WSRC-TR-93-321

Keywords: Reactors
Water Chemistry
Stainless Steel
Corrosion
PTERM
CAMEL

Retention: Permanent

**REACTOR MATERIALS PROGRAM
ELECTROCHEMICAL POTENTIAL MEASUREMENTS BY ORNL WITH
UNIRRADIATED AND IRRADIATED STAINLESS STEEL SPECIMENS (U)**

by

E. W. Baumann
G. R. Caskey, Jr.

ISSUED: July, 1993

Authorized Derivative Classifier

Date

SRTC

SAVANNAH RIVER TECHNOLOGY CENTER, AIKEN, SC 29808
Westinghouse Savannah River Company
Prepared for the U. S. Department of Energy under Contract DE-AC09-89SR18035

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

875

DOCUMENT: WSRC-TR-93-321

TITLE: REACTOR MATERIALS PROGRAM
ELECTROCHEMICAL POTENTIAL MEASUREMENTS BY
ORNL WITH UNIRRADIATED AND IRRADIATED
STAINLESS STEEL SPECIMENS (U)

APPROVALS

R. L. Sindelar, TECHNICAL REVIEWER
MATERIALS TECHNOLOGY SECTION

DATE: 8-17-93

N. C. Iyer, ACTING MANAGER
MATERIALS APPLICATIONS & CORROSION
MATERIALS TECHNOLOGY SECTION

DATE: 8/17/93.

T. L. Capeletti, MANAGER
MATERIALS TECHNOLOGY SECTION

DATE: 8/17/93

INTRODUCTION

One of the primary differences between the Type 304 Stainless Steel of SRS reactor tanks and that of the piping of the primary system is the result of neutron irradiation. Irradiation of the stainless steel alters the mechanical properties and microstructure and also causes activation of the material.

For the Reactor Materials Program^[1], specimens of stainless steel were made from R-Reactor piping material for corrosion and mechanical testing. Some of the specimens underwent reactor irradiations to simulate conditions of the reactor tank stainless steel.

Mechanical testing of the specimens has established that the continued safe operation of the reactors is not limited by radiation-induced degradation of the mechanical properties of the tank material.^[2,3] Constant Extension Rate Tensile (CERT) tests have indicated that the susceptibility to Intergranular Stress Corrosion Cracking (IGSCC) was not significantly different for irradiated specimens.^[4,5] However, neutron activation produces gamma emitters such as Co-60 that cause changes in the aqueous environment of the stainless steel, through radiolysis. This effect was not explored in the previous work.

One monitor of this change in environment is the electrochemical potential (ECP) of the stainless steel. A final task of the Reactor Materials Program was to compare the ECP values of the unirradiated and irradiated stainless steel specimens in an aqueous environment typical of reactor moderator.^[6] These experiments were planned to supplement previous work in identifying effects of the irradiation of stainless steel on corrosion susceptibility.

ECP measurements with unirradiated and irradiated specimens have been completed by Oak Ridge National Laboratory (ORNL). Unfortunately, the experiments were limited by budgetary constraints, and the data must be regarded as preliminary. However, the experiments provide a starting point for a more thorough study of corrosion of activated stainless steel at some future time. The purpose of this memorandum is to transmit the ORNL results and to place them in the context of other ECP measurements made in the Reactor Materials Program.

SUMMARY AND RECOMMENDATIONS

The effect of irradiation of stainless steel on the Electrochemical Potential (ECP) was investigated by measurements in dilute nitric acid and hydrogen peroxide solutions, conditions simulating reactor moderator. The electrodes were small disks made from [unirradiated or irradiated, unsensitized or sensitized] specimens from R-reactor piping, prepared as part of the Reactor Materials Program. The results were inconclusive because of experimental anomalies that could not be resolved because of budgetary restrictions. Also, it was concluded that the dose rate from the specimens may have been too small to produce a significant radiolytic effect.

A comprehensive description of the effects of irradiation on corrosion susceptibility continues to be open. However, neither the earlier CERT corrosion susceptibility tests nor the present ECP measurements revealed a pronounced effect of irradiation on the susceptibility of the stainless steel to IGSCC. This benign behavior should be remarked in view of the absence of IGSCC in the stainless steel of the SRS reactor tanks, except for the knuckle area of C-Reactor tank. The knuckle was sensitized by hot working, in contrast to normal sensitization levels associated with fusion welding to join plates in the tank walls.

The availability of a matched set of irradiated/unirradiated sensitized/unsensitized CERT specimens provides a unique opportunity for comparison of ECP values through structured experiments. Future experiments should include the following:

- Provision for using significantly larger stainless steel electrodes, to provide a radiation dose rate of 10^6 to 10^7 R/hr.
- Chemical analysis of all solutions for oxygen, nitrate, hydrogen peroxide, conductivity, and pH.
- Electrochemical instrumentation and electrode construction that is demonstrably reliable.
- Measurements of irradiated and unirradiated specimens made in separate vessels.

DISCUSSION

BACKGROUND

Reactor Materials Program

Stainless steel specimens for mechanical and corrosion testing in the Reactor Materials Program were prepared from stainless steel taken from R-Reactor piping.^[7] This provided 1950's vintage Type 304 Stainless Steel representative of the material of construction of SRS reactors. Some specimens were subjected to neutron irradiation in the ORNL High Flux Isotopes Reactor (HFIR) to exposures that simulated SRS reactor service. Tests of corrosion susceptibility were made with unirradiated and irradiated specimens. Electrodes used in all previous ECP measurements were made from unirradiated, unsensitized specimen stainless steel.

ECP measurements were an integral part of the experiments performed for the Reactor Materials Program by General Electric Nuclear Energy, San Jose, CA. The effect of the aqueous environment on susceptibility to Intergranular Stress Corrosion Cracking (IGSCC) was investigated with statistically designed Constant Extension Rate Tensile (CERT) tests performed in solutions that simulated SRS conditions. Some of the test solutions included hydrogen peroxide (H_2O_2), which is a primary product from the gamma radiolysis of water. One set of tests used unirradiated CERT specimens,^[4] and the other set used irradiated CERT specimens.^[5] The same model equation described IGSCC susceptibility for both unirradiated and irradiated specimens.

The results from the CERT model studies demonstrated that H_2O_2 was a key parameter in the corrosion of stainless steel. The model showed that the presence of H_2O_2 increased IGSCC susceptibility, but there was no dependence on concentration in the range from 400 to 5000 ppb. The presence of H_2O_2 did not affect IGSCC at chloride concentrations <11 ppb.

Online ECP measurements were made in K-Reactor during more than a year of reactor operation.^[8] The electrodes were located in a loop external to the tank and upstream of the heat exchangers, only a few seconds out of the reactor. The ECP responded to startup, shutdown, and discharging/charging operations. The observed ECP values were in agreement with the values observed during the CERT tests that contained H_2O_2 .

Role of Irradiation

The indigenous gamma field of the irradiated stainless steel constitutes a difference between the environment of the reactor vessel stainless steel and the environment of the piping. The presence of a gamma field, although at a lower level, continues even when the reactor is shut down. Whether this environmental difference has significance in terms of corrosion susceptibility has not been established with certainty.

The gamma radiation from neutron activation products, such as Co-60, causes radiolysis of water with production of many species, including short-lived, highly reactive free radicals like H and OH and the primary radiolysis products H_2 and H_2O_2 . The localized production of these species at irradiated stainless steel surfaces may affect corrosion susceptibility.

The primary electroactive species from the radiolysis is H_2O_2 . Addition of H_2O_2 in the solution increases the ECP of stainless steel and decreases that of platinum.^[9] The effect, if any, of free radicals on the ECP has not been identified because the short half-lives have eliminated them from the previous experiments. For the online ECP measurements in K-Reactor moderator, which were made outside the reactor tank, deuterium peroxide (D_2O_2), but not free radicals, would have been present. For the CERT tests the primary effect of radiation on the system was simulated by addition of H_2O_2 to the solutions.

The CERT tests explored the effects of neutron exposure as well as aqueous environment on IGSCC susceptibility of the stainless steel. Figure 1 shows the %IGSCC and the ECP for unirradiated and irradiated specimens. The CERT test results for the irradiated specimens would include effects, if any, on the metal condition from neutron exposure and on the aqueous environment from the induced gamma field. It is noted, however, that the accompanying ECP values were measured with electrodes of unirradiated, unsensitized stainless steel and hence reflected only the aqueous environment and not the metal condition. The results show the expected trend to higher %IGSCC at higher temperatures, but show no pronounced differences between the unirradiated and the irradiated specimens. It is interesting, but perhaps not significant, that none of the four irradiated specimens showed 100% IGSCC at 105°C, whereas most of the unirradiated specimens did. Also, the two irradiated unsensitized specimens showed 0% IGSCC, i.e., ductile failure, at 105°C.

Experimental Verification

Both the online K-Reactor and the CERT ECP measurements used stainless steel specimens that were neither sensitized nor irradiated, conditions that do not match reactor service. Detecting the effect on ECP (and hence on corrosion) from the localized radiation environment and from sensitization was the focus of the ORNL experiments.

Irradiated and unirradiated, sensitized and unsensitized specimens from the Reactor Materials Program were available, and ORNL had the capability for making ECP measurements with radioactive specimens. Therefore scoping studies to compare ECP values of these four selected specimens in simulated SRS reactor environment were contracted with ORNL.^[6]

ORNL ECP MEASUREMENTS

Experimental Details

Details of the procedure, apparatus, and instrumentation are given in the Attachments 1 and 2. The set of experiments shown in Table 1 was designed to compare ECP of irradiated and unirradiated specimens in different aqueous environments. Experiments were made with deaerated and aerated water. After the initial measurement, the water was sequentially spiked for further measurements: first to produce 10 micromolar nitric acid, and then to produce 5 ppm hydrogen peroxide in addition to the nitric acid. The ECP measurements were made at ambient temperature.

The four stainless steel CERT specimens are described in Table 2. Disks fabricated from these specimens for the ECP measurements were made as small as practical to minimize radiation exposure. Flat disks, 3 mm diameter and 0.25 mm thick, such as used for Transmission Electron Microscopy were fabricated. The discs had radiation dose rates of 800 to 900 mR/hour at Cutie Pie contact. The calculated contact dose rate is between 10^3 and 10^4 R/hour.

The discs were electropolished and then mounted in a holder, Figure 2, with an exposed area of 3 mm². The holders were then placed in the five-neck flask shown in Figure 3. The photograph shows only one holder in the flask. All four specimens, unirradiated and irradiated, were present in the flask during the measurements, as shown in Figure 4. The specimens were about 5 cm apart, and the irradiated specimens were in the center positions. The saturated calomel electrode and the platinum electrode were inserted at the sparge tube position.

Absolute electrochemical potentials were measured versus a saturated calomel electrode. The results were converted to the Standard Hydrogen Electrode (SHE) reference, the standard method of reporting ECP values, by adding 241 mV to the observed reading.

Results

As described in Attachment 1, the first two sets of results were questionable. The results of the third set are presented in Table 3 and in Figures 5 and 6. Selected data from the CERT tests are included for comparison.

The ECP values for the stainless steel, Figure 5, showed no strong trends among the specimens or between deaerated and aerated solutions. In the six sets of measurements, the irradiated sensitized specimen 2A85 had the highest ECP in five sets and the unirradiated sensitized specimen 2A22 had the lowest in four sets. Some higher ECP values (more oxidizing conditions) are evident for the deaerated system that contained nitrate and peroxide. The aerated systems with additions had lower potentials than the deaerated systems. Potentials are in the general range of those reported from the CERT tests.

The ECP values for the platinum, Figure 6, were higher for the deaerated solution than for the aerated solution, and were not changed significantly by the nitrate and peroxide additions. Addition of H_2O_2 did not produce the expected lowering of the potential^[9], seen in the CERT tests.

Discussion of Results

Unirradiated vs. Irradiated Specimens

The dose rate from the small specimens used may not have been high enough to cause significant radiolytic effects.

The effect on the overall aqueous environment of having all specimens in the same container was considered because general radiolytic production of H_2O_2 could affect all ECP values. The dose rate at 5 cm is reduced substantially below the estimated contact dose rate of 10^3 to 10^4 R/hour. Even the contact dose rate may be too low to produce much peroxide. According to previous studies of gamma radiolysis of micromolar nitric acid solutions^[10], a dose rate of 10^6 R/hour produced only about 0.1 ppm H_2O_2 , independent of total dose up to 5×10^6 R. Analyses of the final solution from the aerated ORNL experiments showed 3.6 and 5.5 ppm H_2O_2 in duplicate samples, comparable to the expected 5 ppm. This confirmed that H_2O_2 was not building up in the bulk solution.

The ORNL experimental conditions were compared with those of Clarke^[11] who found that the ECP of stainless steel increased in the presence of a gamma radiation field. In experiments in oxygenated water at power reactor temperatures (288°C), the potential of a stainless steel electrode in water increased when a radiation source approached within 1.5 inches. The radiation source was the broken half of a CERT specimen made of Type 304 Stainless Steel that had received a fluence ($E_n > 1$ MeV) of 3×10^{21} n/cm² in power reactor service. This fluence is approximately three times that estimated for the HFIR irradiated specimens, Table 2. The dimensions of the broken CERT specimen were estimated to be 0.5 in. x 0.5 in. x 0.1 in.

The relative doses in the Clarke and the ORNL experiments were compared through the proportionality relationship:

$$\text{Dose} \propto (\text{volume of source})(\text{neutron exposure of source})/(\text{distance})^2$$

It was assumed that decay times were the same for both specimens. The neutron energy spectrum of the HFIR is similar to power reactors, and a difference in decay times of as much as 5 years (approximately one half-life of Co-60) would not be significant for this estimate. With these assumptions, the contact dose rate of the Clarke specimen was calculated to be about 700 times that of the ORNL specimens.

Dose rates were compared for the Clarke threshold (1.5 inches) and the distance between specimens for the CRNL configuration (5 cm). The calculations showed that the estimated dose rate received by the unirradiated specimens from the irradiated specimens in the ORNL configuration was about 1000 times less than the threshold at 1.5 inches found in the Clarke experiments. The threshold dose rate to give a measurable ECP change, from the Clarke experiments, is lower than the dose rate where measurable buildup of peroxide concentration was found to occur in earlier experiments.^[10]

Another difference between the unirradiated and the irradiated specimens would be the localized effect at the surface. Again, the estimated dose rate of 10^3 to 10^4 R/hour may not have been high enough to cause significant radiolysis, even at the surface. The larger irradiated specimens used for the CERT tests, but not for the accompanying ECP measurements, were reported to have dose rates "on contact" (Cutie Pie?) between 90 and 150 R/hour.^[5]

Aerated or Deaerated Condition

Comparison with CERT test results indicates that oxygen may have been present in the ORNL "deaerated" solutions.

The ECP of the platinum electrode in the ORNL experiments, Figure 6, showed no significant difference between the initial aerated and deaerated waters. This apparent anomaly was reviewed by comparing the results with the ECP values from the CERT tests^[4], which are shown in Figure 7 as a function of oxygen concentration. The low potentials at 5 ppb O_2 were measured in solutions that contained neither nitrate nor peroxide. The other CERT values are for peroxide-free solutions with nitrate and oxygen plus small concentrations of chloride and other ions. Temperatures varied from 40 to 105°C.

The initial deaerated water of the ORNL experiment would be expected to exhibit ECP values similar to that of the 5 ppb oxygen solution from the CERT testing. However, high potentials of the platinum electrode were observed in the initial deaerated waters, as well as similar high potentials after nitric acid and hydrogen peroxide were added. This behavior suggests that oxygen was present in the deaerated solutions of the ORNL tests.

pH of Solution

Lack of knowledge about the pH trends in the test solutions contributes to the uncertainty about the significance of the results.

The pH of 10 micromolar nitric acid is 5.0, but the aerated water analyzed after the final measurements showed a pH of 8.3. This unexpectedly high pH indicates that the solution analyzed did not contain nitric acid, although nitrate salts may well have been

present. The dilute unbuffered acid of the test solutions can be easily neutralized by tramp impurities or leaching.

Whether the pH change occurred before or after the sample was taken for analysis is not known. Higher pH would decrease the ECP, according to Pourbaix^[12]; acid nitrate solutions are more oxidizing than neutral or alkaline nitrate solutions. A higher pH in the aerated solutions could be responsible for the observed potential values, which were unexpectedly lower than those for the deaerated solutions.

Conclusions

The ORNL measurements failed to provide definitive results, although some differences among the specimens and between the conditions are evident. While the experimental ECP measurements were simple in principle, implementation was complicated by the radioactivity, which required miniaturization of the specimens. The dose rate may have been reduced to a level too low for detectable radiation effects. Also, the compositions of the dilute solutions required for the experiment, which are difficult to maintain, needed confirmation by periodic analyses throughout the experiments. Experimental difficulties were compounded by limited resources that forced shortcuts in experimentation and precluded detailed trouble-shooting of the measurements themselves.

REFERENCES

1. N. G. Awadalla, G. R. Caskey, and R. S. Ondrejcin, Reactor Materials Program, DPST-84-820 (December 1984).
2. R. L. Sindelar and G. R. Caskey, Reactor Materials Program. Mechanical Properties of Irradiated Types 304 and 304L Stainless Steel Weldment Components(U), WSRC-TR-91-11 (December 1991).
3. R. L. Sindelar, Reactor Materials Program. Microstructural and Mechanical Response of Types 304, 304L, and 308 Stainless Steels to Low Temperature Neutron Irradiation(U), WSRC-TR-93-196 (June 1993).
4. R. S. Ondrejcin and G. R. Caskey, Assessment of Susceptibility of Type 304 Stainless Steel to Intergranular Stress Corrosion Cracking in Simulated Savannah River Reactor Environments(U), WSRC-RP-90-1230 (December 1, 1989).
5. G. R. Caskey, R. S. Ondrejcin, R. L. Sindelar, and E. W. Baumann, Effect of Neutron Irradiation on Stress Corrosion Cracking of Type 304 Stainless Steel in Simulated Savannah River Reactor Environments, WSRC-TR-91-12 (January 1991).
6. Reactor Materials Program. Specification SRP-SL-1111, Revision 2. Specification for Shipping, Testing, and Analysis of Irradiated Stainless Steel Mechanical and Corrosion Specimens (Retained in NRTSC Task 90-023-1 files) (October 31, 1991)
7. K. J. Stoner, Reactor Materials Program. Materials Source History for Type 304 Stainless Steel Testing Program, DPST-88-1010 (December 1988).
8. E. W. Baumann and R. S. Ondrejcin, Electrochemical Potentials of Stainless Steel and Platinum in K-Reactor Moderator, DPST-88-621 (July 6, 1988).
9. B. M. Gordon and H. S. Mehta, Stress Corrosion Cracking Literature Review and Analysis for the Savannah River Plant, General Electric NEDC-30837. pp 2-16 ff (December 1984).
10. E. W. Baumann, Chemical Effects in Micromolar Nitric Acid from Gamma Radiation and Heated Aluminum. DP-1205 (January 1970).
11. W. L. Clarke and A. J. Jacobs, Effect of Radiation Environment on SSC of Austenitic Materials, Proceedings of the International Symposium on Environmental Degradation of Materials in Nuclear Power Systems--Water Reactors, National Association of Corrosion Engineers, Myrtle Beach, SC, p 451. (August 22-25, 1983).
12. M. Pourbaix, Atlas of Electrochemical Equilibria in Aqueous Solutions (Translated from the French). Houston, National Association of Corrosion Engineers (1974).

TABLE 1
DESCRIPTION OF EXPERIMENT

Measure potential of each of the 4 ECP specimens versus saturated calomel electrode in:

- Solution (1): Aerated or Deaerated Water
- Solution (2): Solution (1) + 10^{-3}M HNO_3 to produce 10^{-5}M HNO_3
- Solution (3): Solution (2) + 0.15% H_2O_2 to produce 5 ppm H_2O_2

TABLE 2
DESCRIPTION OF ECP SPECIMENS

Specimen	Irradiated ^a	Sensitized ^b
2A6	yes	no
2A7	no	no
2A22	no	yes
2A85	yes	yes

a From HFIR(4C) irradiations[5]:

- Fast Fluence ($E_n > 0.1 \text{ MeV}$) = $2.5 \times 10^{21} \text{ n/cm}^2$
- Fast Fluence ($E_n > 1 \text{ MeV}$) = $9.8 \times 10^{20} \text{ n/cm}^2$
- Thermal Fluence = $3.9 \times 10^{21} \text{ n/cm}^2$

b Solution annealed, heat treated at 600°C for 15 hours, furnace cooled.

TABLE 3
SUMMARY OF RESULTS

WSRC-TR-93-321

SOURCE	SPECIMEN	T,°C	uMolar HNO3	FPB H2O2	FPB O2(a)	FPB CL	FPB SO4	pH	mV ECP(SS)	mV ECP(Pt)
<u>No Nitrate or Peroxide</u>										
GE/CERT	--	80	0	0	5	0	0	--	20-30	-300
ORNL	2A7	~35	0	0	0	0	0	--	295	721
(deaerated)	2A6*	~35	0	0	0	0	0	--	292	
	2A22+	~35	0	0	0	0	0	--	227	
	2A85*+	~35	0	0	0	0	0	--	363	
ORNL	2A7	~35	0	0	8000	0	0	--	328	677
(aerated)	2A6*	~35	0	0	8000	0	0	--	269	
	2A22+	~35	0	0	8000	0	0	--	361	
	2A85*+	~35	0	0	8000	0	0	--	416	
<u>Nitrate Only</u>										
ORNL	2A7	~35	10	0	0	0	0	--	322	711
(deaerated)	2A6*	~35	10	0	0	0	0	--	322	
	2A22+	~35	10	0	0	0	0	--	297	
	2A85*+	~35	10	0	0	0	0	--	392	
GE/CERT	B2 38	55	10	0	244	8	17	4.9-5.3	258	600
GE/CERT	2A 98	55	10	0	3755	8	17	4.9-5.3	391	679
ORNL	2A7	~35	10	0	8000	0	0	--	361	
(aerated)	2A6*	~35	10	0	8000	0	0	--	294	
	2A22+	~35	10	0	8000	0	0	--	164	
	2A85*+	~35	10	0	8000	0	0	--	352	
<u>Peroxide Only</u>										
GE/CERT	--	80	0	100	100	30	50	5.6	411-560	314-449
<u>Nitrate and Peroxide</u>										
ORNL	2A7	~35	10	5000	0	0	0	--	406	667
(deaerated)	2A6*	~35	10	5000	0	0	0	--	511	
	2A22+	~35	10	5000	0	0	0	--	396	
	2A85*+	~35	10	5000	0	0	0	--	446	
GE/CERT	D2 24	55	10	845	62	8	17	4.9-5.3	444	503
GE/CERT	D4	55	10	3703	129	8	17	4.9-5.3	577	497
ORNL	2A7	~35	10	4550	8000	0	0	8.32	413	
(aerated)	2A6*	~35	10	4550	8000	0	0	8.32	299	
	2A22+	~35	10	4550	8000	0	0	8.32	290	
	2A85*+	~35	10	4550	8000	0	0	8.32	422	

a. Assume deaerated = 0 ppb O2; aerated = 8000 ppb O2
ppb=parts per billion

*=irradiated material
+=sensitized material

FIGURE 1. ECP of 304 SS and % IGSCC from Unirradiated and Irradiated CERT Tests

Holder with specimen in place (at right)

Placing specimen in holder (below)

FIGURE 2. Photograph Showing Placement of Specimen in Holder

FIGURE 3. Photograph of Five-Neck Flask Test Cell
(Specimen holder in place)

- *Electrodes about 5 cm apart*
- *Saturated Calomel Electrode in sparge tube position*
- *Platinum wire about 10 mils diameter wedged in hole with Saturated Calomel Electrode*

FIGURE 4. Configuration of Electrodes in Flask
(Top view; not to scale)

FIGURE 5. Electrochemical Potentials of Stainless Steel in Nitrate and Peroxide Solutions (Temperature 35 to 55°C)

FIGURE 6. Electrochemical Potentials of Platinum in Nitrate and Peroxide Solutions
(Temperature 35 to 55°C)

FIGURE 7. ECP of SS and PT in Oxygenated Solutions
(ca. 10 μ M HNO₃; 8-68 ppb Cl)

ATTACHMENT 1. Description and Results of ORNL Requirements

- 1.1 Letter S. J. Pawel to R. L. Sindelar, Differential Electrochemical Potential Measurements, April 2, 1993
- 1.2 Sketch of configuration in measurement cell
- 1.3 Analysis results

MARTIN MARIETTA ENERGY SYSTEMS, INC.POST OFFICE BOX 2009
OAK RIDGE, TENNESSEE 37831

April 2, 1993

Mr. Robert Sindelar
Westinghouse Savannah River Company
Savannah River Site
P.O. Box 616
Aiken, South Carolina 29802

Differential Electrochemical Potential Measurements

Dear Bob:

In phone conversations on March 17 and 19, Graham Bell discussed with you several unexpected difficulties in collecting the differential electrochemical potential (dECP) data for irradiated and unirradiated stainless steel specimens with which we were working (Task 89-023-G-1). As you know, we consumed the available time and funding for this effort during our attempts to overcome these difficulties. The purpose of this correspondence is to document the tests attempted and the results obtained to serve as a starting point for continued effort at some time in the future.

Experimental. All TEM-sized specimens were briefly polished (5 sec at 5 v in a solution near 90% acetic acid and 10% perchloric acid) prior to loading into acrylic specimen holders designed to expose a well-defined 3 mm² surface area. The specimens were then immediately loaded into the 5-neck cell containing a known volume of aerated water. The water was obtained from a purifying system delivering demineralized and deionized water of resistivity 14-18 Mohm-cm at the tap. The water was allowed to stand in a loosely-capped sterilized polyethylene container almost 48 h in order to aerate.

All four specimens were exposed together in the test cell. Each pair of specimens (2A6/2A7 and 2A85/2A22) were positioned such that the exposed specimen surface faced the other member of the pair for which dECP measurements were to be made. As a result of using the 5-neck cell, this procedure placed the specimen pairs roughly 3-4 cm apart during the dECP measurements. For each dECP measurement, the unirradiated specimen was made the working electrode and the irradiated specimen the counter electrode. An EG&G Model 273 potentiostat was used as a galvanostat to fix the net current flow between specimens at zero. With no net current flow, a Keithley 610C electrometer was then used to make dECP measurements free of an iR drop.

Following completion of the dECP measurements in aerated water, a sufficient volume of 0.001 M nitric acid was added to the water to generate a 0.00001 M nitric acid solution. The dECP measurement procedure was then repeated for each pair of specimens. Subsequently, a sufficient volume of 0.15% hydrogen peroxide was added to the mixture to generate a 0.00001 M nitric acid solution containing 5 ppm hydrogen peroxide and the dECP measurements were repeated. At this point, specimens of the test solution were collected for analysis. Next, the specimen holders were removed, rinsed with water, and placed directly in another cell containing 14 Mohm-cm water and deaerated with helium for two hours. Again, dECP data was collected for each pair of specimens.

Attachment 1

Results. The results of the first set of dECP measurements are summarized in Table 1. Figure 1 is representative of the dECP value as a function of time as recorded by the EG&G unit for the aerated solutions. In the deaerated solutions, a well-defined value for dECP was not established after 120 s; Figure 2 is representative of this observation.

Table 1. Summary of dECP values measured with Keithley 610C electrometer with the unirradiated specimen the working (positive lead) electrode. Galvanostat set at 0.000 microamperes net current.

	<u>2A7(+) vs 2A6</u>	<u>2A22(+) vs 2A85</u>
(1) aerated water	+234 mv	-145 mv
(2) addition to (1) to make 0.00001 M nitric acid	+145 mv	-170 mv
(3) addition to (2) to make 5 ppm hydrogen peroxide	+220 mv	-155 mv
(4) deaerated water	+210 mv	-215 mv

The dECP values in Table 1 do not follow anticipated trends. Of particular note, the unirradiated specimen exhibited the higher potential in one set of experiments (2A7 vs 2A6) and the lower potential in the other set (2A22 vs 2A85). Further, solution additions expected to change the oxidizing strength of the environment did not result in regular changes in the dECP values. Early in the test set-up, the potential of each specimen in an acrylic holder was measured against a platinum wire in aerated water. The platinum was a steady +240 mv to specimen 2A6; the platinum was near +50 mv and rising steadily (over a 30 second wait or so) compared to the other three specimens. At the time this measurement was completed, it was deemed that this was sufficient indication that the specimen holders had not leaked. If leaking were significant, it was envisioned (based on our experience in more conductive solutions) that the potential difference between platinum and each specimen would be very nearly zero since a leak would expose the platinum lead wires. However, the results in Table 1 suggested that, even if the specimen holders were not leaking at the beginning of the experiment, there was reason to suspect some leakers at the conclusion of the day's effort.

As a result of this concern, a saturated calomel electrode (SCE) was used to measure the absolute potential of each stainless steel specimen. These values (Table 2) indicated that for three of the specimens, the potential of the stainless steel was inordinately high, suggesting a leak exposing the platinum wire and generating a "mixed potential" of stainless steel and platinum. Only specimen 2A6 indicated a potential similar to 2A9 (known to be free of leaks or mixed potential value).

Table 2. Absolute measurement of potential of stainless steel (positive lead) measured against saturated calomel electrode in deaerated water. Corresponding values for similar stainless steel (2A9) in situation known to be free of leaks and bare platinum included for comparison.

platinum	+480 mv
annealed 2A9	-115 mv
sensitized 2A9	-060 mv
2A7	+153 mv
2A6	-115 mv
2A22	+087 mv
2A85	+340 mv

Potential measurements for each TEM specimen (undisturbed in aerated water overnight) were repeated the following morning and results confirmed the values given in Table 2. Repeated attempts to reseal the acrylic specimen holders did not improve this situation (although it is curious that no obvious leaking was evident).

It appears that several factors may have contributed to leaking specimen holders, the most obvious of which was that both of the irradiated specimens had a "knick" on the edge that compromised the sealing ability of the holder. In addition, the unirradiated specimens were originally too thick for the holders and these were mechanically ground in an attempt to thin them by 50% or more. This was performed using a special wax to affix the specimens to a large polishing block, but specimen flatness after this procedure is questionable.

Revised System/Results. An attempt was made to remount the samples to eliminate leaking and the resulting mixed (or distorted) potentials. Polished stainless steel in epoxy mounts (with a contact lead extending from the rear of the large block of epoxy) was used as the base for the makeshift specimen holders. Each TEM specimen was affixed to the large polished stainless steel surface with conducting silver paint. After a short period for drying, a non-conducting lacquer was used to cover the exposed stainless steel from the base mount and the extreme edges of the TEM specimen. A resistance probe was used to confirm a low resistance contact joint for the TEM specimen so mounted.

The mounts were hung in the test solutions with the solution line intersecting the epoxy mount such that only the TEM specimen (not the contact lead) was exposed to solution. Further attempts to

make dECP measurements were hindered by the fact that the Keithley electrometer began to malfunction (and/or was susceptible to interference). At this point in the testing, use of the Keithley electrometer was discontinued and the EG&G 273 was used to make all potential measurements.

Attempts to make dECP measurements with the "modified" specimen holders proved unsuccessful in the test solutions of interest. The EG&G unit was incapable of control as a galvanostat to generate zero (0.000 microamperes) net current flow; current overload indications on the instrument panel suggest a current (or voltage) larger than the capability of the EG&G was required. As a result, it was determined to collect absolute potential measurements vs SCE. Table 3 contains this data. This data, with few exceptions, conforms to the trends expected for changes in potential with oxidizing additions to the solution.

Table 3. Absolute potential measurements (mv) for stainless steel (working electrode) vs saturated calomel reference electrode as measured with EG&G 273 potentiostat. In a few cases, values reported are mean values for an unsteady potential that varies by several mv in each direction.

	<u>2A7</u>	<u>2A6</u>	<u>2A22</u>	<u>2A85</u>	<u>Pt</u>
(1) aerated water	+087	+028	+120	+175	+436
(2) addition to (1) to make 0.00001 M nitric acid	+120	+053	-077	+111	
(3) addition to (2) to make 5 ppm hydrogen peroxide	+172	+058	+049	+181	
(4) deaerated water	+054	+051	-014	+122	+480
(5) addition to (4) to make 0.00001 M nitric acid	+081	+081	+056	+151	+470
(6) addition to (5) to make 5 ppm	+165	+270	+155	+205	+426

Even increasing the net current allowance of the galvanostat setting to 10 microamperes did not improve the situation. It was found, however, that increasing the conductivity of the solution (we "randomly" chose to increase the nitric acid concentration by a factor of 5-6) allowed the EG&G to gain control at a net current setting of 2.5 microamperes. Figure 3 records the dECP values for each pair of materials as a function of time during this experiment; clearly, a "control" situation was eventually established but the dECP value of 900-1500 mv is an extreme value inconsistent with experience.

Our cumulative effort to this point consumed the available funds for the task and, based on Graham Bell's phone conversations with you, this document was prepared for the Project File. We are disappointed that we were unable to generate more complete results. Perhaps, at some time in the future, we could use these results as a starting point and study this system much more thoroughly.

Please do not hesitate to contact me if you have questions.

Sincerely,

Steven J. Pawel

SJP:gsn

Enclosures: 1. Fig. 1
2. Fig. 3
3. Fig. 3

c/encs: A. M. Ammons
G. E. C. Bell
J. R. DiStefano
W. G. Northcutt, Jr.
G. M. Slaughter
D. F. Wilson
File - SJP - NoRC

Fig. 2. Representative dECP vs time curves for deaerated solutions using acrylic specimen holder and 0.000 microampere galvanostat setting.

Fig. 3. Differential ECP data using revised specimen holder arrangement and galvanostat setting of 2.5 microamperes.

for measurements utilizing a saturated calomel electrode, the sparge tube was removed and temporarily replaced with an SCE. When a platinum wire was utilized, it was of small diameter (if memory serves, 10 mils thick) and wedged in the hole holding the SCE (if present). Potential measurements on 2A9 specimens made by temporarily removing 2A7 specimen from this arrangement.

Oak Ridge National Laboratory
Analytical Chemistry Division
Results of Analyses
2026 Radiochemical Analysis

IPA6983

Page

Customer Name
Request Number
Frequency
Sample Matrix

PAWEL
IPA6983

Date Received
Charge Number
Series
Date of Report

17-Mar-1993 17:13
34700894

Approved By J. L. Bell
Date 4/20/93

Customer ID
CONTROL 1

Analy No.	930317-089	H2O2
		0.195
		IN

CONTROL 2

Analy No.	930317-090	H2O2
		0.10
		IN

SAMPLE A

Analy No.	930317-091	H2O2	PH
		0.00055	8.44
		IN	

SAMPLE B

Analy No.	930317-092	H2O2	PH
		0.00036	8.21
		IN	

Each of these "control" specimens was taken from the same master batch and were intended to be 0.15% H₂O₂

each of these are "identical" specimens taken after aerated tests (after addn of nitric acid and peroxide to aim values of 10 μ M HNO₃ and 5ppm H₂O₂)

5ppm H₂O₂ = .0005%

These indicate no significant H₂O₂ generation (at least as measured by bulk analysis).

End of Data for Request IPA6983

Total Pages = 1

Printed Using Format: 8

Cust. Copy File Copy

ATTACHMENT 2. ORNL Standard Operating Guidelines

- 2.1 Standard Operating Procedure for Electrochemical Testing of Irradiated Materials**
- 2.2 Electrochemical Testing in Aqueous Environments**

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 1 of 22

STANDARD OPERATING GUIDELINES

Title: Standard Operating Procedure for Electrochemical Testing of Irradiated Materials

Prepared by:

G. E. C. Bell
G. E. C. Bell, Staff Member

Date

08 JUN 92

Reviewed by:

J. R. DiStefano
J. R. DiStefano

Group Leader, Corrosion Science and Technology

Date

6/19/92

A. F. Rowcliffe
A. F. Rowcliffe

Group Leader, Structural Materials

Date

6-20-92

L. T. Gibson
L. T. Gibson

Laboratory Supervisor

Date

6/9/92

M. C. Vande
M. C. Vande
QAS M&C

Date

6/16/92

C. A. Maples
C. A. Maples
M&C DSO and RSO

Date

6/16/92

Paul E. Col
Area Health Physicist

Date

6-19-92

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 2 of 22

STANDARD OPERATING GUIDELINES

Title: Standard Operating Procedure for Electrochemical Testing of Irradiated Materials

1. SCOPE/PURPOSE

Electrochemical (a.k.a. e'chem) testing techniques can be used to give insight into corrosion and other chemical reactions which involve charge exchange. This document gives guidelines for safety and precautions to be used making absolute and differential electrochemical corrosion potential (AECF and DECP) and single-loop electrochemical potentiokinetic reactivation (SL-EPR) measurements on irradiated materials. General safety procedures and precautions for electrochemical testing are covered in MET-CST-SOP-036 which is given in Appendix A.

This equipment is installed in and around the fume hoods in room 242. Bldg. 4508: the hoods are enclosed by a Regulated Area complete with protective clothing, monitoring equipment, barriers and step-off pads.

The specimens to be tested are in the form of flat, metal alloy specimens (usually TEM disks 3 mm diam x 0.25 mm thick) which have been irradiated in various fission reactors.

These specimens are β/γ emitters. The primary source of radiation is ^{60}Co at the μC level. Other shorter lived isotopes such as ^{59}Fe and ^{54}Mn also may play a minor role.

The specimens are transported to and from the 3025 hot cells or 3112 storage area in lead pigs in accordance with SMG-SOP-007.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016

Revision 1
Date: 08 June 1992
Page: 4 of 22

STANDARD OPERATING GUIDELINES

- 2.5 Before working on radioactive specimens, make sure that the process and abandoned hot drains are plugged to prevent the ingress of radioactive liquids. Disposal of liquids from the polishing and testing operations is covered elsewhere. It is expected that approximately 2 liters of low-level liquid waste (LLW) per specimen will be generated by these operations.
- 2.6 At the completion of the e'chem testing, the disposable blotter paper should be removed; LLLW disposed of properly and the area in the hood should be checked for contamination. Disposal of LLLW is described elsewhere. The entire area should be surveyed by Health Physics on a routine, weekly basis.
- 2.7 In the event of a spill, off-scale DRPD, detection of personal contamination, or any other problem relating to radiation protection, contact the Laboratory Supervisor (L. T. Gibson, x6-6640) and the area Health Physicist (P. Cox, x-4-4349) for assistance.
- 2.8 The general procedure for transportation and storage of irradiated specimens are described elsewhere. A maximum of four specimens in lead pigs should be stored in the lead-lined cabinet in the Regulated Area.
- 2.9 When leaving the regulated area, place shoe covers and gloves in the yellow drum provided. Monitor hands, clothing and shoes for contamination. If significant contamination, as indicated by a red "High-Level" light on the front panel of the "pancake" meter at the step-off pad, is detected, re-enter regulated area and contact the Laboratory Supervisor (L. T. Gibson, x6-6640) and the area Health Physicist (x-4-4349) for assistance.
- 2.10 Contaminated solid waste (blotter paper, gloves, shoe covers, paper towels, etc.) shall be placed in the compactible (yellow) trash can in the regulated area. Solvent (alcohol and acetone) rags should be disposed of in the red, mixed-waste trash can in the regulated area.

3. RADIOCHEMICAL GUIDELINES

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016

Revision 1
Date: 08 June 1992
Page: 5 of 22

STANDARD OPERATING GUIDELINES

Two sets of radiochemical guidelines, described in the ORNL Health Physics Manual, are used to set the standards of radiological protection.

3.1 Guidelines for Radiochemical Laboratories (RP-2.16)

These guidelines are applicable to radiochemical laboratories where radionuclides are processed or handled. The dominant isotope to be handled in this laboratory is ⁶⁰Co. This isotope is classified as a Class 2 high radiotoxicity isotope in Appendix A of RP-2.16. Appendix B of RP-2.16 defines the type of laboratory required to handle any given isotopic quantity and level of toxicity.

The fume hoods in Room 242 are classified as Type C facilities. The amount of radioactive material allowed in a Type C facility is specified in Appendix B of RP-2.16. The amount of material allowed may be increased or decreased depending upon the type and complexity of the operation being carried out and the physical form of the isotope. We have chosen to conservatively define these electrochemical operations in Room 242 as "normal chemical operations." The allowable quantities of ⁶⁰Co in each fume hood based on the ORNL Health Physics Manual Guidelines is 10 mCi in solid form, and 0.1 mCi dissolved in solution.

3.2 ALARA Policy

This policy requires the reduction of radiation exposure by personnel to the lowest levels commensurate with sound economics, available technology, and good operating procedures. Control of direct radiation exposure is achieved through the intelligent use of distance, shielding, and minimization of exposure time. Control of contamination is achieved through careful planned physical procedures and daily cleanup. Measurement of radiation levels is achieved through personal dosimetry and monitoring by Health Physics personnel.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 6 of 22

STANDARD OPERATING GUIDELINES

The maximum whole body and extremities radiation levels allowable without a Radiation Work Permit (RWP) are 20 mrem and 200 mrem per work shift, respectively. In addition, the maximum dose rates to the whole body and unprotected extremities without an RWP are 100 mrem/h and 1 rem/h, respectively.

4. RADIOLOGICAL CONTROLS

4.1 Before beginning work on a specimen, the strength of the gamma field must be measured and the isotopic content of the specimen estimated.

4.2 The specimen monitoring station located in the regulated area is equipped with an RSO-5 ionizing meter capable of measuring fields between 0.1 and 1000 mrem/h. This instrument should be used to measure the γ -field at a distance of 30 cm. This measurement is used to generate a plot of field versus distance for each individual specimen; this plot provides the operator with a guide to the approximate radiation field associated with each stage of the e'chem procedure.

4.3 The approximate isotopic content of ^{60}Co and ^{54}Mn is determined from the γ -field measurement at 30 cm from the following relationship:

$$\text{No. of mCi} = (\text{mrem/h at 30 cm}) \times (6 \times 10^{-2})$$

Approximately 75% of the radioactivity is ^{60}Co and 25% is

^{54}Mn . The basis for these relationships is fully described in SMG-SOG200. "Radiological Control and Protection for Bldg. 3025 Specimen Preparation Laboratory."

4.4 The measurement of the γ -field for each specimen is the basis for minimizing direct exposure by using distance and local shielding, if warranted. The measurement of the isotopic content of the specimens is used to ensure that the

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SQG-016Revision 1Date: 08 June 1992Page: 7 of 22

STANDARD OPERATING GUIDELINES

Type C radiological limits are not exceeded and also to estimate the isotopic content of the low level liquid waste.

5. TYPICAL RADIOLOGICAL ASSESSMENT

5.1 The specimens used for electrochemical measurements are typically stainless steel discs 3 mm diam x 0.25 mm thick weighing approximately 14 mg. As a result of neutron irradiation, the γ -field at 30 cm from a single disc will range between <1 and 10 mrem/h. A specimen at the high end of the range contains, from the relationships given in section 4.3, approximately 0.6 mCi of ^{60}Co . The specific activity is 4×10^{-2} mCi/mg.

5.2 The electropolishing operation removes approximately 1 mg of material from the specimen and a total of 40 μCi of ^{60}Co goes into the electropolishing solution. Thus, for specimens at the high end of the activity range, the allowable concentration of ^{60}Co in solution will be exceeded after three (3) electropolishing operations; the electrolyte must then be disposed of according to approved procedures. Proportionally more electropolishing operations on lower-activity specimens can be performed before disposing of the electrolyte.

5.3 The allowable concentrations of ^{60}Co for storage in a fume hood is 10 mCi. Up to 17 specimens each containing 0.6 mCi of ^{60}Co may be stored.

5.4 The greatest potential for direct gamma field exposure in these operations occurs during the handling of the discs with tweezers. Typically the fingers are as close as 5 cm from the source for periods of 1 to 2 minutes per specimen. A specimen having a field of 10 mrem/h at 30 cm will have a field of 360 mrem/h at the fingers (5 cm) during tweezer handling. The maximum handling time during a work shift involving five (5) specimen handling operations is

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 8 of 22

STANDARD OPERATING GUIDELINES

approximately 10 minutes. The maximum finger dose for these five operations with specimens on the high end of the activity range is 60 mrem, well below the allowable limit of 200 mrem.

5.5 The maximum activity specimen that can be handled without an RWP is a disc having a gamma field of 25 mrem/h at 30 cm since the field at the handling distance of 5 cm would approach 1 rem/h. This maximum activity specimen contains ~1.5 mCi of ⁶⁰Co; the allowable level of ⁶⁰Co in solution (0.1 mCi) would be reached in one electropolishing operation.

6. RESPONSIBILITIES

Control and responsibility for the regulated area belongs to L. T. Gibson and A. F. Rowcliffe. Do not perform e'chem testing without prior authorization from Gibson or Rowcliffe. The equipment for e'chem testing belongs to G. E. C. Bell and who is also an authorized operator.

7. CALIBRATIONS

7.1 Calibration of the PARC 273: The PARC 273 is user calibrated before each test. From front panel of PAR-273, input "Function 21" and then push "enter" button to perform an internal calibration. Record calibration in logbook. If the result of the internal calibration is an error code. Cease operation and have the PARC 273 unit returned to the factory for calibration. Bi-annual factory calibration is suggested but not necessary.

7.2 Calibration of the Keithley 610C: The unit shall be calibrated by ORNL Instrumentation and Control every year.

8. EQUIPMENT AND APPARATUS

8.1 Keithley 610C Solid-State Electrometer (K610C) with associated wires and connections.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 9 of 22

STANDARD OPERATING GUIDELINES

- 8.2 Princeton Applied Research Model 273 (PAR-273) Potentiostat/Galvanostat with associated electrometer, wires and connections
- 8.3 1-10 liter Nalgene bottle filled with demineralized, de-ionized distilled water from Aqueous Chemistry Groups facility in Bldg. 4500S, room R-227.
- 8.4 1 liter Nalgene wide-mouth beakers or 5-neck polarization flask w/ temperature control.
- 8.5 I-CHEM Protocol-A Amber water sample bottles, Pipettes and syringes for water analyses
- 8.6 Necessary reagents to make up solutions for measurements
9. TEST CELL PREPARATION AND EQUIPMENT WARM-UP
 - 9.1 Transfer 500 to 700 ml of desired or starting test solution to polarization cell.
 - 9.2 If necessary, begin purging the test solution with high-purity helium gas through Matheson Oxisorb cartridge at a rate of 0.3 ft³/h and continue purging throughout the test.
 - 9.3 Ensure that electrometer for PAR-273 is in "dummy" position and turn on power switch while holding local button down (this re-initializes the PAR-273).
 - 9.4 From front panel of PAR-273, input "Function 21" and then push "enter" button to perform an internal calibration. Record calibration in logbook.
 - 9.5 Turn on K610C and allow unit to warm-up for at least 30 minutes.
 - 9.6 If necessary, install counter and reference electrodes in beaker or polarization cell.
 - 9.7 Measure and record temperature of test solution.
10. ELECTROPOLISHING SPECIMENS

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 10 of 22

STANDARD OPERATING GUIDELINES

GENERAL COMMENTS:

To obtain meaningful and reproducible electrochem results it is generally necessary to electropolish the specimen surface prior to testing. The electrolyte used is generally a 90% acetic/10% perchloric acid solution at room temperature, unless otherwise specified in the logbook.

10.1 Place the lead pig on a clean surface and remove the top; to avoid possible contamination, do not put the pig in the fume hood. Holding the specimen vial with tweezers, remove the cap with the tool provided.

10.2 Transfer the vial to the fume hood and tap the specimen out into a dish.

10.3 Select the surface to be polished and, using tweezers, place the specimen on the platinum tray.

10.4 Open leak valve.

10.5 Open building air supply valve.

10.6 Slowly close leak valve to begin flow of electrolyte in downcomer to the polishing stage.

10.7 Adjust flow rate as desired, using regulator.

10.8 Open leak valve and allow electrolyte to flow back into bottom reservoir.

10.9 Place the platinum tray with the specimen on the polishing stage with long-handled tongs and position specimen under nozzle of downcomer.

10.10 Connect positive lead wire from the power supply to tray and negative lead to downcomer platinum lead wire.

10.11 Turn main power switch of power supply on and set desired voltage.

10.12 Close leak valve to re-establish flow of electrolyte to polishing stage.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 11 of 22

STANDARD OPERATING GUIDELINES

- 10.13 Turn current on for the required polishing time.
- 10.14 Turn current off.
- 10.15 Open leak valve to stop flow of electrolyte. Record polishing voltage, current and time in logbook.
- 10.16 Remove tray from stage with tongs and drop specimen in long-handled rinsing basket.
- 10.17 Rinse specimen in alcohol, then in acetone with basket and drop specimen onto plastic petri dish lined with clean filter paper.
- 10.18 Pick up disk with tongs, place on camera stage, and examine specimen surface with monitor. If the surface condition is unsatisfactory, return to 10.7 and adjust voltage and flow rate as required.
- 10.19 Transfer specimen to loading station.
- 10.20 Turn main power switch of power supply off and close building air supply valve.
- 10.21 Leave leak valve open.

11. LOADING THE SPECIMEN(S) INTO THE CELL

- 11.1 Mount the specimen(s) on the working electrode holder, either acrylic TEM or PAR-5/8" disk.
- 11.2 Check the connection between the specimen(s) and the electrode holder with an ohmmeter.
- 11.3 Wet the specimen surface(s) with the test solution using squeeze bottle.
- 11.4 Transfer the electrode holders to the polarization cell.
- 11.5 Connect lead wires to electrodes.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 12 of 22

STANDARD OPERATING GUIDELINES

11.6 Move PAR-273 electrometer switch from "DUMMY" to "EXT."
If the potentiostat displays "overload," place electrometer
in "DUMMY" position. remove the electrode holder from the
polarization cell. and return to 11.1

12. PROCEDURE FOR ECP MEASUREMENTS

12.1 Differential ECP Measurement

A schematic diagram of the experimental set-up for DECP measurements is shown in figure 12.1.1. The PAR-273 is used as a galvanostat with current set to zero and the potential difference between the working and counter electrodes, in the solutions of interest, is measured using the K610C. Note: There is no reference electrode connection for DECP measurements. If samples of solutions are to be chemically analyzed, notify Marion Ferguson of Analytical Chemistry prior to starting measurements.

12.1.1 At this time, the electrodes should already be in the desired or starting solution (section 5). Ensure that PAR-273 electrometer is in "DUMMY" position and K610C meter switch is in "power-on/cell-off" position. Connect positive (+) connection of K610C to working electrode specimen and negative (-) connection of K610C to counter electrode specimen. Note connections in logbook.

12.1.2 Ensure that "cell-enable" light is "off" on PAR-273.

12.1.3 Select galvanostat mode and 100 nA range from front panel and enter applied current as 0.000. Push Cell-enable to on.

12.1.4 Select voltage range on K610C and center zero; lock zero-check and zero meter; take zero-check off

12.1.5 Switch electrometer on PAR-273 to "EXT" and record voltage on K610C. Monitor DECP reading for 120 s and record variations as they occur. If necessary, adjust voltage scale and re-zero.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 13 of 22

STANDARD OPERATING GUIDELINES

12.1.6 Switch electrometer on PAR-273 to "DUMMY" and turn cell-enable "off". If necessary, sample solution with pipette or syringe and deposit in I-CHEM-Protocol A cleaned amber glass bottle. Record time, date and solution additions on bottle label and in logbook. Have bottles checked by HP and delivered to Analytical Chemistry as soon as possible.

12.1.7 If required, make additions to solution and repeat steps 12.1.2-12.1.7.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016

Revision 1
Date: 08 June 1992
Page: 14 of 22

STANDARD OPERATING GUIDELINES

Figure 12.1.1: Schematic of DECP Measurement

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 15 of 22

STANDARD OPERATING GUIDELINES

12.2 Absolute ECP (AECF) Measurement

A schematic diagram of the experimental set-up for AECF measurements is shown in figure 12.2.1. Either, or both, the PAR-273 or K610C can be used to measure the AECF. Note: There is a reference electrode connection for AECF measurements and this requires that you install an electrode. This procedure assumes that Princeton Applied Research -type reference electrodes (e.g. part no. GO-093) with Vycor™ tipped probes are used. If samples of solutions are to be chemically analyzed, notify Marion Ferguson of Analytical Chemistry prior to starting measurements.

12.2.1 Submerge the tip of the reference electrode probe in test solution.

12.2.2 Ensure that PAR-273 electrometer is in "DUMMY" position and K610C meter switch is in "power-on/cell-off" position. Ensure that "cell-enable" light is "off" on PAR-273. Connect positive (+) connection of K610C to working electrode specimen and negative (-) reference electrode specimen. Note connections in logbook.

12.2.3 Switch electrometer on PAR-273 to "EXT" and record voltage on K610C and from front panel of PAR-273. If necessary, adjust voltage scale and re-zero of K610C.

12.2.4 Switch electrometer on PAR-273 to "DUMMY" and turn cell-enable "off".

12.2.5 If necessary, sample solution with pipette or syringe and deposit in I-CHEM-Protocol A cleaned amber glass bottle. Record time, date and solution additions on bottle label and in logbook. Have bottles checked by HP and delivered to Analytical Chemistry as soon as possible.

12.2.6 If required, make additions to solution and repeat steps 12.2.2-12.2.6.

MAR-12-1993 11:45 FROM ORNL/M & C-BLDG 5500
Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

TO 42582 P.017/034
Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 16 of 22

STANDARD OPERATING GUIDELINES

Figure 12.2.1: Schematic of AECP Measurement

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 17 of 22

STANDARD OPERATING GUIDELINES

13. PROCEDURE FOR SL-EPR MEASUREMENTS

A schematic diagram of the experimental set-up for EPR measurements is shown in figure 13.1.1. The counter electrode for EPR measurements should be a platinum "flag." There is a reference electrode connection for EPR measurements and this requires that you install an electrode. This procedure assumes that Princeton Applied Research -type reference electrodes (e.g. part no. GO-093) with Vycor™ tipped probes are used. The test solution for EPR testing is deaerated 1NH₂SO₄ + 0.01MKSCN maintained at 30°C ± 1°C.

13.1.1 Turn on the CRT display, computer, and printer. Change directories from root to "PARC"("CD\PARC"<CR>) and enter "M342C<CR>" to enter e'chem data acquisition software. Input the required information as prompted by computer menus

13.1.2 Set the electrometer switch to "EXT." and allow potential to stabilize (~5- 10 minutes). Once a stable potential is displayed, record potential in logbook.

13.1.3 Conduct EPR Measurement in accordance with the Procedures Described in MODEL 342 SOFTCORR CORROSION MEASUREMENT SOFTWARE OPERATING MANUAL. Chapter 14.

13.1.4 Use computer to calculate reactivation charge and determine breakdown potential. Print results and record in logbook.

MAR-12-1993 11:46 FROM ORNL/M & C-BLDG 5500
Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

TO 42582 P.019/034
Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 18 of 22

STANDARD OPERATING GUIDELINES

Note: EPR Solution is 1 N H₂SO₄ + 0.01 M KSCN

Figure 13.1.1: Schematic of EPR Measurement

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036
SMG-SOG-016
Revision 1
Date: 08 June 1992
Page: 19 of 22

STANDARD OPERATING GUIDELINES

14. CLEANING OF TEST CELL. SPECIMEN UNLOADING AND STORAGE

- 14.1.1 Ensure that the exit tube is firmly connected to drain bottle. vent is open on top of the bottle. that the bottle is properly labeled for the solutions to be disposed of and that bottle is no more than one third full before beginning this operation.
- 14.1.2 Set the electrometer switch to "DUMMY". turn "cell-enable" to off and disconnect lead wires from the electrodes.
- 14.1.3 Stop nitrogen gas purging.
- 14.1.4 Empty polarization cell of the test solution through the drain line to bottle by opening the electric valve and opening the in-line mechanical valve.
- 14.1.5 Close electric valve and mechanical valve.
- 14.1.6 Fill the test cell with deionized water by turning on the pump.
- 14.1.7 Stir the water by gas bubbling for 5 min.
- 14.1.8 Dispose of the water through the drain by opening the electric and mechanical valves.
- 14.1.9 Repeat Steps 14.1.6 through 14.1.9. Follow approved procedures for LLLW waste disposal.
- 14.1.10 Dry the specimen holder(s) by purging polarization cell with gas at a rate of 0.8 ft³/h for ~5 min.
- 14.1.11 Remove the working electrode(s) from the cell.
- 14.1.12 Remove the specimen(s) from the holder and decontaminate the specimen by rinsing in alcohol.
- 14.1.13 If it is necessary to examine the specimen surface using the metallograph. transfer the disk to a Petri dish. Using tongs. carry the Petri dish to the metallograph stage; using tweezers transfer the specimen to a glass slide and position it over the objective lens. After examination.

MAR-12-1993 11:47 FROM ORNL/M & C-BLDG 5500

TO

42582 P.021/034

Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

SMG-SOG-016

Revision 1
Date: 08 June 1992
Page: 20 of 22

STANDARD OPERATING GUIDELINES

return specimen via Petri dish to the hood for packaging and storage.

14.1.14 Using tongs, place the specimens in labeled vials back in the lead pig; tape lid securely and return the pig to the lead storage cabinet.

Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-036SMG-SOG-016Revision 1Date: 08 June 1992Page: 21 of 22

STANDARD OPERATING GUIDELINES

Re-review Record:

- Required every two (2) years.
- Signatures indicate that procedure or guidelines are still adequate for this activity.

1st Rereview:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

_____ Date _____
Structural Materials Group
Group Leader

_____ Date _____
DSO/RSO

2nd Rereview:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

_____ Date _____
Structural Materials Group
Group Leader

_____ Date _____
DSO/RSO

MAR-12-1993 11:48 FROM ORNL/M & C-BLDG 5500
Corrosion Science and Technology Group
Structural Materials Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

TO

42582 P.023/034

Document Nos. CST-SOP-036
SMG-SOG-016

Revision 1

Date: 08 June 1992

Page: 22 of 22

STANDARD OPERATING GUIDELINES

APPENDIX A:

STANDARD OPERATING PROCEDURE FOR ELECTROCHEMICAL TESTING
IN AQUEOUS ENVIRONMENTS
MET-CST-035

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 1 of 10

STANDARD OPERATING GUIDELINES

Title: Electrochemical Testing in Aqueous Environments

Prepared by:

G. E. C. Bell

G. E. C. Bell, Staff Member

Date

24 JUN 92

Reviewed by:

J. R. DiStefano

J. R. DiStefano

Group Leader, Corrosion Science and Technology

Date

M. Vance
QAS M&C

Date

C. A. Maples
M&C DSO and RSO

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 2 of 10

STANDARD OPERATING GUIDELINES

1. SCOPE/PURPOSE

Electrochemical testing techniques can be used to give insight into corrosion and other chemical reactions which involve charge exchange. Since the nature of the test are experimental in nature, the specifics of a particular test will not be given in this procedure, rather general safety precautions and guidelines will be discussed. The electrochemical equipment is located in B-147 in building 4500S and consists of an EG&G PARC model 273 potentiostat/galvanostat and 5208EC lock-in amplifier with an IBM PS/2 30 computer with EG&G models 342 and 378 software, recirculating temperature control system, and associated glassware, deaeration systems and reference cells as required. Many of the standard electrochemical/corrosion tests are outlined in manuals for the equipment. In addition to the standard tests given in the manuals, the operator can create his/her own electrochemical procedures using the software "HEAD START."

2. ENVIRONMENTAL, HEALTH, AND SAFETY REQUIREMENTS

- 2.1 Safety glasses should be worn at all times in the test area and protective gloves and lab coats should be worn when caustic and/or acidic solutions are used.
- 2.2 No smoking, eating, or drinking is allowed in B-147.
- 2.3 The Model 273 potentiostat/galvanostat has the capacity to provide 1 ampere at 100 V-DC. CARE MUST BE TAKEN TO NEVER HANDLE THE TEST LEADS (RED, GREEN, AND BLACK ALLIGATOR CLIPS) WHEN THE SWITCH ON THE ELECTROMETER IS IN THE "EXT" POSITION. ALWAYS SWITCH PULL-TOGGLE SWITCH ON ELECTROMETER TO "DUMMY" BEFORE HANDLING THE TEST LEADS.

3. RESPONSIBILITIES

G. E. C. Bell has control and responsibility for activities in B-147. Do not perform e'chem testing without prior authorization from Bell. All operators are required to read the manuals for the equipment prior to beginning initial operation. Details of many standard e'chem tests and procedures are in the PARC 342 software manual. Operators shall review

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 3 of 10

STANDARD OPERATING GUIDELINES

to familiarize themselves with the steps in the manual prior to performing a particular type of test.

The following is a list of the authorized operators of the electrochemical equipment in B-147. 4500S as of the date shown above:

G. E. C. Bell
G. L. Edgemon
D. F. Wilson
M. Howell

Amendments to this list are at the back of this procedure in Appendix A. Amendments to the operator list above require only the signature of the CS&T Group leader.

4. CALIBRATIONS

- 4.1 Calibration of the PARC 273: The PARC 273 is user calibrated before each test. From front panel of PARC 273, input "Function 21" and then push "enter" button to perform an internal calibration. Record calibration in logbook. If the result of the internal calibration is an error code. Cease operation and have the PARC 273 unit returned to the factory for calibration. Bi-annual factory calibration is suggested, but not necessary.

5 GENERAL DESCRIPTION AND COMMENTS:

5.1.1 Specimen or Working Electrode (WE) Geometry

Almost any specimen shape can be tested. The primary consideration should be that the maximum output of the 273 is 1 ampere and the area of the specimen should be such that this maximum is not exceeded. On the other hand, the resolution of the 273 is on the order of 1 - 5 nanoamperes and there must be sufficient area to produce this detectable current at the maximum potential of the test. Typical wetted specimen areas are usually 1 cm² or less. Specimen holders for TEM and 5/8" disks are available. Otherwise, materials are usually mounted in a metallographic resin with a lead wire to obtain electrical contact.

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 4 of 10

STANDARD OPERATING GUIDELINES

5.1.2 Electrochemical Polarization Cell (EPC)

The "standard EPC in B-147 provides a constant temperature bath volume of approximately 600 to 700 ml for electrochemical measurements. The volume is accessed via a removable ground glass 5-neck head which sits atop the EPC. The standard head has five 24/40 or four 24/40 and one 45/50 ground glass tapered joints for access. Temperature in the reaction vessel is controlled by circulating water around the perimeter of the volume from a recirculating chiller/heater system. The reaction vessel is removable from the temperature bath via a ground glass joint to allow easy disposal of the electrolyte. (Electrolytes from each series of experiments should be collected in a labeled container marked "for disposal"). NOTE: ELECTROLYTES MUST BE PROPERLY STORED AND DISPOSED OF VIA APPROVED PROCEDURES. ROOM B-147 HAS A DESIGNATED SATELLITE WASTE ACCUMULATION AREA FOR SEGREGATION AND TEMPORARY STORAGE OF ELECTROLYTES FOR DISPOSAL. DO NOT POUR ELECTROLYTES DOWN THE PROCESS OR OTHER DRAIN. THIS WOULD BE A VIOLATION OF STATE AND/OR FEDERAL LAW AND MAY BE PUNISHABLE BY FINE AND/OR IMPRISONMENT.

5.1.3 Reference Electrodes (RE)

Electrochemical measurements require a standard half-cell to reference the applied voltages. The standard RE in B-147 is a Saturated Calomel Electrode (SCE). The RE is typically electrically contacted with the EPC via a Lugin probe/Agar+KCl salt bridge. This is done to reduce chloride contamination of the electrolyte in the cell.

5.1.4. Counter Electrode (CE)

The counter electrode completes the electrical circuit (i.e. collects the current generated from the specimen during the electrochemical test) and is typically made of an inert material, such as platinum or graphite.

5.1.5. Deaeration and Mechanical Stirring

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 5 of 10

STANDARD OPERATING GUIDELINES

Oxygen is normally not desired in electrochemical testing and it is best to stir while testing. Deaeration is accomplished by sparging the test solution with solution wetted nitrogen gas. Stirring is accomplished with magnetic stirring bars.

5.1.6. Differential Electrometer (DE)

The DE is the means by which the connections to the working, counter and reference electrodes are made and through which the current and voltage are applied/measured. The DE has a pull-toggle switch with "dummy" and "ext" positions. In the "dummy" position, the leads from the front of the DE are disconnected from the potentiostat and the potentiostat output is directed across a 100 K ohm resistor. In the "ext" position, the leads from the front of the DE are connected to the potentiostat and the potentiostat output is directed across the red and green leads. CARE MUST BE TAKEN TO NEVER HANDLE THE TEST LEADS (RED, GREEN AND BLACK ALLIGATOR CLIPS) WHEN THE SWITCH ON THE ELECTROMETER IS IN THE "EXT" POSITION. ALWAYS SWITCH FULL-TOGGLE SWITCH ON ELECTROMETER TO "DUMMY" BEFORE HANDLING THE TEST LEADS.

The following steps in the guideline represent general lab technique. Selection of a specific testing environment and electrochemical method are left to the operator/experimenter. Three basic steps are involved: (1) preparation of the specimen, test cell and equipment. (2) the experiment is run. (3) the system must be cleaned, waste disposed of and apparatus re-assembled.

5.2 Preparation of EPC

5.2.1 Prepare electrolyte solution and fill reaction vessel of EPC with 500 to 600 ml of electrolyte.

5.2.2 Install reaction vessel in EPC and place 5-neck head on top and secure with elastic straps.

5.2.3 Insure that hose connections to temperature bath are tight and turn on recirculating unit and adjust LED to

STANDARD OPERATING GUIDELINES

desired temperature (-30 to 130°C). Note: the standard solution in the recirculator if demineralized water. Temperatures below 10°C or above 90°C are required. drain recirculator and re-fill with 50/50 antifreeze/water mixture.

5.2.4 If deaeration is desired, install sparging and stirring rods and initiate nitrogen flow. Note: In order to wet the nitrogen and avoid loss of electrolyte from sparging, the nitrogen must be bubbled through a mixture of the same electrolyte prior to entering the EPC. For most solutions, deaeration can be accomplished in less than an hour. However, some specimen materials (materials which have very stable oxides) may take several days and require ultra pure nitrogen to achieve oxide free surfaces.

5.2.5 Install Lugin probe and draw deaerated solution into salt bridge.

5.2.6 Install counter and working electrodes.

5.2.7 With the pull-toggle switch of the differential electrometer in the "dummy" position, connect the leads to the working, counter and reference electrodes. CARE MUST BE TAKEN TO NEVER HANDLE THE TEST LEADS (RED, GREEN, AND BLACK ALLIGATOR CLIPS) WHEN THE SWITCH ON THE ELECTROMETER IS IN THE "EXT" POSITION. ALWAYS SWITCH PULL-TOGGLE SWITCH ON ELECTROMETER TO "DUMMY" BEFORE HANDLING THE TEST LEADS.

5.3 Operation of the Potentiostat and Computer

5.3.1 Depress the Local button on the lower left corner of the potentiostat and turn potentiostat power switch on (lower right) and watch LCD for System Re-initialized message. This can also be done by turning power on and entering function 10 from the front panel. This clears any previous instruction which may have been stored during the last use.

5.3.2 Depress "CELL ON" switch on potentiostat and move pull-toggle switch on DE from "dummy" to "ext" and the free

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 7 of 10

STANDARD OPERATING GUIDELINES

corrosion potential should be displayed on the LCD. If I or E overload indications are shown, there is a connection problem. Switch DE to "dummy" before checking connections. CARE MUST BE TAKEN TO NEVER HANDLE THE TEST LEADS (RED, GREEN, AND BLACK ALLIGATOR CLIPS) WHEN THE SWITCH ON THE ELECTROMETER IS IN THE "EXT" POSITION. ALWAYS SWITCH FULL-TOGGLE SWITCH ON ELECTROMETER TO "DUMMY" BEFORE HANDLING THE TEST LEADS.

- 5.3.3 If AC impedance is to be performed, switch power of 5208EC Lock-in amplifier on.
- 5.3.4 Turn on printer, monitor, and computer. The computer is self-booting and menu driven. Follow menu instructions to launch appropriate electrochemical software (PARC 342 or PARC 378).
- 5.3.5 The EG&G software is also menu driven. Follow the instructions of software.
- 5.3.6 Once the experiment is selected and parameters entered and "R" runs the experiment, ctrl-p will pass a step in the electrochemical procedure (conditioning, delay, etc.) and a ctrl-a will abort the experiment.
- 5.4 Break-down, clean-up and shut-down of equipment.
 - 5.4.1 After completing experiment, switch DE to "dummy" and remove leads and electrodes from solution.
 - 5.4.2 Remove and rinse electrodes.
 - 5.4.3 Store RE in salt solution.
 - 5.4.4 Drain EPC into disposal container and rinse with distilled water. If further testing is required, reassemble EPC and return to section 5.1.
 - 5.4.5 Exit to DOS from program and shut down computer, 273 and 5208EC.

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 8 of 10

STANDARD OPERATING GUIDELINES

Re-review Record:

- Required every two (2) years.
- Signatures indicate that procedure or guidelines are still adequate for this activity.

1st Rereview:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

_____ Date _____
M&C DSO/RSO

2nd Rereview:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

_____ Date _____
M&C DSO/RSO

MAR-12-1993 11:53 FROM ORNL/M & C-BLDG 5500

TO

42582 P.033/034

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 9 of 10

STANDARD OPERATING GUIDELINES

APPENDIX A:

AMENDMENTS TO AUTHORIZED OPERATOR LIST

Corrosion Science and Technology Group
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

Document Nos. CST-SOP-035
Revision 1
Date: 08 June 1992
Page: 10 of 10

STANDARD OPERATING GUIDELINES

The following is a list of the authorized operators of the electrochemical equipment in B-147, 4500S as of the date shown above:

G. E. C. Bell
G. L. Edgemon
D. F. Wilson
M. Howell

Amendments to the operator list above require only the signature of the CS&T Group leader and are shown below this line.

1st Amendment:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

2nd Amendment:

_____ Date _____
Corrosion Science and Technology Group
Group Leader

END

**DATE
FILMED**

11/01/93