CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

CORRECTIVE ACTION REQUEST

CAR No: 2005-3 **Associated AR, SR, NCR No:** AR 2005-1

PART A: DESCRIPTION OF CONDITION ADVERSE TO QUALITY

Contrary to the requirements of the CQAM, Section 12, Rev.4, Chg. 3, inadequate labeling or other non-compliance(s) to calibration requirements were noted:

1. Fourteen thermocouples used in the CLST laboratory were calibrated, but found without labels. (Section 12.4 (4))

The following apply to the slow-rate strain testing performed in Division 18, Building 90 Annex:

2. Load cell and linear displacement transducers (LVDT) calibrated by Division 18 but not labeled as to status. (Section 12.4 (4))
3. Thermocouple calibration by Division 18 not documented. (Section 12.4 (6))
4. Identification of the digital voltmeter used to take voltage potential across specimen not noted in the test documentation. Traceability not available to evaluate subsequent out of tolerance conditions. (Section 12.7)

Initiated by: R. Weber **Responsible Individual:** D. Dunn
Date: May 18, 2005 **Response Due:** June 15, 2005

PART B: PROPOSED ACTION

1) Extent of Condition:
1. In addition to the 14 thermocouples, other equipment used in testing identified to be without mandatory calibration information include: (i) temperature controllers used to both control and measure test cell temperature, (ii) a metallurgical microscope used to measure localized corrosion penetration depth and (iii) two colorimeters used to conduct microbial influenced corrosion investigations
2. The load cell, LVDT, thermocouple, and electrometer used in slow strain rate tests in Building 90 annex are the only are instruments owned by Division 18 that are presently used in CNWRA slow strain rate tests. Additional load cells and LVDTs owned by Division 18 have been used in previous stress corrosion cracking tests.

2) Root Cause:
1. The labeling of individual thermocouples was not considered to be practical by CNWRA staff, instead, separate calibration status information was relied upon.
2. The Division 20 staff are not clear on how to control work using equipment and personnel from other SwRI Divisions and comply with the applicable QA requirements.

3) Remedial Action: **Proposed Completion Date:** July 1, 2005

1. Individual calibration labels were added to the group of 14 thermocouples.
2. A calibrated thermocouple meter was added to record the temperature of test cells.
3. Per QAP-019, the LVDT and the load cell require calibration before use. The details of the calibration procedures for these instruments will be documented in the appropriate scientific notebook. Labels will be added to the equipment to indicate calibration status (CALIBRATE BEFORE USE).
4. The fine focus knob of the metallurgical microscope used to measure localized corrosion penetration depth was calibrated (i.e., verified to have 1 micrometer graduations) using a calibrated caliper. The verification of the performance was recorded in scientific notebook 308.
5. CALIBRATE BEFORE USE stickers will be added to the colorimeters used in the microbial Influenced corrosion studies.

4) Corrective Action to Preclude Recurrence: **Proposed Completion Date:** July 30, 2005

1. Staff shall be retrained on QAP-001 and QAP-019. The requirements of these procedures has been summarized in an training guide with specific suggestions for initial and in process entries for scientific notebooks. The guidance document provides specific instructions on the use of equipment from other Divisions.
2. Initial scientific notebook entries for ongoing tests will be revised to include additional information on test equipment calibration as required by QAP-001. The revised initial entries will be reviewed and approved by the element manager.
3. Test equipment and instrumentation in Division 18 laboratories will be classified as calibrate before use. Scientific notebook initial entries will specifically identify equipment not owned by Division 20 and the calibrations applied to that equipment. Per QAP-019, details of the calibrations or performance verifications will be documented in the scientific notebooks.
4. Staff will discontinue using the provision listed in CQAM Section 12.4.1(5) allowing calibration labels to be placed on other locations such as equipment containers when it is impractical to attach the calibration labels to the calibrated item. Instead, each calibrated item will be labeled with status information.

Manager/Director: [signature] **Date:** 6/1/2005

PART C: APPROVAL
Comments/Instructions

FORM QAP-14 (06/2004)

CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

CORRECTIVE ACTION REQUEST

CAR No: 2005-3 **Director of QA:** [signature]	**Associated AR, SR, NCR No:** AR 2005-1 **Date:** 6/1/2005
PART D: VERIFICATION OF CORRECTIVE ACTION IMPLEMENTATION see below **Verified by:** [signature] **Date:** 7/27/05	**Distribution:** **Original-CNWRA/QA DIRECTOR QA Records** **~~ORIGINATOR~~** **PRINCIPAL INVESTIGATOR** D. DUNN **MANAGER** V. JAIN **ASSISTANT DIRECTOR** S. MOHANTY **CNWRA PRESIDENT** GED V.P.

Remedial action:

*
1. Individual calibration tags verified on remaining thermocouples.
2. 4 Thermocouple meters are now calibrated and able to measure the temperature of test cells.
3. Calibration Before Use labels added,
4. Fine focus adjustment performance verification is documented in Scientific Notebook #308 Page 195.
5. "Calibrate Before Use" stickers have been placed on the colorimeters.

Corrective action to preclude recurrence

Corrective action to preclude recurrence was performed on 6/6/05 (attached)

1. Training on QAP-001 & 015 was performed
2. Training was performed on QAP-001 (Snd Ed 1). Initial Entries were revised (attached) & Remedially stored (attached)
3. Slow strain rate calibration gage blocks ~~were calibrated~~ as evidence of calibration was verified.
4. Calibrate labels were used for each thermocouple (example attached)

* Remedial actions verified by: MLE SwRI QA 25-27 July 05

FORM QAP-14 (06/2004)

CAR 2005-2 Corrective Action Item 1
CAR 2005-3 Corrective Action Item 1

GEOSCIENCES AND ENGINEERING DIVISION
MEETING ATTENDANCE

SUBJECT OF MEETING: Training on Procedures QAP-001, QAP-009, QAP-016, QAP-017, QAP-018, QAP-019, and TOP-012

DATE: June 6, 2005 **LOCATION: Bldg. 189**

PERSON	ORGANIZATION	TITLE/FUNCTION	TELEPHONE NUMBER
Kuang-Tsan Chiang	GED	Sr. Res. Scientist	210-522-2308
Lietai Yang	GED	Sr. Res. Engineer	210 522 2483
Xihua He	GED	Res. Scientist	210-522-5194
PAVAN SHUKLA	GED	Res. Engineer	210-522-6534
Yi-Ming Pan	GED	Principal Engineer	210-522-6640
VIJAY JAIN	"	Manager, CSPE	210-522-5439
DARRELL DUNN	GED	Principal Engineer	210 522 6090

Form AP-5

CAR 2005-3 Corrective Action Item 1
CAR 2005-3 Corrective Action Item 2
CAR 2005-3 Corrective Action Item 3

Scientific Notebook Initial and In Process Entries for Laboratory Activities

From QAP-001

Initial Entries. Scientific notebook initial entries shall be made in the beginning of the notebook and whenever substantive changes to the objectives, approach, or methods are made. Initial Entries shall include:

- Title of the experiment, field investigation, computer simulation, or other technical task.
- Names of the individuals performing the activity.
- Description of the objectives of the task and the proposed approach or procedure for achieving the objectives.
- Special personnel training or qualification requirements

Laboratory and Field Activity initial entries shall also include, as appropriate:

- Equipment and materials to be employed during the experiment, including any necessary design or fabrication of experimental equipment and any material/chemical characterizations.
- Measurement parameters and test equipment calibration, accuracy, and precision requirements.
- As applicable, description of suitable and controlled environmental conditions.
- Potential sources of uncertainty and error.

In process entries. Scientific notebook in-process entries shall be made at the time work is performed. Laboratory and field activity in-process entries shall include, as appropriate:

- Evidence that experiment/test prerequisites have been met (i.e., special environmental conditions, equipment calibrations, etc).
- Description of the experiment or field investigation, including detailed description of the step-by-step process followed, either by reference to a Technical Operating Procedure (TOP) or industry Standard Method, or by description in the scientific notebook.
- Variance from Standard Methods shall be clearly identified as such and documented.
- Description of any conditions which may adversely affect the results.
- Identification of samples used and any additional equipment and materials not included as initial entries. Measuring and test equipment shall be identified by item and its calibration status documented.
- Significant data taken and a brief description of the results, to include notation of any unaccepted results.
- Any interim conclusions reached, as appropriate.

Suggested Format for Initial Entries

Title of the experiment, investigation, or other technical task

This can be short if it is an entry for a specific type of testing. Examples:
1. Crevice Chemistry Experiments for Type 316L Stainless Steel
2. Oxidation Rate Tests for Alloy 22

Names of the individuals performing the activity

As indicated, put the names of the individuals. All staff and consultants making entries should have completed COI evaluation. Also all persons making entries should sign the front of the notebook

Description of the task objectives, proposed approach or procedure to achieve objectives

List the objectives of the testing. Examples:
1. Determine the chemistry inside crevices during active crevice corrosion
2. Determine the oxidation kinetics and characterize intergranular oxidation of Alloy 22

The proposed approach must include specific information on how the tests will be conducted. A test plan and a test matrix should be considered.

The procedure for conducting the tests should be listed or described. If a standard test procedure is used (e.g. ASTM) the procedure number should be listed along with any deviations from the procedure. In many cases a combination of standard ASTM procedures are used along with procedures developed specifically for conducting the types of tests required. All of this information should be included. A reference to a paper or report describing the test procedure may be considered.

Special personnel training or qualification requirements

For many initial entries, this may not be necessary. This is training above and beyond the QA and standard laboratory training. Nevertheless make sure all persons making entries into the notebook have received training on QAP-001. Examples of specialty training such as radiation safety or laser safety training. Consider what type of objective evidence will be required to demonstrate training was obtained.

Equipment

List the equipment that will be used in the tests. This must include any necessary design or fabrication of experimental equipment. Consider the measurement parameters that are required. All parameters recorded should be obtained from instruments that are either calibrated or the performance of the equipment has been verified. If no calibration or performance verification sticker is present, the instrument is not calibrated and cannot be used for the measurement of critical parameters. Status of equipment with the following labels:

CALIBRATION NOT REQUIRED: This means that the instrument is not calibrated. Unless a performance verification has been conducted and documented, these instruments cannot be used for measuring critical parameters.

CALIBRATE BEFORE USE: The instrument must either be calibrated (SwRI Calibration Laboratory) or the performance must be verified and documented prior to use.

CALIBRATION OVERDUE: The calibration is no longer valid and the instrument needs to be returned to the SwRI Calibration Laboratory for calibration.

Materials to be employed during the experiment

This must include any necessary design or fabrication of test specimens and any material/chemical characterizations. The following must be included:

- Material specifications (e.g. Type 316L Stainless Steel)
- Heat Number (e.g. P80746)
- Test Specimen Description (e.g. CNWRA Drawing or ASTM standard)

Measurement parameters, test equipment calibration, accuracy, and precision requirements

List parameters that will be measured (e.g. Current density, Potential, pH, Temperature) and identify the instruments that will be used. Specific serial numbers may not be required. If specific serial numbers are included in the initial entry, then every time a different instrument is used, a explanation of the deviation from the initial entry will likely be necessary. The instruments used should either be calibrated or the performance of the instruments will need to be verified. Verification of instrument performance requires documentation with data and should use standard test methods (ASTM), accepted procedures from the manufacturer, or control tests using calibrated instruments or components. The procedure for verifying potentiostat performance (TOP-022) is a good example. A calibrated resistor is used to verify the performance of the entire system.

The equipment used must be compatible with the requirements. When in doubt, check the instrument specifications. Avoid over specification (e.g. specifying ± 0.5 °C when ± 2 °C is acceptable for the tests to be conducted) as several nonconformance reports have been written because parameters were over specified.

Equipment that is not owned by Division 20 should specifically be identified. This equipment should either be calibrated by the SwRI calibration lab or be calibrate before use. In either case, the calibration or performance verification information must be placed in the appropriate scientific notebook. At a minimum the calibration or performance verification must be conducted prior to and after the test data are acquired. Routine scheduled calibration or performance verification must be conducted for equipment that is used for extended periods. In general a 6 month interval should be used but an interval of up to 1 year may be necessary for equipment used in long term tests. The calibration status of the instrument must be clearly visible and must reference the scientific notebook where the calibration or performance verification information is documented.

As applicable, description of suitable and controlled environmental conditions

Include relevant conditions that must be controlled (e.g. test temperature) and specifications of the control. Consider how compliance with the controlled conditions will be determined and documented.

Potential sources of uncertainty and error

As applicable. This will vary significantly from one tests to the next.

Suggestions for In Process Entries

A test specific form that can be completed for each tests has many advantages and is recommended. Test forms can reduce the number of omission mistakes such as missing heat numbers and missing equipment calibration. There are many examples of test forms that have been used successfully. These may serve as examples.

While each type of test has different requirements the following information is typical of routine corrosion tests.

- Test Name
- Material and Heat Number
- Significant Material Preparation (provide a notebook number and page number if applicable)
- Test Solution Preparation including:
  - Concentrations,
  - Measured reagent weights,
  - Reagent lot numbers,
  - Volume of solution prepared,
  - Any stock solutions used (provide notebook number and page number),
  - Starting and ending pH.
- Other environmental conditions such as any gasses used
- Test temperature
- Initial conditions such as
  - corrosion potentials
  - potential of Pt counter electrodes
- Instruments and calibration status
  - model number
  - serial number
  - Calibration or Performance Verification Date
  - Calibration or Performance Verification Due Date
- Values of parameters that were measured in the test
- Electronic data files collected in the test

Include any additional information necessary. If the test results are invalid, the scientific notebook entry should clearly indicate this and why the tests are invalid.

CAR 2005-3 Corrective Action Item 3 SN#695

Details of Equipment Calibration and Performance Verification.

SSRT Instrument Calibration for Slow Strain Rate Tests per QAP-019.

Institute Calibration Laboratory Instruments Processed:

Action Performed: CHECKIN **Date:** 5/19/2005

Asset	Charge Code	Workorder	Model	Manufacturer	Description	Serial Number
011659	00751.006 1.20	303064334	TYPE K	DURO-SENSE	THERMOCOUPLE	0003

Total Number of Instruments Processed: 1

MARCH 19, 2004

THE GAGE BLOCKS LISTED BELOW WERE MEASURED IN BUILDING #162 LOCATED ON SOUTHWEST RESEARCH INSTITUTE CAMPUS. THE TEMPERATURE WAS 70 DEGREES AND HUMIDITY OF 60 PERCENT. 0-1 DIGITAL MICROMETER (ASSET# 009976, SERIAL #2191790) WAS USED TO TAKE THE MEASUREMENTS AND HAS A CALIBRATION DUE DATE OF JAN. 15, 2005.

GAGE BLOCK #	DIMENSION MEASURED
1/16 P&W X369A	.0625
1/16 P&W X379A	.0624
.125 P&W Y242A	.12505
.250 P&W Y455A	.2500
.500 P&W Y694A	.5001
1.000 P&W Y189B	1.0000

SwRI 24 QA

WALTER L. SMITHSON
IQA Sr. INSPECTOR

K.T. Chiang
6/15/06

SOUTHWEST RESEARCH INSTITUTE
CALIBRATION LABORATORY
MEMORANDUM

To: DARRYL WAGAR DIV18 B128

From: Walt Hill, Metrology Group Leader
Institute Calibration Laboratory

Subject: Status of Calibration Supplier

Manufacturer/Model: MOREHOUSE 10,000 LBF
Description: PROVING RING
Serial Number: 3668C-709
Asset Number: 000748
Work Order Number: 444061883
Date Calibrated: December 30, 2004
Supplier: MOREHOUSE, YORK PA - A2LA - 717 843-0081
Remarks: Morehouse Report # 3668CL3004.

(✓) Supplier is on the Approved Suppliers List (ASL).

( ) Supplier is not on the Approved Suppliers List.

(✓) Calibration is ISO 17025 accredited.

( ) Calibration is not ISO 17025 accredited.

( ) There is no known supplier to meet ISO 17025 accreditation at this time.

Please notify the Institute Calibration Laboratory, extension 5215, of any discrepancies with the item or calibration documentation.

Attachment(s) 9

K. T. Chiang
6/15/06

"As Returned"

Certificate of Calibration

and Traceability to the

United States National Institute of Standards & Technology

MOREHOUSE INSTRUMENT CO.
SERIES 100: PROVING RING, SERIAL NO. 3668C
10,000 LBF CAPACITY, TENSION & COMPRESSION

The above identified instrument was calibrated in accordance with Section 7 of the American Society for Testing and Materials (ASTM) specification E74-02 entitled, "Standard Practice of Calibration of Force-Measuring Instruments...". We could not provide an "As Received" calibration due to the condition of the Proving Ring when it was returned to us. This calibration is in conformance with the requirements of Morehouse QAM Rev.7, dated 12/04/00, ISO/IEC 17025, and ANSI/NCSL Z540.1.

The result of this calibration as determined by statistical analysis according to Section 8 of ASTM E74-02, is as follows:

	Uncertainty	Resolution
Tension:	**1.57 Lbf**	**0.60 Lbf**
Compression:	**1.61 Lbf**	**0.58 Lbf**

This is within the Manufacturer's tolerance of 5.00 Lbf.

Calibration data is correct for a temperature of 23 degrees C. For a temperature of t degrees C, the values of the calibration factor should be corrected for temperature using the following formula.

$d_{23} = d_t - .00027 \times ((t-23) \times d_t)$

Where: d_{23} = deflection at a temperature of 23 degrees C.
d_t = deflection at a temperature of t degrees C.
t = temperature, degrees Celsius.

This calibration is certified traceable to the United States National Institute of Standards & Technology according to the following documentation and calibration apparatus used:

Dead Weight Force Machine S/N M-4644 NIST Lab No. 822/255038-95

Uncertainty of Force Standard used to perform this calibration did not exceed +/- 0.002% of applied load

Calibrated By:

MOREHOUSE INSTRUMENT CO. MI-CO

Date Calibrated:
February 16, 2004
Report No: 3668CB1604

MOREHOUSE INSTRUMENT COMPANY, INC.
FORCE CALIBRATION LABORATORY
1742 SIXTH AVENUE
YORK, PA 17403-2675 U.S.A.
PHONE: 717 / 843-0081
FAX: 717 / 846-4193
WEB : www.morehouseinst.com

K. T. Chiang
6/15/06

SOUTHWEST RESEARCH INSTITUTE

6220 Culebra Road
San Antonio. TX. 78228

Progam name: Program Number: 0
Date: 03/01/105 Time: 11:35 Page: 1

Description	Value	Tolerance
DISTANCE Plane (Feature # 1) to Point (Feature # 2)		
DISTANCE	0.0625	1/16 P+W X369A

SOUTHWEST RESEARCH INSTITUTE

6220 Culebra Road
San Antonio. TX. 78228

Progam name: Program Number: 0
Date: 03/01/105 Time: 16:56 Page: 1

Description	Value	Tolerance
DISTANCE Plane (Feature # 1) to Point (Feature # 2)		
DISTANCE	0.0625	1/16 P+W 379A
DISTANCE Plane (Feature # 1) to Point (Feature # 3)		
DISTANCE	0.1251	.125 P+W Y242A
DISTANCE Plane (Feature # 1) to Point (Feature # 4)		
DISTANCE	0.2500	.250 P+W Y455A
DISTANCE Plane (Feature # 1) to Point (Feature # 5)		
DISTANCE	0.5001	.500 P+W 694A
DISTANCE Plane (Feature # 1) to Point (Feature # 11)		
DISTANCE	1.0001	1.000 P+W Y189B

Bldg # 162
TEMP. 70°
Humidity 48%
CMM 900900871 DUE 1-18-05

MAR - 1 2005

K. T. Chiang 6/15/06

Load Cell Calibration

Frame No. 1 **Date:** 4/4/2005

Load Cell S/N: 732329

Morehouse Proving Ring S/N: 3668C-709

Last Cal.: 12/30/2004 **Next Cal:** 12/30/2006

Load	Moorehouse Ring	Adj. Moorehouse Ring Wts.	Meter	Computer	Chart Table Wts.
0	4.0	0.0	0.0	-3.7	0
600	54.2	50.2	599.7	594.7	593
1200	105.7	101.7	1198.0	1194.6	1201.7
1800	155.7	151.7	1791.4	1789.2	1794.2
2400	204.3	200.3	2376.1	2376.2	2371.6
3000	252.3	248.3	2951.4	2952.4	2943.2
3600	300.5	296.5	3522.6	3524.1	3518.5
4200	348.0	344.0	4091.0	4093.0	4086.9
4800	395.0	391.0	4652.7	4655.7	4650.7
5000	425.0	421.0	5010.7	4995.0	5011.3
4800	400.3	396.3	4716.0	4719.0	4714.4
4200	361.0	357.0	4237.3	4239.2	4242.7
3600	313.7	309.7	3697.4	3699.2	3676.3
3000	264.0	260.0	3081.6	3083.1	3082.7
2400	212.5	208.5	2465.4	2465.4	2469.1
1800	160.6	156.6	1849.4	1847.5	1852.3
1200	108.0	104.0	1232.5	1229.5	1228.9
600	56.0	52.0	615.1	610.1	614.2
0	4.0	0.0	2.4	1.5	0.0

Load Cell Calibration

Frame No. 2 **Date:** 4/4/2005

Load Cell S/N: 732328%

Morehouse Proving Ring S/N: 3660C-709

Last Cal.: 12/30/2004 **Next Cal:** 12/30/2006

Load	Moorehouse Ring	Adj. Moorehouse Ring Wts.	Meter	Computer	Chart Table Wts.
0	4.0	4.0	0.7	2.1	0
600	51.3	51.3	563.2	562.1	558.6
1200	100.6	100.6	1137.1	1136.5	1141.3
1800	148.4	148.4	1711.4	1710.3	1707.6
2400	197.0	197.0	2283.4	2282.4	2284.7
3000	245.0	245.0	2856.1	2855.1	2856.1
3600	293.3	293.3	3429	3427.2	3432.5
4200	340.0	340.0	4001.2	3999.1	3991.1
4800	387.0	387.0	4575.2	4572.3	4554.7
5000	420.0	420.0	4958.8	4955.8	4951.1
4800	385.0	385.0	4578.3	4575.3	4500.6
4200	339.9	339.9	4007.3	4004.8	3870.2
3600	292.0	292.0	3435.3	3433.5	3417
3000	243.3	243.3	2862.6	2861.5	2835.9
2400	195.0	195.0	2286	2288.6	2260.9
1800	148.0	148.0	1718.5	1716	1702.8
1200	100.6	100.6	1142.2	1140.7	1141.3
600	52.0	52.0	567.3	561.6	566.9
0	4.0	4.0	0	2	0

K. T. Chiaf
6/15/06

Load Cell Calibration

Frame No. 3 **Date:** 4/4/2005

Load Cell S/N: 789126

Morehouse Proving Ring S/N: 3668C-709

Last Cal.: 12/30/2004 **Next Cal:** 12/30/2006

Load	Moorehouse Ring	Adj. Moorehouse Ring Wts.	Meter	Computer	Chart Table Wts.
0	4.0	0	0.0	-2.0	0.0
600	53.3	49.3	575.3	574.5	582.5
1200	103.6	99.6	1157.8	1156.7	1176.8
1800	153.5	149.5	1740.7	1739.3	1768.1
2400	200.1	196.1	2322.7	2319.0	2321.6
3000	247.3	243.3	2840.1	2839.4	2883.6
3600	287.3	283.3	3304.5	3304.2	3360.8
4200	332.4	328.4	3830.5	3830.4	3900.1
4800	355.6	351.6	4102.2	4102.2	4178.0
5000	374.8	370.8	4328.4	4328.9	4408.2
4800	367.6	363.6	4244.2	4243.1	4321.9
4200	342.7	338.7	3949.7	3949.9	4023.4
3600	302.0	298	3481.8	3481.7	3536.5
3000	250.3	246.3	2910.0	2909.1	2919.3
2400	203.8	199.8	2327.2	2325.6	2365.6
1800	153.3	149.3	1743.7	1742.8	1765.7
1200	103.4	99.4	1161.6	1160.6	1174.4
600	53.5	49.5	577.9	577.0	584.6
0	4.0	0	-0.9	-1.5	0.0

Load Cell Calibration

Frame No. 4 **Date:** 4/4/2005

Load Cell S/N: 789127

Morehouse Proving Ring S/N: 3668C-709

Last Cal.: 12/30/2004 **Next Cal:** 12/30/2006

Load	Moorehouse Ring	Adj. Moorehouse Ring Wts.	Meter	Computer	Chart Table Wts.
0	4	0.0	0.4	0.2	0.0
600	48.3	44.3	532.6	531.6	523.2
1200	96.5	92.5	1091.6	1090.6	1092.8
1800	143.0	139.0	1640.7	1639.7	1643.5
2400	188.3	184.3	2184.9	2182.4	2181.3
3000	234.5	230.5	2727.5	2726.4	2731.0
3600	279.5	275.5	3265.1	3268.1	3267.7
4200	324.0	320.0	3803.3	3802.6	3799.6
4800	367.0	363.0	4332.4	4331.6	4314.7
5000	396.2	392.2	4672.2	4671.3	4665.1
4800	370.5	366.5	4389.4	4389.3	4356.7
4200	336.4	332.4	3944.0	3943.0	3948.0
3600	298.4	294.4	3480.5	3479.3	3493.4
3000	252.6	248.6	2945.2	2944.7	2946.7
2400	202.4	198.4	2349.0	2347.0	2349.0
1800	149.0	145.0	1758.0	1761.1	1714.7
1200	102.0	98.0	1158.0	1156.5	1157.9
600	51.5	47.5	561.3	560.4	561.0
0	4.0	0.0	2.3	1.6	0.0

K. J. Chiang
6/15/06

Initial Scientific Notebook Entry for Corrosion Resistant Material Potentiostatic Tests, Potentiodynamic Tests, Slow Strain Rate Tests

Title: Potentiaostatic tests, polarization tests, slow strain rate tests (SSRT).

Test Performed by: Kuang-Tsan Kenneth Chiang, Walter Machowski, Brian Derby

Objective: Study the effect of Bicarbonate [HCO_3^-], Carbonate [CO_3^{2-}] Nitrate [NO_3^-] to Chloride [Cl^-] concentration ratio in simulated groundwater on stress corrosion cracking susceptibility of mill-annealed Alloy 22 and Alloy 22 weldments using slow strain rate tests.

Equipment: EG&G Versastat Serial Number 20104. EG&G Model 352 corrosion software. NEC 586 computer. Keithley Electrometer Model 614 SN 55538 or equivalent. ASTM G-5 Polarization Cell. The SSRT equipment and instrumentation in Div. 18 laboratory is classified as calibrate before use. The SSRT system includes B&B Motor Control Type MSH-54RL Motor, Ohio Gear Model D3 Gear Box, Sensotec LVDT Model VL7A, Bodine DC Motor Speed Control (SN L4904600), 440 multichannel potentiostats with National Instruments Labview data acquisition software 4.1. CNWRA Computer Property No. 2330. Calibration and due dates are provided in data sheets for each test.

Materials: Mill-annealed Alloy 22, gas metal-arc welded Alloy 22, gas tungsten-arc welded Alloy 22.

Specimen Specifications: Cylindrical CPP specimens 1.195"x.250" and slow strain rate specimens drawings 20-06002-01-321-001 and 20-06002-01-322-002. SSRT per ASTM G129 procedure.

Measurement Parameters: Current and Potential as described in TOP-008. Temperature of solution ±2°C.

Required Level of Accuracy: Potentials ±5mV. Current ±0.1 microamps.

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

K.T. Chiang
7/20/05

CAR 2005-3

Corrective Action Item 4

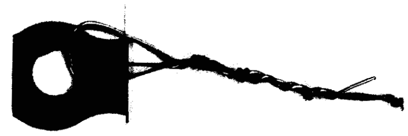
thermocouple #16

SwRI Cal Lab By:blt

Cal: Apr 06, 05 Due: Apr 05, 06

An:000261 Sn:293376W

See Attache Data - Tested at 0.0 a



GEOSCIENCES AND ENGINEERING DIVISION

M E M O R A N D U M

TO: QA Files

FROM: Vijay Jain, Manager - CSPE

SUBJECT: Review of revised initial entries in active lab notebooks

I have reviewed the revised initial entries in the following active scientific laboratory notebooks and found them to have adequately incorporated revised format. This completes the actions required by Item #2 of the Corrective Action to Preclude Reoccurrence for CAR 2005-3.

- SN 670
- SN 706
- SN 718
- SN 715
- SN 695
- SN 713
- SN 617
- SN 641 (closed)
- SN 659
- SN 675
- SN 533

Notebook No. 695

Initial Scientific Notebook Entry for Corrosion Resistant Material Potentiostatic Tests, Potentiodynamic Tests, Slow Strain Rate Tests

Title: Potentiaostatic tests, polarization tests, slow strain rate tests (SSRT).

Test Performed by: Kuang-Tsan Kenneth Chiang, Walter Machowski, Brian Derby

Objective: Study the effect of Bicarbonate [HCO_3^-], Carbonate [CO_3^{2-}] Nitrate [NO_3^-] to Chloride [Cl^-] concentration ratio in simulated groundwater on stress corrosion cracking susceptibility of mill-annealed Alloy 22 and Alloy 22 weldments using slow strain rate tests.

Equipment: EG&G Versastat Serial Number 20104. EG&G Model 352 corrosion software. NEC 586 computer. Keithley Electrometer Model 614 SN 55538 or equivalent. ASTM G-5 Polarization Cell. The SSRT equipment and instrumentation in Div. 18 laboratory is classified as calibrate before use. The SSRT system includes B&B Motor Control Type MSH-54RL Motor, Ohio Gear Model D3 Gear Box, Sensotec LVDT Model VL7A, Bodine DC Motor Speed Control (SN L4904600), 440 multichannel potentiostats with National Instruments Labview data acquisition software 4.1. CNWRA Computer Property No. 2330. Calibration and due dates are provided in data sheets for each test.

Materials: Mill-annealed Alloy 22, gas metal-arc welded Alloy 22, gas tungsten-arc welded Alloy 22.

Specimen Specifications: Cylindrical CPP specimens 1.195"x.250" and slow strain rate specimens drawings 20-06002-01-321-001 and 20-06002-01-322-002. SSRT per ASTM G129 procedure.

Measurement Parameters: Current and Potential as described in TOP-008. Temperature of solution ±2°C.

Required Level of Accuracy: Potentials ±5mV. Current ±0.1 microamps.

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

K.T. Chiang
7/20/05

Initial Scientific Notebook Entry for Dry-Air Oxidation, Humid Air Oxidation, Passive Film.

Title: Isothermal oxidation tests, cyclic oxidation tests, dry-air oxidation with deposited salt, oxygen 16/oxygen 18 experiments, microstructure analysis.

Test Performed by: Kuang-Tsan Kenneth Chiang

Objective: Study the oxidation kinetics, oxide morphology, and mechanism of passive film formation in dry air, humid air and thermal cycling conditions. Study the effect of fabrication processes such as welding and post-weld heat treatment on passive film formation.

Equipment: High Temperature Microbalance TherMax 700, Lindberg Furnace, Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Analysis (EDX), X-ray Diffraction (XRD), Secondary Ion Mass Spectroscopy (SIMS) of oxygen 16/oxygen 18 oxidized specimens will be performed by a qualified supplier through a purchase order.

Materials: Alloy C-22, Alloy 825

Specimen Specifications: ASTM G54-77 for determining total depth of attack for static oxidation testing.

Measurement Parameters: Temperature of oxidation treatment, weight change after oxidation and thermal cycling.

Required Level of Accuracy: Temperature ±3°C for oxidation studies, weight measurement ±.005 mg.

Uncertainty and Source of Error: Duplicated oxidation specimens will be tested for each oxidation conditions.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by K. T. Chiang	7/21/05

Notebook No. 713

Initial Scientific Notebook Entry for Corrosion Resistant Material Potentiostatic and Potentiodynamic Tests.

Title: Potentiaostatic tests, polarization tests, crevice repassivation tests, passive current density tests, critical pitting temperature tests, critical repassivation temperature tests.

Test Performed by: Kuang-Tsan Kenneth Chiang, Darrell S. Dunn, Brian K. Derby.

Objective: Study the effect of Bicarbonate [HCO_3^-] and Carbonate [CO_3^{2-}] to Chloride [Cl^-] concentration ratios on corrosion resistance of candidate nuclear waste package materials.

Equipment: EG&G Versastat Serial Number 20104. EG&G Model 352 corrosion software. NEC 586 computer. Keithley Electrometer Model 614 SN 55538 or equivalent. ASTM G-5 Polarization Cell. Large 2 L glass cells with Teflon tops. Electrochemical Impedance Spectroscopy system including Solaritron 1260 FRA and Solaritron 1287 Potentiostat. ESC 440 multichannel potentiostats with National Instruments Labview data acquisition software or Strawberry Tree data acqusition software. Calibration and due dates are provided in data sheets for each test.

Materials: Alloy C-22

Specimen Specifications: Cylindrical CPP specimens 1.195"x.250" and Crevice repassivation specimens with Teflon crevice washers attached to surface.

Measurement Parameters: Current and Potential as described in TOP-008. Temperature of solution ±2°C.

Required Level of Accuracy: Potentials ±5mV. Current ±0.1 microamp.

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

K. T. Chiang
7/21/05

Book 675

Revised Initial Entries:

Title: Microbially Influenced Corrosion of Container Materials

Names of the individuals performing the activity:
Lietai Yang, and Brian Derby, Roger Dykstra, Geri Becker and Stuart Birnbaum (All signatures are in the back of cover page of this book).

Objectives and the proposed approach or procedure:
Microbially influenced corrosion of alloys in solutions containing different microbes. Two tests will be conducted, one is in electrochemical cell and the other is the total immersion test in vials. In the electrochemical cell, both coupons and coupled multielectrode array sensor (MAS) probes will be used. In the total immersion test, only coupons will be used. The coupons will be taken out at different time intervals for examination of corrosion evidence.

Equipments and measurement parameters:
Coupling Currents from MAS probes will be measured with the Keithly 2182 nanovolt meter and Keithley 7001 Mainframe Switch. The potentials from electrodes will be measured with a Keithley 617 electrometer SN0679628. The pH will be measured with Orion EA940, SN 2330, Temperature will be measured with a non-contact thermometer (SN 2332580201-0007). Two colorimeters (SN: 46580 (on loan from UTSA) and DR/890 SN: 031090028183) will be used as a quick means to determine rough growth of bacteria. Calibration must be performed before use. Results from these meters will not be used in any report or papers. Only the results from the cultivation method will be used in the reports.

Software:
In-house developed visual basic code was used to control the two Keithley meters and to store the data in a computer hard drive. The visual basic code was verified (see page 45, Scientific Note Book # 423).

Test equipment calibration, accuracy, and precision requirements:
The Keithley meters (7001 and 2182) and the visual program were verified on a regular basis. The latest verification was performed on 8/26/2004. See page 294, Scientific Note Book # 604 for details.
Keithley 617 electrometer: SN0679628 Cal 09/13/04, Due 09/13/05;
Orion EA940 pH meter, SN 2330, Cal 721/04, Due 7/21/05. Calibration with proper standards is needed before use;
Non-contact thermometer SN 2332580201-0007 Cal 5/13/05, Due 5/13/06;
Two colorimeters (SN: 46580 (on loan from UTSA) and DR/890 SN: 031090028183):
Calibration before use is required.

Required level of accuracy:
Temperature: ±3 oC, pH: ±0.5, Potential ±5 mV; Currents: ±10 nA.

Potential Source of Uncertainty:
Temperature in room may change with climate. Heaters may be required in room during the winter season.

Special personnel training or qualification requirements:
None

Material/chemical:
Varies (see in-progress entries).

L. Yang
7/22/05

Book #533

Revised Initial Entries for Under-salt Corrosion Tests:

Title: Corrosion of Alloy 22 in Salt Environment at Elevated Temperatures

Names of the individuals performing the activity:
Lietai Yang, Steve Young
Objectives and the proposed approach or procedure:
Measurement of non-uniform corrosion rate of Alloy 22 in the liquid and vapor phases of a system containing mixed salts (NaCl-NaNO3-KNO3) at elevated temperatures. Coupling multielectrode array sensor (MAS) probes will be used in the test.
Equipments and measurement parameters:
The signal from the MAS probes will be measured with a Keithly 2182 nanovolt meter and Keithley 7001 Mainframe Switch. The salts will be placed in an electrochemical cell which will be heated by a temperature controller. The temperature will be measured with a calibrated thermocouple (SN 293376W) and a calibrated temperature meter (SN:T-94140)

Software:
In-house developed visual basic code was used to control the two Keithley meters and to store the data in a computer hard drive. The visual basic code was verified (see page 45, Scientific Note Book # 423).
Test equipment calibration, accuracy, and precision requirements:
The two Keithley meters and the visual program were verified on a regular basis. The latest verification was performed on 8/26/2004. See page 294, Scientific Note Book # 604 for details. This instrument has been used continuously for the microbially influenced corrosion test. The verification will be conducted as soon as the present test is completed and by 8/26/2005.
Thermocouple: SN293376W, Cal'd: Apr 05, 2005, Due: Apr 05, 2006
Temperature Meter: SN: T-94140, Cal'd: May 12, 2005, Due: Nov 11, 2005
Required level of accuracy:
Temperature: ±3 oC, Currents: ±10 nA.
Potential Source of Uncertainty:
Noise from power line or nearby equipment may affect readings from the probes. Pumps and heaters should not be close to the test cell.
Special personnel training or qualification requirements:
None
Material/chemical:
Probe will be made of Alloy 22 wire, solution annealed at 1121 oC for 10 minute followed by water quenching.
Special personnel training or qualification requirements:
None
Material/chemical:
Varies (see in-progress entries).

L. Yang
7/22/05