen was polished with 240 grit and finished with 600 grit paper.

③ De-aeration was according to G61 using argon. Specimen was immersed for one hour prior to starting polarization.

④ The Pt potential 10 min before polarization : -27 mV vs SCE
Specimen potential at start : -686 mV " "

⑤ SCE reference was #2.
Comparison with calibration ΔmV = 2

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE Walter J. Machowski DATE 2/16/90

DISCLOSED TO AND UNDERSTOOD BY N. Sridhar DATE 3/14/90 WITNESS DATE

HASTELLOY C-22, MILL SURFACE - 20KV,WD=32 (after cleaning)
Standardless Analysis
20.0 KV 43.7 Degrees

Chi-sqd = 0.92

Element	Rel. K-ratio	Net Counts
Ni-K	0.57807 +/- 0.00615	36361 +/- 387
Ni-L	0.05512 +/- 0.00273	2224 +/- 110
Cr-K	0.12656 +/- 0.00229	15323 +/- 277
Mo-L	0.08882 +/- 0.00262	15448 +/- 455
W -L	0.06333 +/- 0.01419	1508 +/- 338
W -M	0.02863 +/- 0.00278	2599 +/- 252
Fe-K	0.04321 +/- 0.00227	3870 +/- 203
Co-K	0.01123 +/- 0.00244	849 +/- 184
Al-K	0.00504 +/- 0.00077	1011 +/- 155

ZAF Correction 20.00 kV 43.69 deg
No.of Iterations = 2

Element	K-ratio	Z	A	F	Atom%	Wt%
Ni-K	0.624	0.971	1.026	0.994	65.14	61.84
Cr-K	0.137	0.996	1.041	0.931	15.68	13.19
Mo-L	0.096	1.074	1.267	0.998	8.39	13.02
W -M	0.031	1.211	1.371	0.998	1.72	5.12
Fe-K	0.047	0.991	1.041	0.883	4.70	4.25
Co-K	0.012	1.009	1.030	0.996	1.32	1.25
Al-K	0.005	0.929	2.639	0.998	3.05	1.33
					Total=	100.00%

TITLE Surface & Bulk Analysis of Hastelloy alloy C-22 by SEM-EDX, 0.5" Plate

PROJECT NO. 3704-042

BOOK NO.

HASTELLOY C-22, MILL SURFACE - 20KV, WD=33
Standardless Analysis
20.0 KV 44.2 Degrees

Chi-sqd = 0.90

Element	Rel. K-ratio	Net Counts
Ni-K	0.58754 +/- 0.00616	37396 +/- 392
Ni-L	0.05219 +/- 0.00266	2141 +/- 109
Cr-K	0.13248 +/- 0.00230	16232 +/- 282
Mo-L	0.09003 +/- 0.00256	15877 +/- 451
W -L	0.05428 +/- 0.01415	1308 +/- 341
W -M	0.02622 +/- 0.00270	2417 +/- 249
Fe-K	0.04148 +/- 0.00225	3760 +/- 204
Co-K	0.01002 +/- 0.00243	766 +/- 186
Al-K	0.00576 +/- 0.00076	1171 +/- 155

ZAF Correction 20.00 kV 44.20 deg
No.of Iterations = 2

Element	K-ratio	Z	A	F	Atom%	Wt%
Ni-K	0.626	0.972	1.026	0.995	65.04	62.13
Cr-K	0.141	0.997	1.040	0.931	16.11	13.63
Mo-L	0.096	1.075	1.262	0.998	8.31	12.98
W -M	0.028	1.213	1.370	0.998	1.55	4.63
Fe-K	0.044	0.992	1.041	0.882	4.43	4.03
Co-K	0.011	1.010	1.030	0.996	1.15	1.11
Al-K	0.006	0.930	2.626	0.998	3.41	1.50
					Total=	100.00%

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE N. Sridhar DATE 3/14/90

DISCLOSED TO AND UNDERSTOOD BY [signature] DATE 3/14/90 WITNESS DATE 3/14/90

HASTELLOY C-22, SUBSURFACE - 20KV, WD=33
Standardless Analysis
20.0 KV 44.2 Degrees

Chi-sqd = 0.97

Element	Rel. K-ratio			Net Counts		
Ni-K	0.54517	+/-	0.00584	36031	+/-	386
Ni-L	0.05362	+/-	0.00256	2284	+/-	109
Cr-K	0.20660	+/-	0.00257	26287	+/-	327
Mo-L	0.08113	+/-	0.00245	14857	+/-	449
W -L	0.04237	+/-	0.01355	1060	+/-	339
W -M	0.02153	+/-	0.00254	2061	+/-	243
Fe-K	0.04195	+/-	0.00218	3948	+/-	205
Co-K	0.00549	+/-	0.00231	436	+/-	183
Al-K	0.00214	+/-	0.00069	451	+/-	146

ZAF Correction 20.00 kV 44.20 deg
No.of Iterations = 2

Element	K-ratio	Z	A	F	Atom%	Wt%
Ni-K	0.580	0.974	1.032	0.996	60.10	58.05
Cr-K	0.220	0.998	1.036	0.939	24.93	21.32
Mo-L	0.086	1.076	1.244	0.997	7.30	11.52
W -M	0.023	1.213	1.352	0.998	1.24	3.75
Fe-K	0.045	0.993	1.051	0.900	4.56	4.19
Co-K	0.006	1.011	1.038	0.997	0.63	0.61
Al-K	0.002	0.931	2.608	0.998	1.24	0.35
					Total=	100.00%

TITLE Surface & bulk analyses for Incoloy alloy 825, 0.5" Plate

PROJECT NO.

BOOK NO.

SQ: QUANTIFY

INCOLOY 825, MILL SURFACE - 20KV,WD=31
Standardless Analysis
20.0 KV 43.2 Degrees

Chi-sqd = 1.23

Element	Rel. K-ratio			Net Counts		
Fe-K	0.43716	+/-	0.00432	38339	+/-	379
Ni-K	0.33066	+/-	0.00526	20368	+/-	324
Ni-L	0.02093	+/-	0.00237	823	+/-	93
Cr-K	0.17066	+/-	0.00253	20232	+/-	300
Ti-K	0.00415	+/-	0.00114	630	+/-	173
Cu-K	0.01407	+/-	0.00387	712	+/-	196
Mo-L	0.01530	+/-	0.00198	2600	+/-	336
Si-K	0.00494	+/-	0.00090	1098	+/-	200
Al-K	0.00213	+/-	0.00085	417	+/-	166

ZAF Correction 20.00 kV 43.18 deg
No.of Iterations = 3

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.442	1.006	1.031	0.948	43.47	43.46
Ni-K	0.334	0.987	1.070	1.000	33.58	35.29
Cr-K	0.172	1.010	1.017	0.890	16.94	15.77
Ti-K	0.004	1.010	1.040	0.918	0.47	0.40
Cu-K	0.014	1.035	1.053	1.000	1.36	1.55
Mo-L	0.015	1.086	1.229	0.996	1.29	2.06
Si-K	0.005	0.912	2.053	0.999	1.86	0.93
Al-K	0.002	0.939	2.691	0.999	1.12	0.54
					Total=	100.00%

See next page (back of this page) for bulk analysis.

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE N. Sridhar DATE 3/14/90

DISCLOSED TO AND UNDERSTOOD BY DATE 3/14/90 WITNESS DATE 3/

SQ: QUANTIFY

INCOLOY 825, SUBSURFACE - 20KV,WD=26
Standardless Analysis
20.0 KV 40.5 Degrees

Chi-sqd = 1.06

Element	Rel. K-ratio			Net Counts		
Fe-K	0.28830	+/-	0.00347	29613	+/-	356
Ni-K	0.39378	+/-	0.00510	28419	+/-	368
Ni-L	0.03191	+/-	0.00219	1429	+/-	98
Cr-K	0.23609	+/-	0.00258	32766	+/-	358
Ti-K	0.00926	+/-	0.00109	1644	+/-	194
Cu-K	0.01397	+/-	0.00373	928	+/-	221
Mo-L	0.02326	+/-	0.00180	4574	+/-	354
Si-K	0.00277	+/-	0.00076	716	+/-	197
Al-K	0.00068	+/-	0.00071	154	+/-	161

ZAF Correction 20.00 kV 40.48 deg
No.of Iterations = 3

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.293	1.005	1.047	0.939	29.26	28.97
Ni-K	0.401	0.986	1.063	1.000	40.35	42.00
Cr-K	0.240	1.009	1.021	0.917	24.63	22.70
Ti-K	0.009	1.010	1.044	0.919	1.08	0.91
Cu-K	0.014	1.034	1.047	1.000	1.37	1.54
Mo-L	0.024	1.086	1.234	0.996	1.86	3.16
Si-K	0.003	0.912	2.101	0.999	1.08	0.54
Al-K	0.001	0.939	2.777	0.999	0.38	0.18
					Total=	100.00%

TITLE Surface & Bulk Analyses for AISI 304L S.S., 0.5" Plate

PROJECT NO.

BOOK NO.

AISI 304L, MILL SURFACE - 20KV, WD=33
Standardless Analysis
20.0 KV 44.2 Degrees

Chi-sqd = 0.92

Element	Rel. K-ratio			Net Counts		
Fe-K	0.67387	+/-	0.00669	33661	+/-	354
Ni-K	0.08241	+/-	0.00457	3063	+/-	170
Cr-K	0.21779	+/-	0.00375	15582	+/-	268
Si-K	0.00663	+/-	0.00112	891	+/-	150
Al-K	0.00155	+/-	0.00104	184	+/-	123
Mn-K	0.01545	+/-	0.00323	957	+/-	200
Cu-K	0.00232	+/-	0.00380	71	+/-	116

ZAF Correction 20.00 kV 44.20 deg
No.of Iterations = 1

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.672	1.003	1.032	0.989	67.05	68.73
Ni-K	0.082	0.984	1.103	1.000	8.28	8.92
Cr-K	0.217	1.006	1.009	0.861	19.90	18.99
Si-K	0.007	0.908	1.953	0.999	2.27	1.17
Al-K	0.002	0.935	2.535	0.999	0.74	0.37
Mn-K	0.015	1.023	1.004	0.992	1.55	1.57
Cu-K	0.002	1.032	1.079	1.000	0.22	0.26
					Total=	100.00%

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

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AISI 304L, SUBSURFACE - 20KV, WD=23
Standardless Analysis
20.0 KV 38.7 Degrees

Chi-sqd = 1.00

Element	Rel. K-ratio	Net Counts
Fe-K	0.67995 +/- 0.00681	35024 +/- 351
Ni-K	0.08170 +/- 0.00461	2957 +/- 167
Cr-K	0.21359 +/- 0.00375	14861 +/- 261
Si-K	0.00807 +/- 0.00108	1042 +/- 139
Al-K	0.00069 +/- 0.00096	79 +/- 109
Mn-K	0.01358 +/- 0.00327	818 +/- 197
Cu-K	0.00242 +/- 0.00397	72 +/- 115

ZAF Correction 20.00 kV 38.75 deg
No.of Iterations = 3

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.674	1.003	1.034	0.989	67.47	69.22
Ni-K	0.081	0.984	1.115	1.000	8.24	8.89
Cr-K	0.212	1.007	1.011	0.862	19.46	18.59
Si-K	0.008	0.908	2.051	0.999	2.89	1.49
Al-K	0.001	0.935	2.701	0.999	0.35	0.17
Mn-K	0.013	1.022	1.004	0.992	1.36	1.37
Cu-K	0.002	1.031	1.088	1.000	0.23	0.27
					Total=	100.00%

AISI 316L SS, MILL SURFACE - 20KV,WD=27
Standardless Analysis
20.0 KV 38.7 Degrees

Chi-sqd = 0.82

Element	Rel. K-ratio	Net Counts
Fe-K	0.67730 +/- 0.00677	35020 +/- 350
Ni-K	0.08648 +/- 0.00473	3142 +/- 172
Cr-K	0.19446 +/- 0.00365	13581 +/- 255
Si-K	0.00510 +/- 0.00110	661 +/- 142
Al-K	0.00115 +/- 0.00102	131 +/- 116
Mn-K	0.01972 +/- 0.00326	1193 +/- 197
Cu-K	0.00229 +/- 0.00389	68 +/- 116
Mo-L	0.01351 +/- 0.00238	1326 +/- 234

ZAF Correction 20.00 kV 38.75 deg
No.of Iterations = 3

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.670	1.001	1.033	0.989	67.63	68.52
Ni-K	0.086	0.982	1.113	1.000	8.79	9.36
Cr-K	0.192	1.005	1.013	0.863	17.94	16.92
Si-K	0.005	0.907	2.052	0.999	1.84	0.94
Al-K	0.001	0.934	2.697	0.999	0.58	0.29
Mn-K	0.020	1.021	1.005	0.992	1.99	1.99
Cu-K	0.002	1.029	1.088	1.000	0.22	0.25
Mo-L	0.013	1.080	1.211	0.996	1.00	1.74
					Total=	100.00%

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE N. Sridhar DATE 3/14/90

DISCLOSED TO AND UNDERSTOOD BY [illegible] DATE 3/14/90 WITNESS DATE

AISI 316L SS, SUBSURFACE - 20KV,WD=27.
Standardless Analysis
20.0 KV 38.7 Degrees

Chi-sqd = 0.82

Element	Rel. K-ratio	Net Counts
Fe-K	0.67730 +/- 0.00677	35020 +/- 350
Ni-K	0.08648 +/- 0.00473	3142 +/- 172
Cr-K	0.19446 +/- 0.00365	13581 +/- 255
Si-K	0.00510 +/- 0.00110	661 +/- 142
Al-K	0.00115 +/- 0.00102	131 +/- 116
Mn-K	0.01972 +/- 0.00326	1193 +/- 197
Cu-K	0.00229 +/- 0.00389	68 +/- 116
Mo-L	0.01351 +/- 0.00238	1326 +/- 234

ZAF Correction 20.00 kV 38.75 deg
No.of Iterations = 1

Element	K-ratio	Z	A	F	Atom%	Wt%
Fe-K	0.670	1.001	1.033	0.989	67.63	68.51
Ni-K	0.086	0.982	1.113	1.000	8.79	9.36
Cr-K	0.192	1.005	1.013	0.863	17.94	16.92
Si-K	0.005	0.907	2.051	0.999	1.84	0.94
Al-K	0.001	0.934	2.697	0.999	0.58	0.29
Mn-K	0.020	1.021	1.006	0.991	2.00	1.99
Cu-K	0.002	1.030	1.087	1.000	0.22	0.25
Mo-L	0.013	1.080	1.211	0.996	1.00	1.74
					Total=	100.00%

Pages 50 Through 51 Are Intentionally Left Blank

TITLE Calibration of Reference Electrode PROJECT NO. BOOK NO. 52

Keithley 602 Electrometer. 2/9/90.

Calomel electrode

Ref. electrode

"Reference" electrode (red coned) vs

①	− 3.0 mV	(black)
②	+ 3.5 mV	
③	+ 2.3 mV	
④ new	+ 27 mV.	
EG&G.	− 14 mV.	
Blue	− 110 mV.	(Agcl?)

SCIENTIFIC BINDERY PRODUCTS CHICAGO 60605

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS DATE

REVIEW OF SCIENTIFIC NOTEBOOK

"PRELIMINARY EXPERIMENTS AND PROCEDURE DEVELOPMENT
FOR NRC POLARIZATION TESTS"

COMMENTS:

1. Entry corrections were not made with a single line through incorrect data, initialed, and dated. pages- Table of Contents; # 28 & 29, 15, 19, 37, 40, 42, and the backside of pages 31, 32, 34, and 35.

2. Initial entries were not made describing the objectives of the experiments or tests, the experiment/test apparatus, test and measuring equipment requirements.

3. Test and measuring equipment and their respective calibration status was not provided, for example; balances, pH meters, electrometer, potentiostat, dimensional measuring equipment for specimens.

4. The data captured on magnetic media is also test data, so it should be more clearly identified and its storage location indicated. If possible, a copy of the data (on disk) should be included in the notebook so the notebook can be a stand-alone document.

don't have these #'s

5. Specimens must be identified by heat number and/or receiving log number to maintain traceability to material analysis documentation.

6. Reagents used for standards should be identified by lot number and/or receiving lot number for traceability.

7. In-process entries entries should provide a more thorough explanation of activities and why method changes were made, for example;

The basis for the formulation of the simulated J-13 water is not referenced.

Silicic acid was deleted from the J-13 water formulation after Test SWRI-27, however an explanation for this method change was not provided.

After Test SwRI-27, the all specimen areas were 10 cm. sq., however , earlier tests measured and calculated areas for each specimen individually.

Entry on page 42 indicates that it was the first test after repair of the potentiostat. If prior tests indicated a problem with the equipment, it should have been discussed in the notebook.

8. The SEM analysis is identified as "Standardless". Without traceability to recognized (NIST/NBS) standards, data can be used "for information only". Initial entries for this set of tests should clearly indicate any qualification to use of the data.

3/15/90
KBurt

ADDITIONAL INFORMATION FOR SCIENTIFIC NOTEBOOK #: 012

Document Date:	05/01/1989
Availability:	Southwest Research Institute® Center for Nuclear Waste Regulatory Analyses 6220 Culebra Road San Antonio, Texas 78228
Contact:	Southwest Research Institute® Center for Nuclear Waste Regulatory Analyses 6220 Culebra Road San Antonio, TX 78228-5166 Attn.: Director of Administration 210.522.5054
Data Sensitivity:	■"Non-Sensitive" □ Sensitive □"Non-Sensitive - Copyright" □ Sensitive - Copyright
Date Generated:	1992
Operating System: (including version number)	DOS
Application Used: (including version number)	EG&G 342C
Media Type: (CDs, 3 1/2, 5 1/4 disks, etc.)	1- 3 ½ disk, 1 CD
File Types: (.exe, .bat, .zip, etc.)	ASCII
Remarks: (computer runs, etc.)	Media contains: Corrosion data files.