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CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES
QUALITY ASSURANCE
SURVEILLANCE REPORT

PROJECT NO.: 20-3602 | REPORT NO.: C-89-008 | PAGE 1 OF 3

SURVEILLANCE SCOPE: Peer Review Meeting for The Integrated Waste Package Experiments

REFERENCE DOCUMENTS: IWPE Project Plan, Agenda

STARTING DATE: 7/27/89 ENDING DATE: 7/27/89

QA REPRESENTATIVE: Bruce Mabrito

PERSONS CONDUCTING ~~TEST / EXAM~~ ACTIVITY: Dr. P. Nair, Dr. Bryan Wilde, Dr. R. Mason, Dr. H. Manaktala, Mr. Fred Lyle. Presented To: Dr. M. Streicher, Dr. S. Yukawa, Mr. O. Siebert.

SATISFACTORY FINDINGS: Good overview of The IWPE work by Dr. Nair by viewgraphs (Attached). Presentations by F. Lyle, R. Mason, B. Wilde with slides and viewgraphs with questions by Peer Reviewers. H. Manaktala presented Long Term material stability information. There was a detailed question and answer session between Peer Reviewers and IWPE staff following presentations. Dr. W. Murphy was called in to brief The reviewers on The potential geologic setting.

UNSATISFACTORY FINDINGS: None, Dr. Nair conducted an informative, productive and efficient Peer Review meeting within The stated requirements.

NONCONFORMANCE REPORT NO.: Not applicable

ATTACHMENTS: Meeting Agenda (page 2 of 2); Meeting Attendance (page 3 of 3)

RECOMMENDATIONS / ACTIONS

None.

APPROVED: [signature]
CENTER DIRECTOR OF QUALITY ASSURANCE

DATE: 7/27/89

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CNWRA FORM QAP-8

AGENDA

PEER REVIEW

INTEGRATED WASTE PACKAGE EXPERIMENTS PLAN

on

Thursday, July 27, 1989
at

CNWRA

San Antonio, Texas

8:00 a.m.	Introduction and Agenda Review	Nair
8:15 a.m.	IWPE Project Overview - Programatic Background - Technical Scope	Nair
9:00 a.m.	Corrosion Related Studies	Lyle
10:15 a.m.	Statistical Considerations	Mason
10:30 a.m.	Break	
10:45 a.m.	Hydrogen Diffusion Studies	Wilde
11:15 a.m.	Long-term Material Stability	Manaktala
11:45 a.m.	Lunch	
1:00 p.m.	IWPE Plan Discussion	Peer Rev. & IWPE Staff
2:00 p.m.	Peer Review Members' Executive Meeting	Peer Rev.
3:30 p.m.	Adjourn	

CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

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MEETING ATTENDANCE

SUBJECT OF MEETING: Peer Review for IWPE Plan

DATE: 7-27-89 LOCATION: CNWRA - San Antonio - Conf. Rm #1

PERSON	ORGANIZATION	TITLE/FUNCTION	TELEPHONE NUMBER
M. A. STREICHER	CONSULTANT		(302) 478-7725
O. W. SIEBERT	CONSULTANT		(314) 227-3583
S. Yukawa	Consultant		(303) 530-2969
B. F. WILDE	CONTRACTOR		614-292-6255
J. R. Weeks	Consultant to NRC. BNL.	Sr. Metallurgist	516-282-2617
Mel Silberberg	NRC	Chief, Wast. Mgt/RES	(301)-492-3810
Paul Shewmon	Ohio State U.	NRC/ACNW consultant	614-292-5864
ROBERT L. MASON	SWRI	STATISTICIAN	512-522-2671
HERSH MANAKTALA	CNWRA	SRE	522-5210
RICK WELLER	NRC	LEADER, MATERIALS SECTION	301-492-3458
BRUCE MABRITO	CNWRA	Quality Assurance	512/522-5149
PRASAD K. NAIR	CNWRA	MANAGER, EBS	512-522-5150
FRED F. LYLE, JR	CNWRA/SwRI	STAFF CORROSION ENGR	512-522-2508

PEER REVIEW MEETING

INTEGRATED WASTE PACKAGE EXPERIMENTS PROJECT PLAN

JULY 27, 1989

CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

Members:
- Mr. Oliver W. Siebert
- Dr. Michael A. Streicher
- Dr. Sumio Yukawa

Presentations by:
- Dr. Prasad K. Nair
- Mr. Fred F. Lyle, Jr.
- Dr. Bryan E. Wilde
- Dr. Robert L. Mason
- Dr. Hersh K. Manaktala

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PURPOSE OF PEER REVIEW

GENERAL

- Independent Assessment of IWPE Project Plan
  - Project Direction
  - Technical Approach
  - Reasonableness and Adequacy
- Provide Guidance for Plan Improvements

SPECIFIC

- Review Use of Reference Materials in the Test Matrix
- Review the Statistical Approach Taken for Testing

IWPE PROJECT OVERVIEW PRESENTATION OUTLINE

PROGRAMMATIC BACKGROUND

- Regulatory Framework
- Implications of Regulations to Waste Package Performance
- Integrated Waste Package Experiment Project Approach
  - Uncertainty Reduction Concepts
  - Controlled Test Environments
  - Stepwise Testing Strategy
  - Baseline Evaluations
  - Reference Material – Hastelloy C-22

TECHNICAL SCOPE

- Specific Objectives
- Technical Program
- Technical Approach

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REGULATORY FRAMEWORK

10CFR60.113(a)(ii)

Containment of HLW within the waste packages will be substantially complete for a period to be determined by the Commission taking into account the factors specified in 60.113(b), provided that such period shall be not less than 300 years nor more than 1000 years after permanent closure of the geologic repository;

10CFR60.21(c)(1)(ii)(D)

. . . The analysis shall also include a comparative evaluation of alternatives to the major design features that are important to waste isolation, with particular attention to the alternatives that would provide longer radionuclide containment and isolation

IMPLICATIONS OF THE REGULATIONS

10CFR60.113(a)(ii) Related

- Technical Interpretation of "Substantially Complete" Containment
  - Waste Package Material/Design Evaluation
    - Mechanisms of Degradation (Qualitative)
    - Performance Assessment (Quantitative)
  - Technical Criteria to be Met by DOE
    - Guidance Requirements
- Compliance Determination Strategy for Containment
  - Containment Period Rationale (300-1000 yrs)
    - Technical
    - Regulatory
  - Criteria to be Met by DOE

IMPLICATIONS OF THE REGULATIONS (CONT'D)

<u>10CFR60.21(c)(1)(ii)(D) Related</u>

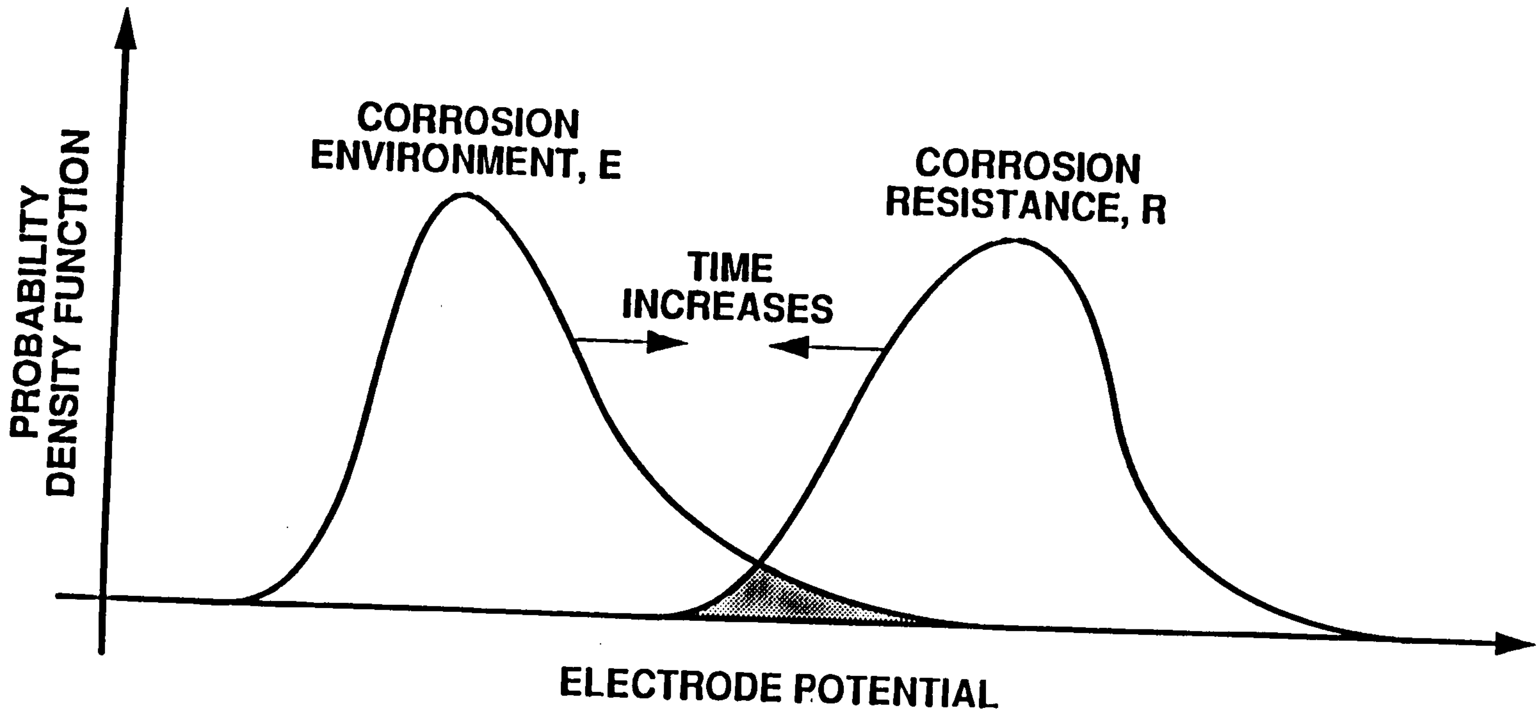
- Comparative Evaluation of Alternatives
  - Technical Basis of Comparison
  - Standards of Comparison (Reference Material/Design)
- Alternatives Requiring "Longer Radionuclide Containment"
  - A Technical Requirement Incorporating Material Selection and Design of Waste Packages.
  - Requires a Technical Approach to Bound Alternatives

INTEGRATED WASTE PACKAGE EXPERIMENTS

- Regulatory Basis
  - 10CFR60.113
  - 10CFR60.21(c)(1)(ii)(D)
- Uncertainty Reduction Concepts
- Controlled Test Environments
- Stepwise Testing Strategy
- Baseline Evaluations

PROBABILISTIC CORROSION PERFORMANCE ASSESSMENT

R = R (MATERIAL CHARACTERISTICS)
E = E (PH, TEMPERATURE, CHLORINE CONCENTRATION, . . .)
R, E ARE RANDOM VARIABLES



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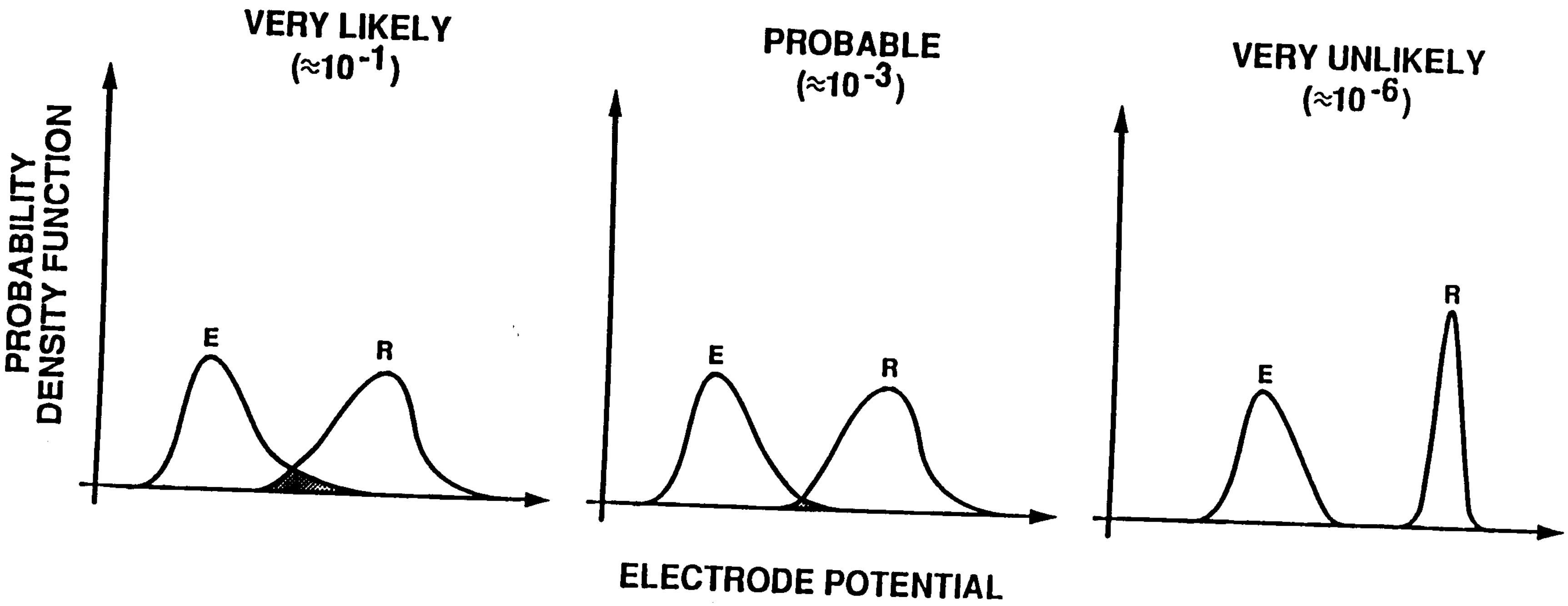
EXAMPLES OF PROBABILISTIC PERFORMANCE ASSESSMENT



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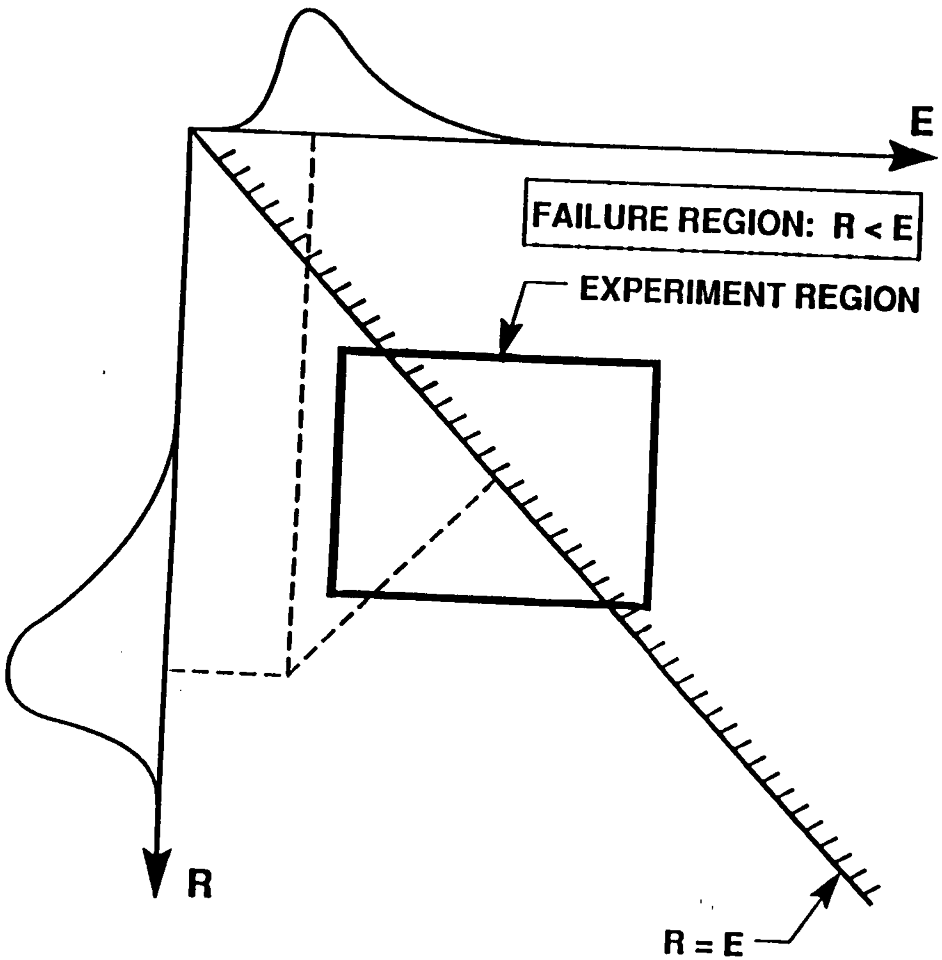
SELECTION OF EXPERIMENTAL REGION TO SUPPORT PERFORMANCE ASSESSMENT



APPROACHES:

- EXPERIMENTS CONCENTRATE AT CRITICAL REGIONS
- RESULTS DIRECTLY CORRELATE WITH PROBABILITY OF FAILURE
- IDENTIFY CRITICAL PARAMETERS

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CONTROLLED TEST ENVIRONMENTS

Constituents (Molal)	Field		EQ3/EQ6 Calculated				
	Yucca Mountain Vicinity	J_{13}	EQ3 25°C	EQ3 70°C	EQ3 95°C	EQ3 25°C Magnetite	EQ6 25°C Fe
Na^+	6.1×10^{-4} to 1.4×10^{-2}	2.0×10^{-3}	2.0×10^{-3}	2.0×10^{-3}	2.0×10^{-3}	2.0×10^{-3}	2.0×10^{-3}
Cl^-	2.0×10^{-5} to 3.2×10^{-3}	1.8×10^{-4}	2.0×10^{-4}	2.0×10^{-4}	2.0×10^{-4}	2.0×10^{-4}	2.0×10^{-4}
HCO_3^-		2.7×10^{-3}	1.7×10^{-3}	1.5×10^{-3}	1.3×10^{-3}	1.7×10^{-3}	1.7×10^{-3}
fCO_2	$10^{-3.5}$ – $10^{-0.8}$	$10^{-1.8}$	$10^{-3.5}$	$10^{-3.5}$	$10^{-3.5}$	$10^{-3.5}$	$10^{-3.5}$
fO_2		Oxidizing	0.2 (bar)	0.2 (bar)	0.2 (bar)	0.2 (bar)	0.2 (bar)
pH	6.6 to 9.1	6.9	8.5	8.8	8.9	8.5	8.5

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STEPWISE TESTING STRATEGY

- Scoping Tests
  - Literature Assessment
  - Other NRC/DOE Programs
  - Select Tests
- Short Term
  - Uncertainty Reduction Need Based
  - Baseline Tests
  - Performance Assessment and Statistically Valid Tests
- Long Term
  - Performance Confirmatory Tests

BASELINE EVALUATIONS

- Baseline Material Selection Criteria
  - Represents Similar Metallurgical Characteristics
  - Predictable Behavior in Environments of Interest
  - Stable Performance in a Range of Environments
  - Quantification of Performance should be Possible
- Develop Data/Confidence in Baseline Material
  - Assist in Reducing Future Testing Needs of Similar Class of Materials
- Provide a Relative Upper Bound on Degradation Processes
  - Accommodate Changes in DOE Programs
  - Assist in Reviewing Alternatives for "Longer Radionuclide Containment"

HASTELLOY C-22 BACKGROUND

- Austenitic Based Material (High Ni, Cr, Mo)
- Superior Corrosion Resistance
- Resists Sensitization (Low Carbon)
- Applications in:
  - Reducing Environment
  - Oxidizing Environment
- Industries
  - Chemical
  - Oil-Drilling
  - Paper

RESULTS IN SEVERAL SOLUTIONS WITH A "WIDE" CREVICE (72-H EXPOSURES)

TEST	AVERAGE PENETRATION IN ATTACKED AREA, mmpy			
	TYPE 316	ALLOY 825	ALLOY 625	HASTELLOY ALLOY C
5% $FeCl_3$, 50 C	410	200	0.04	0.02
5% $CuCl_2$, 50 C	23, 950	200	0.003	0.01
5% NaOCl, 50 C	73	46	0.003	0.006
5% $FeCl_3$, RT	320	32	0.002	0.02

WHY HASTELLOY C-22?

Technical

- Demonstrated Corrosion Resistance in Aggressive Environments
- Low Sensitization Material
- Stable Performance for a Range of Parameters
- Provides an Upper Bound on Performance

WHY HASTELLOY C-22? (CONT'D)

Programmatic

- Reduces NRC Schedule Risks for Development of Materials Evaluation Data on Individual Materials in Light of Uncertainty in DOE's Current Program.
- Provides Early Guidance to DOE on Waste Package Performance Issues
- Supports a Proactive Research Approach Consistent with Requirements of 10CFR60.21(c)(1)(ii)(D)
- Provides a Firm Foundation for Developing Rational Quantitative Estimates of Waste Package Material Performance
- Will Provide Long-Term Cost Savings in Testing by Limiting Data Information Needs
- DOE is Expected to Include Hastelloy C-22 in an "Alternate Materials Program" for Waste Packages.

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SPECIFIC OBJECTIVES

- TO ASSESS THE STATE OF KNOWLEDGE FOR CORROSION AND OTHER POTENTIAL WASTE PACKAGE MATERIALS DEGRADATION PROCESSES IN THE YUCCA MOUNTAIN PROJECT (YMP) TUFF ENVIRONMENT AND THE METHODOLOGIES USED TO PREDICT LONG-TERM MATERIALS PERFORMANCE

- TO CONDUCT EXPERIMENTAL PROGRAMS TO IDENTIFY AND UNDERSTAND KEY FACTORS AFFECTING LONG-TERM MATERIALS PERFORMANCE

- TO ASSESS EXPERIMENTALLY YMP SELECTED MATERIALS AND DESIGNS AND PROVIDE INDEPENDENT EVALUATION TO ASSURE LONG-TERM PERFORMANCE

- TO FACILITATE TECHNICAL INTEGRATION SUPPORT IN THE AREA OF WASTE PACKAGE PERFORMANCE

TECHNICAL PROGRAM

TESTS CATEGORIES

CONFIRMATORY

- CONDUCTED TO DETERMINE REPRODUCIBILITY AND ADEQUACY OF EXPERIMENTAL RESULTS REPORTED BY YMP AND OTHERS

EXPLORATORY

- CONDUCTED TO PROVIDE ADDITIONAL INFORMATION IN AREAS PREVIOUSLY INVESTIGATED BY YMP, e.g., CORROSION TESTS FOR LONGER PERIODS OF TIME THAN THOSE REPORTED BY YMP TO PROVIDE INPUT DATA FOR KINETIC MODELS

- CONDUCTED TO PROVIDE DATA IN AREAS WHICH THE CNWRA PROJECT TEAM BELIEVES ARE NEEDED FOR THE PROPER PERFORMANCE OF NRC'S LICENSING FUNCTION BUT WHICH HAVE NOT BEEN INVESTIGATED BY YMP, e.g., THE EFFECTS OF HYDROGEN ON CONTAINER MATERIALS

(CONTINUED)

TECHNICAL PROGRAM (CONTINUED)

TEST MATERIALS

METALLIC ALLOYS PROPOSED IN YMP SITE CHARACTERIZATION PLAN (SCP)

- TYPE 304L STAINLESS STEEL (REFERENCE ALLOY)
- TYPE 316L STAINLESS STEEL
- INCOLOY 825
- COPPER ALLOY CDA 102 (OXYGEN-FREE, HIGH-CONDUCTIVITY COPPER)
- COPPER ALLOY CDA 613 (7-8 % ALUMINUM BRONZE)
- COPPER ALLOY 715 (70% COPPER-30% NICKEL)

ADDITIONAL CNWRA REFERENCE MATERIAL

- HASTELLOY C-22

TECHNICAL PROGRAM (CONTINUED)

ALTERNATIVE MATERIALS PROPOSED BY YMP

- ALTERNATIVE MATERIALS OR COMBINATIONS OF MATERIALS HAVE NOT BEEN IDENTIFIED BY YMP, ALTHOUGH AN ALTERNATIVE MATERIALS PROGRAM IS IDENTIFIED IN THE SCP

  IF PURSUED, THIS PROGRAM IS EXPECTED TO INCLUDE:

  - MORE HIGHLY CORROSION RESISTANT MATERIALS THAN THE SIX ALLOYS CURRENTLY PROPOSED
  - BIMETALLIC CONTAINERS (e.g., CORROSION-RESISTANT ALLOY CLADDING OR WELD OVERLAY ON CARBON STEEL OR OTHER LESS CORROSION-RESISTANT STRUCTURAL ALLOY
  - CERAMIC CONTAINERS, e.g. Al_2O_3; AND/OR
  - MULTI-LAYER COMPOSITE CERAMIC AND, METALLIC CONTAINERS, e.g., A CERAMIC CONTAINER PLACED INSIDE A CORROSION-RESISTANT METALLIC CONTAINER

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TECHNICAL APPROACH

ASSESS STATE OF KNOWLEDGE

- DEVELOP INFORMATION/DATA BASE -- YMP REPORTS; NRC REPORTS AND ONGOING WORK OF OTHER NRC CONTRACTORS; OPEN LITERATURE; OTHER COUNTRIES; AND CNWRA EXPERIENCE
- EVALUATE TECHNOLOGY WITH RESPECT TO YMP CURRENT WASTE PACKAGE PLANS

MAJOR TOPICAL AREAS

- DEFINITION OF REPOSITORY ENVIRONMENTS CONTAINERS ARE EXPECTED TO ENCOUNTER IN FIRST 1,000 YEARS -- INCLUDING LIQUID AND VAPOR PHASES AND GAMMA RADIATION
- CORROSION OF CONTAINER MATERIALS IN REPOSITORY ENVIRONMENTS -- UNIFORM CORROSION, PITTING, CREVICE CORROSION, STRESS CORROSION CRACKING (SCC), DEALLOYING, GALVANIC CORROSION, AND INTERGRANULAR CORROSION
- METALLURGICAL STABILITY -- e.g., LOW-TEMPERATURE SENSITIZATION (LTS) AND OTHER TIME-TEMPERATURE-DEPENDENT METALLURGICAL CHANGES
- OTHER FAILURE MODES -- e.g., HYDROGEN ATTACK, MICROBIOLOGICAL ACTION, AND FAILURE OF CONTAINER CLOSURES

(CONTINUED)

TECHNICAL APPROACH (CONTINUED)

EXPERIMENTAL PROGRAMS

OBJECTIVES

- DETERMINE FORMS OF CORROSION AND OTHER TYPES OF MATERIALS DEGRADATION THAT CAN OCCUR UNDER POSSIBLE REPOSITORY ENVIRONMENTAL CONDITIONS

- DEVELOP KINETICS DATA FOR CORROSION AND OTHER DEGRADATION MECHANISMS THAT CAN OCCUR IN THE YMP REPOSITORY

- IDENTIFY AND EVALUATE EFFECTS OF METALLURGICAL CHANGES THAT CAN OCCUR AS A RESULT OF FABRICATION HISTORY, THERMAL HISTORY, STRESS AND STRAIN, EXPOSURE TIME, AND ENVIRONMENTAL EXPOSURE

- DEVELOP PREDICTIVE MODELS

(CONTINUED)

TECHNICAL APPROACH (CONTINUED)

EXPERIMENTAL PROGRAMS (CONTINUED)

PROGRAM STRUCTURE

- SCOPING AND SCREENING TESTS
  - ELECTROCHEMICAL CHARACTERIZATION OF MATERIALS IN REPOSITORY ENVIRONMENTS, INCLUDING EFFECTS OF GAMMA RADIATION
  - SLOW-STRAIN-RATE SCC TESTS
  - OTHER TYPES OF TESTS, AS NECESSARY
- SHORT-TERM TESTS (3 TO 12 MONTHS)
- LONG-TERM TESTS (12 MONTHS TO 3 YEARS OR LONGER
- DEVELOP PREDICTIVE MODELS THROUGH DATA ANALYSES
- STUDY HYDROGEN EFFECT
- STUDY WELDING (OR OTHER CLOSURE) EFFECTS
- EVALUATE METALLURGICAL STABILITY OF MATERIALS

(CONTINUED)

TECHNICAL APPROACH (CONTINUED)

ASSESS YMP RECOMMENDED WASTE PACKAGE

- EVALUATE ADEQUACY OF CORROSION AND METALLURGICAL STABILITY MODELING
- PERFORM SMALL-SCALE CONFIRMATORY TESTING
- EVALUATE NEED FOR LARGE-SCALE TESTS AND DEFINE TESTS, IF NEEDED

PROVIDE GENERAL SUPPORT AND COORDINATION

- COORDINATE CNWRA PROGRAM WITH OTHER ONGOING NRC-SPONSORED WASTE PACKAGE RESEARCH PROGRAMS
- PREPARE TECHNICAL REPORTS AND PUBLICATIONS

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CORRORSION-RELATED STUDIES

PURPOSE:

- Identify environmental conditions that promote the corrosion and materials degradation process of candidate alloys

APPROACH:

- Statistical experimental design approach to accommodate a large number of environmental variables

STATISTICAL DESIGN PROCEDURES

PURPOSE:

- Determine the individual chemical species or interactions between them that are associated with the various processes

APPROACH:

- Run test matrix to determine how to minimize experimental test variation
- Apply sequential experimental design technique
- Analyze resultant data

WHY DESIGN EXPERIMENTS?

- Saves time and money
  - Often can learn more with fewer observations and less cost
  - Yields more precise results
- Helps locate important factors
  - Can measure influence of several factors simultaneously on a response
  - Can assess impact of joint factor effects (e.g., interactions)
- Aids in modelling and prediction
  - Can fit response surfaces and find optimum points
  - Can predict future responses with high accuracy
- Guards against unknown biases
  - Uses randomization
- Simplifies data analysis and reporting of results

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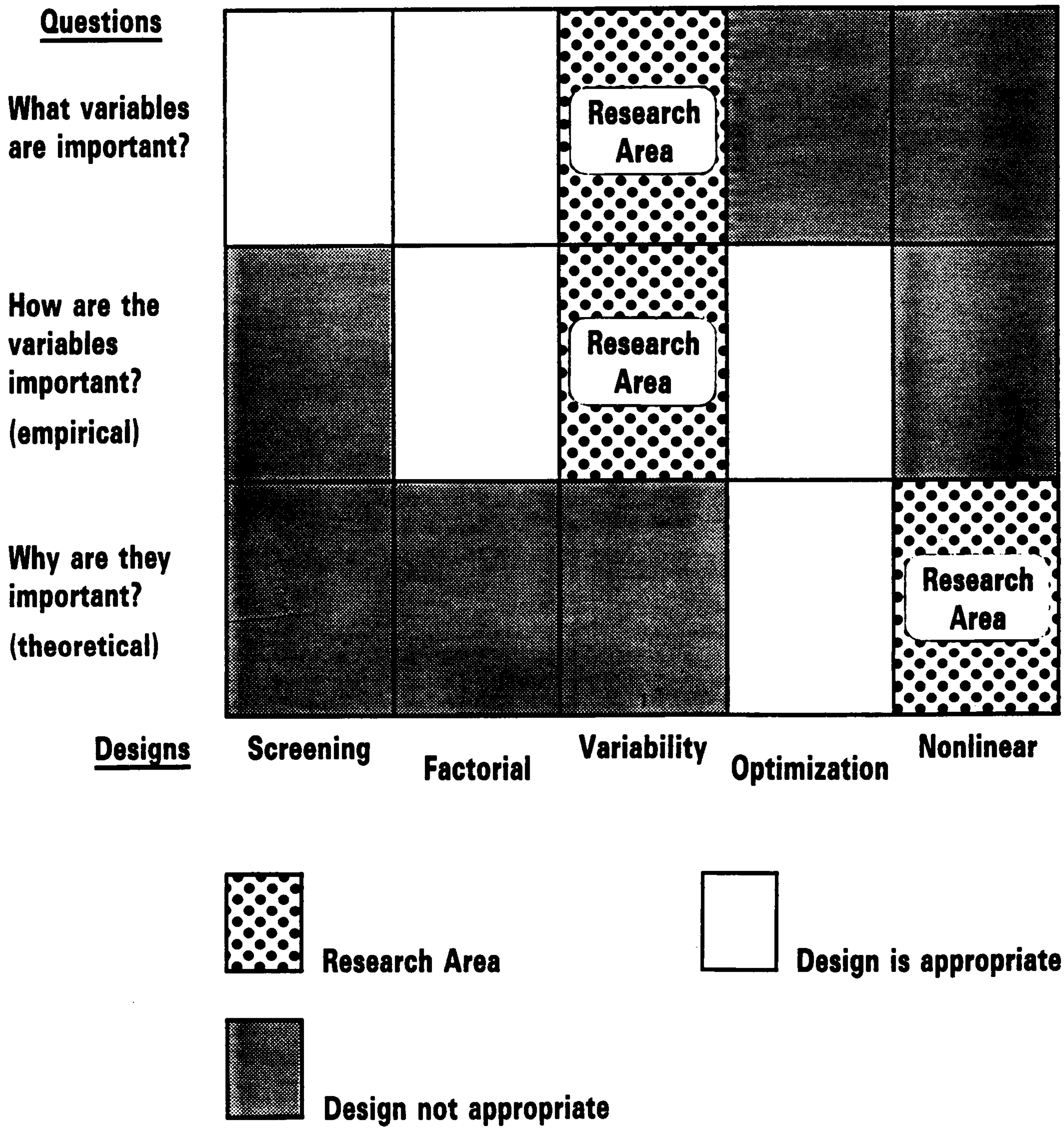
COMMON EXPERIMENTAL PROBLEMS

- Variation in experiment is so large it masks factor effects
- Experimental conclusions are invalidated because key factors are not considered
- Factor effects cannot be measured or experimental units are wasted because improper design concepts are utilized
- Technical objectives may not be achieved with "one-factor-at-a-time" designs


Questions
What variables are important?
How are the variables important? (empirical)
Why are they important? (theoretical)
Research Area
Research Area
Research Area
Designs
Screening
Factorial
Variability
Optimization
Nonlinear
Research Area
Design is appropriate
Design not appropriate

MINIMIZATION OF TEST VARIATION

IDENTIFY POTENTIAL SOURCES OF VARIATION

- Materials
- Test Procedure
- Environmental

DESIGN FACTORIAL EXPERIMENT

- High and low level for each factor
- Five factors with replication
- 35 tests

ANALYZE RESULTS

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TWO-LEVEL FACTORIAL EXPERIMENT DESIGN IN FIVE VARIABLES TO DEFINE VARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS

		A^+				A^-			
		B^+		B^-		B^+		B^-	
		C^+	C^-	C^+	C^-	C^+	C^-	C^+	C^-
E^+	D^+								
	D^-								
E^-	D^+								
	D^-								

CODE	VARIABLE	+ CONDITION	- CONDITION
A	SCAN RATE	3.6 V/hr	0.6 V/hr
B	SOLUTION PREPARATION	6.5 PPM Cl^-	32.5 PPM Cl^-
C	ELECTRODE-GASKET	IMMERSED	ABOVE LIQUID
D	MAXIMUM CURRENT	5,000 µA	2,250 µA
E	AUX. ELECTRODE	IN TEST CELL	IN SEPARATE COMPARTMENT

REPLICATIONS -- 3 OF 1 TEST COMBINATION, RANDOMLY SELECTED

TOTAL TESTS -- 35

SEQUENTIAL EXPERIMENTAL DESIGN

TEST PROCEDURE

- Minimize variation

SCREENING DESIGN

- Two-level (high and low) design
- Highly fractionated factorial
- Only concern is main effects
- Reduce large number of variables to more manageable group

DETAILED EXPERIMENT

- May be two- or three-level design
- Less fractionated than screening design
- Concerned with both main effects and interactions
- Complements screening design
- Can repeat as necessary

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SCREENING DESIGNS

- Large number of factors available
- Often seeking identification of critical factors, or desiring to control for external sources of variability
- Interactions are nonexistent or negligible relative to main effects
- Often preliminary to expanded design for critical factors
- Use relatively few experimental tests and highly fractionated
- Less fractionated designs preferred if interactions are not negligible

DESIGN RESOLUTION

- Goal is to construct fractional factorial experiments in which high-order interactions are confounded with other high-order interactions

- Designs where main effects and low-order interactions are not confounded with each other are called high-resolution designs

- Computer design packages produce designs of the highest possible resolution

HIGHLY FRACTIONATED DESIGNS

- At screening stage, large portion of experimental variation may be due to small proportion of factors
- Highly fractionated designs are a reasonable approach to locate the important effects
- These designs avoid expense of replication at each design point
- Useful in identifying both main effects as well as dispersion effects

ANALYTIC APPROACH

IDENTIFY RESPONSE VARIABLE

- E_{cor} - Corrosion Potential
- E_{pit} - Pitting Potential
- E_{rp} - Repassivation Potential

ANALYZE DATA

- Regression analysis and ANOVA techniques
- Express each response variable in terms of environmental variables
- Use actual values as well as target values

ANALYSIS

- Experimental factors may be either qualitative or quantitative
- Analysis-of-variance (ANOVA) techniques are used with both types provided one has used a proper experimental design
- Computations often are facilitated by making use of the fact that ANOVA models are special cases of multiple linear regression models
- When data are not collected in a designed experiment, multiple regression analysis is the recommended approach
- When the factors are all quantitative, regression techniques are usually preferred

CRITIQUE OF APPROACH

ADVANTAGES

- Can determine influence of test procedures on test results
- Use is made of small screening experiments to identify major variables
- Detailed experimentation limited to use only when necessary for identification of interaction

DISADVANTAGES

- Results are limited to range of conditions used in test procedures
- Unknown how lab tests will relate to real-world environment
- Statistical conclusions are only as accurate as the collected data

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The Hydrogen Absorption and Potential Embrittlement of Candidate High Level Nuclear Waste Package Containers in the Proposed Tuff Repository Environment

Prepared for Peer Review at SwRI on July 27, 1989

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The Hydrogen absorption and potential embrittlement of candidate High Level Nuclear Waste Package containers in the proposed Tuff repository environment.

SwRI Project No.20020698.

B.E.Wilde,Principal Investigator.

Prepared for Peer Review at SwRI on 27,July,1989.

Anticipated repository conditions 0-300years

* Influx of ground water of general chemical composition similar to that of J-13 well water.
* Evaporative drying/scaling of canister wall because radioactive decay heat maintains outer wall temperature >J-13 boiling point at atmospheric pressure.
* Canister wall becomes covered with solid deposit containing known corrosives at temperatures above the dew point.
* Gamma radiolysis of steam produces transient species including atomic hydrogen on the canister wall.

Anticipated repository conditions 300-1000years.

* As decay heat decreases, wall temperatures fall below the boiling point and condensation occurs.
* At scale/wall interface a saturated solution will be formed.
* Gamma radiolysis of condensed water also produces atomic hydrogen on metal surface.

Situations leading to hydrogen embrittlement.

* The occlusion of hydrogen into a material either pre- or post stressed is a necessary precursor for HE.

* Common sources of hydrogen:

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Metal production.

Hot metal + H_2O --- H_{abs}.

Hydrogen gas storage.

Metal + High Pressure H_2 --- H_{abs}.

Electroplating.

M^{n+} + ne --- nM^o.

H^+ + e --- H_{ads}.

H_{ads} --- H_{abs}.

Cathodic Protection.

H^+ + e --- H_{ads}.

H_{ads} --- H_{abs}.

Corrosion.

M^{n+} + ne --- nM

H^+ + e --- H_{ads}.

H_{ads} --- H_{abs}.

Potential new situation leading to HE. in Waste Disposal Canisters.

* Gamma radiolysis of steam/water at metal surface.

$$\gamma \text{ ray} + H_2O \text{ ---- } H_{ads}.$$

$$H_{ads} \text{ ---- } \underline{H_{abs}}.$$

NOTE: NO CORROSION IS NECESSARY FOR THE GENERATION OF THE CONDITIONS NECESSARY FOR HE.

ALSO THE PRESENCE OF AN UNCONTROLLED AMOUNT OF STRESS AT THE THICK SECTION WELD.

Program structure.

* **Phase 1:**

Modification of ambient temperature and pressure electrochemical hydrogen permeation procedures to allow use under simulated repository conditions.

* **Phase 2:**

QA certification of procedures developed in phase 1.Use procedures to evaluate any hydrogen absorption produced by cathodic polarization of candidate materials as a function of a)temperature.b)environment composition,c)state of surface stress,d)alloy composition and e)microstructure eg.base metal versus weld metal.

* **Phase 3:**

Use new procedure to evaluate hydrogen absorption at temperature under the influence of gamma radiation rather than cathodically polarization.

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PERMEATION CURVE FOR 1018 STEEL IN
1 N NA3PO4 SOLN. @ 93±2 C

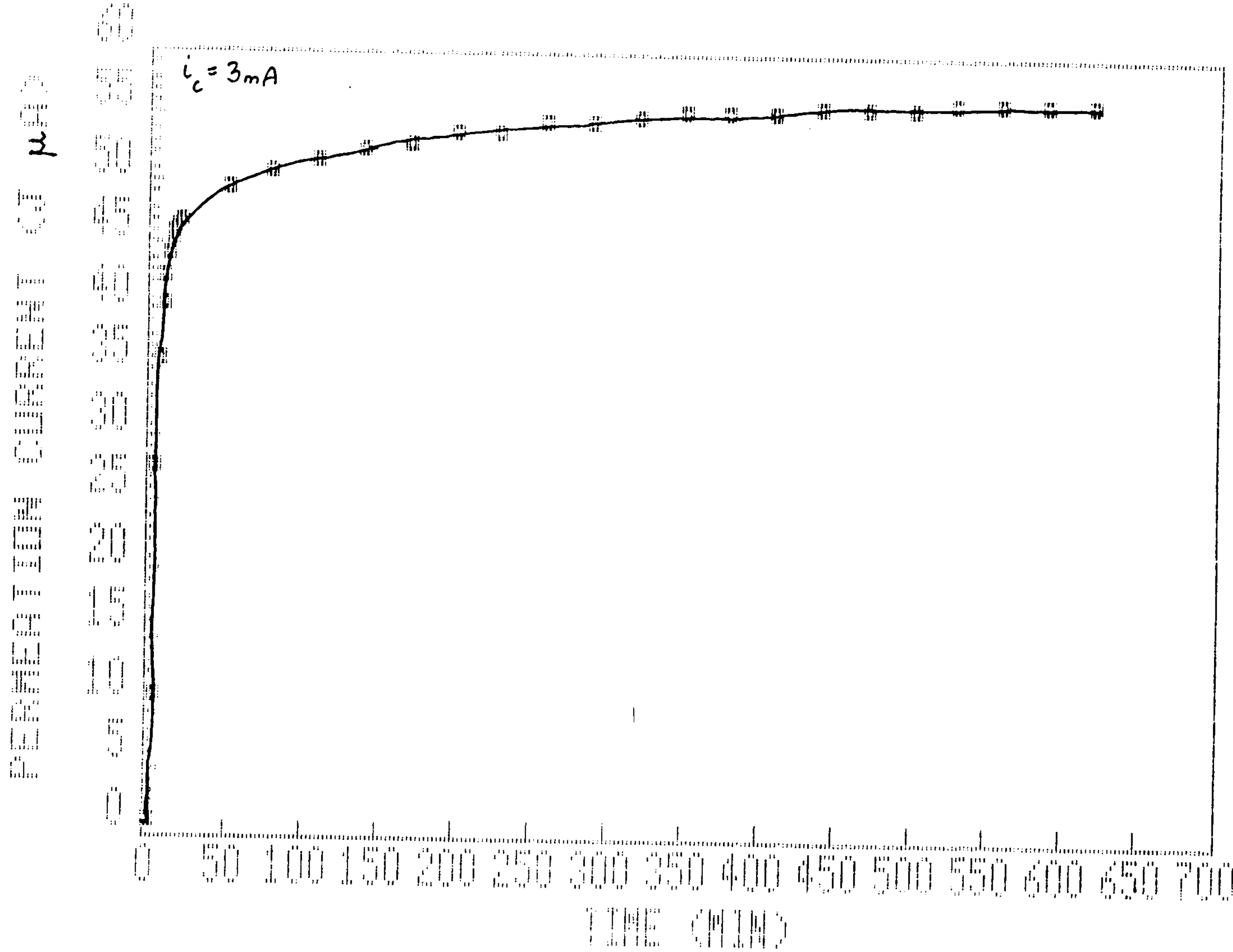

FIGURE 3. HYDROGEN PERMEATION TRANSIENT AT 93 °C.

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Program schedule.

* Phase 1:

Method to be developed and tested on each of the six candidate materials by 30,September,1989.

CURRENT STATUS: PROCEDURES HAVE BEEN DEVELOPED FOR PURE COPPER,90%Cu/10%Ni,INCOLLOY 825,TYPE 316L STAINLESS STEEL,TYPE 304 L STAINLESS STEEL and HASTELLOY C-22. PHASE 1 WILL BE COMPLETED ON SCHEDULE.

* Phase 2:

To be initiated after QA approval of procedures developed in Phase 1.When initiated,Phase 2 will also include in addition to the alloy test matrix, further modification of phase 1 procedures to allow use on hollow tensile specimen to evaluate the effects of stress.

* Phase 3:

Will be initiated toward the third quarter of 1990.

Progress in the first year - September 1988 to date.

* Major task; to develop a surface treatment for the oxidation side of the membrane to resist corrosion at elevated temperatures whilst maintaining sensitivity to hydrogen detection eg at least 1×10^{-12} moles $/cm^2/sec$. 94/155

  eg. $\sum i_{ox} = i_{oxH} + i_{oxM}$.

  max.sensitivity when $i_{oxM} \lll i_{oxH}$.

  STATUS: EFFORTS TO DEVELOP A PORE FREE PALLADIUM PLATE INCLUDED i)ELECTROPLATE,ii)ELECTROLESS PLATE AND SPUTTER DEPOSITION.ALL WERE SUSCEPTIBLE TO GALVANIC CORROSION OF THE SURFACE WHICH MASKED THE HYDROGEN PERMEATION CURRENT.

* Successful treatments were developed using simple corrosion inhibitors rather than noble metal plating.The following have been found effective:

  COPPER and COPPER ALLOYS ----BENZOTRIAZOLE.

  STAINLESS STEELS ------- TRISODIUM PHOSPHATE.

  NICKEL ALLOYS -------- SODIUM SULFATE.

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CORROSION-RELATED STUDIES

PURPOSE:

- Identify environmental conditions that promote the corrosion and materials degradation process of candidate alloys

APPROACH:

- Statistical experimental design approach to accommodate a large number of environmental variables

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STATISTICAL DESIGN PROCEDURES

PURPOSE:

- Determine the individual chemical species or interactions between them that are associated with the various processes

APPROACH:

- Run test matrix to determine how to minimize experimental test variation
- Apply sequential experimental design technique
- Analyze resultant data

WHY DESIGN EXPERIMENTS?

- Saves time and money
  - Often can learn more with fewer observations and less cost
  - Yields more precise results
- Helps locate important factors
  - Can measure influence of several factors simultaneously on a response
  - Can assess impact of joint factor effects (e.g., interactions)
- Aids in modelling and prediction
  - Can fit response surfaces and find optimum points
  - Can predict future responses with high accuracy
- Guards against unknown biases
  - Uses randomization
- Simplifies data analysis and reporting of results

COMMON EXPERIMENTAL PROBLEMS

- Variation in experiment is so large it masks factor effects
- Experimental conclusions are invalidated because key factors are not considered
- Factor effects cannot be measured or experimental units are wasted because improper design concepts are utilized
- Technical objectives may not be achieved with "one-factor-at-a-time" designs

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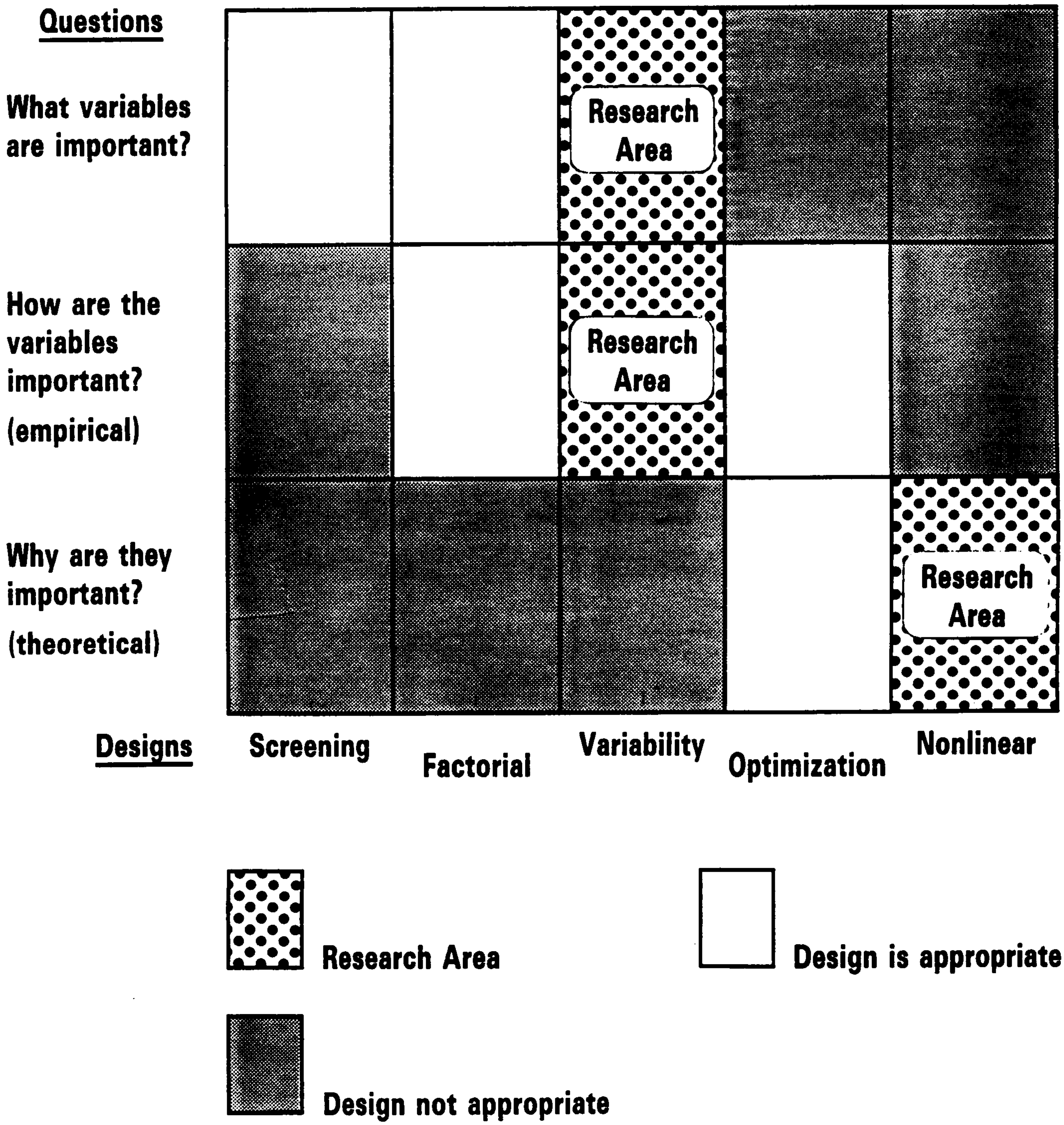

Questions
What variables are important?
How are the variables important? (empirical)
Why are they important? (theoretical)
Research Area
Research Area
Research Area
Designs
Screening
Factorial
Variability
Optimization
Nonlinear
Research Area
Design is appropriate
Design not appropriate

MINIMIZATION OF TEST VARIATION

IDENTIFY POTENTIAL SOURCES OF VARIATION

- Materials
- Test Procedure
- Environmental

DESIGN FACTORIAL EXPERIMENT

- High and low level for each factor
- Five factors with replication
- 35 tests

ANALYZE RESULTS

TWO-LEVEL FACTORIAL EXPERIMENT DESIGN IN FIVE VARIABLES TO DEFINE VARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS

		A^+				A^-			
		B^+		B^-		B^+		B^-	
		C^+	C^-	C^+	C^-	C^+	C^-	C^+	C^-
E^+	D^+								
	D^-								
E^-	D^+								
	D^-								

CODE	VARIABLE	+ CONDITION	- CONDITION
A	SCAN RATE	3.6 V/hr	0.6 V/hr
B	SOLUTION PREPARATION	6.5 PPM Cl^-	32.5 PPM Cl^-
C	ELECTRODE-GASKET	IMMERSED	ABOVE LIQUID
D	MAXIMUM CURRENT	5,000 μA	2,250 μA
E	AUX. ELECTRODE	IN TEST CELL	IN SEPARATE COMPARTMENT

REPLICATIONS -- 3 OF 1 TEST COMBINATION, RANDOMLY SELECTED

TOTAL TESTS -- 35

SEQUENTIAL EXPERIMENTAL DESIGN

TEST PROCEDURE

- Minimize variation

SCREENING DESIGN

- Two-level (high and low) design
- Highly fractionated factorial
- Only concern is main effects
- Reduce large number of variables to more manageable group

DETAILED EXPERIMENT

- May be two- or three-level design
- Less fractionated than screening design
- Concerned with both main effects and interactions
- Complements screening design
- Can repeat as necessary

SCREENING DESIGNS

- Large number of factors available
- Often seeking identification of critical factors, or desiring to control for external sources of variability
- Interactions are nonexistent or negligible relative to main effects
- Often preliminary to expanded design for critical factors
- Use relatively few experimental tests and highly fractionated
- Less fractionated designs preferred if interactions are not negligible

DESIGN RESOLUTION

- Goal is to construct fractional factorial experiments in which high-order interactions are confounded with other high-order interactions
- Designs where main effects and low-order interactions are not confounded with each other are called high-resolution designs
- Computer design packages produce designs of the highest possible resolution

HIGHLY FRACTIONATED DESIGNS

- At screening stage, large portion of experimental variation may be due to small proportion of factors
- Highly fractionated designs are a reasonable approach to locate the important effects
- These designs avoid expense of replication at each design point
- Useful in identifying both main effects as well as dispersion effects

ANALYTIC APPROACH

IDENTIFY RESPONSE VARIABLE

- E_{cor} - Corrosion Potential
- E_{pit} - Pitting Potential
- E_{rp} - Repassivation Potential

ANALYZE DATA

- Regression analysis and ANOVA techniques
- Express each response variable in terms of environmental variables
- Use actual values as well as target values

ANALYSIS

- Experimental factors may be either qualitative or quantitative
- Analysis-of-variance (ANOVA) techniques are used with both types provided one has used a proper experimental design
- Computations often are facilitated by making use of the fact that ANOVA models are special cases of multiple linear regression models
- When data are not collected in a designed experiment, multiple regression analysis is the recommended approach
- When the factors are all quantitative, regression techniques are usually preferred

CRITIQUE OF APPROACH

ADVANTAGES

- Can determine influence of test procedures on test results
- Use is made of small screening experiments to identify major variables
- Detailed experimentation limited to use only when necessary for identification of interaction

DISADVANTAGES

- Results are limited to range of conditions used in test procedures
- Unknown how lab tests will relate to real-world environment
- Statistical conclusions are only as accurate as the collected data

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CNWRA INTEGRATED WASTE PACKAGE CORROSION EXPERIMENTS

PRESENTATION SUMMARY

- BACKGROUND
- SUMMARIES OF DATA FROM OTHER SOURCES
  - YMP/DOE
  - CORTEST COLUMBUS
  - MARSH ET AL.
- CNWRA ELECTROCHEMICAL LOCALIZED CORROSION STUDIES
- PROPOSED STUDY OF VARIABILITY IN CYCLIC POTENTIODYNAMIC POLARIZATION TEST PROCEDURE
- SUMMARY OF PLANNED CNWRA SCREENING TEST PROGRAM
- SUMMARY OF CNWRA PLANNED SHORT-TERM TEST PROGRAM

TECHNICAL PROGRAM

TEST MATERIALS IN CNWRA CORROSION TEST PROGRAM

- TYPE 304L STAINLESS STEEL (REFERENCE ALLOY)
- TYPE 316L STAINLESS STEEL
- INCOLOY 825
- COPPER ALLOY CDA 102 (OXYGEN-FREE, HIGH-CONDUCTIVITY COPPER)
- COPPER ALLOY CDA 613 (7-8 % ALUMINUM BRONZE)
- COPPER ALLOY 715 (70% COPPER-30% NICKEL)

ADDITIONAL CNWRA REFERENCE MATERIAL

- HASTELLOY C-22

TECHNICAL PROGRAM (CONTINUED)

TEST ENVIRONMENTS

- WITH AND WITHOUT GAMMA RADIATION
    - AQUEOUS SOLUTIONS SIMULATING LIQUID PHASES IN EQUILIBRIUM WITH TUFF ROCK (e.g., VADOSE WATERS AND J-13 WELL WATER) AT T≤95°C
    - CONCENTRATED SALT SOLUTIONS SIMULATING:
        - SOLUTIONS CONCENTRATED BY BOILING OF AQUEOUS SOLUTIONS AND SUBSEQUENT CONDENSATION OF WATER VAPOR AT TEMPERATURES OF 95°C OR LOWER
        - ENVIRONMENTS WITHIN OCCLUDED CELLS
    - SOLUTIONS IN WHICH SPECIES DETRIMENTAL TO SPECIFIC ALLOYS ARE CONCENTRATED, e.g., SIMULATED J-13 WELL WATER + 1,000 PPM CHLORIDE ION FOR COMPARING PITTING, CREVICE CORROSION, AND SCC RESISTANCE OF STAINLESS STEELS AND Cr-Ni-Fe ALLOYS
    - AIR-STEAM VAPOR PHASE AT 150 TO 250°C TO SIMULATE CONDITIONS ANTICIPATED TO BE PRESENT IN THE TUFF REPOSITORY DURING THE FIRST 200 TO 300 YEARS WHEN TEMPERATURES ARE ABOVE THE BOILING POINT OF AQUEOUS SOLUTIONS

CHEMICAL COMPOSITIONS OF ACTUAL AND SIMULATED J-13 WELL WATER SOLUTIONS*

Species	Actual J-13 Well Water (ppm)	Simulated J-13 Well Water (ppm)
Cations		
Na +	45.0	46.0
K +	5.30	5.50
Mg + +	1.76	1.70
Ca + +	11.5	12.0
Subtotal	63.56	65.2
Anions		
F -	2.10	1.70
Cl -	6.40	6.40
HCO3 -	143	121
NO3 -	10.1	12.4
SO4 - -	18.1	19.2
Subtotal	179.7	160.7
Other		
SiO2	64.2	64.2
Tot. Dis. Solids	307.5	290.1
pH	6.9	7.0 ± 0.2

*FROM CORTEST COLUMBUS QUALITY ASSURANCE DOCUMENT QA003, REVISION D, BASED ON NUREG/CR-4955 BMI-2155, NOVEMBER 1987, BATTLE COLUMBUS LABORATORIES, QUALITY ASSURANCE PROCEDURE WF-PP-34, REVISION O

SOURCES OF DATA PERTINENT TO CORROSION OF METALS IN THE YMP TUFF REPOSITORY ENVIRONMENT

- OPEN TECHNICAL LITERATURE
- YMP/DOE REPORTS, TECHNICAL PAPERS, AND PRESENTATIONS
- NRC CONTRACTOR REPORTS, TECHNICAL PAPERS, AND PRESENTATIONS, PRIMARILY CORTEST COLUMBUS
- OHIO STATE UNIVERSITY
- OTHER COUNTRIES

SUMMARY OF CORROSION DATA IN YMP/DOE SITE CHARACTERIZATION PLAN

GENERAL CORROSION

COUPONS OF STAINLESS STEELS WERE EXPOSED FOR 3,800 TO 11,500 HRS IN J-13 WATER AT TEMPERATURES OF 50, 80, 100, AND 150°C. VARIATION OF RATE WITH TIME WAS NOT INVESTIGATED

WORST-CASE RESULT WAS A CORROSION RATE OF 0.211μm/yr FOR INCOLOY 825 AT 50°C.

CONCLUSION: A CANISTER MADE OF ANY OF THE ALLOYS WOULD NOT BE PENETRATED IN 1,000 YRS.

(CONTINUED)

SUMMARY OF CORROSION DATA IN YMP/DOE SITE CHARACTERIZATION PLAN (CONTINUED)

STRESS CORROSION CRACKING (SCC)

U-BEND SPECIMENS

SENSITIZED TYPE 304 STAINLESS STEEL EXPOSED IN MOIST AIR AND GAMMA RADIATION AT 50°C DEVELOPED INTERGRANULAR CRACKS IN 1-MO. AND 3-MO. TESTS.

SENSITIZED TYPE 304 STAINLESS STEEL EXPOSED AT 90°C DEVELOPED CRACKS IN MOIST AIR AND IN J-13 WATER IN CONTACT WITH TUFF ROCKS.

TYPE 304L STAINLESS STEEL SPECIMENS DEVELOPED TRANSGRANULAR SCC IN SOME TESTS.

SLOW-STRAIN-RATE TEST SPECIMENS (STRAIN RATE = 2×10^{-7}/SEC)

SENSITIZED TYPE 304 STAINLESS STEEL SPECIMENS TESTED IN J-13 WATER AT 150°C CRACKED INTERGRANULARLY

MILL-ANNEALED TYPE 304 AND TYPE 304L STAINLESS STEELS DID NOT CRACK UNDER THE SAME CONDITIONS.

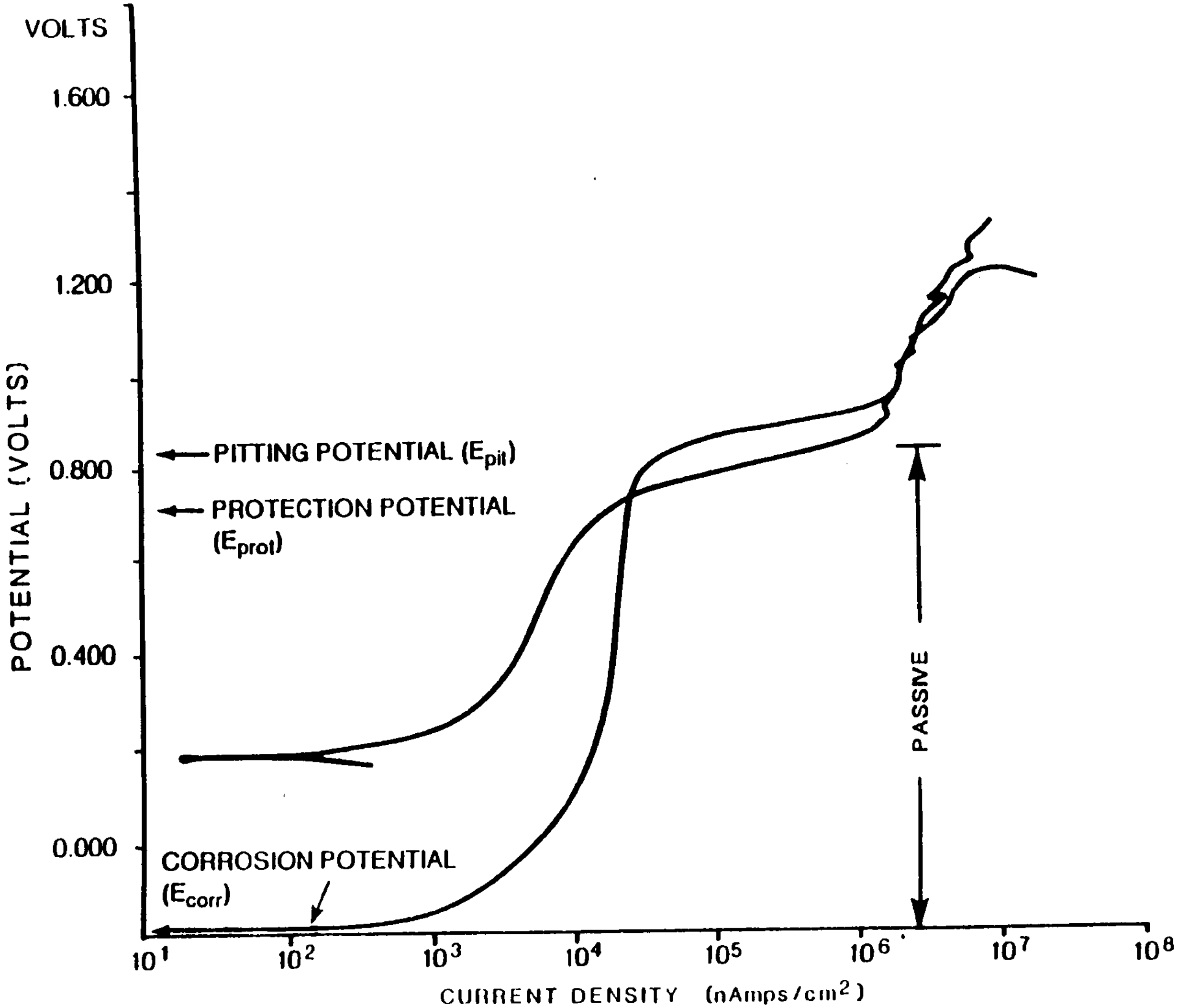

Figure 7-15. Potentiodynamic anodic polarization curve for AISI 304L stainless steel in well J-13 water at 90°C. Scan rate was 1 mV/s. Scan starts from E_{corr}. Line marked passive indicates that stainless steel remains passive until the pitting potential is reached. Modified from Glass et al. (1984)

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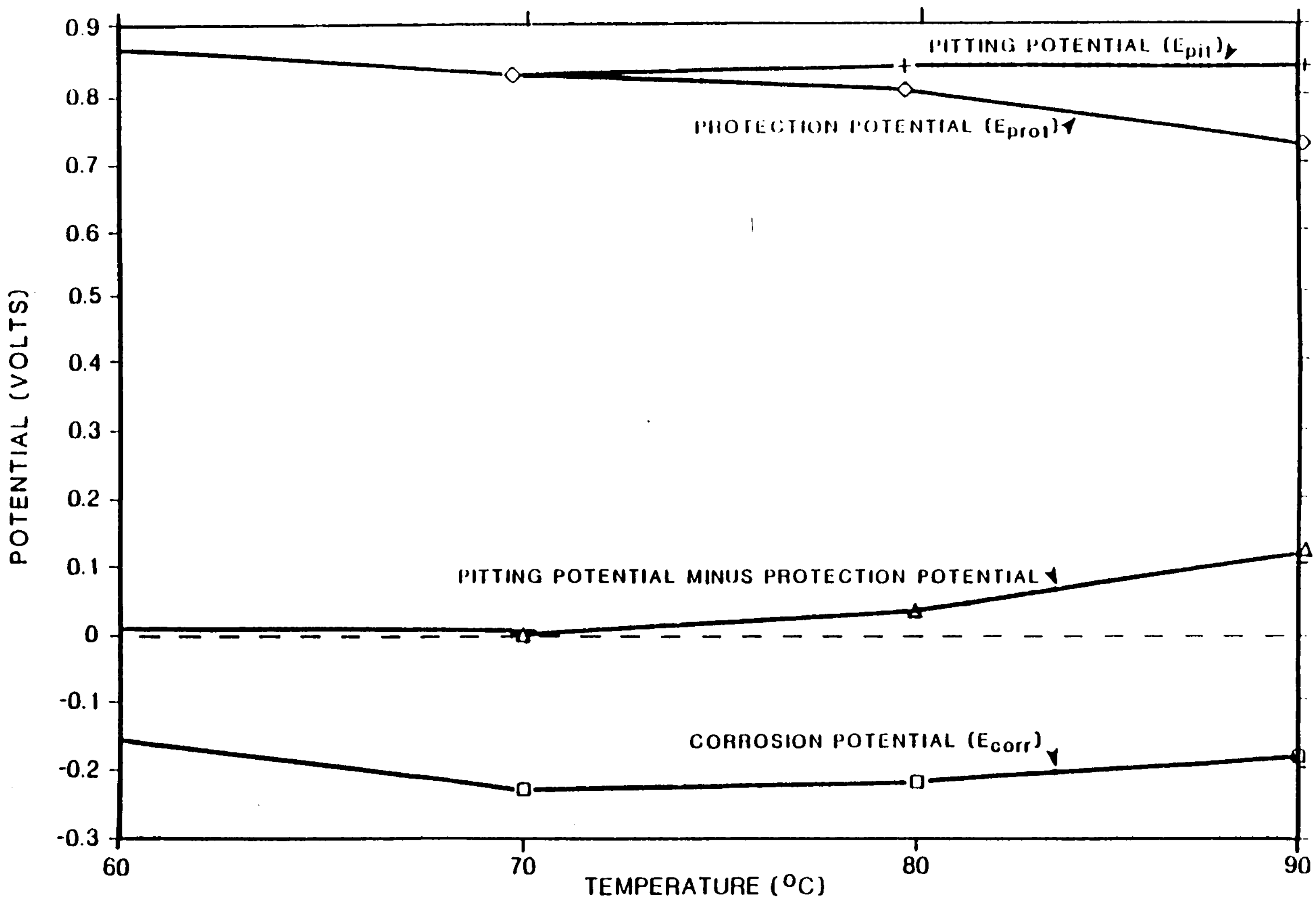

Figure 7-16. Electrochemical parameters for AISI 304L stainless steel in tuff conditioned water from well J-13 as a function of temperature. All potentials are referenced to a saturated calomel electrode at 25°C. Modified from Glass et al. (1984)

11/7/85

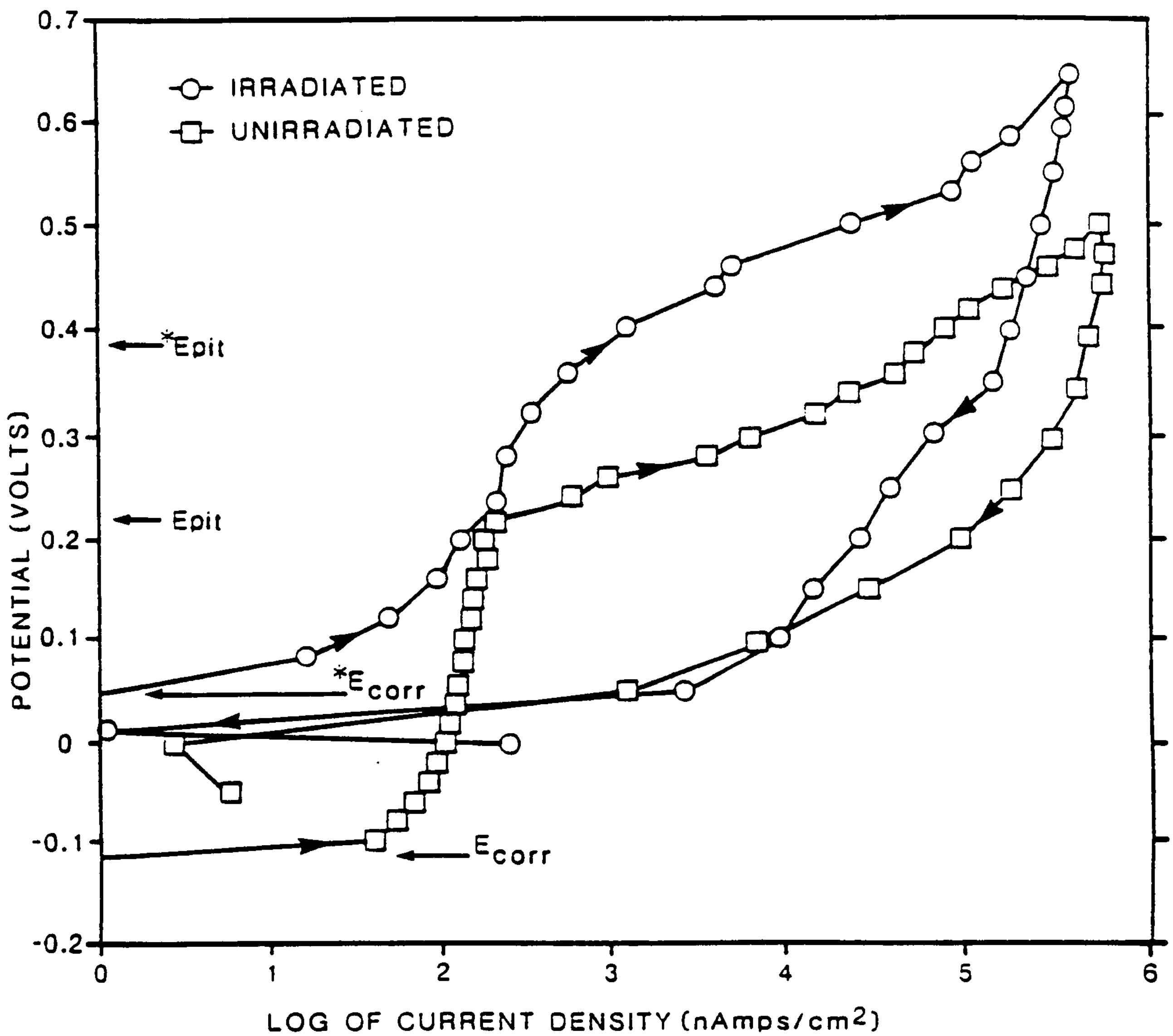

Figure 7-18. Comparison of the potentiostatic anodic polarization behavior for 316L stainless steel in 650 ppm chloride solution in deionized water with and without gamma irradiation. All potentials are referenced to a saturated calomel electrode. (The polarization curves were scanned anodically starting from the corrosion potential in each case. Upon reaching the anodic limit, the scans were reversed to more negative potentials. In this figure, E_{corr} and E_{pit} represent values of the corrosion potential and pitting potential, respectively, for the nonirradiated case. The corresponding values for the irradiated experiment are indicated on the figure as $^*E_{corr}$ and $^*E_{pit}$). Modified from Glass et al. (1985).

OUTLINE OF CORTEST COLUMBUS CYCLIC POTENTIODYNAMIC POLARIZATION TEST PROGRAM

- A TWO-LEVEL STATISTICAL DESIGN EXPERIMENT IS BEING CONDUCTED TO DETERMINE THE EFFECTS OF ENVIRONMENTAL SPECIES, pH, AND TEMPERATURE ON:

  - PITTING POTENTIALS (Epit)
  - PROTECTION POTENTIALS (Eprot)
  - CORROSION POTENTIALS (Ecorr)

- APPROACH -- 36 CYCLIC POTENTIODYNAMIC POLARIZATION CURVES ARE DEVELOPED FOR EACH ALLOY STUDIED. THE TEST MATRIX INCLUDES 32 COMBINATIONS OF ENVIRONMENTS PLUS 4 REPLICATIONS OF A 33rd (MIDPOINT) ENVIRONMENT.

  STATISTICAL ANALYSIS OF THE RESULTS ARE USED TO DETERMINE THE MAIN EFFECTS OF EACH OF THE 16 VARIABLES.

  EFFECTS OF INTERACTIONS BETWEEN TWO OR MORE VARIABLES ARE NOT DETERMINED.

(CONTINUED)

OUTLINE OF CORTEST COLUMBUS CYCLIC POTENTIODYNAMIC POLARIZATION TEST PROGRAM

- ENVIRONMENT -- SIMULATED J-13 WATER MADE IN ACCORDANCE WITH THE BATTELLE PROCEDURE AND MODIFIED BY VARIATIONS IN LEVELS OF CHEMICAL SPECIES
  - 13 CHEMICAL SPECIES -- 2 LEVELS OF EACH
  - pH -- 5 AND 10
  - TEMPERATURE -- 50 AND 90°C
- ALLOYS -- 4 0F 6 BEING CONSIDERED BY YMP/DOE:
  - TYPE 304L STAINLESS STEEL
  - INCOLOY 825
  - CDA 102
  - CDA 715
- TEST PROCEDURE -- DETAILS NOT REPORTED. SPECIMEN SIZE IS APPROXIMATELY 5 cm^2. SCAN RATE PROBABLY IS 0.6V/hr.
- RESULTS -- REPORT NOT RELEASED

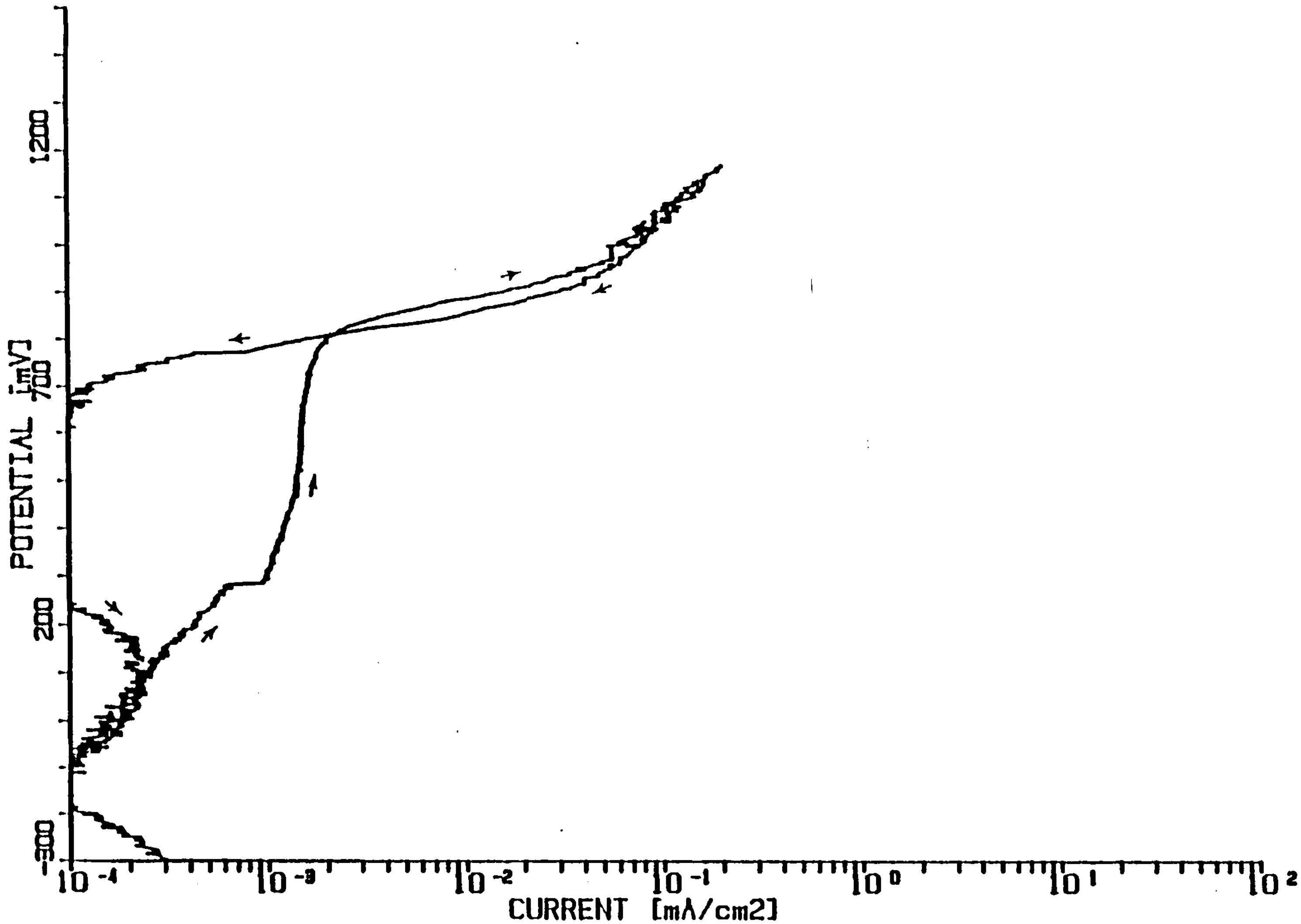

Figure 1. Polarization Curve For Type 304L Stainless Steel In Simulated J-13 Well Water At 90°C.

CORTEST COLUMBUS

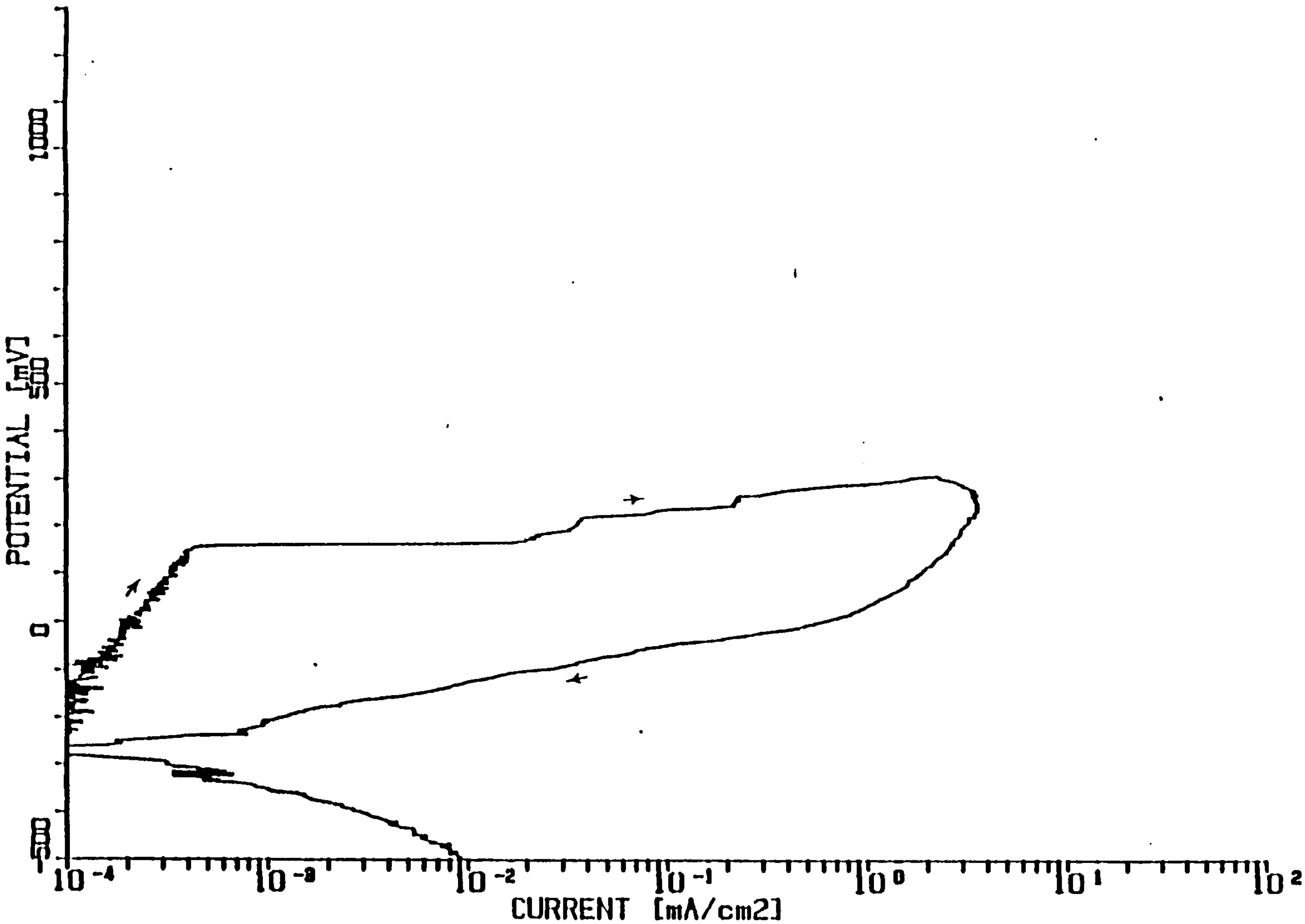

Figure 2. Polarization Curve For Type 304L Stainless Steel In Simulated J-13 Well Water With 1,000mg/l Cl Added At 90°C.

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List Of Variables Included In the Resolution IV Matrix Of Potentiodynamic Polarization Tests In Task 2.

Variable No.	Variable Name	Origin	Test Matrix High Concentration, mg/l	Test Matrix Low Concentration, mg/l	Nominal Concentration Of J-13, mg/l
1	Si	J-13	100	1	58
2	HCO_3	J-13	2000	10	125
3	F	J-13	200	1.0	2.2
4	Cl	J-13	1000	5.0	6.9
5	NO_3	J-13	1000	5.0	9.6
6	NO_2	Radiolysis	200	0	-
7	H_2O_2	Radiolysis	200	0	-
8	Ca	J-13	20	0.1	12
9	Mg	J-13	20	0.1	1.9
10	Al	J-13	20	0.01	0.01
11	P	J-13	20	0.1	0.12*
12	Oxalic	Radiolysis	200	0	-
13	O_2	Open Repository and Radiolysis	30**	5**	-
14	Temp	J-13	90+	50+	-
15	pH	J-13	10	5	7.6

* McCright, R.D. FY 1985 Status Report, UCID-20509, September 30, 1985.

** Volume %.

\+ Degrees Centigrade.

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ELECTROCHEMICAL TEST DATA FOR TYPE 304L STAINLESS STEEL IN 300 PPM CHLORIDE SOLUTION (ADAPTED FROM MARSH ET AL., CORROSION SCIENCE, VOL. 26, NO. 11, PP. 971-982, 1986)*

Environment		Corrosion Potential (Ecorr) (mv vs SCE)	Pitting Potential (Epit) (mV vs SCE)	Repassivation Potential (Erp) (mV vs SCE)	Epit - Erp (mV vs SCE)
Unirradiated		95	340	180	160
Unirradiated		118	810	250	560
Unirradiated		-17	740	170	570
Unirradiated		45	190	140	50
Unirradiated		-10	695	220	475
Unirradiated		-3	275	280	-5
Unirradiated		-75	565	260	305
Unirradiated		-55	615	250	365
Unirradiated		-218	570	175	395
Unirradiated		-210	600	270	330
	Mean and Std. Dev.	-33 ± 113	540 ± 206 (655 ± 93)**	220 ± 49 (228 ± 41)**	320 ± 199 (429 ± 108)**
Irradiated***		264	650	220	430
Irradiated		230	690	150	540
Irradiated		252	700	310	390
Irradiated		260	590	250	340
Irradiated		355	645	220	425
Irradiated		325	610	190	420
Irradiated		370	710	260	450
Irradiated		378	730	210	520
Irradiated		407	660	200	460
Irradiated		360	680	230	450
	Mean and Std. Dev.	320 ± 63	667 ± 45	224 + 43	443 ± 58

Notes:

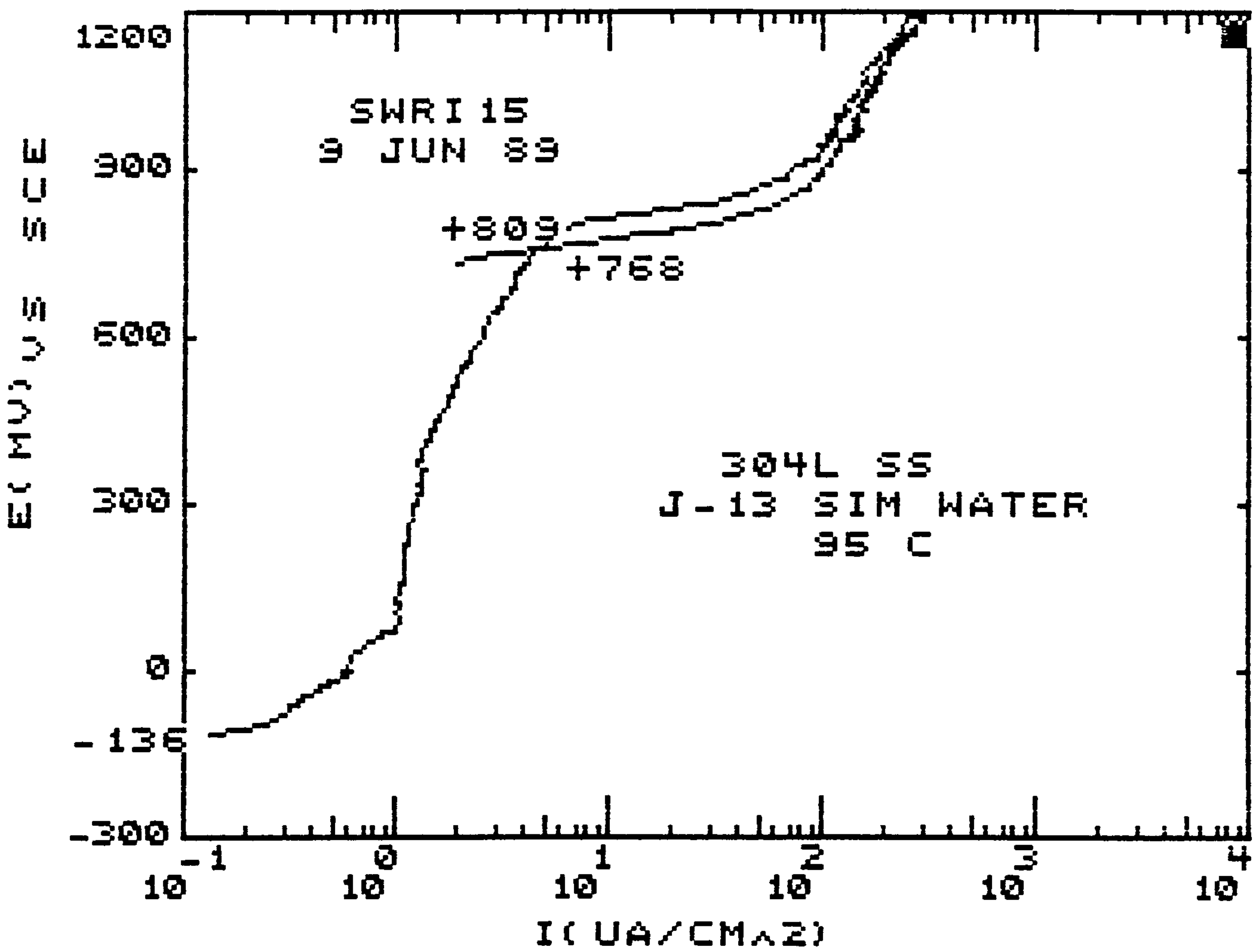
* Scan rate = 0.5 mV/sec; Temperature = 40°C; Argon cover gas; Specimen Area = 2.5 cm2

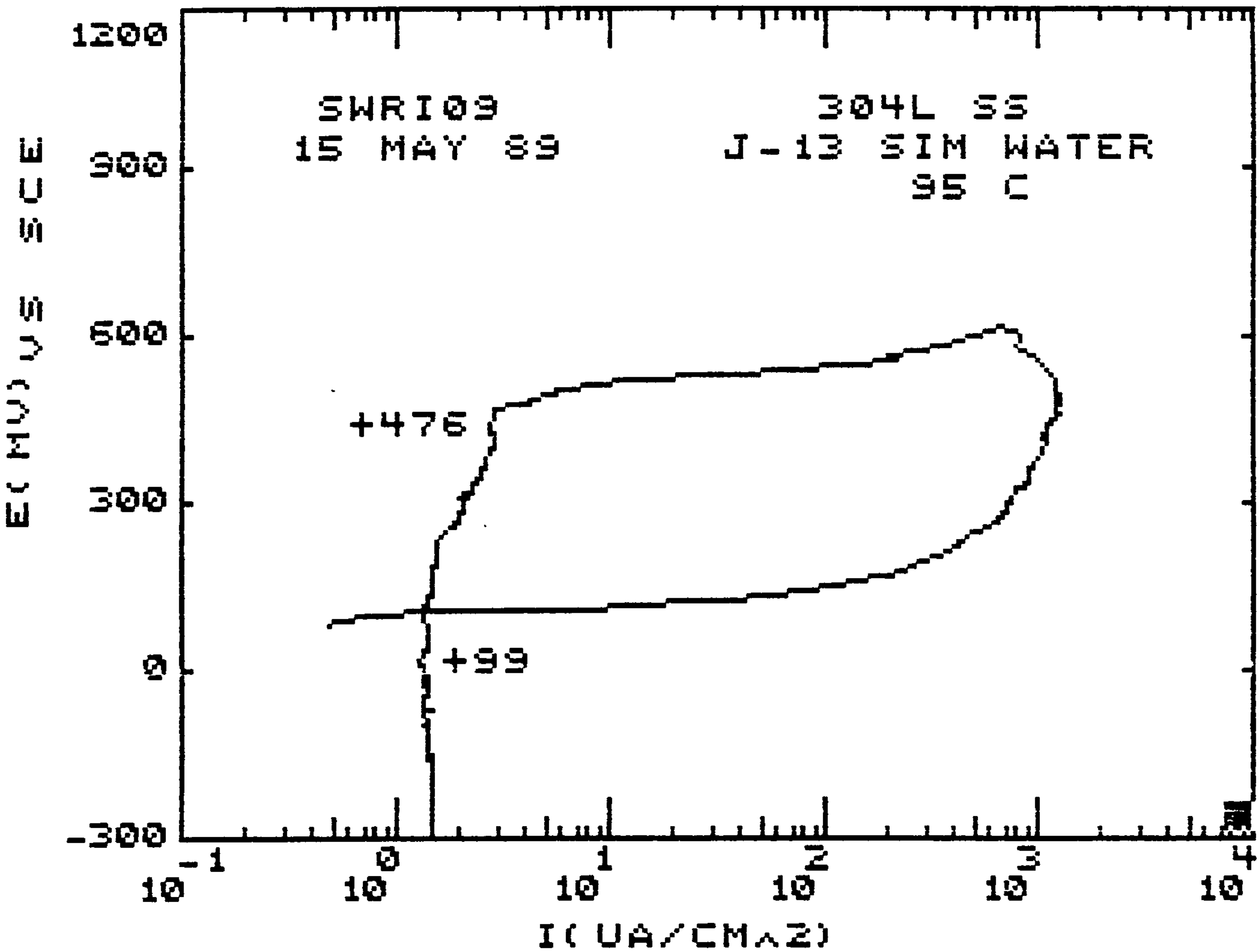
** 1st, 4th, and 6th data points disregarded

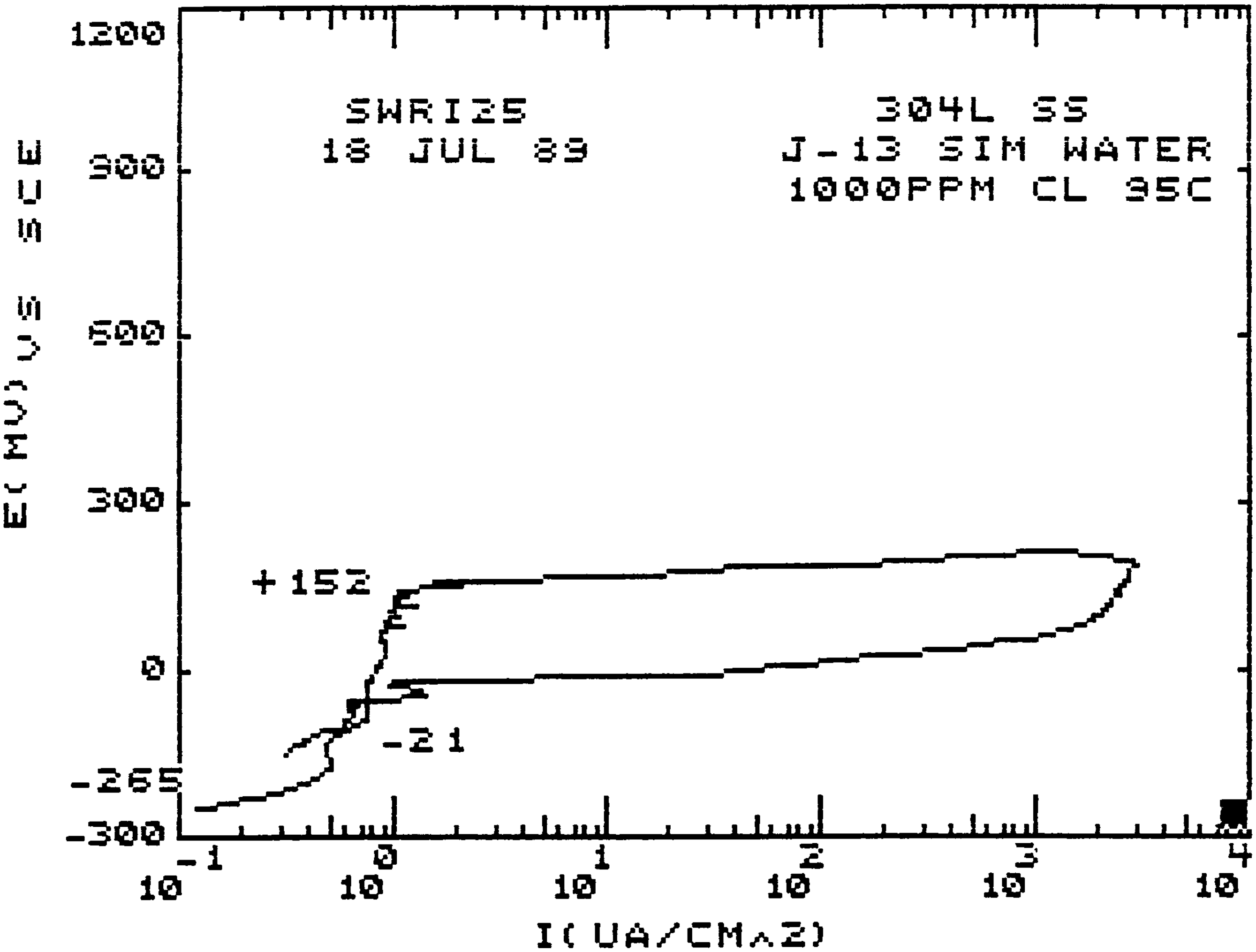
*** Gamma Radiation; Co-60 Source; 1 to 2 E+3 Sv/hr

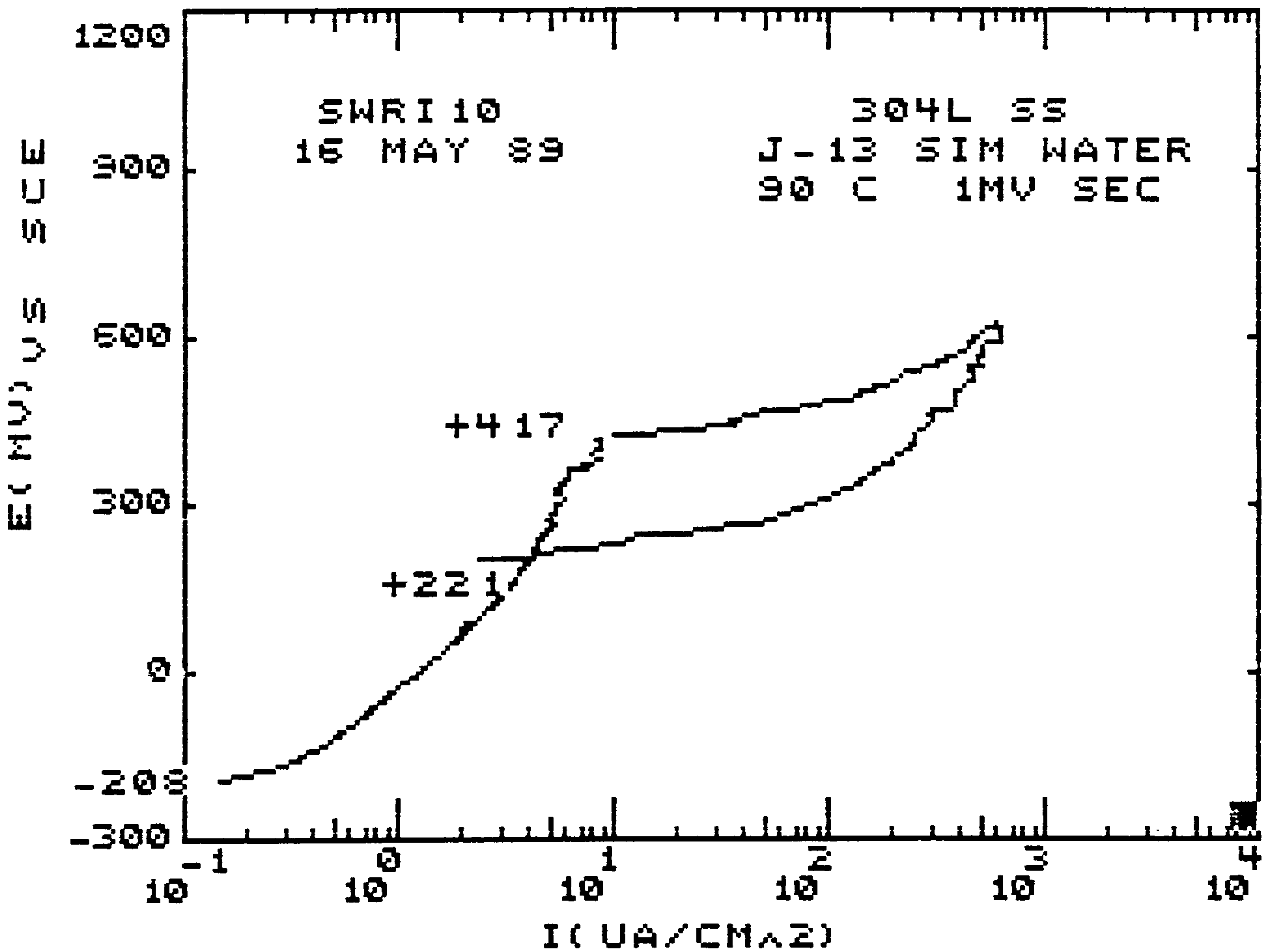
CNWRA ELECTROCHEMICAL LOCALIZED CORROSION TEST PARAMETERS

- TEST PROCEDURE -- CYCLIC POTENTIODYNAMIC POLARIZATION CURVES GENERATED IN ACCORDANCE WITH TEST PROCEDURE GIVEN IN ASTM G-61, "CONDUCTING CYCLIC POTENTIODYNAMIC POLARIZATION MEASUREMENTS FOR LOCALIZED CORROSION"
  - SCAN RATE -- 0.6 V/hr (0.17 mV/sec) EXCEPT FOR 2 TESTS CONDUCTED AT 3.6 V/hr (1.0 mV/sec)
  - THRESHOLD CURRENT -- 5,500 μA
  - ELECTRODE SIZE -- CYLINDRICAL ELECTRODE, 10 cm^2 EXPOSED SURFACE AREA
  - SOLUTION VOLUME -- 900 ml
- ALLOYS STUDIED -- 3: TYPES 304L AND 316L STAINLESS STEEL AND INCOLOY 825
- TEST SOLUTIONS -- SIMULATED J-13 WATER AND J-13 WATER + 1,000 PPM Cl^-
- TEMPERATURES -- 95°C EXCEPT FOR 2 TESTS CONDUCTED AT 90°C


SWRI 15
9 JUN 89
+809
+768
-136
304L SS
J-13 SIM WATER
95 C
E(MV) VS SCE
I(UA/CM^2)
1200
900
600
300
0
-300
10^-1
10^0
10^1
10^2
10^3
10^4


SWRI09
15 MAY 89
304L SS
J-13 SIM WATER
95 C
+476
+99
1200
900
600
300
0
-300
E(MV) VS SCE
10^-1
10^0
10^1
10^2
10^3
10^4
I(UA/CM^2)


SWRI25
18 JUL 89
304L SS
J-13 SIM WATER
1000PPM CL 95C
E(MV) VS SCE
1200
900
600
300
0
-265
-300
+152
-21
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
10^{4}
I(UA/CM^2)


SWRI 10
16 MAY 89
304L SS
J-13 SIM WATER
90 C 1MV SEC
+417
+221
-208
1200
900
600
300
0
-300
E(MV) VS SCE
10^-1 10^0 10^1 10^2 10^3 10^4
I(UA/CM^2)

PRELIMINARY ELECTROCHEMICAL TEST DATA -- TYPE 304L STAINLESS STEEL

(See Notes 1-5)

Environment	Solution Deaerated?	Initial Potential (mV vs SCE)	Corrosion Potential (Ecorr) (mv vs SCE)	Pitting Potential (Epit) (mV vs SCE)	Repassivation Potential (Erp) (mV vs SCE)	Epit - Erp (mV vs SCE)	Solution pH		pHi - pHf
							Initial	Final	
Simulated J-13 Water	Yes	-427	-427	512	140	372	6.88	9.14	2.26
Simulated J-13 Water	Yes	-200	-478	451	107	344	6.83	9.10	2.27
Simulated J-13 Water	Yes	-200	-515	514	128	386	7.06	7.55	0.49
Simulated J-13 Water	Yes	-200	-742	476	99	377	6.97	9.22	2.25
Simulated J-13 Water	No	-140	-140	809	768	41	7.18	9.12	1.94
				552 ± 146 (488 ± 30)*	248 ± 291 (118 ± 19)*	304 ± 148 (370 ± 18)*	6.98 ± 0.14	8.83 ± 0.71	1.84 ± 0.77 (2.18 ± 0.16)*
Simulated J-13 Water (Scan Rate: 1.0 mV/sec; Temp: 90°C)	No	-208	-208	417	221	196	6.91	8.11	1.20
Simulated J-13 Water + Crevice (Initial pH adjusted with CO2)	No	-228	-228	837	824	13	6.92	8.65	1.73
Sim. J-13 Water + 1,000 ppm Cl-	Yes	-693	-693	-48	-129	81	7.17	8.97	1.80
Sim. J-13 Water + 1,000 ppm Cl- (Initial pH adjusted with CO2)	No	-265	-265	152	-21	173	6.86	8.59	1.73

Notes:

(1) Initial pH adjusted with 0.1 N HCl, unless otherwise indicated

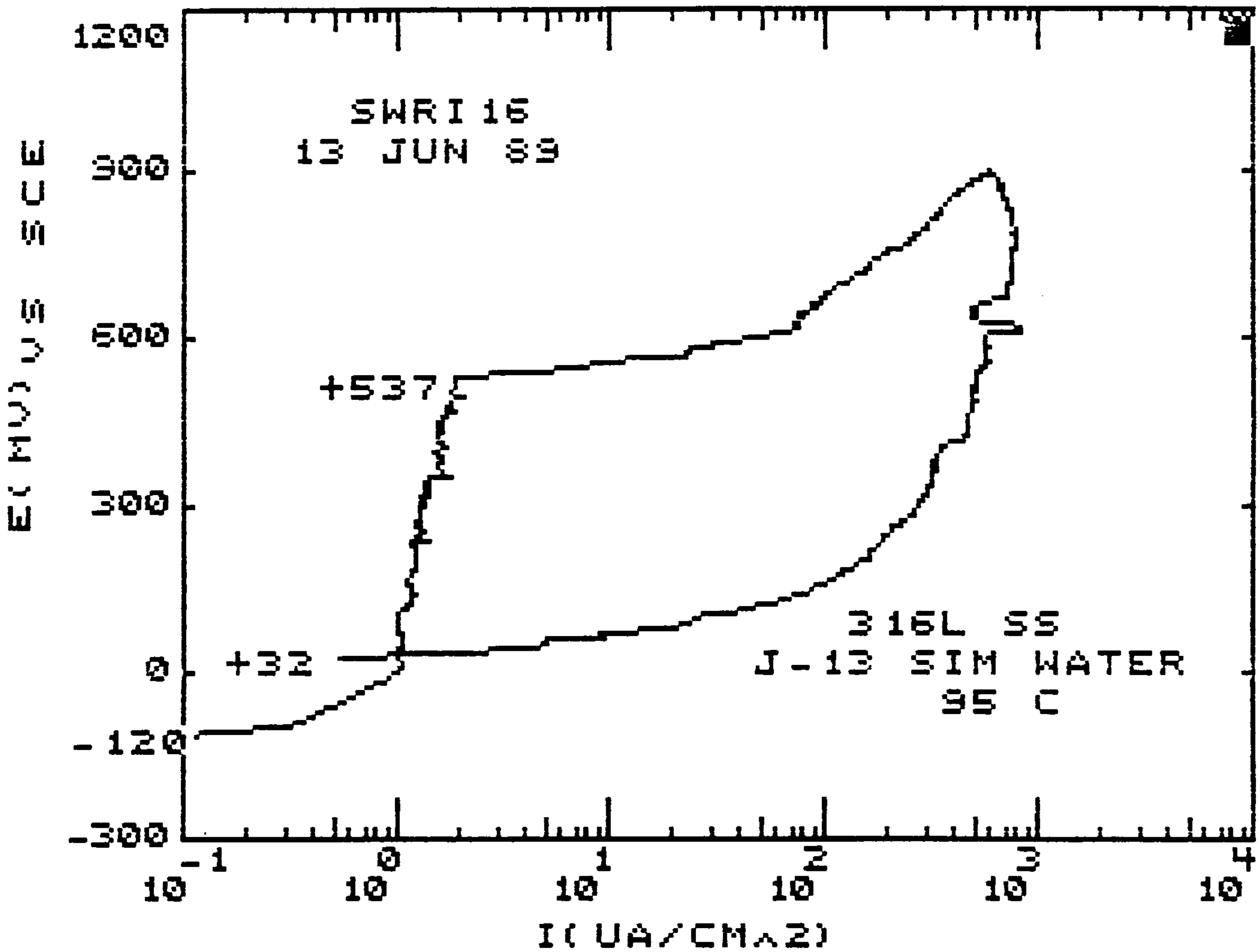
(2) Potential held at Ecorr for 60 minutes prior to scan

(3) Scan rate = 0.17 mV/sec; Temp. = 95°C, unless otherwise indicated

(4) Electrode partially immersed in solution

(5) Current threshold = 5,500 μA

* Based on four values (extreme value eliminated)



SWRI 16
13 JUN 89
316L SS
J-13 SIM WATER
95 C
+537
+32
-120
1200
900
600
300
0
-300
E(MV) VS SCE
10^-1
10^0
10^1
10^2
10^3
10^4
I(UA/CM^2)

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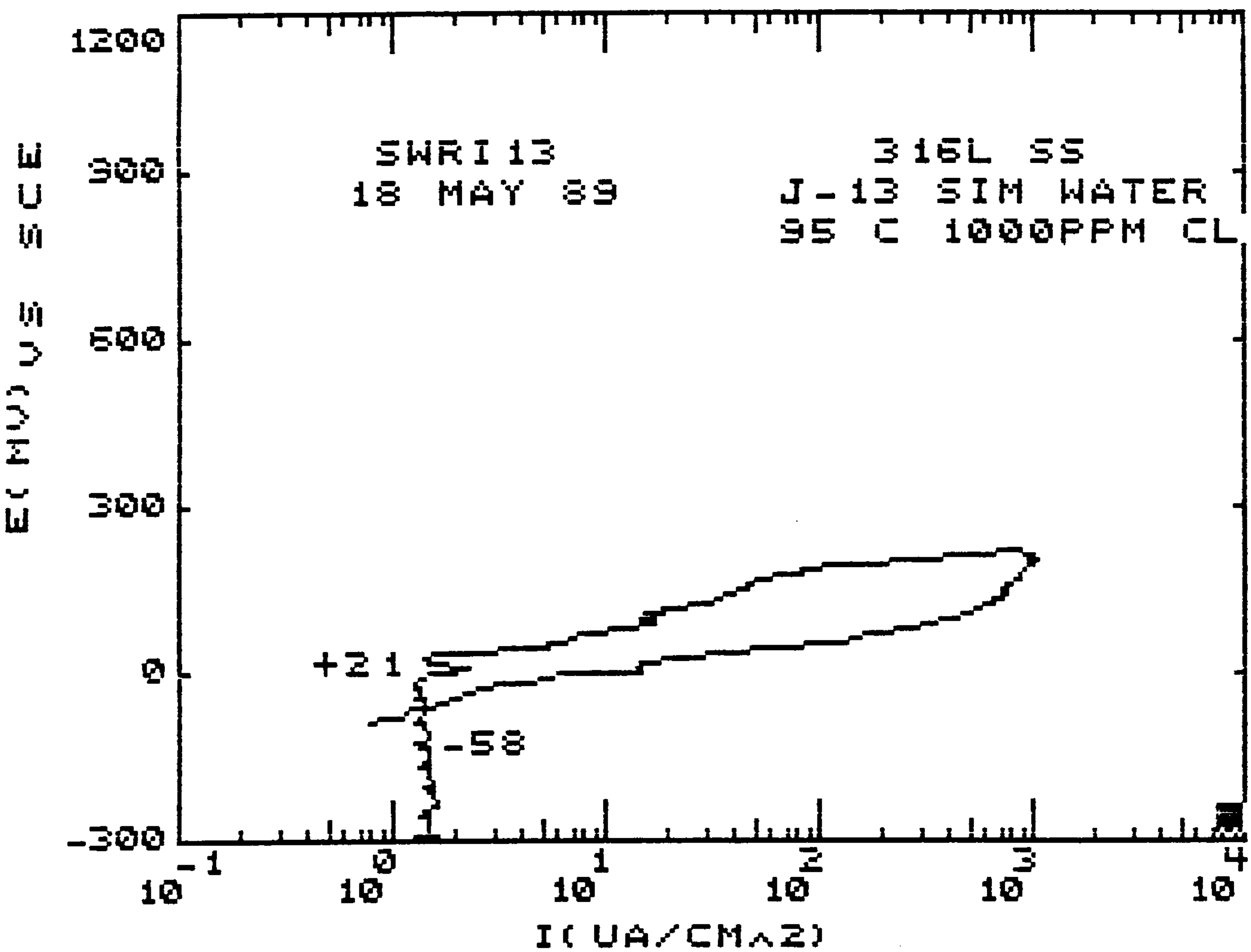

SWRI 13
18 MAY 89
316L SS
J-13 SIM WATER
95 C 1000PPM CL
+215
-58
E(MV) VS SCE
1200
900
600
300
0
-300
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
10^{4}
I(UA/CM^2)

PRELIMINARY ELECTROCHEMICAL TEST DATA -- TYPE 316L STAINLESS STEEL
(See Notes 1-5)

Environment	Solution Deaerated?	Initial Potential (mV vs SCE)	Corrosion Potential (Ecorr) (mv vs SCE)	Pitting Potential (Epit) (mV vs SCE)	Repassivation Potential (Erp) (mV vs SCE)	Epit - Erp (mV vs SCE)	Solution pH Initial	Solution pH Final	pHi - pHf
Simulated J-13 Water	No	-250	-402	417	129	288	7.18	8.33	1.15
Simulated J-13 Water	Yes	-730	-730	464	131	333	7.03	9.53	2.50
Simulated J-13 Water	Yes	-200	-720	511	110	401	7.13	8.90	1.77
Simulated J-13 Water	No	-212	-212	497	229	268	7.02	8.63	1.61
				472 ± 42	150 ± 54	322 ± 59	7.09 ± 0.08	8.85 ± 0.51	1.76 ± 0.56
Simulated J-13 Water (Scan Rate:1.0 mV/sec; Temp: 90°C)	No	-162	-162	423	267	156	6.89	7.19	0.30
Sim. J-13 Water + 1,000 ppm Cl-	Yes	-250	-718	21	-58	79	6.88	9.11	2.23

Notes:

(1) Initial pH adjusted with 0.1 N HCl

(2) Potential held at Ecorr for 60 minutes prior to scan

(3) Scan rate = 0.17 mV/sec; Temp. = 95°C

(4) Electrode partially immersed in solution

(5) Current threshold = 5,500 μA

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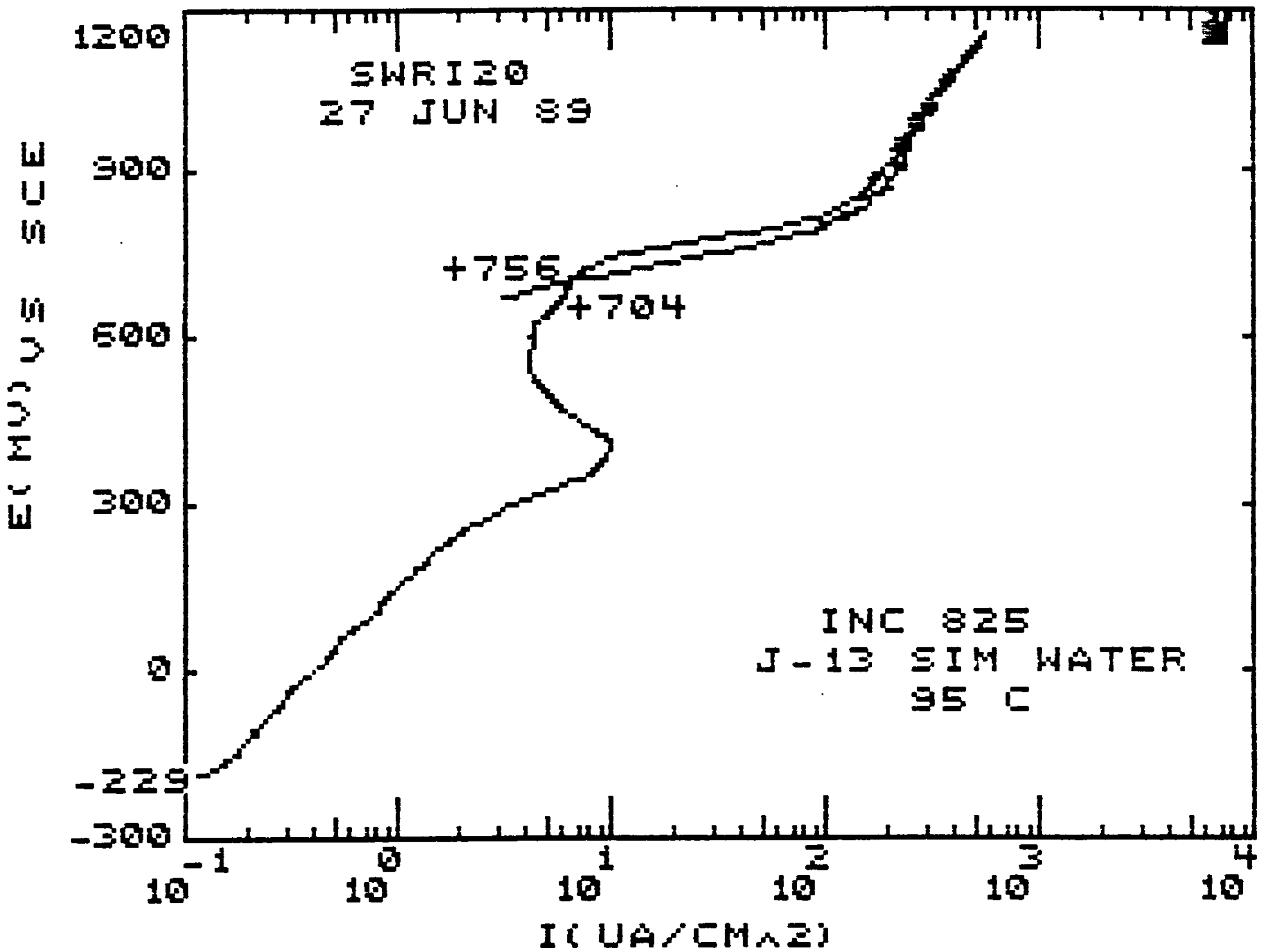
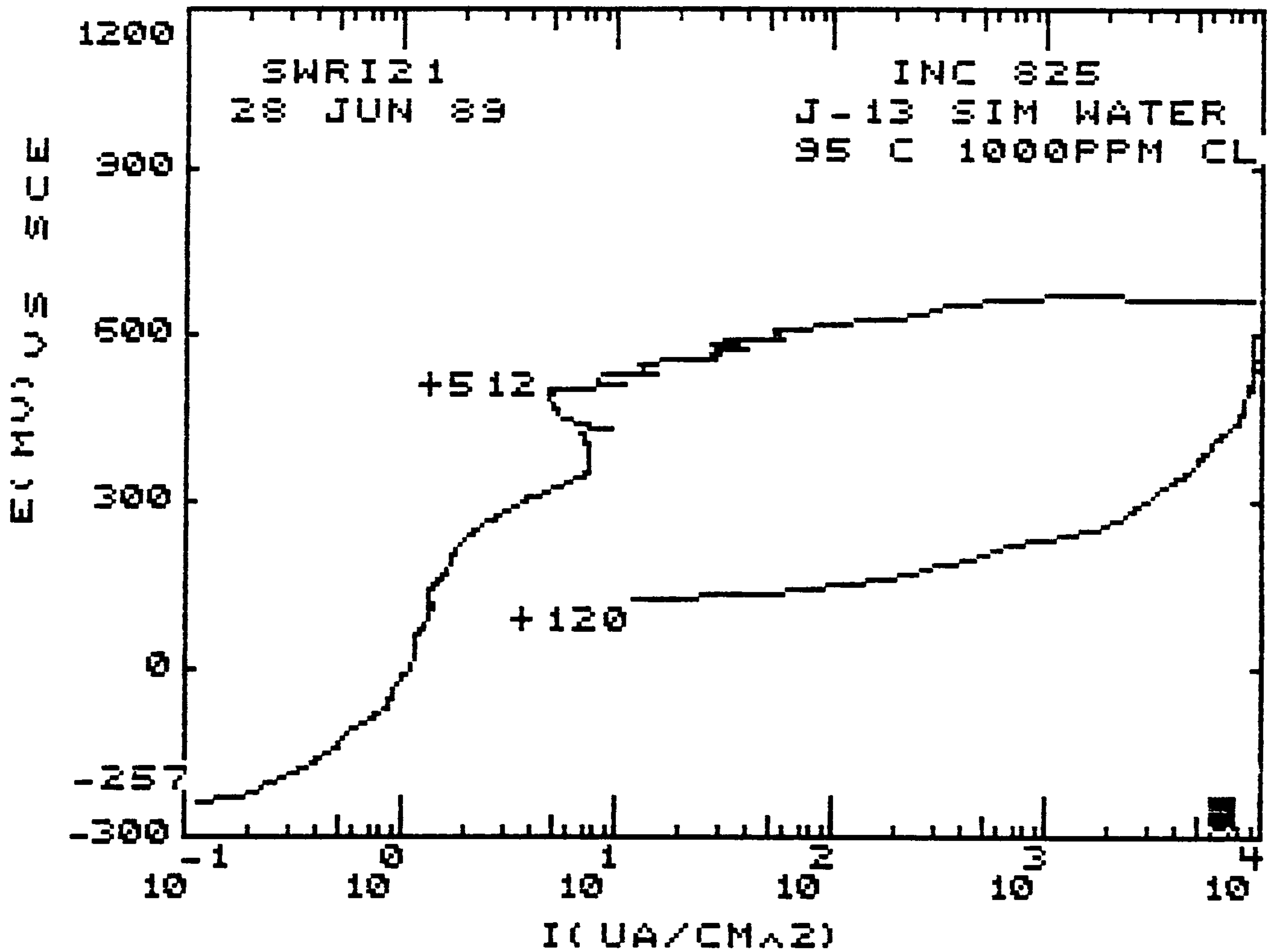

SWRI20
27 JUN 89
+756
+704
INC 825
J-13 SIM WATER
95 C
-229
E(MV) VS SCE
1200
900
600
300
0
-300
I(UA/CM^2)
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
10^{4}


SWRI21
28 JUN 89
INC 825
J-13 SIM WATER
95 C 1000PPM CL
E(MV) VS SCE
1200
900
600
300
0
-257
-300
+512
+120
-1
10
0
10
1
10
2
10
3
10
4
10
I(UA/CM^2)

PRELIMINARY ELECTROCHEMICAL TEST DATA -- INCOLOY 825

(See Notes 1-5)

Environment	Solution Deaerated?	Initial Potential (mV vs SCE)	Corrosion Potential (Ecorr) (mv vs SCE)	Pitting Potential (Epit) (mV vs SCE)	Repassivation Potential (Erp) (mV vs SCE)	Epit - Erp (mV vs SCE)	Solution pH Initial	Solution pH Final	pHi - pHf
Simulated J-13 Water (Initial pH adjusted with 0.1 N HCl)	No	-180	-180	833	833	0	7.04	8.42	1.38
Simulated J-13 Water	No	-229	-229	756	704	48	7.06	9.31	1.84
Simulated J-13 Water + Crevice	No	-231	-231	790	790	0	6.93	8.60	1.67
Sim. J-13 Water + 1,000 ppm Cl-	No	-257	-257	512	120	392	7.15	8.90	1.75
Sim. J-13 Water + 1,000 ppm Cl- + Crevice	No	-261	-261	484	168	316	6.92	8.46	1.54

Notes:

(1) Initial pH adjusted with CO_2, unless otherwise indicated

(2) Potential held at Ecorr for 60 minutes prior to scan

(3) Scan rate = 0.17 mV/sec; Temp. = 95°C

(4) Electrode partially immersed in solution

(5) Current threshold = 5,500 μA

SUMMARY OF AVAILABLE ELECTROCHEMICAL TEST RESULTS

- CNWRA DATA SHOW SIGNIFICANT VARIABILITY IN VALUES OF PITTING POTENTIAL AND REPASSIVATION POTENTIAL OBTAINED FOR TYPE 304L AND TYPE 316L STAINLESS STEELS IN SIMULATED J-13 WATER

- DATA OF MARSH ET AL. FOR TYPE 304L STAINLESS STEEL EXPOSED IN 300 PPM Cl^- SOLUTION AT 40°C SHOW SIGNIFICANT VARIABILITY IN VALUES OF PITTING POTENTIAL OBTAINED IN UNIRRADIATED WATER.

  THESE DATA ALSO SHOW A LARGE INCREASE IN THE CORROSION POTENTIAL AND PITTING POTENTIAL IN IRRADIATED WATER, AND THAT THE CORROSION POTENTIAL IN IRRADIATED WATER IS ABOVE THE REPASSIVATION POTENTIAL.

- YMP/DOE REPORTS AND THE SCP DO NOT ADDRESS THE REPRODUCIBILITY OF ELECTROCHEMICAL TEST DATA.

  THE YMP/DOE DATA INDICATE THAT GAMMA RADIATION SIGNIFICANTLY INCREASES THE CORROSION AND PITTING POTENTIALS OF TYPE 316L STAINLESS STEEL EXPOSED IN A 650 PPM Cl- SOLUTION AT 30°C, AND THAT THE CORROSION POTENTIAL IN THE IRRADIATED SOLUTION IS ABOVE THE REPASSIVATION POTENTIAL.

- CORTEST AND OHIO STATE UNIVERSITY -- DATA NOT YET RELEASED

QUESTIONS RAISED BY ELECTROCHEMICAL TEST RESULTS

WHAT IS THE SIGNIFICANCE OF THE VARIABILITY IN ELECTROCHEMICAL POLARIZATION TESTS NOTED IN THE CNWRA TESTS AND THE WORK OF MARSH ET AL.?

DOES THE OBSERVED VARIATION IN RESULTS MEAN THAT THERE IS SIGNIFICANT VARIATION IN THE SUSCEPTIBILITY OF THE ALLOYS TESTED TO PITTING AND CREVICE CORROSION IN THE ENVIRONMENTS EXAMINED?

IS SIGNIFICANT VARIATION INHERENT IN THE TEST PROCEDURE, AND, IF SO, WHAT ARE THE FACTORS RESPONSIBLE FOR THE VARIATION AND CAN IT BE REDUCED?

WHAT ARE THE RAMIFICATIONS OF THE OBSERVED VARIATION IN RESULTS WITH RESPECT TO THE CORTEST AND YMP/DOE STUDIES IN WHICH REPRODUCIBILITY OF TEST DATA MAY NOT HAVE BEEN EXAMINED?

WHAT ARE THE LIMITATIONS OF THESE TYPES OF TESTS WITH RESPECT TO THE PREDICTION OF LONG-TERM CORROSION PERFORMANCE OF ALLOYS?

RATIONALE FOR DETERMINING VARIATION PRESENT IN CYCLIC POTENTIODYNAMIC POLARIZATION TEST

- TO UNDERSTAND THE USEFULNESS AND LIMITATIONS OF THE TEST METHOD IN ORDER:
  - TO EVALUATE DATA DEVELOPED BY YMP/DOE AND OTHERS
  - TO DEVELOP A Q/A-QUALIFIED SCREENING PROCEDURE FOR USE IN THE PLANNING AND CONDUCT OF CORROSION AND MATERIALS EVALUATIONS IN THE CNWRA EXPERIMENTAL PROGRAM
  - TO PROVIDE APPROPRIATE PRELICENSING GUIDANCE TO YMP/DOE; AND
  - TO EVALUATE AND JUDGE THE ADEQUACY OF THE YMP/DOE LICENSE APPLICATION

POTENTIAL SOURCES OF ERROR IN PROCEDURE FOR THE PREPARATION OF SIMULATED J-13 WELL WATER*

FROM SECTION 5.0, "PROCEDURE FOR PREPARATION OF STOCK SOLUTIONS"

THIS SECTION STATES A PROCEDURE FOR MIXING ONE OF TWO STOCK SOLUTIONS FROM WHICH SIMULATED J-13 WATER IS PREPARED SUBSEQUENTLY. CHEMICALS MIXED IN DEIONIZED WATER ARE $CaSO_4$, $CaNO_3 \cdot 4H_20$, KCl, and $MgCl_2 \cdot 6H_2O$.

SUBSECTION 5.1.3 STATES: "*FILTER* SOLUTION, IF ANY *SOLIDS* REMAIN."

FROM SECTION 6.0, "PREPARATION OF SIMULATED TUFF GROUNDWATER:

THIS SECTION ADDRESSES FINAL PREPARATION OF SIMULATED J-13 WATER FROM TWO STOCK SOLUTIONS.

SUBSECTION 6.3 STATES: "CHECK pH. SHOULD BE 7.0 ± 0.2 AT ROOM TEMPERATURE. IF NECESSARY, ADJUST pH WITH *A FEW DROPS OF 0.1 N HCl* OR NaOH."

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*FROM CORTEST COLUMBUS QUALITY ASSURANCE DOCUMENT QA003, REVISION D, BASED ON NUREG/CR-4955 BMI-2155, NOVEMBER 1987, BATTLE COLUMBUS LABORATORIES, QUALITY ASSURANCE PROCEDURE WF-PP-34, REVISION O

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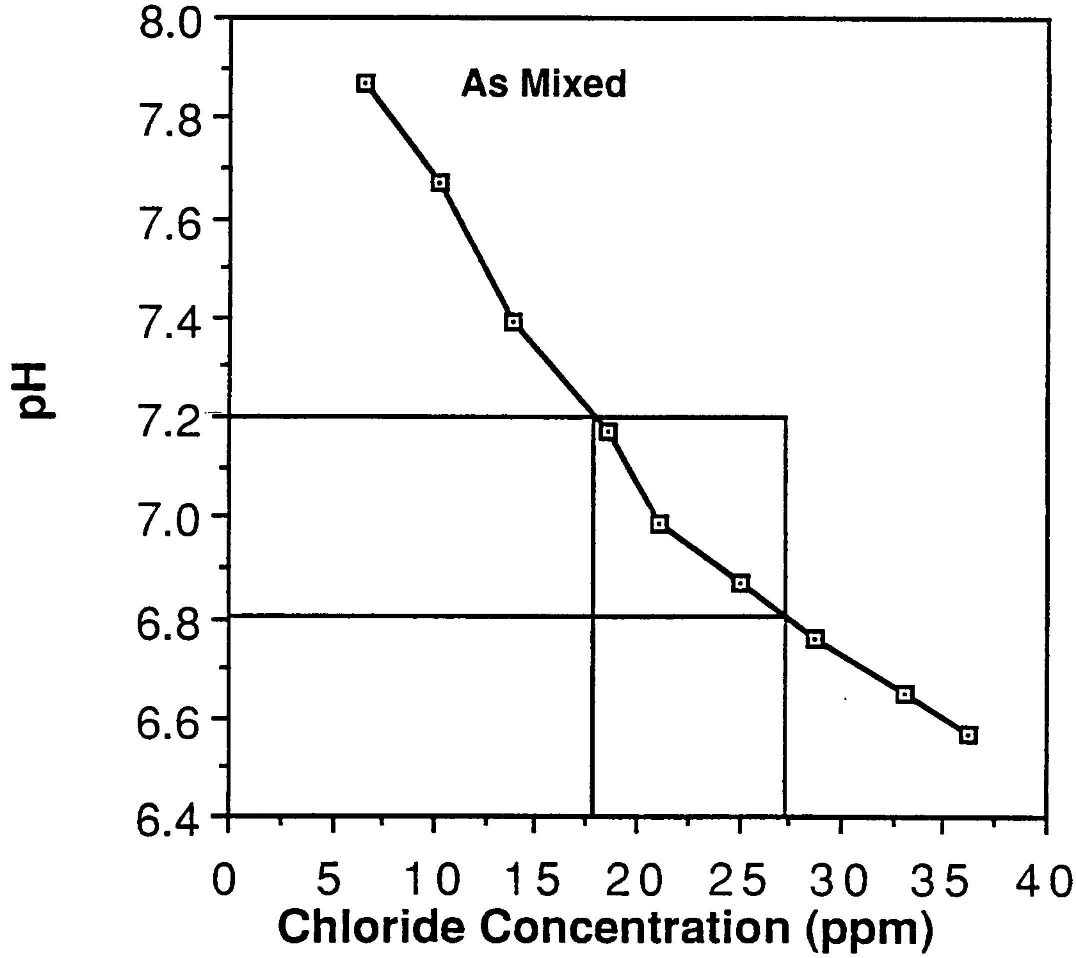
Chloride Concentration Range for Simulated J-13 Water Produced by Battelle Procedure



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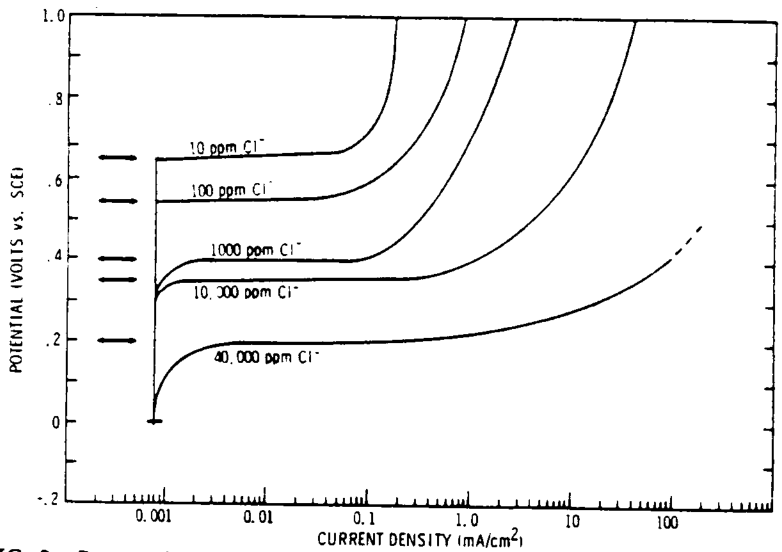

FIG. 2—*Potentiodynamic polarization curves of Type 304 in various chloride ion concentrations using NaCl as the salt.*

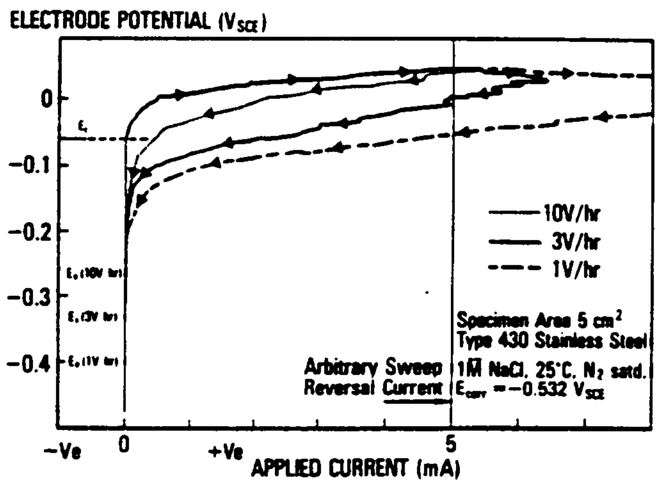

FIGURE 9 – "Cyclic" polarization behavior of Type 430 stainless steel in 1M NaCl indicating the effect of pit propagation time on the magnitude of E_p.

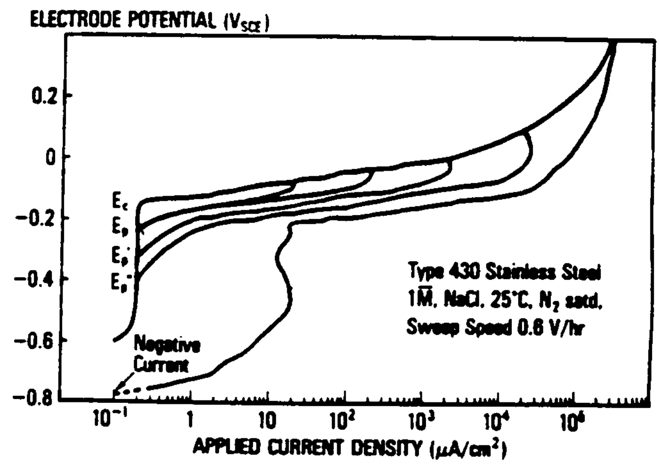

FIGURE 10 – "Cyclic" polarization behavior of Type 430 stainless steel in 1M NaCl demonstrating the marked effect of pit propagation on E_p.

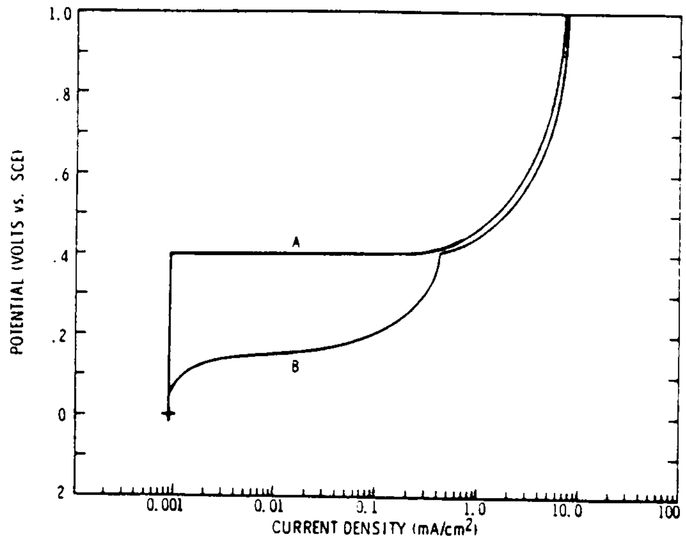

FIG. 3—*Potentiodynamic polarization curves of Type 304 in chloride solution showing a normal breakthrough (A) and a crevice initiated breakthrough (B).*

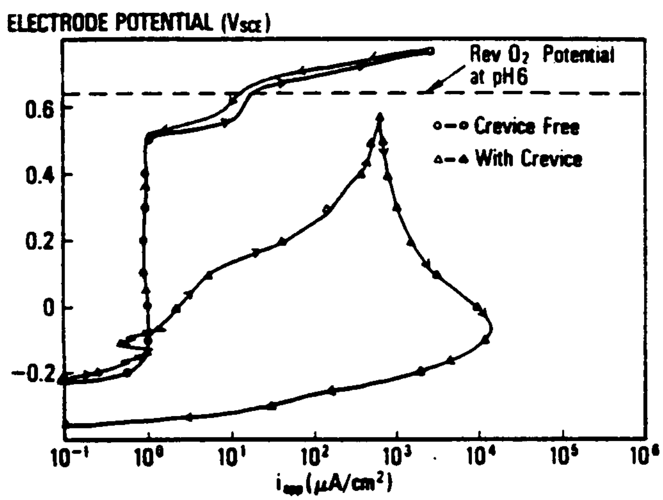

FIGURE 4 — "Cyclic" potentiodynamic anodic polarization curves for a 30Cr-3Mo-bal Fe alloy in nitrogen saturated 1M NaCl at 25 C sweep speed 0.6 V/hr.

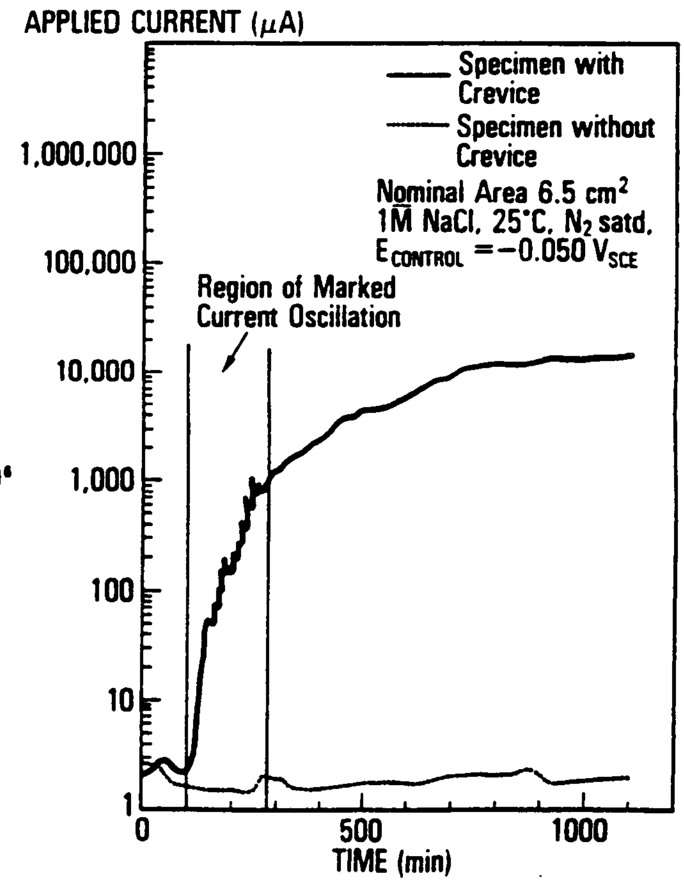

FIGURE 6 — Controlled potential/time behavior of a 30Cr-3Mo-bal Fe alloy in 1M NaCl, with and without a crevice on the specimen.

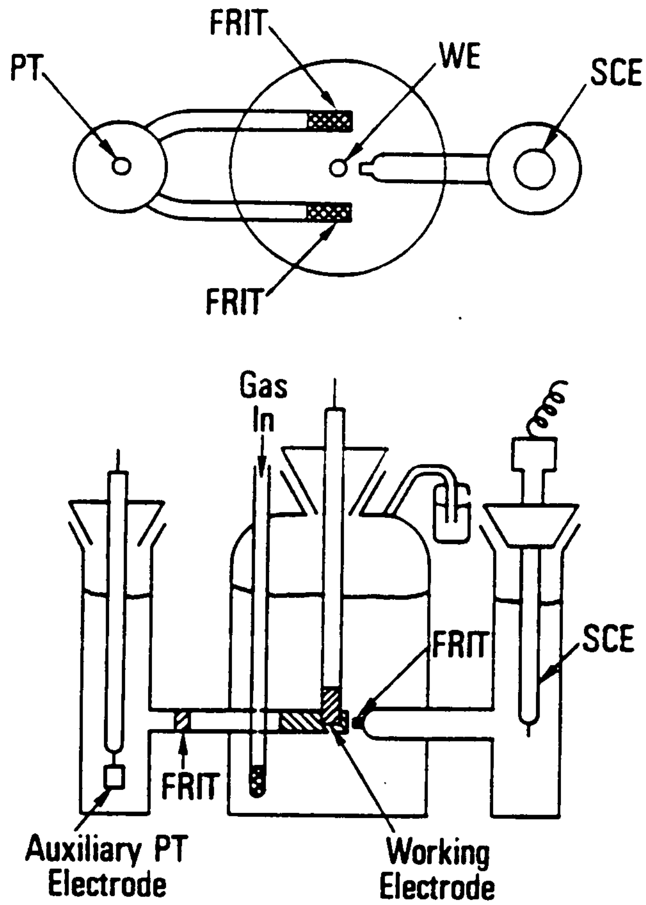

FIGURE 2 — Schematic of polarization cell.

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SUMMARY OF THE PRINCIPAL SOURCES OFVARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS

MATERIALS

- ALLOY COMPOSITION
- SPECIMEN SURFACE FINISH

TEST PROCEDURE

- INSTRUMENTATION
- SCAN RATE
- MAXIMUM APPLIED CURRENT
- TEST ELECTRODE HOLDER
    - DESIGN
    - LOCATION OF ELECTRODE/GASKET INTERFACE

ENVIRONMENTAL

- TEMPERATURE
- SOLUTION COMPOSITION
    - SOLUTION PREPARATION PROCEDURE
    - AERATION
    - SOLUTION VOLUME/SPECIMEN AREA RATIO
    - AUXILIARY ELECTRODE LOCATION

PROPOSED CONDITIONS FOR CNWRA STUDY OF VARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS

APPROACH: TWO-LEVEL FACTORIAL EXPERIMENT DESIGN (FIVE VARIABLES WITH REPLICATION -- 32 TEST COMBINATIONS PLUS 3 REPLICATIONS)

MATERIALS

- ALLOY COMPOSITION -- 1; TYPE 304L SS
- SPECIMEN SURFACE PREPARATION -- 1; 600 GRIT

TEST PROCEDURE

- INSTRUMENTATION -- 1 PAR 273 POTENTIOSTAT
- SCAN RATES -- 2
  - 3.6 V/hr (1.0 mV/sec) -- USED BY DOE
  - 0.6 V/hr (0.17 mV/sec) -- ASTM G-61 STANDARD
- MAXIMUM APPLIED CURRENTS -- 2
  - 5,000 μA -- ASTM G-61 STANDARD
  - 2,500 μA
- TEST ELECTRODE HOLDER
  - DESIGN -- 1; AS IN ASTM G-5 (AREA = 10 cm^2)
  - ELECTRODE/GASKET INTERFACE LOCATIONS -- 2
    - IMMERSED IN SOLUTION (CREVICE PRESENT) -- AS IN ASTM G-5
    - ABOVE SOLUTION (NO CREVICE PRESENT)

(CONTINUED)

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PROPOSED CONDITIONS FOR CNWRA STUDY OF VARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS (CONTINUED)

ENVIRONMENTAL

- TEMPERATURE -- 1; 90°C (USED BY DOE)
- SOLUTION COMPOSITION
  - AERATION -- 1; IN EQUILIBRIUM WITH AIR AT 90°C
  - SOLUTION PREPARATIONS -- 2
    - SIMULATED J-13 WATER; PREPARED IN ACCORDANCE WITH BATTELLE PROCEDURE, BUT WITH pH ADJUSTED WITH CO_2; FINAL Cl^- = 6.5 ppm
    - SIMULATED J-13 WATER; PREPARED IN ACCORDANCE WITH BATTELLE PROCEDURE, i.e., WITH pH ADJUSTED WITH 0.1 N HCl; FINAL $Cl^- \approx$ 26 ppm Cl^-)
  - SOLUTION VOLUME/SPECIMEN AREA RATIO -- 1; 900 ml/10 cm^2
  - AUXILIARY ELECTRODE LOCATIONS -- 2
    - IN CELL WITH TEST ELECTRODE
    - IN SEPARATE COMPARTMENT
- REPLICATIONS -- 3 RANDOMLY SELECTED REPLICATIONS OF 1 OF 32 COMBINATIONS OF VARIABLES

TOTAL TESTS -- 35

TWO-LEVEL FACTORIAL EXPERIMENT DESIGN IN FIVE VARIABLES TO DEFINE VARIATION IN CYCLIC POTENTIODYNAMIC POLARIZATION TESTS

		A^+				A^-			
		B^+		B^-		B^+		B^-	
		C^+	C^-	C^+	C^-	C^+	C^-	C^+	C^-
E^+	D^+								
	D^-								
E^-	D^+								
	D^-								

CODE	VARIABLE	+ CONDITION	- CONDITION
A	SCAN RATE	3.6 V/hr	0.6 V/hr
B	SOLUTION PREPARATION	6.5 PPM Cl^-	32.5 PPM Cl^-
C	ELECTRODE-GASKET	IMMERSED	ABOVE LIQUID
D	MAXIMUM CURRENT	5,000 µA	2,250 µA
E	AUX. ELECTRODE	IN TEST CELL	IN SEPARATE COMPARTMENT

REPLICATIONS -- 3 OF 1 TEST COMBINATION, RANDOMLY SELECTED

TOTAL TESTS -- 35

SUMMARY OF SCREENING TESTS PLANNED FOR CNWRA PROGRAM

- CONDUCT 35-TEST TWO-LEVEL STATISTICAL DESIGNED EXPERIMENT AND ANALYZE RESULTS TO DEFINE AND MINIMIZE VARIATION IN CYCLIC POTENTIO-DYNAMIC POLARIZATION TESTS

- USE OPTIMIZED TEST METHOD AND SMALLER TWO-LEVEL OR OTHER STATISTICALLY-BASED EXPERIMENT DESIGNS:

  - TO EVALUATE EFFECTS OTHER VARIABLES, SUCH AS SURFACE ROUGHNESS, TEMPERATURE, AND EFFECTS OF CREVICES

  - TO EVALUATE ALLOYS NOT INCLUDED IN YMP AND CORTEST PROGRAMS, i.e., TYPE 316L STAINLESS STEEL, CDA 613, AND CNWRA REFERENCE ALLOY HASTELLOY C-22

  - TO EVALUATE SELECTED ALLOYS IN OTHER PERTINENT ENVIRONMENTS, SUCH AS CONCENTRATED SALT SOLUTIONS

  - TO CONDUCT CONFIRMATORY TESTS FOR SELECTED ALLOY-ENVIRONMENT COMBINATIONS STUDIED IN YMP/DOE AND CORTEST PROGRAMS

(CONTINUED)

SUMMARY OF SCREENING TESTS PLANNED FOR CNWRA PROGRAM (CONTINUED)

- MODIFY PROCEDURE AS NECESSARY TO CONDUCT TESTS IN HIGH-LEVEL GAMMA RADIATION FIELDS, AND EVALUATE EFFECTS OF HIGH-LEVEL GAMMA RADIATION FIELDS IN TESTS ON SELECTED ALLOY-ENVIRONMENT COMBINATIONS

- DEVELOP SLOW-STRAIN-RATE SCC TEST PROCEDURE

  - DETERMINE SUSCEPTIBILITY OF CANDIDATE ALLOYS TO SCC IN SELECTED VAPOR AND LIQUID ENVIRONMENTS

  - MODIFY PROCEDURE AS NECESSARY TO CONDUCT TESTS IN HIGH-LEVEL GAMMA RADIATION FIELDS AND EVALUATE EFFECTS OF GAMMA RADIATION ON CANDIDATE ALLOYS IN SELECTED VAPOR AND LIQUID ENVIRONMENTS

SUMMARY OF SHORT-TERM TESTS PLANNED FOR CNWRA PROGRAM

CONFIRMATION OF RESPASSIVATION POTENTIALS FOR SELECTED MATERIALS

OBJECTIVES -- FOR TWO OR THREE ALLOYS INDICATED BY POLARIZATION TESTS TO HAVE THE BEST LOCALIZED CORROSION RESISTANCE:

- INCREASE ACCURACY OF REPASSIVATION POTENTIALS MEASURED IN POTENTIODYNAMIC POLARIZATION TESTS BY EXAMINING EFFECT OF EXPOSURE TIME; AND

- DETERMINE IF THERE ARE DIFFERENCES BETWEEN: THE REPASSIVATION POTENTIAL FOR PITTING ON SMOOTH SURFACES AND THE REPASSIVATION POTENTIAL FOR SPECIMENS UNDERGOING CREVICE CORROSION. (SMOOTH CYLINDRICAL SPECIMENS WITH A COLLAR AROUND THE MIDDLE WILL BE USED FOR CREVICES SPECIMENS).

PROCEDURE -- THE PROCEDURE GIVEN IN ASTM F 746, "STANDARD TEST METHOD FOR PITTING AND CREVICE CORROSION OF METALLIC SURGICAL IMPLANTS," WILL BE FOLLOWED.

- SPECIMENS WILL BE EXPOSED FOR 30 MINUTES AT A POTENTIAL ABOVE THE PITTING POTENTIAL TO INITIATE PITS, AS INDICATED BY HIGH CURRENT VALUE, OR UNTIL A CURRENT 500 $\mu A/cm^2$ IS ACHIEVED.

(CONTINUED)

SUMMARY OF SHORT-TERM TESTS PLANNED FOR CNWRA PROGRAM (CONTINUED)

CONFIRMATION OF RESPASSIVATION POTENTIALS FOR SELECTED MATERIALS (CONTINUED)

- THE POTENTIAL THEN WILL BE REDUCED TO A VALUE 100 TO 150 mV ABOVE THE EXPERIMENTALLY DETERMINED REPASSIVATION POTENTIAL, AND THE CURRENT WILL BE MONITORED FOR 15 MINUTES. REPASSIVATION IS INDICATED BY A RAPID DROP IN CURRENT TO A VALUE CONSISTENT WITH PASSIVITY.

  - IF REPASSIVATION IS NOT ACHIEVED AT THE POTENTIAL SELECTED, THE POTENTIAL WILL BE REDUCED IN 50 mV STEPS UNTIL REPASSIVATION OCCURS.

  - WHEN REPASSIVATION IS ACHIEVED, PITTING WILL BE REINITIATED BY RAISING THE POTENTIAL ABOVE THE PITTING POTENTIAL; AND

    THE PROCESS WILL BE REPEATED. THE REPASSIVATION POTENTIAL WILL BE TAKEN AS THE HIGHEST POTENTIAL AT WHICH CURRENT IS REDUCED TO A LEVEL CONSISTENT WITH PASSIVITY.

SUMMARY OF SHORT-TERM TESTS PLANNED FOR CNWRA PROGRAM (CONTINUED)

DEFINITION OF OCCLUDED CELL ENVIRONMENTS FOR SELECTED MATERIALS

OBJECTIVE: TO DEFINE ENVIRONMENTAL CONDITIONS PRESENT INSIDE OCCLUDED CORROSION CELLS (PITS, CREVICES, AND CRACKS) FOR TWO OR THREE ALLOYS INDICATED BY POLARIZATION TESTS TO HAVE THE BEST LOCALIZED CORROSION RESISTANCE FOR VARIOUS BULK SOLUTIONS.

THE ENVIRONMENTAL CONDITIONS THUS DEFINED THEN WILL BE USED TO DEVELOP LONG-TERM PROPAGATION DATA FOR LOCALIZED CORROSION.

PROCEDURE: A TWO-COMPARTMENT TEST CELL WILL BE USED. THE OCCLUDED CELL WILL BE SIMULATED BY A SMALL-AREA ELECTRODE IN A SMALL VOLUME OF DEAERATED TEST SOLUTION. CONDITIONS AT FREELY EXPOSED SURFACES WILL BE SIMULATED BY A LARGE-AREA ELECTRODE OF THE SAME ALLOY IN A LARGE VOLUME OF AERATED TEST SOLUTION.

THE ELECTRODES WILL BE CONNECTED EXTERNALLY THROUGH A POTENTIOSTAT, WITH THE VOLTAGE DIFFERENCE BETWEEN THE TWO ELECTRODES SET AT ZERO. A POROUS PLUG BETWEEN THE COMPARTMENTS WILL PERMIT IONIC CURRENT FLOW.

(CONTINUED)

SUMMARY OF SHORT-TERM TESTS PLANNED FOR CNWRA PROGRAM (CONTINUED)

DEFINITION OF OCCLUDED CELL ENVIRONMENTS FOR SELECTED MATERIALS (CONTINUED)

THE CURRENT BETWEEN THE ELECTRODES, THEIR INDIVIDUAL POTENTIALS AND THE MIXED POTENTIAL, AND THE pH's IN THE TWO COMPARTMENTS WILL BE MONITORED UNTIL EQUILIBRIUM IS ACHIEVED.

EQUILIBRIUM WILL BE DEFINED AS NO SIGNIFICANT CHANGE IN ANY OF THE MONITORED VALUES OVER A PERIOD OF AT LEAST 24 HOURS. A TEST WILL BE TERMINATED WHEN EQUILIBRIUM HAS BEEN ACHIEVED.

THE SOLUTION IN THE OCCLUDED-CELL COMPARTMENT THEN WILL BE ANALYZED TO DETERMINE ITS CHEMICAL COMPOSITION.

THE pH, POTENTIAL, AND CHEMICAL COMPOSITION THUS DETERMINED WILL BE USED IN LONG-TERM TESTS TO MEASURE PROPAGATION OF LOCALIZED CORROSION.

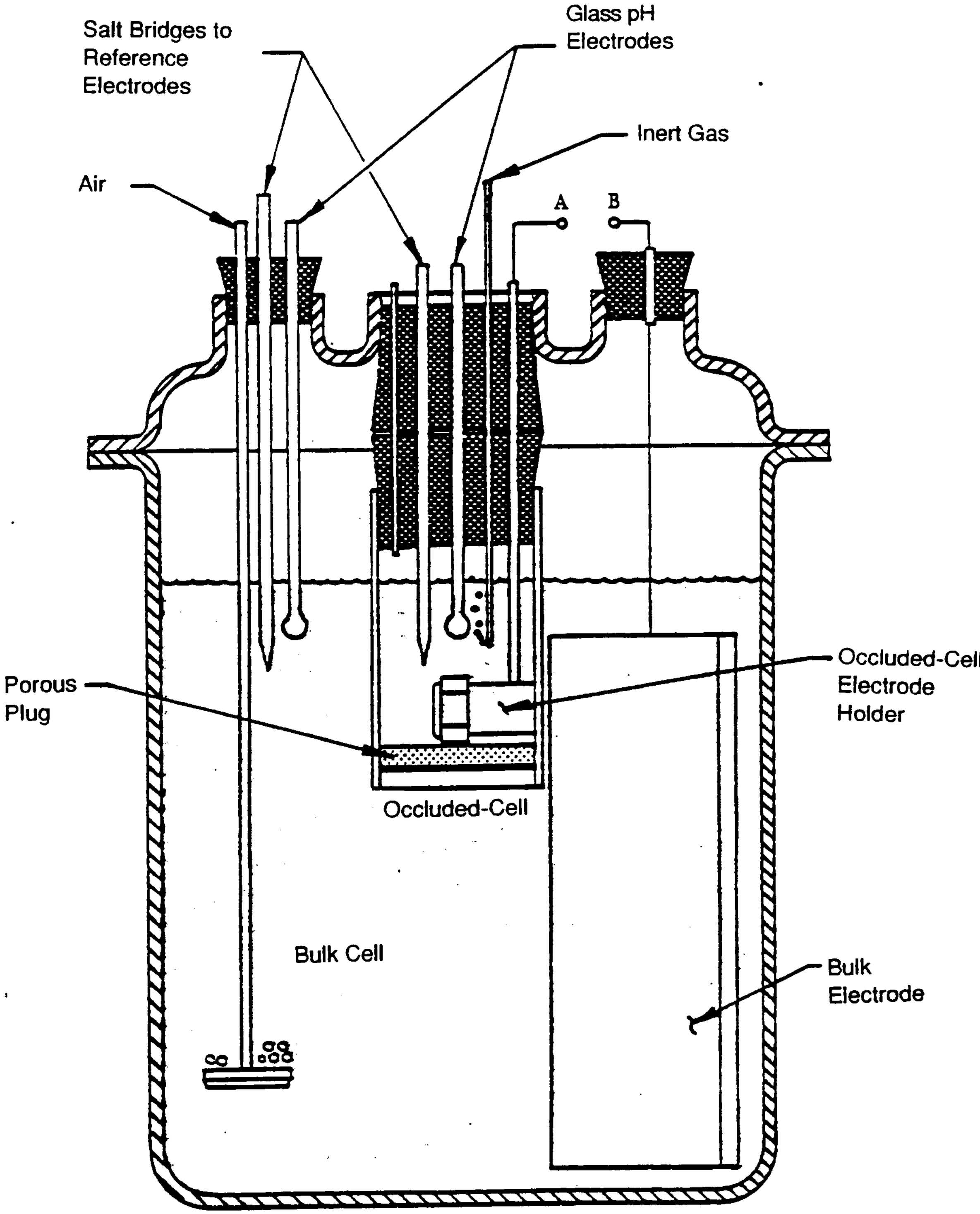

FIGURE 1. SCHEMATIC ILLUSTRATION OF OCCLUDED-CELL TEST CELL