Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

We put science to work.™

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

A U.S. DEPARTMENT OF ENERGY NATIONAL LABORATORY • SAVANNAH RIVER SITE • AIKEN, SC

SRNL Report for the Tank Waste Disposition Integrated Flowsheet: Corrosion Testing

R. B. Wyrwas

September 30, 2015

SRNL-STI-2015-00506

SRNL.DOE.GOV

DISCLAIMER

Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2015-00506
Revision 0

Keywords: *corrosion, chloride, DFLAW*

Retention: *permanent*

SRNL Report for Tank Waste Dispostion Integrated Flowsheet: Corrosion Testing

R.B. Wyrwas

September 30, 2015

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

APPROVALS/TASK TECHNICAL REQUEST IDENTIFICATION

REVIEWS AND APPROVALS

AUTHORS:

____

R.B. Wyrwas, SRNL-MST-C&MP Date

TECHNICAL REVIEW:

____

R. E. Fuentes, SRNL-MST-C&MP Date

APPROVAL:

____

B. J. Wiersma, Manager Date
SRNL-MST-C&MP

____

S. T. Arm, Washington River Protection Solutions Date

ACKNOWLEDGEMENTS

A special acknowledgement to Mitchell Vogatsky who performed a majority of the laboratory testing as an intern in the Material Science & Technology Internship Program at the Savannah River National Laboratory.

EXECUTIVE SUMMARY

A series of cyclic potentiodynamic polarization (CPP) tests were performed in support of the Tank Waste Disposition Integrated Flowsheet (TWDIF). The focus of the testing was to assess the effectiveness of the SRNL model for predicting the amount of nitrite inhibitor needed to prevent pitting induced by increasing halide concentrations. The testing conditions were selected to simulate the dilute process stream that is proposed to be returned to tank farms from treating the off-gas from the low activity waste melter in the Waste Treatment and Immobilization Plant.

The results of the CPP tests indicated 'Category 1' and 'Category 1 with minor pitting' behavior as defined by the CPP test protocol. Category 1 behavior indicates no pitting susceptibility. Category 1 with minor pitting is resolved utilizing the ASTM G-192 method per the approved CPP test protocol. The results of the G 192 tests performed by DNV-GL for two of the test conditions indicated that the protection potential (E_{prot}) is at a large electropositive value (i.e., greater than +600 mV vs. SCE reference electrode). Estimates of the E_{prot} determined from the CPP curves performed at SRNL showed good correlation with the E_{prot} determined by G 192. An initial review of the CPP scans performed at SRNL indicated that at all tested conditions the values of the estimated E_{prot} are greater than +550 mV vs. SCE reference electrode. The difference between the estimated E_{prot} and the zero current potential (E_{zc}) was utilized as an initial assessment of the likelihood that the pit-like indications would propagate. A large difference, between E_{prot} and E_{zc}, greater than +400 mV, was observed on the CPP curves. This result suggests that the indications that were observed during the CPP test are not propagating and that the test conditions are relatively benign with respect to pitting. Characterization of the pits by SEM and EDS on one of the samples further suggested that the pit-like inclusions were due to etching of manganese sulfide inclusions rather than the development of propagating pits.

These results indicated that the SRS chloride inhibition equation over estimates the amount of inhibitor needed for the anticipated WTP return stream conditions. Future testing will delineate the conditions where pitting susceptibility is indicated and provide corrosion control limits that are suited for the expected return stream compositions. The testing will also more accurately determine the difference between the protection and long term open circuit potentials to allow for an understanding of any borderline cases. Until these limits are defined, the process flow sheet group may use the SRS equation realizing the limitations of the extrapolation of the model and that the new limits will be defined in the future.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

A	amps
CPP	cyclic potentiodynamic polarization
DFLAW	Direct-Feed Low Activity Waste
DST	Hanford Double Shell Tank
EDS	energy dispersive x-ray spectroscopy
E_{max}	peak current potential
E_{pit}	pitting potential
E_{prot}	protection potential
E_{trans}	transpassive potential
E_{zc}	zero current cotential
HLW	high-level waste
i_{cor}	corrosion current density
i_{max}	peak current density
i_{pas}	passive current density
LAWPS	Low Activity Waste Pretreatment System
M	molarity, or moles per liter
mV	millivolts
RIE	Rapid Improvement Event
SEM	scanning electron microscope
SCE	saturate calomel electrode
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TWDIF	Tank Waste Disposition Integrated Flowsheet
WRPS	Washington River Protection Solutions
WTP	Hanford Waste Treatment and Immobilization Plant

1.0 Introduction

The Hanford site stores several million gallons of radioactive waste in underground storage tanks. The Hanford Waste Treatment and Immobilization Plant (WTP) is being designed to treat the high-level waste (HLW) and low-activity waste (LAW) in the tank. Integration of processes across the tank farm, 242-A evaporator, WTP, interim storage facilities, and effluent handling is a challenge given the number of different facilities and contractors involved in the overall program. The Tank Waste Disposition Integrated Flowsheet (TWDIF) is tasked with developing an integrated flowsheet for the stabilization of the Hanford waste by defining and managing the interfaces between facilities. In addition, the TWDIF task is designed to identify gaps and opportunities facility interfaces and to develop plans to close the gaps and realize opportunities.

The TWDIF program for FY15 includes evaluation of the corrosion controls needed to allow transfer of Direct-Feed Low Activity Waste (DFLAW) effluents to the Hanford Double Shell Tank (DST) system and evaporation of these effluents in the 242-A evaporator. These effluents originate from the Low Activity Waste Pretreatment System (LAWPS) and the WTP LAW facility. The returns from LAWPS include the solids slurry from cross-flow filtration, chemical cleaning of the filters, and elution cycle effluents from ion exchange including concentrated cesium eluent. These streams are expected to be handled by the current corrosion control protocols for the DST system.

The WTP LAW effluent stream will be generated by condensation and scrubbing of the LAW melter off-gas stream. A portion of this stream, which will contain substantial amounts of chloride, fluoride, ammonia, and sulfate ions, and potentially minor concentrations of mercury, may be returned to the tank farms for storage and evaporation [1].

At present the tank farm facility has no corrosion control measures for the waste tanks or the 242-A evaporator that address the halides and sulfate anions. The Savannah River Site (SRS) has data on chlorides and sulfates that may apply to the Hanford waste tanks [2]. However, application of the data at Hanford would require addition of a significant quantity of inhibitor and dilution water to first reduce the halide and sulfate concentrations into the range for which the SRS data was developed. The volume of returned effluent is anticipated to reduce operational flexibility in the tank farms as waste is also concurrently retrieved from single shell tanks.

On 2/17/2015 through 2/19/2015 a Lean Rapid Improvement Event (RIE) was conducted to address these issues. The work reported herein is a response to the recommendations that came from the event. Testing was recommended that will extend over the next 3 years and a statement of work addressing corrosion control tasks to be initiated in FY15 and performed through FY17 was prepared. The testing conducted by Savannah River National Laboratory (SRNL) is the focus of this report and summarizes the results of the testing in support of the issues identified in the RIE.

Washington River Protection Solutions (WRPS) requested a review and testing to determine if the following equation can be used to provide corrosion control limits for halide concentrations up to 0.1 M in the return off-gas stream from the LAW melter.

$$[NO_2^-]_{min} = 104 * [Halide]^{1.34}$$

Since the stream is dilute, the most likely mechanism of attack for carbon steel is pitting. Originally, the model equation was used to determine the minimum nitrite requirement necessary to prevent pitting due to chloride (i.e., did not consider fluoride) as shown in Figure 1 [2]. The pH was 10 and the maximum temperature is 30 °C. Chloride concentrations were varied up to 0.05 M in the original tests. However prior to beginning the test program, the test temperature was increased to 35 °C based on inputs from the WTP design contractor. For 35 °C the coefficient for the equation changes to 166. Ideally WRPS would like to be able to demonstrate that the equation with the lower coefficient is applicable since this would require less inhibitor addition and dilution of the stream. Therefore, although the tests will be conducted at 35 °C, the test matrix was planned and the results were evaluated based on the equation that assumes the 30 °C temperature.

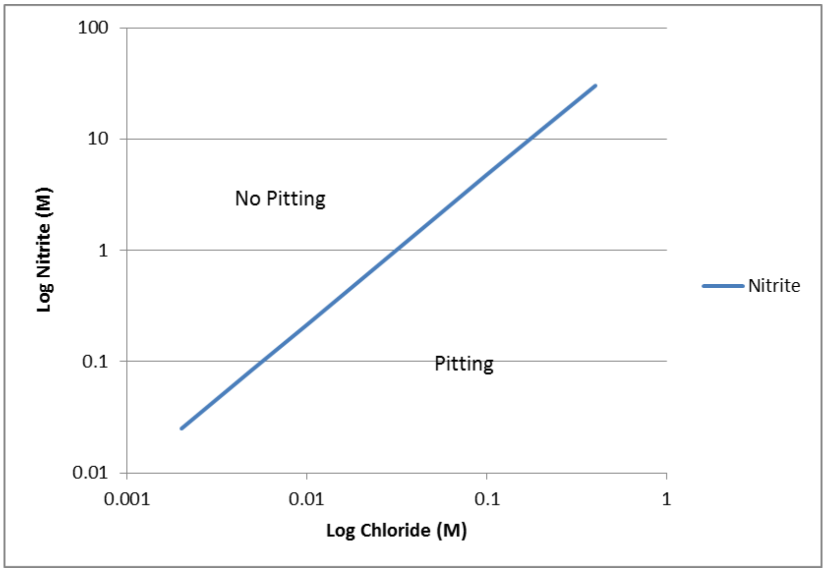


Figure 1. Minimum nitrite required to mitigate pitting corrosion due to chloride.

WRPS needed a "quick experiment" to determine the confidence in the equation as a predictor for minimum nitrite as a function of the halide concentration (i.e., chloride and fluoride). The quick evaluation testing investigated the validity of the equation at a composition of 0.1 M halide. If the equation, or a substitute, cannot be implemented, then dilution of the stream will be required, which could result in additional waste volume for tank farm storage and processing.

For this work the objectives were:

1) Determine if SRS equation for chloride can be utilized as a predictor for minimum nitrite as a function of the halide concentration.
2) To extend the concentration range for the testing from 0.05 M to at least 0.1 M halide.

2.0 Experimental

A series of cyclic potentiodynamic polarization (CPP) tests were performed to assess the effectiveness of the equation in predicting pit/no pit regimes. The tests were performed using EL-400 working electrodes (area of 4.75 cm^2) from Metal Samples Company constructed of TCR-128 rail car steel provided by

WRPS. All potentials are reported in relation to the saturated calomel electrode (SCE) that was used as a reference electrode in this testing. Thirty replicated tests were performed according to the pitting protocol established by the Expert Panel Oversight Committee [4]. The simulant contained the anions shown in Table 1 at concentrations within the envelope indicated. The tests were conducted at a temperature of 35 °C and a pH of 10. Three species were varied: chloride, fluoride, and nitrite. Each of these were varied between anticipated minimum and maximum values for the DFLAW off-gas stream. Other anions and cations were set at constant values as shown in Table 1. The justification for each constant value is listed in Table 1 while the ranges are based on information presented by WRPS during the RIE. An OLI™ simulation was performed to determine the actual carbonate and bicarbonate concentrations such that the pH is 10.

Table 1. Initial Anion Concentration Test Range

Concentrations in moles/liter

	Minimum	Maximum	Comments
Sulfate	0.1		Sulfate/Nitrate ratio less than 0.3 reduces likelihood of interaction
Chloride	0.01	0.15	Range to test
Fluoride	0.01	0.05	Anticipated free fluoride maximum based on solubility
Halide	0.02	0.2	Summation of halide ions
Nitrite	1	5	Range to test
Nitrate	0.5		Started to see no additional benefit for adding more nitrite
pH	10		Lowest pH; Adjusted with carbonate and bicarbonate
TIC	0.01		Midpoint of range
Ammonium	0.01		Lower end of range due to solubility questions
Phosphate	0.003		Midpoint of range
Aluminum	0.002		Midpoint of range
Chromium	0.004		Midpoint of range
Potassium	0.003		Midpoint of range
Temperature (°C)	30		

The 30 tests are depicted by the blue diamonds in Figure 2, which shows the halide and corresponding nitrite concentration for each test. The black, solid line is the actual minimum nitrite concentration determined by testing above which pitting is expected to occur. The black, dashed line represents a conservative 50% safety margin on the test data and is used for corrosion control purposes. This line also represents the model that is being investigated in the quick evaluation. The lines of the model and the 50% margin lines are plotted at 30℃ or a scaling factor of 69 for the model line and 104 for the 50% margin line. The two red lines represent the region from 0.05 to 0.1 M halide, which will extend the model into a useful range. Approximately, half the tests are performed in this region. Several tests are also designed at higher halide concentrations. Previously it has been observed that above a certain concentration of aggressive species [5], the minimum nitrite required for inhibition becomes independent of the concentration of the aggressive species. These tests were designed to investigate whether this is true for the halide species.

The chloride and fluoride were also varied in these tests. Figure 3 shows the distribution of the fluoride and chloride concentrations that were used. Preliminary thermodynamic calculations were performed to assess the solubility of the fluoride species [6]. Although the material balances indicated high fluoride concentrations (i.e. up to 0.7 M), the amount of soluble fluoride was on the order of 0.05 M or less. It is the free, soluble fluoride that will contribute to corrosion. In order to optimize the return stream volume, other factors regarding the corrosion inhibitors should be considered in further testing.

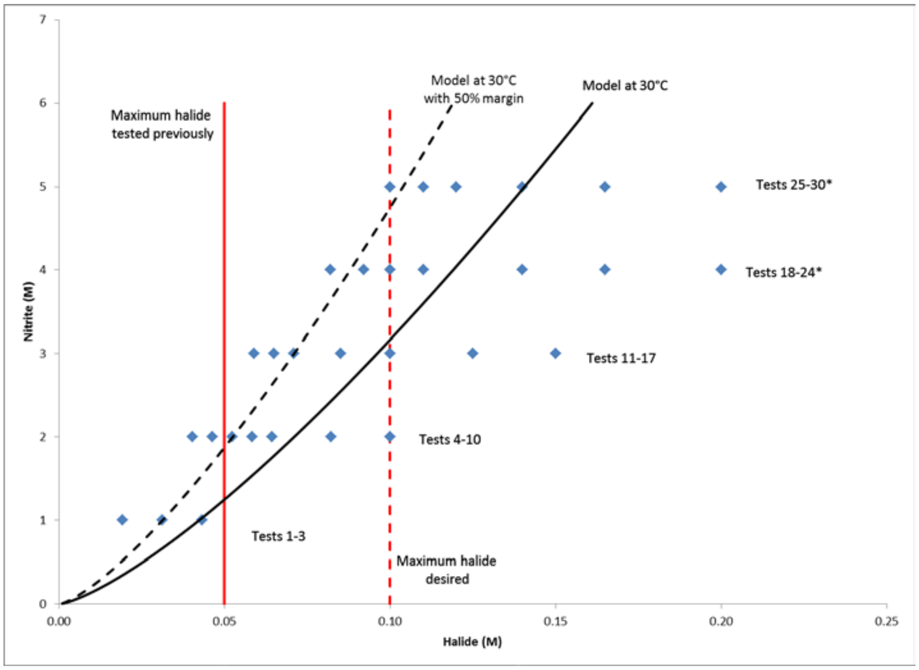


Figure 2. Test matrix for the 30 CPP tests. Blue diamonds indicate the nitrite and halide concentrations that were used for testing.

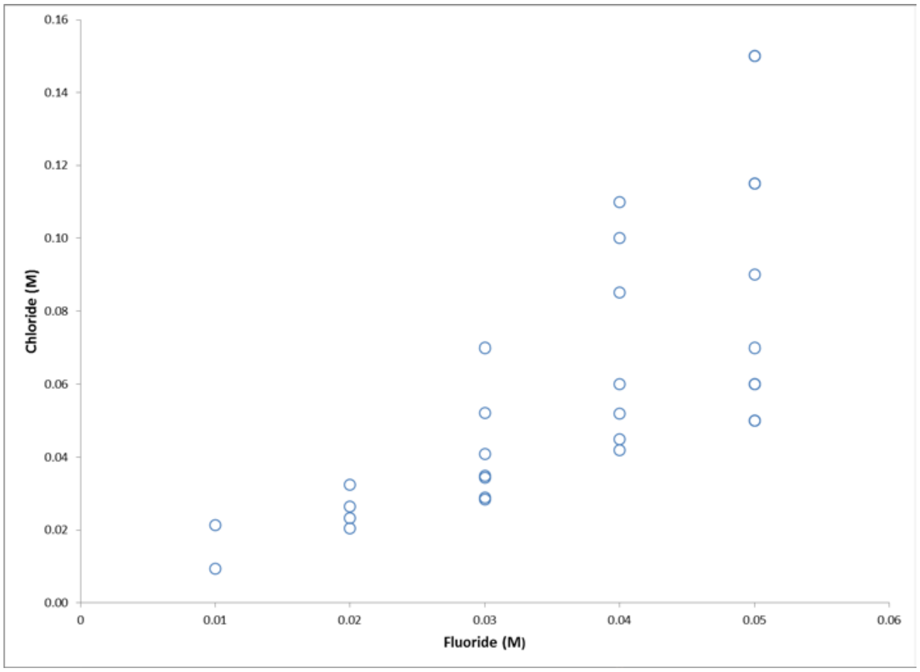


Figure 3. Chloride and fluoride concentrations that were utilized for the 30 CPP tests.

3.0 Interpretation of protocol results

During 2014, the EPOC also standardized an approach for interpreting the results of the CPP tests [4]. Important aspects of this approach are summarized as a reference here since they will be utilized in the discussion of the results. Figure 4 shows a schematic of an idealized CPP curve along with experimental parameters that are measured from the curve.

Definitions for these polarization parameters are:

E_{zc} = Zero Current Potential:	The potential at zero current, measured on the forward scan.
E_{max} = Peak Current Potential:	The potential at the active peak prior to passivation.
E_{pit} = Pitting Potential:	The potential at which stable pits initiate on the forward scan. The increase in current at this potential may not be associated with pitting. The potential may be the result of other anodic reactions (e.g., oxygen evolution). In that case the potential may be referred to as the transpassive potential (E_{trans}). A transpassive potential is often observed for samples that have negative hysteresis.
E_{prot} = Protection Potential:	The potential at which pits (if they occur) passivate and stop growing on the reverse scan or the potential where passivation is reestablished.

i_{cor} = Corrosion Current Density: The corrosion current density, which is related to the corrosion rate by Faraday's law.

i_{max} = Peak Current Density: The current density at the active peak prior to passivation.

i_{pas} = Passive Current Density: The current density in the passive range.

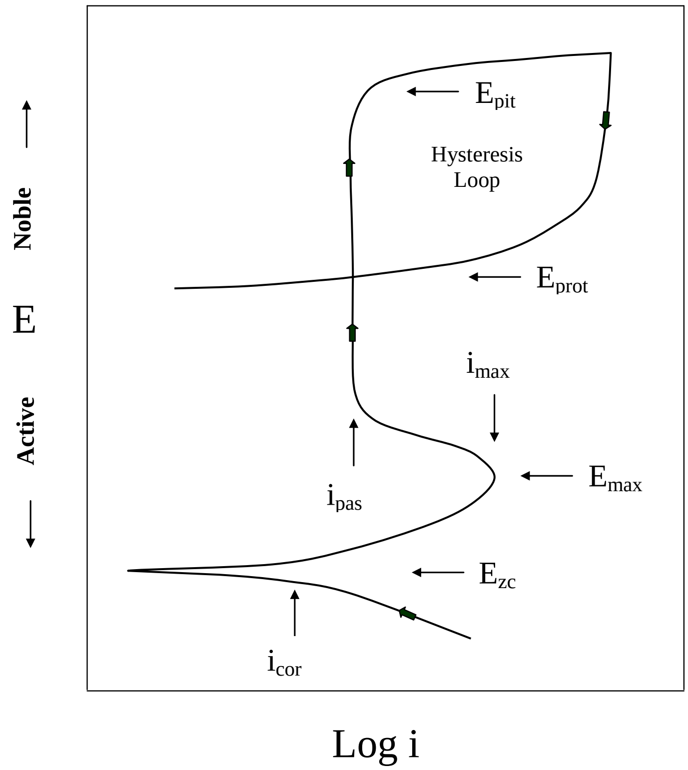


Figure 4. Schematic of an idealized (CPP) Curve.

The zero current potential, E_{zc}, taken from CPP curves is the potential at which the current changes polarity from negative to positive on the forward scan. The corrosion potential, E_{cor}, also sometimes referred to as the open circuit potential, is the potential of a specimen measured under open circuit conditions where the specimen is connected solely to a high impedance voltmeter. In a CPP test, E_{cor} is measured for a short time period (e.g., 2 hours) prior to starting the scan and the scan is started at a fixed voltage (e.g, 100 mV) below the measured E_{cor} The E_{zc} may not be the same potential as E_{cor} measured before starting the scan. E_{cor} typically moves in the noble direction with exposure time for passive alloys. Therefore, the E_{cor} value measured prior to starting a CPP scan and E_{zc} typically are more negative than E_{cor} values measured after longer exposure times.

If the sample is corroding actively at E_{zc}, the current will increase exponentially as the potential is scanned upwards from E_{zc}, exhibiting a straight line in the semi-log plot. Samples susceptible to pitting must be passive, so an active/passive transition resulting in a peak current density, i_{max}, will be observed for such samples. Under conditions where the alloy is spontaneously passive, the current reaches a relatively constant value just above E_{zc}, so that i_{max} is not observed. In the passive region, the current, i_{pas}, is usually almost constant, with little dependence on potential.

The pitting potential is the value at which the current increases rapidly owing to the onset of stable pitting. In most instances, pitting potentials are reasonably easy to define by a change in slope and a sharp increase in the corrosion current. The occurrence of positive hysteresis, where the current on the reverse (downward) scan is higher than during the forward scan, is usually indicative of the occurrence of localized corrosion such as pitting or crevice corrosion. For steel samples that do not exhibit localized corrosion, the current will eventually increase above i_{pass} at high applied potentials owing to oxygen evolution by water oxidation. In such a case, during the reverse scan, the current will trace back along the increasing part of the forward scan with no evidence of hysteresis. Often, a negative hysteresis is observed where the passive current on the reverse scan is lower than that on the forward scan. Pitting and crevice corrosion are almost never found in association with such a CPP curve. The potential in this case is referred to as a transpassive potential (E_{trans}) rather than the pitting potential.

For a sample exhibiting pitting and a positive hysteresis, the pits will eventually repassivate during the reverse scan as the potential is lowered. The potential at which this happens is called the protection or repassivation potential (E_{prot}). This is a critical parameter in the assessment of localized corrosion susceptibility because a conservative approach for designing against localized corrosion would be to determine that the corrosion potential would remain well below this value. E_{prot} is often defined as the potential at which the current on the reverse scan falls below that observed on the forward scan. In other words, it is the potential at which the reverse scan crosses the forward scan as shown in Figure 4. However, in some cases, the passive current on the reverse scan is higher than that on the forward scan. In that case, the protection potential is taken as the point at which the current exhibits a sharp decrease. In other cases, the protection potential is below the E_{zc} observed on the forward scan. If the original E_{zc} was used as the final limit for the reverse scan, then the protection potential cannot be definitively determined in this situation.

The severity of pitting corrosion can be ranked based on the shape of the CPP curve according to five categories:

Category 1: Negative hysteresis and no evidence of pitting.

Category 2: Positive hysteresis, but with pitting and protection potentials well above the zero current potential ($E_{prot} >> E_{zc}$).

Category 3: Positive hysteresis with a noble pitting potential, but with the protection potential relatively near the zero current potential (E_{prot} near E_{zc}).

Category 4: Positive hysteresis with the protection potential lower than the zero current potential ($E_{prot} < E_{zc}$).

Category 5: Spontaneous pitting at the zero current potential so that the current increases rapidly upon polarization to potentials above the zero current potential.

These categories are shown graphically in Figures 5 to 9. For these figures, the metal is assumed to be passive at the free corrosion potential so no active-passive transition is shown.

The Category 1 ranking (Figure 5) is the most desirable because it indicates that the environment is not capable of promoting pitting of the alloy. This should be confirmed by a post-test examination of the specimen. Note that the potential associated with the significant increase in current on the forward scan is not called a pitting potential (E_{pit}) for Category 1 because it is not associated with pitting corrosion. The increase in current is associated with water breakdown or transpassive behavior and the potential is referred to as the transpassive potential (E_{trans}) in Figure 5. This case is defined as a “pass” condition and no additional testing is required; the environment is considered to be benign with respect to pitting.

For Categories 3 through 5 (Figures 6 through 9) localized corrosion is likely to occur in service. In the presence of pitting on the sample, these categories are considered a “fail” condition; the environment is considered to be aggressive with regard to pitting.

All other outcomes require additional testing. Examples of other outcomes include:

- Category 1 behavior with pitting on the sample;
- Category 2 behavior (Figure 6) with or without pitting;
- Category 3 through 5 behavior with no pitting;
- Undefined hysteresis with or without pitting; this type of behavior is typified by the reverse scan following close to the forward scan or crossing it several times.

Additional tests include ASTM G192 [7] long-term coupon immersion testing, and in-tank reference electrode measurements to determine E_{cor}. The ASTM G192 protocol is being modified for carbon steels in waste simulants by DNV-GL.

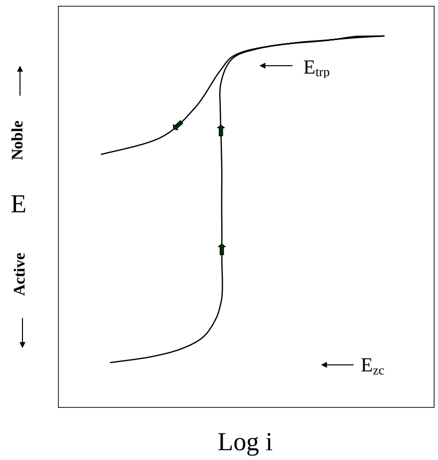


Figure 5. Schematic of Category 1 CPP Curve.

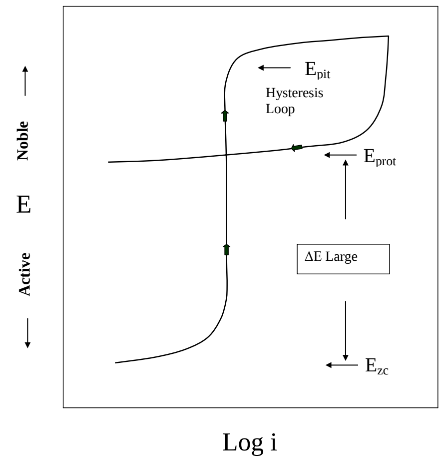


Figure 6 Schematic of Category 2 CPP Curve.

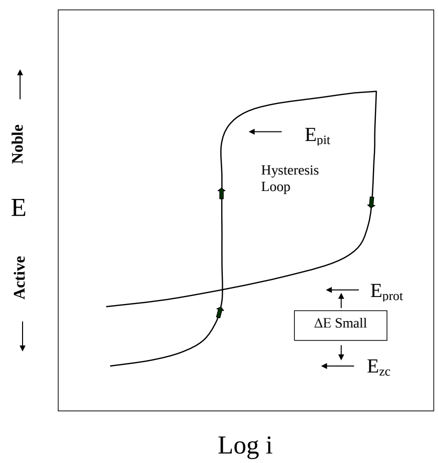


Figure 7. Schematic of Category 3 CPP Curve.

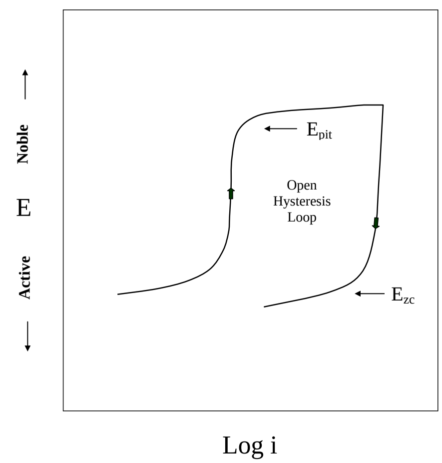


Figure 8. Schematic of Category 4 CPP Curve.

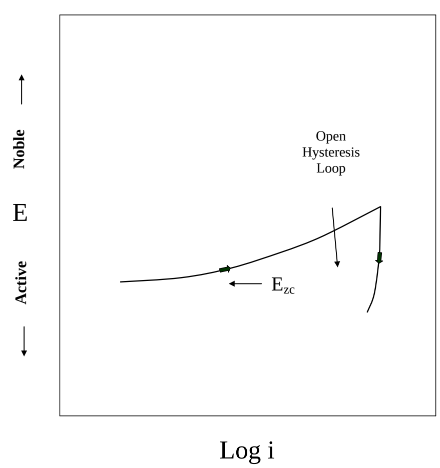


Figure 9. Schematic of Category 5 CPP curve.

4.0 Results and discussion

CPP tests were performed on the 30 test simulant formulas at 35℃ for all the solution compositions presented in Figure 2 and Figure 3. The simulated compositions were prepared by combining the chemical components in a step-wise order based on the solubility of the constituent. The amount of carbonate and bicarbonate required was estimated by OLI™ simulations. A table of the amounts used is presented in Appendix A of this report. The pH of the simulated waste forms was recorded and adjusted to a pH of 10 by adding sodium bicarbonate or sodium hydroxide at the testing temperature of 35℃ before testing and recorded after testing. The pH values measured during solution preparation and testing are included in Appendix A. Table 2 summarizes the test results of the CPP scans. The actual scans are presented in Appendix B. Tests at conditions 1, 4, 11, 19 and 26 were also conducted at DNV, which resulted in a Category 1 with minor pitting in agreement with the SRNL results [8, 9].

The test results indicated either Category 1 or Category 1 with minor pitting for all conditions. Category 1 behavior indicates no pitting susceptibility, while the protocol dictates that Category 1 with minor pitting be further evaluated with tests such as the ASTM G192 method [10]. The ASTM G 192 test performed by DNV-GL measured the E_{prot}. For this test, the potential was scanned to a potential at which the current density was 50 μA/cm^2. This constant current density was applied for 4 hours to allow sufficient time for pit propagation. The potential was then stepped in 10 mV decrements and the current monitored. For each potential step, the current decreased with time (i.e., an indication that pits were not propagating or initiating). The magnitude of the current decreased below the i_{pas} after the eleventh potential step decrement. This potential was reported as E_{prot}.

As an example, test condition 1, which contained the lowest nitrite (inhibitor) at 1M, resulted in a Category 1 scan with minor pitting on the electrode. The G 192 method was conducted at DNV-GL for Test 1[11]. The measured E_{prot} for Test 1 was reported as +664 mV (vs. SCE reference electrode). Similarly, DNV performed the G192 technique for Test 19 conditions and measured +618 mV for the protection potential. In both cases, the results of the G 192 method correlate very well with the CPP scan in that the potential at which the current density equal to i_{pass} during the reverse scan results in nearly the same E_{prot} that was determined from the G192 test This intersection is illustrated in Figure 10 for the CPP of Test 1. From this test, the E_{prot} is + 662 mV which agrees very well with the +664 mV measured in the G 192 test. Table 3 provides the estimates for E_{prot} for other selected tests. Although not within the approved protocol, it appears that this extrapolated value for E_{prot} from the CPP scan, which shows clear negative hysteresis, could provide an estimate of the E_{prot} for an initial evaluation. Given this assumption, Table 3 shows the estimated E_{prot} values were consistently greater than +550 mV. A review of all the plots in Appendix B shows the same general trend.

The protocol set forth in reference 10 dictates that no additional testing is required when the difference between E_{prot} and the long term (i.e., several days or weeks) open circuit potential is greater than 200 mV. The potential for zero current may be used as an initial estimate for the long term open circuit potential. Table 3 shows that difference between the estimated E_{prot} and E_{zc} is on the order of 800-1000 mV. Testing to date in similar waste simulant compositions has shown that the long term open circuit potential is typically 200 to 300 mV more positive than the potential of zero charge for the forward scan [12]. Thus, the difference between the protection potential and the long term open circuit potential is likely on the order of 500-700 mV. This result still suggests that the minor pits that were observed did not propagate and the conditions are benign.

Table 2. CPP Test Results for the Nitrite and Halide Concentrations at pH 10 and 35℃.

Test ID	Nitrite, (M)	Cl^- (M)	F^- (M)	Total halide	Category	Visual Pitting (Y/N)	Notes
1	1	0.01	0.01	0.020	1	Y	
2	1	0.021	0.01	0.031	1	Y	
3	1	0.023	0.02	0.043	1	Y	
4	2	0.020	0.02	0.040	1	Y	10-20µm pits observed under SEM
5	2	0.026	0.02	0.046	1	Y	
6	2	0.032	0.02	0.052	1	Y	
7	2	0.028	0.03	0.058	1	Y	
8	2	0.034	0.03	0.064	1	N	
9	2	0.052	0.03	0.082	1	Y	
10	2	0.070	0.03	0.100	1	Y	
11	3	0.029	0.03	0.059	1	Y*	*under magnification
12	3	0.035	0.03	0.065	1	Y	
13	3	0.041	0.03	0.071	1	Y	
14	3	0.045	0.04	0.085	1	Y	
15	3	0.060	0.04	0.100	1	Y&N	1 shows visual pits, 1 shows no visual pits
16	3	0.085	0.04	0.125	1	N	
17	3	0.110	0.04	0.150	1	N	
18	4	0.100	0.04	0.140	1	Y	
19	4	0.042	0.04	0.082	1	Y	
20	4	0.052	0.04	0.092	1	Y	
21	4	0.050	0.05	0.100	1	Y&N	1 shows visual pits, 1 shows no visual pits
22	4	0.060	0.05	0.110	1	Y&N	1 shows visual pits, 1 shows no visual pits
23	4	0.115	0.05	0.165	1	N	
24	4	0.150	0.05	0.200	1	N	
25	5	0.090	0.05	0.140	1	Y	
26	5	0.050	0.05	0.100	1	Y*	*under magnification
27	5	0.060	0.05	0.110	1	N	
28	5	0.070	0.05	0.120	1	Y&N	1 shows visual pits, 1 shows no visual pits
29	5	0.115	0.05	0.165	1	N	
30	5	0.150	0.05	0.200	1	N	

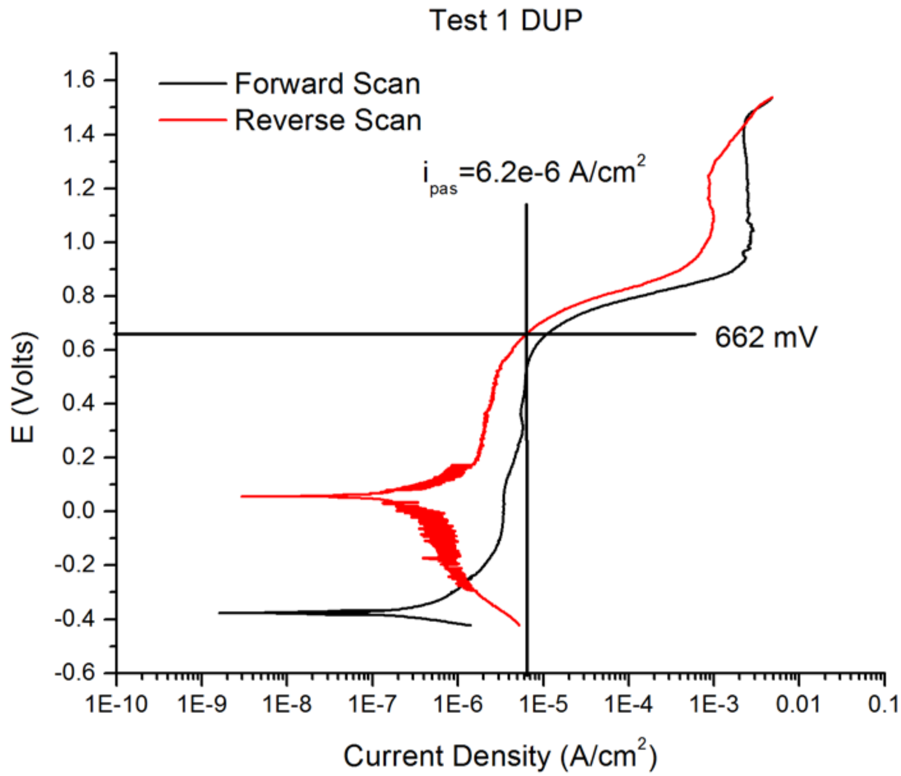


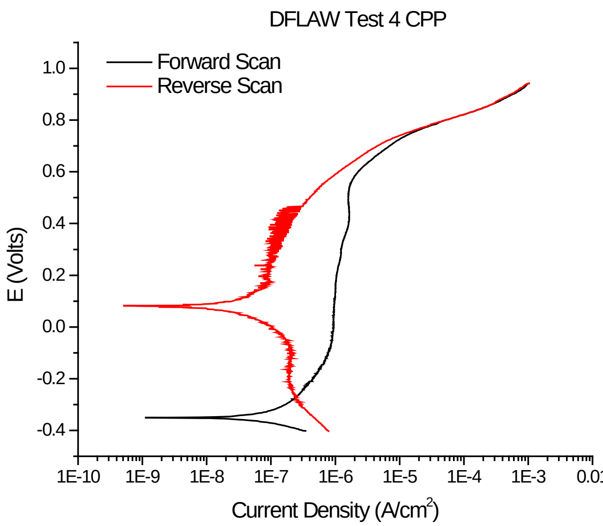
Figure 10. CPP Plot of Test 1 showing the estimated value for E_{prot}.

Table 3. Comparison of E_{prot} values estimated from the CPP scans and E_{prot} determine by the G 192 test.

	Extrapolated E_{prot}	E_{prot} from DNV-GL via G 192 test	E_{prot} - E_{zc}
Test1	631	664	981
	662		1032
Test 4	630		981
	625		884
Test 11	586		897
	605		873
Test 19	608	618	943
	624		986
Test 26	599		918
	623		914

A number of the working electrodes showed what were presumed to have visible pits of various degrees. In some cases, the pits were not observable unless magnified 20X beneath a microscope. CPP results from Test 4 were examined in more detail and are presented in Figure 11 and 12. [Note: Not all coupons were examined under 20x magnification to determine pitting or measure pit diameters via SEM.

However, in an attempt to distinguish between a pit and an etched inclusion, the working electrode from Test 4 was examined more closely.] The CPP curves for this test display a negative hysteresis and have a separation between the E_{prot} and E_{zc} potentials of about 900 mV. The scanning electron microscope images in Figure 12 reveals the inclusions to be about 10-20 μm in diameter. Energy dispersive x-ray spectroscopy indicates these regions to be rich in iron, manganese, and oxygen and residual sodium presumably from the simulated waste. This observation suggests that a manganese sulfide inclusion has been etched and removed rather than the presence of a propagating pit.



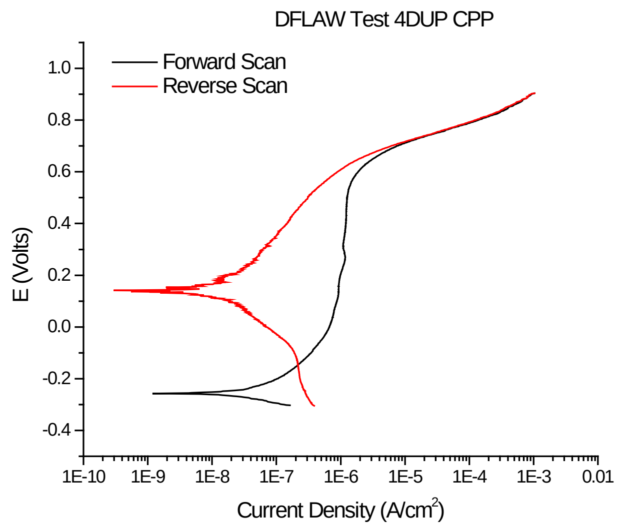
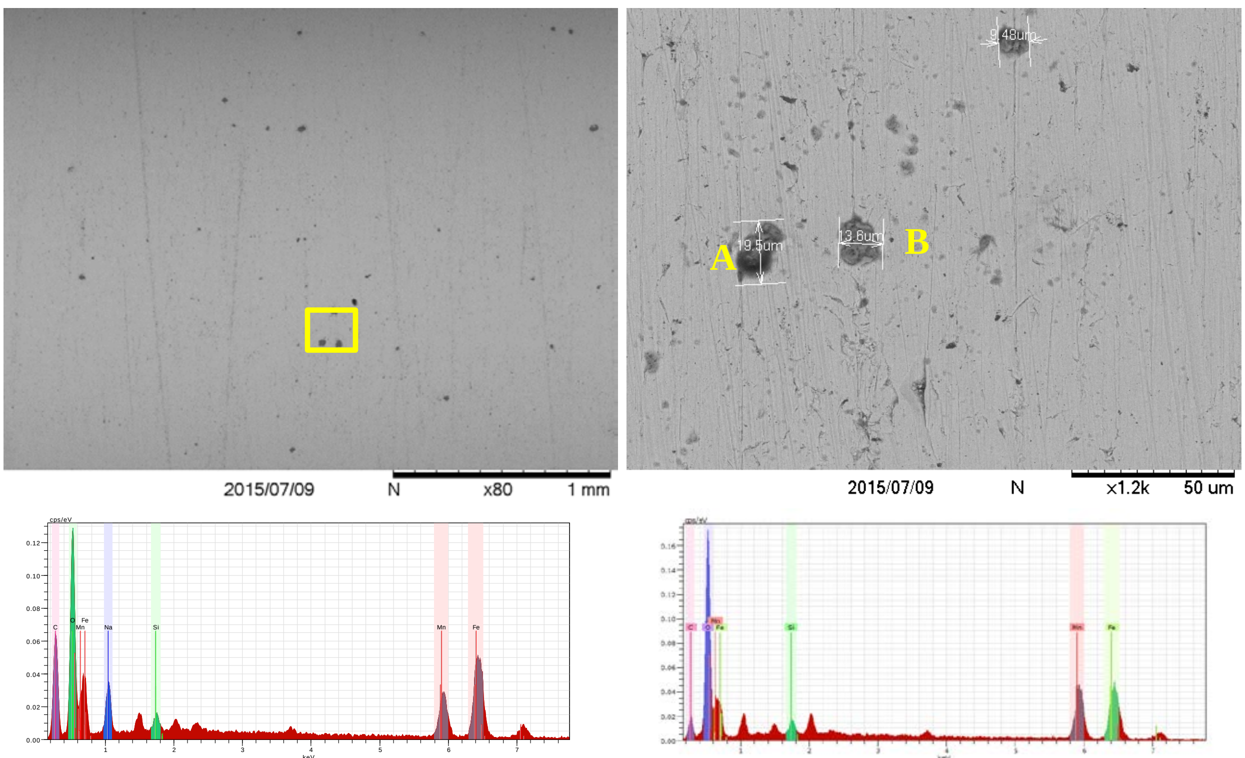


Figure 11. CPP results of Test 4 at 35℃.

The model that was tested (see Figure 2) is an empirical fit to data for test conditions that were performed at halide concentrations up to 0.05 M. These present test conditions were selected to assess if the empirical fit could provide a good prediction of the nitrite concentration needed to inhibit pitting at higher chloride concentrations (i.e., could the empirical fit be extrapolated). The current tests do seem to suggest that at nitrite concentrations greater than 3 M the model overestimates the amount of nitrite that is needed for halide concentrations greater than 0.05 M. The observation of no pitting at halide concentrations that are 50-100% greater than the model predicts are pitting conditions, is an indication that the model is conservative. At the 2 M nitrite concentration, the percentage difference is similar, however, a smaller difference exists between the inhibitor concentration predicted by the model. The data at1 M nitrite concentration provides the least information about the margin of conservatism since the halide and nitrite concentrations are relatively close to the model. While quantifying the degree of conservativism that is assumed by using the extrapolated model is difficult, since no pitting conditions were observed during the tests, the present results seem to suggest that the actual inhibitor concentration needed to mitigate pitting is less than originally thought. Future testing will delineate the conditions where pitting susceptibility is indicated and provide appropriate corrosion control limits.

The purpose of this work was to determine the applicability of the SRS equation for inhibition of pitting corrosion due to chlorides as a means of corrosion control for the WTP off-gas stream that may be returned to the waste tanks. The question may be answered two ways. At halide concentrations greater than 0.05 M, the nitrite concentrations predicted by the model are not applicable to the situation for two reasons. First, the nitrite concentrations predicted by the model quickly approaches the solubility limit for sodium nitrite by itself in solution. For example, at 0.2 M halide the model requires a minimum of 12 M nitrite to inhibit pitting, a composition which likely will not dissolve in the mixture at 35 °C. Secondly the results of the tests indicate that for solutions up to 0.2 M halide are not susceptible to pitting for nitrite concentrations between 2 to 3 M. These concentrations are well below that predicted by the model. These observations point to the limitations of extrapolation of experimental data and perhaps differences between the SRS simulants and the WTP off-gas simulants.

However, the results are also positive in the sense that they suggest that the model overestimates the amount of nitrite necessary for inhibit. Less inhibitor, hence less sodium, may be needed to inhibit the off-gas return stream. Future testing will define these new inhibitor concentration requirements. Until these limits are defined, the process flow sheet group may use the SRS equation realizing the limitations of the extrapolation of the model and that the new limits will be defined in the future.



A B

Figure 12**. SEM images and EDS Spectra of Test 4 Working Electrode. Inclusion sizes are 10-20 µm in diameter.**

5.0 Conclusions

A series of cyclic potentiodynamic polarization (CPP) tests were performed in support of the Tank Waste Disposition Integrated Flowsheet (TWDIF). The focus of the testing was to assess the effectiveness of the SRNL model for predicting the amount of nitrite inhibitor needed to prevent pitting induced by increasing halide concentrations. The testing conditions were selected to simulate the dilute process stream that is proposed to be returned to tank farms from the off-gas stream of the low activity waste melter.

The results of the CPP tests indicated Category 1 and Category 1 with minor pitting behavior as defined by the CPP test protocol. Category 1 behavior indicates no pitting susceptibility. Category 1 with minor pitting is resolved utilizing the ASTM G-192 method per the approved CPP test protocol. The results of the G 192 tests performed by DNV-GL for two of the test conditions indicated that the protection potential (E_{prot}) is at a large electropositive value (i.e., greater than +600 mV vs. SCE reference electrode). Estimates of the E_{prot} determined from the CPP curves performed at SRNL showed good correlation with the E_{prot} determined by G 192. An initial review of the CPP scans performed at SRNL indicated that at all tested conditions the values of the estimated E_{prot} are greater than +550 mV vs. SCE reference electrode. The difference between the estimated E_{prot} and the zero current potential (E_{zc}) was utilized as an initial assessment of the likelihood that the pit-like indications would propagate. A large difference, between E_{prot} and E_{zc}, greater than +400 mV, was observed on the CPP curves. This result suggests that the indications that were observed during the CPP test are not propagating and that the test conditions are relatively benign with respect to pitting. Characterization of the pits by SEM and EDS on one of the samples further suggested that the pit-like indications were due to etching of manganese sulfide inclusions rather than the development of propagating pits.

These results indicated the SRS chloride inhibition equation over estimates the amount of inhibitor needed for the anticipated WTP return stream conditions. Future testing will delineate the conditions where pitting susceptibility is indicated and provide appropriate corrosion control limits. The testing will also more accurately determine the difference between the protection and long term open circuit potentials to allow for an understanding of any borderline cases. Until these limits are defined, the process flow sheet group may use the SRS equation realizing the limitations of the extrapolation of the model and that the new limits will be defined in the future.

6.0 References

1. D. J. McCabe, W. R. Wilmarth, and C. A. Nash, “Waste Pretreatment Technology Process Development Plan for Hanford Waste Treatment plant Low Activity Waste Recycle”, SRNL-STI-2013-00351, July 2013.
2. P. E. Zapp, “Recommended Nitrite Limits for Chloride and Sulfate in ESP Slurries”, WSRC-TR-94-0250, June 6, 1994.
3. “SRNL-Tank Chemistry Testing and Analysis FY2015,” Statement of Work, SRNL Tracking Number 271655, October 2014.
4. T. M. Martin, “Subject: Expert Panel Oversight Committee August 2014 Meeting Outcomes”, RPP-ASMT-59979, Rev. 0, September 30, 2014.
5. E. N. Hoffman, B. L. Garcia-Diaz, and T. B. Edwards, “Effect of Chloride and Sulfate Concentration on Probability Based Corrosion Control for Liquid Waste Tanks – Part IV”, SRNL-STI-2011-00479, July 2012.

6. Sridhar, N., Private communication, DNV-GL Columbus, March 9, 2015.
7. ASTM G192-08, “Standard Test Method for Determining the Crevice Repassivation Potential of Corrosion-Resistant Alloys Using a Potentiodynamic-Galvanostatic-Potentiostatic Technique”, ASTM International, West Conshohocken, PA, 2014.
8. S. Chawla, et. al., “Test Plan and Status Report -06292015”, DNV GL Project No.: PP119843 – WRPS Tank Chemistry Testing, June 29, 2015.
9. S. Chawla, et. al., “Test Plan and Status Report -07272015”, DNV GL Project No.: PP119843 – WRPS Tank Chemistry Testing, July 27, 2015.
10. T.M. Martin, “Expert Panel Oversight Committee March 2015 Meeting Outcomes”, RPP-ASMT-60348, Rev. 0, March 2015.
11. S. Chawla, et. al., “Test Plan and Status Report -08102015”, DNV GL Project No.: PP119843 – WRPS Tank Chemistry Testing, August 10, 2015.
12. S. Chawla, et. al., “Test Plan and Status Report -009082015”, DNV GL Project No.: PP119843 – WRPS Tank Chemistry Testing, September 8, 2015.

Appendix A Supplemental Data

Table A-1. Measured pH values for the test solutions as prepared, as adjusted before testing, post-testing, and the OLI calculated Carbonate quantities used, and bicarbonate available for pH adjustment.

Test ID	$pH_{solution}$ at 35°C	pH_{init} at 35°C	pH_{final} at 35°C	Na_2CO_3, (M) From OLI	$HNaCO_3$,(M) From OLI
1	9.99	9.99	9.8	0.0414	0.0331
2	10.07	9.91	10.01	0.0414	0.0083
3	10.03	9.99	9.98	0.0414	0.0104
4	10.04	10.04	10.11	0.0443	0.0104
5	10.15	9.92	9.96	0.0444	0.0083
6	9.96	9.97	9.98	0.0444	0.0104
7	9.97	9.92	9.93	0.0443	0.0104
8	10.12	10.04	10.06	0.0444	0.0104
9	10.09	10.02	10.07	0.0444	0.0104
10	10.06	10.06	10.11	0.0445	0.0104
11	9.84	9.93	-	0.0438	0.0104
12	10.20	10.02	10.12	0.0438	0.0104
13	10.13	10.03	10.02	0.0438	0.0104
14	10.23	10.03	10.08	0.0438	0.0104
15	10.32	10.04	10.05	0.0438	0.0104
16	10.24	10.03	9.93	0.0439	0.0104
17	10.24	10.04	9.93	0.0439	0.0104
18	10.07	10.04	9.97	0.0409	0.0103
19	9.81	9.99	9.91	0.0409	0.0103
20	10.19	10.00	10.15	0.0409	0.0103
21	10.12	9.98	9.97	0.0408	0.0103
22	10.05	10.05	10.02	0.0408	0.0104
23	10.23	10.04	9.99	0.0408	0.0102
24	10.30	10.04	9.99	0.0408	0.0102
25	9.96	10.04	10.06	0.0365	0.0101
26	9.74	10.02	9.98	0.0366	0.0101
27	9.75	10.00	9.98	0.0366	0.0101
28	9.98	9.96	10.00	0.0366	0.0101
29	10.19	10.03	10.06	0.0365	0.0101
30	10.22	10.06	10.11	0.0365	0.0101

Appendix B Cyclic Potentiodynamic Polarization Scans

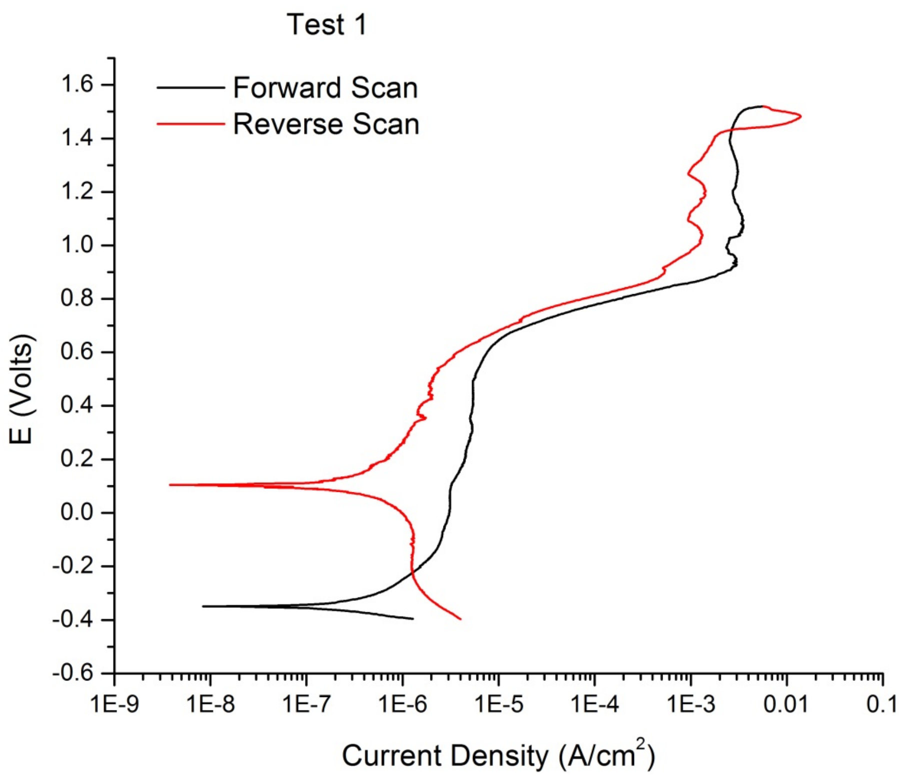


Figure B-1. Cyclic Potentiodynamic Test 1.

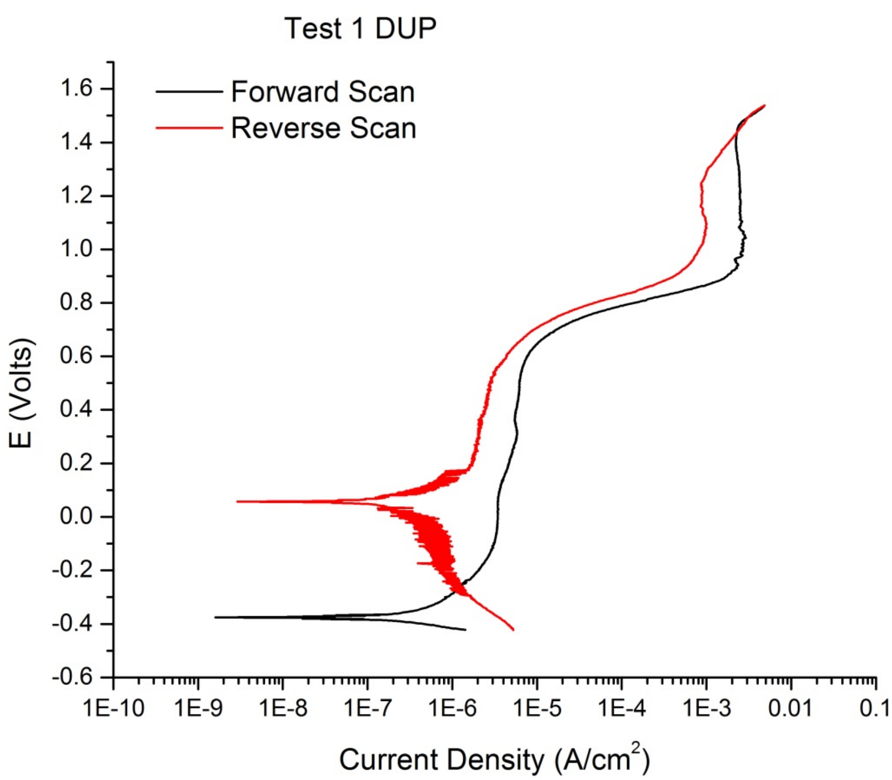


Figure B-2. Cyclic Potentiodynamic Test 1 DUP.

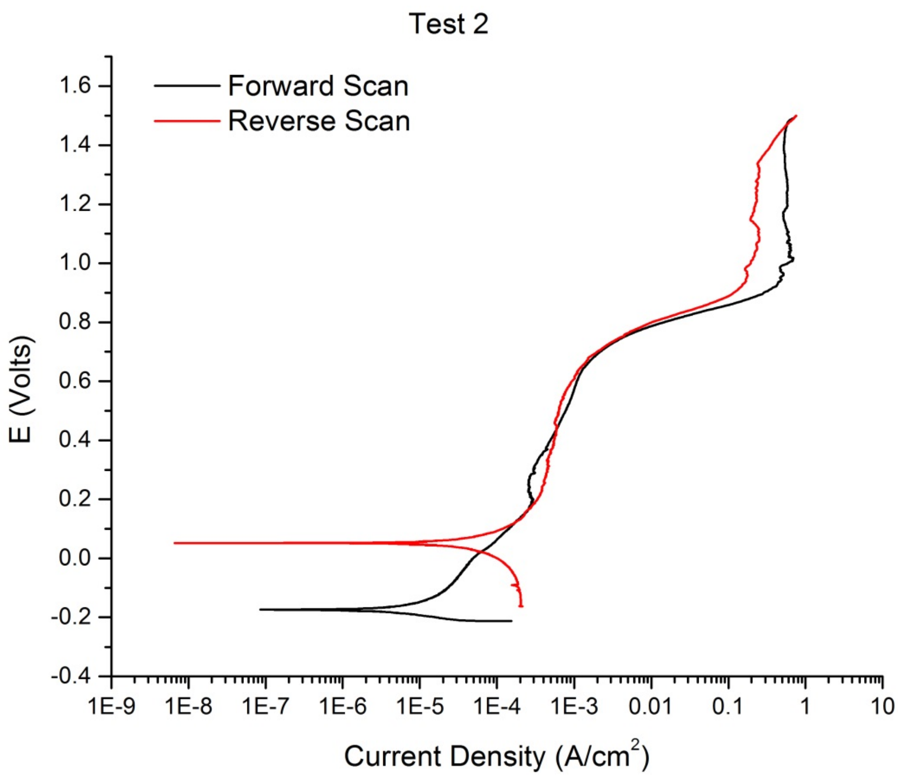


Figure B-3. Cyclic Potentiodynamic Test 2.

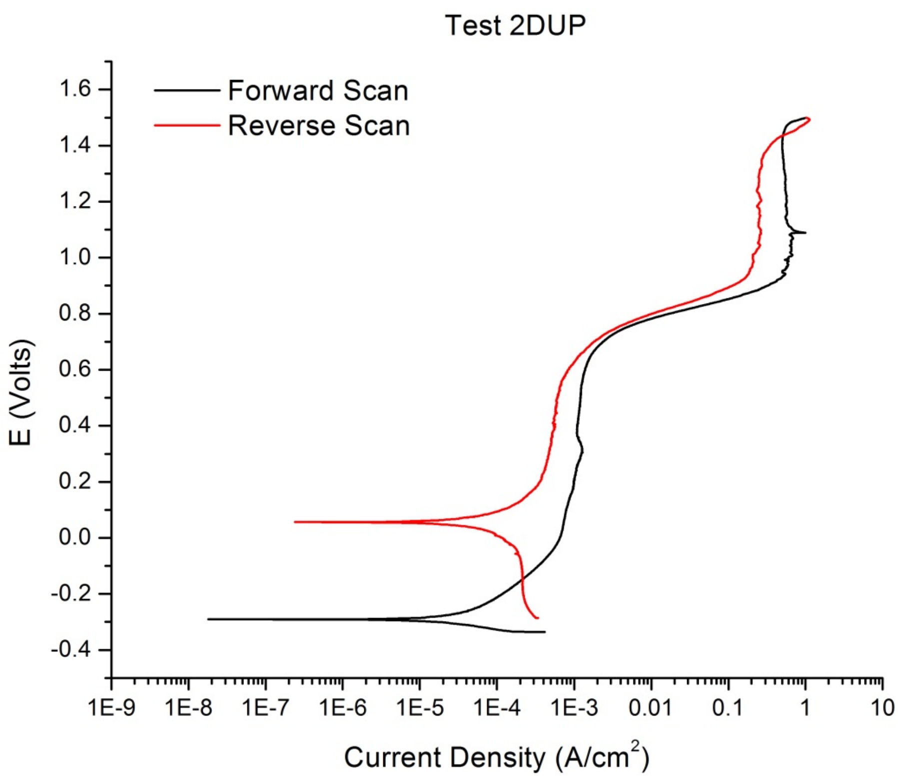


Figure B-4. Cyclic Potentiodynamic Test 2 DUP.

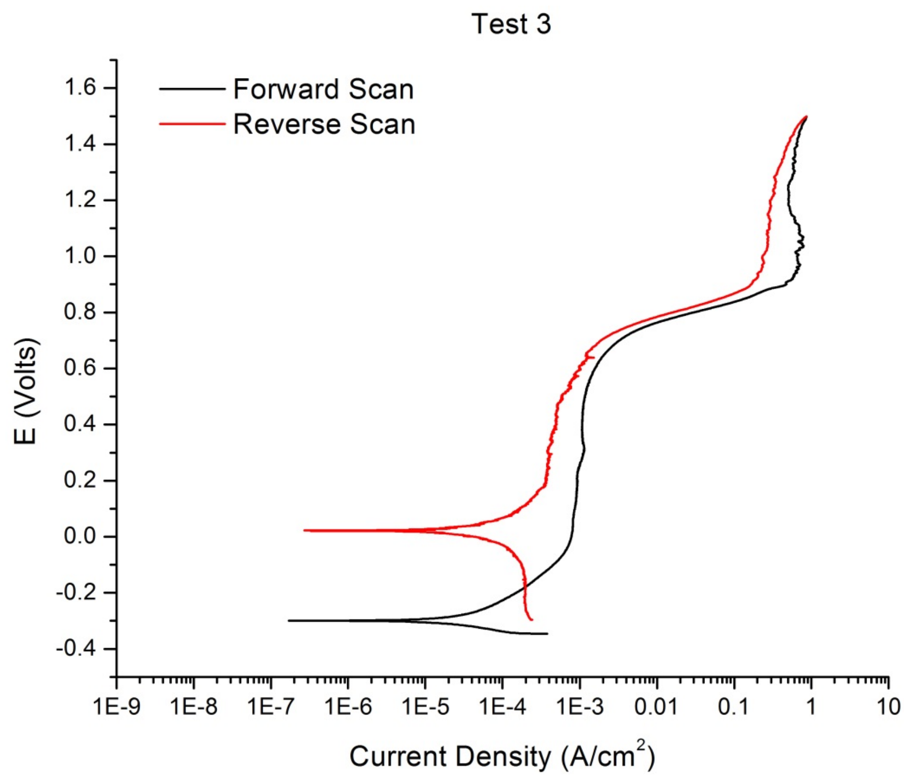


Figure B-5. Cyclic Potentiodynamic Test 3.

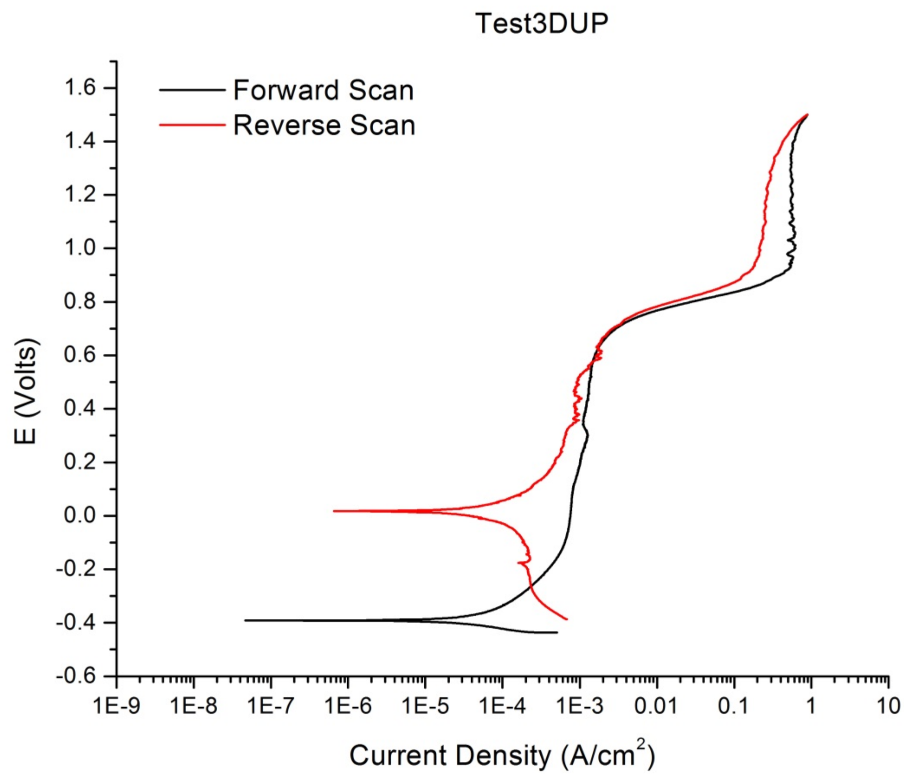


Figure B-6. Cyclic Potentiodynamic Test 3 DUP.

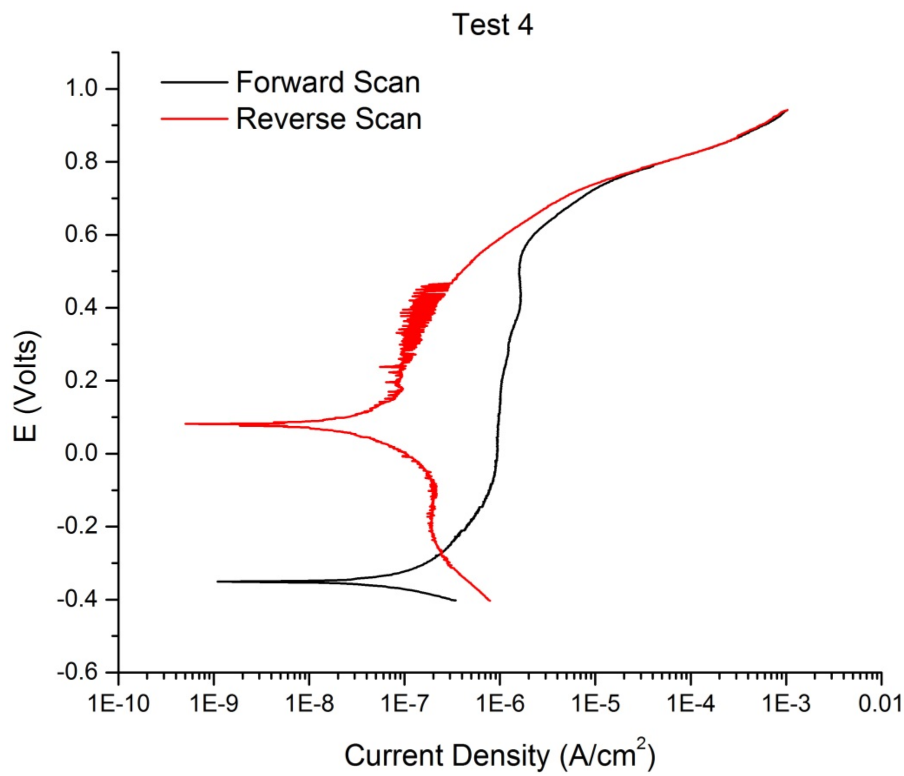


Figure B-7. Cyclic Potentiodynamic Test 4.

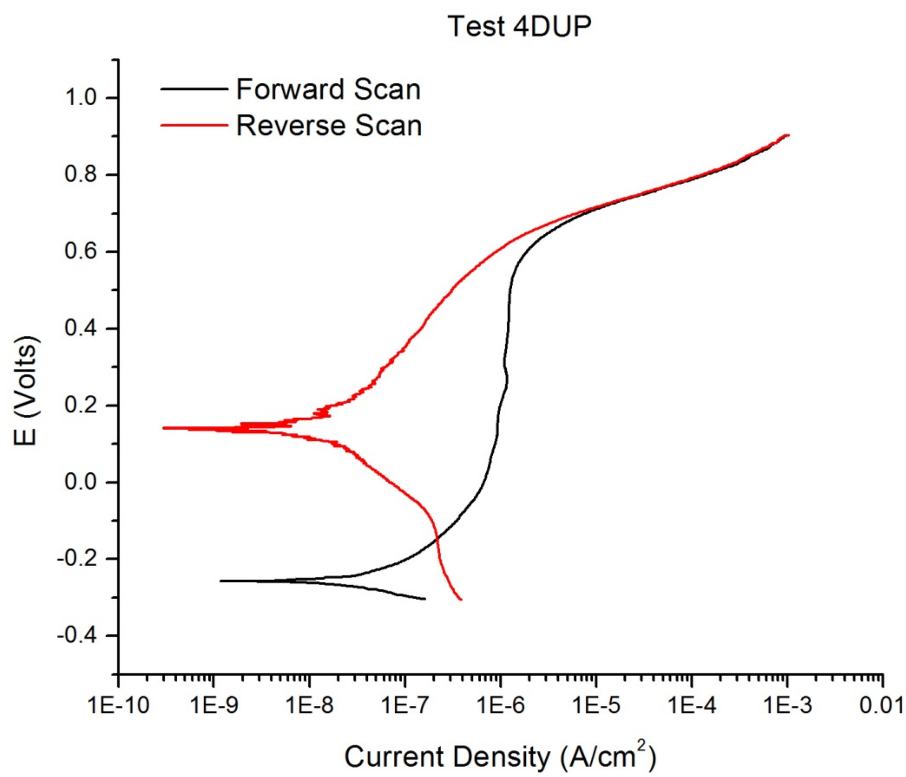


Figure B-8. Cyclic Potentiodynamic Test 4 DUP.

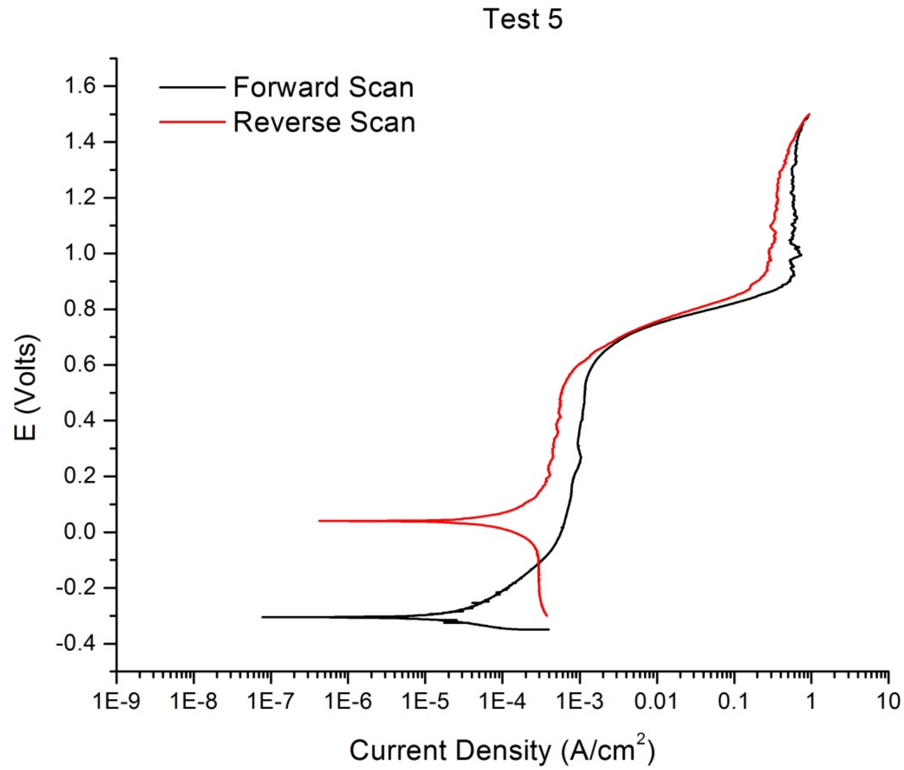


Figure B-9. Cyclic Potentiodynamic Test 5.

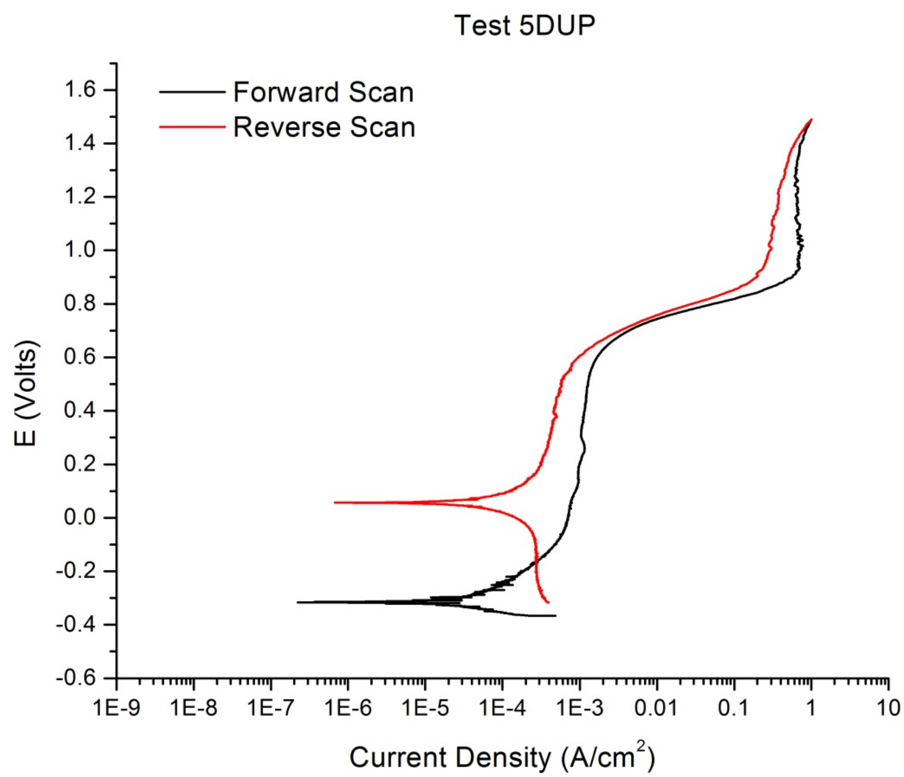


Figure B-10. Cyclic Potentiodynamic Test 5 DUP.

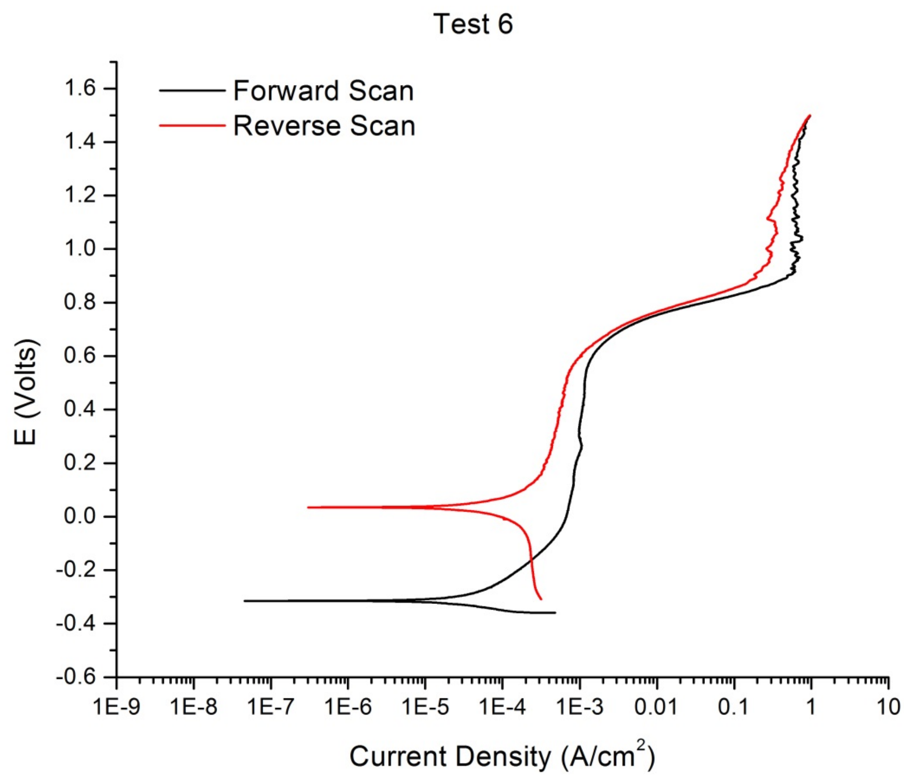


Figure B-11. Cyclic Potentiodynamic Test 6.

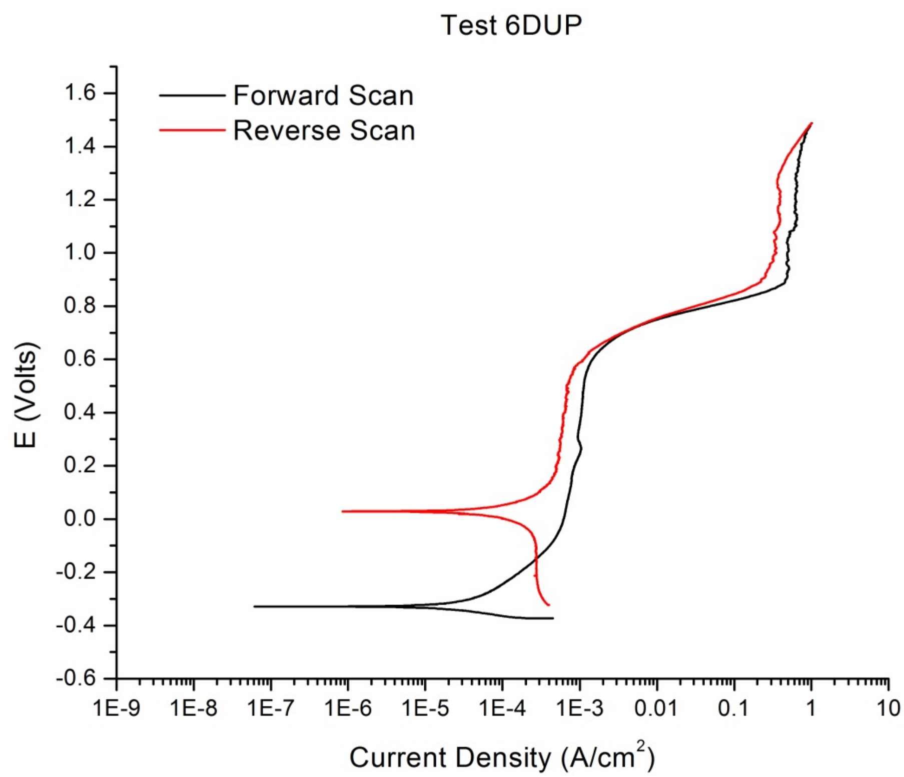


Figure B-12. Cyclic Potentiodynamic Test 6 DUP.

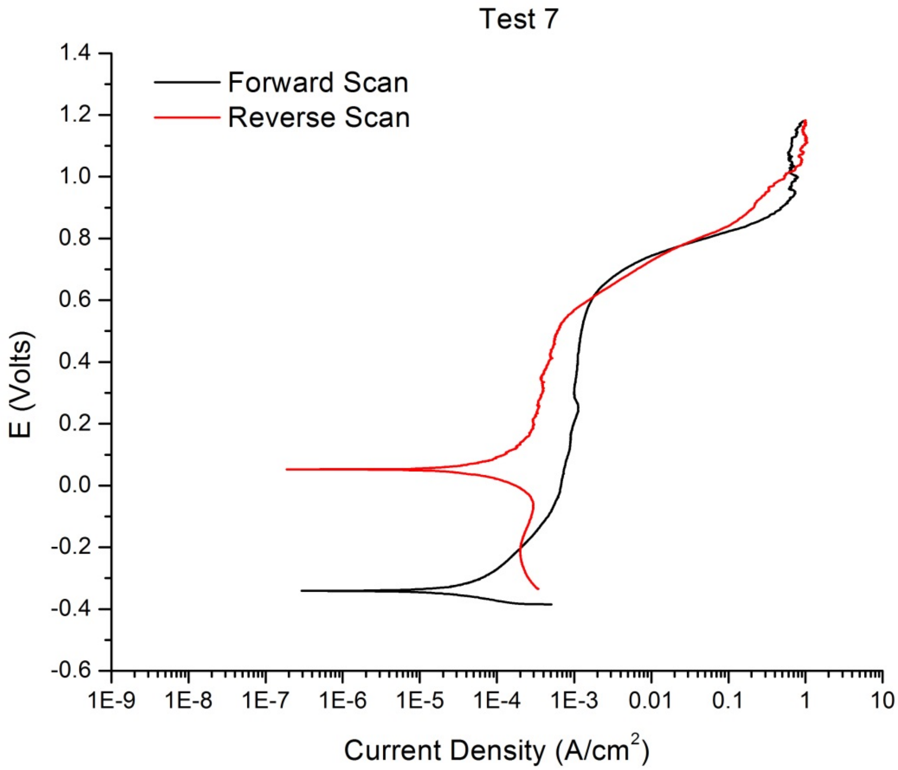


Figure B-13. Cyclic Potentiodynamic Test 7.

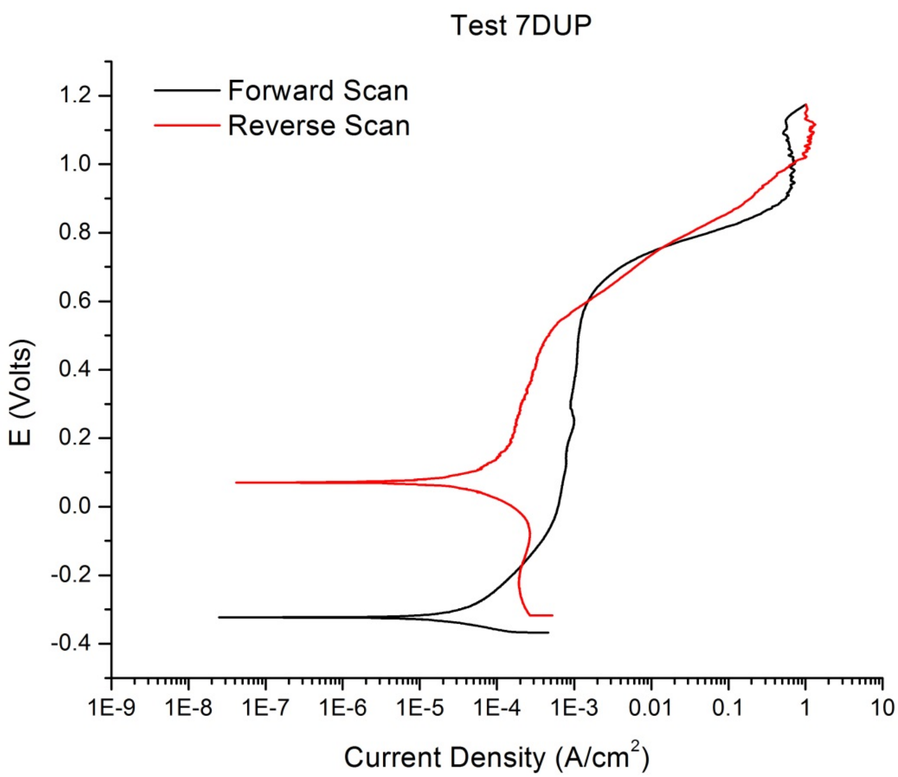


Figure B-14. Cyclic Potentiodynamic Test 7 DUP.

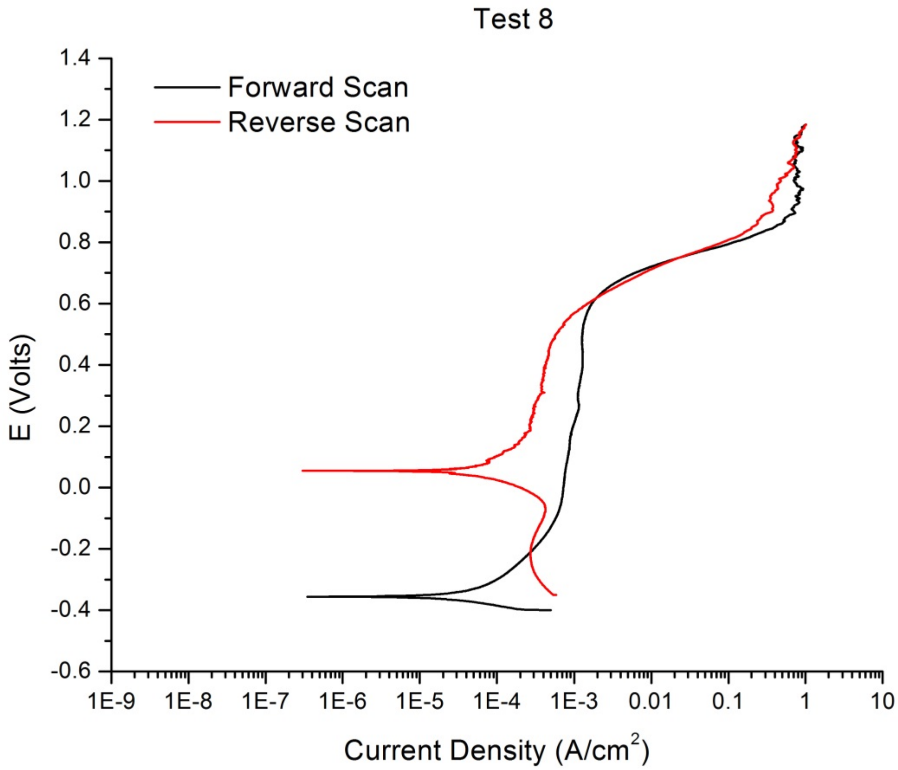


Figure B-15. Cyclic Potentiodynamic Test 8.

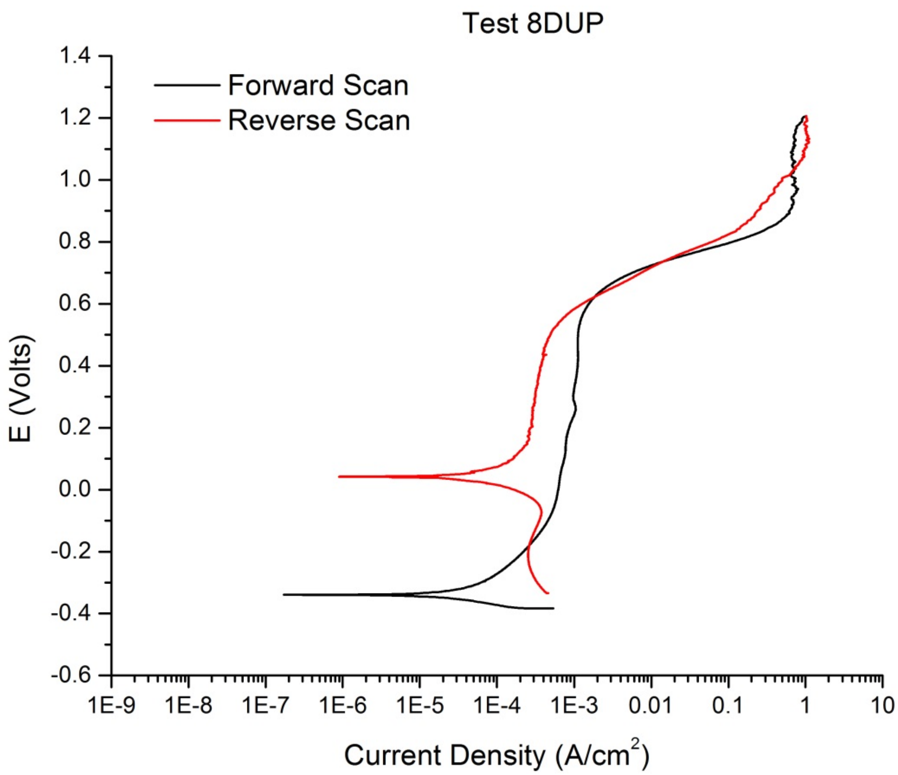


Figure B-16. Cyclic Potentiodynamic Test 8 DUP.

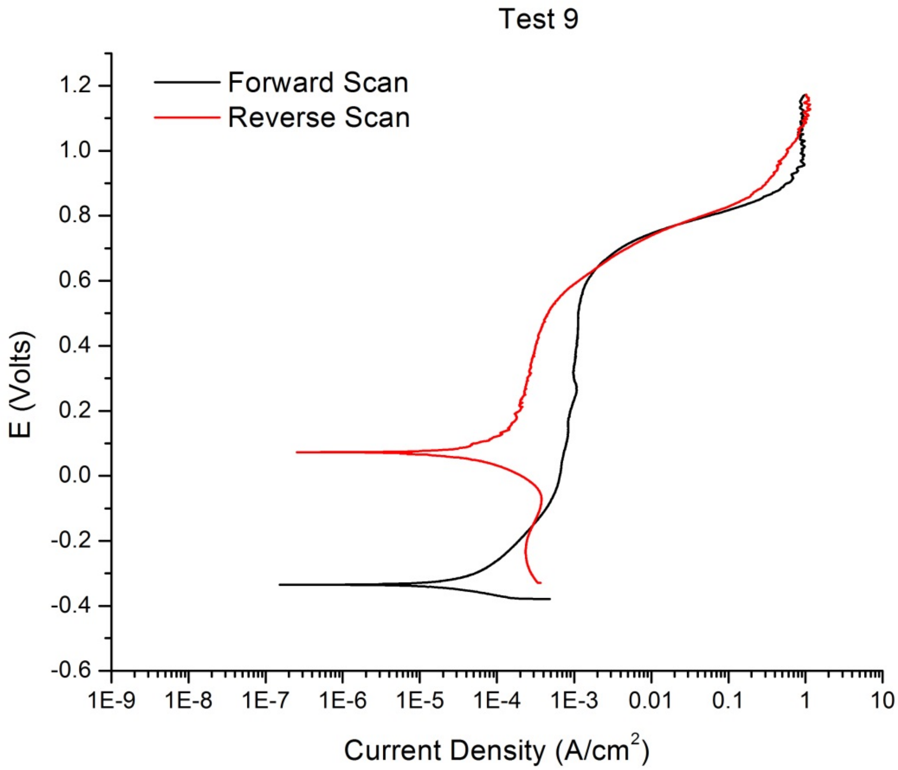


Figure B-17. Cyclic Potentiodynamic Test 9.

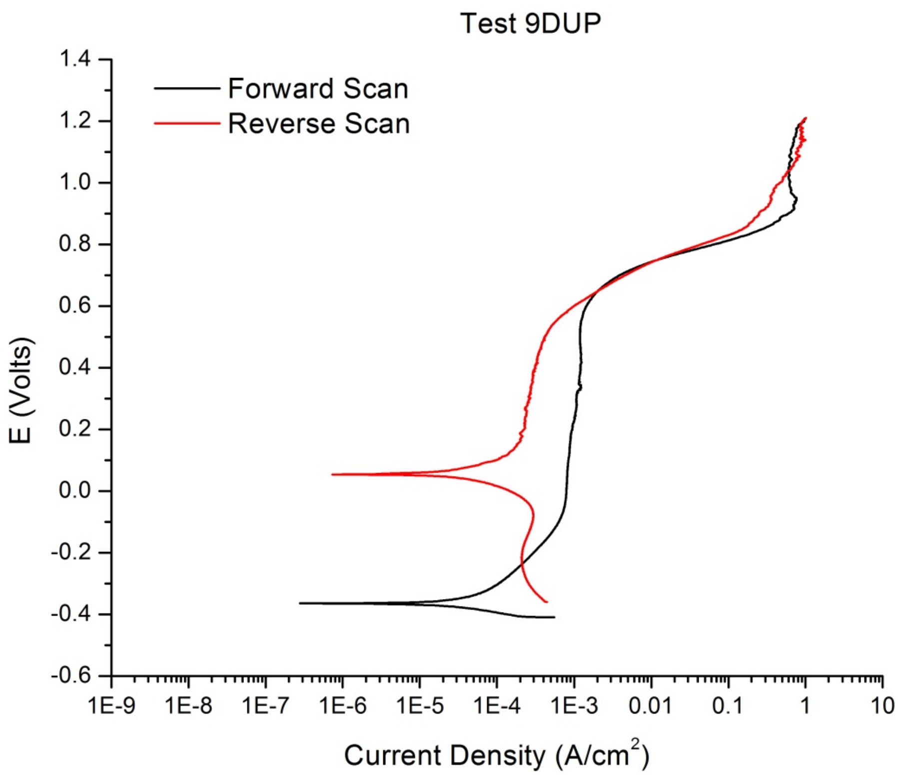


Figure B-18. Cyclic Potentiodynamic Test 9 DUP.

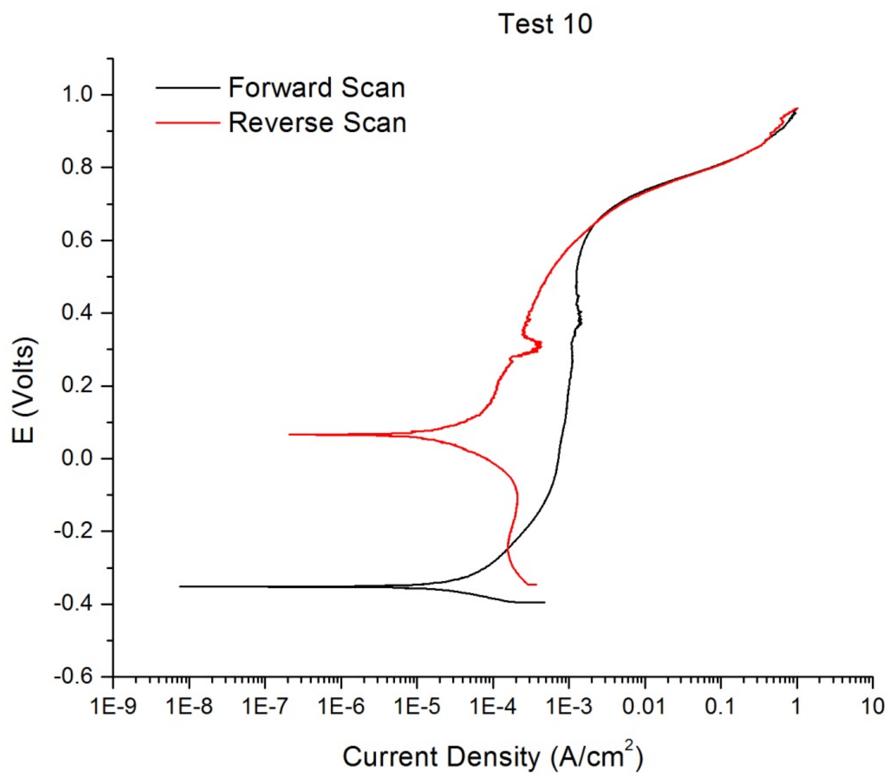


Figure B-19. Cyclic Potentiodynamic Test 10.

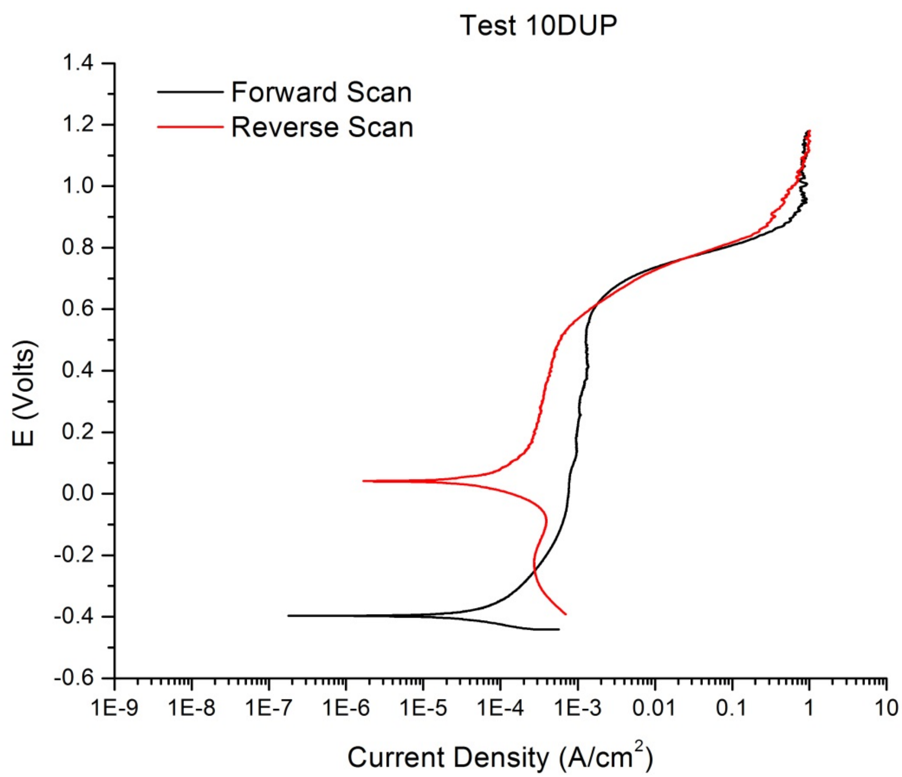


Figure B-20. Cyclic Potentiodynamic Test 10 DUP.

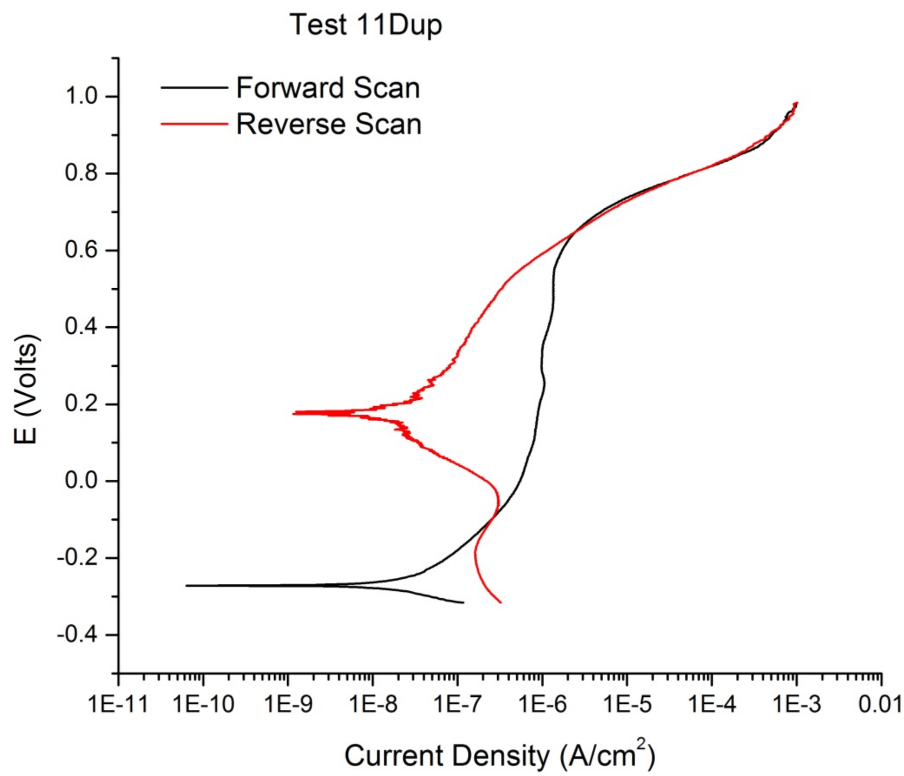


Figure B-21. Cyclic Potentiodynamic Test 11.

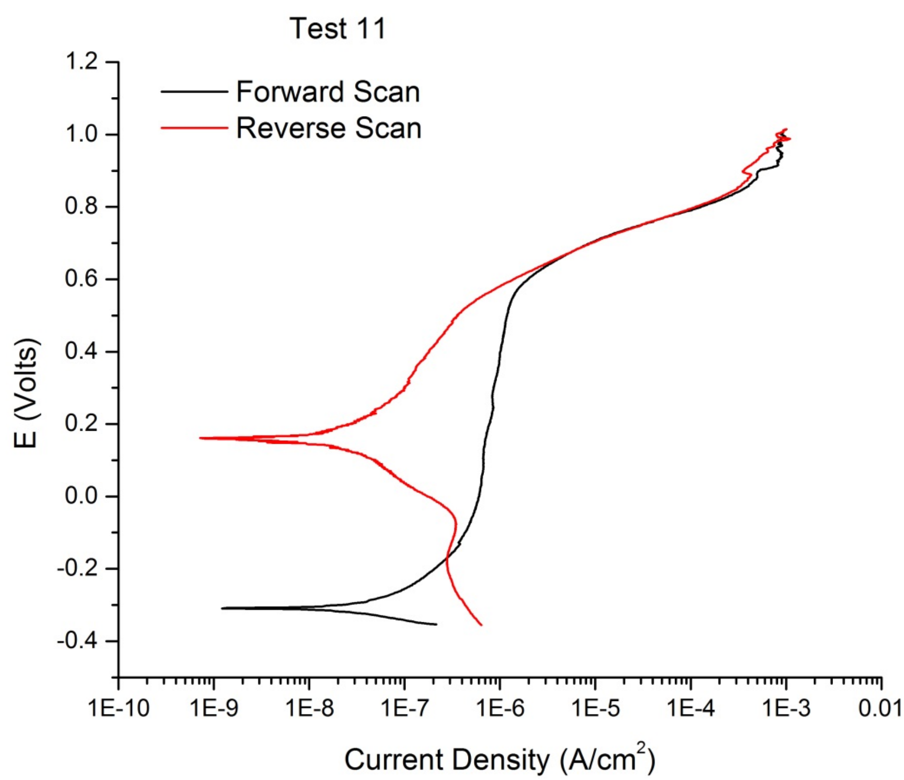


Figure B-22. Cyclic Potentiodynamic Test 11 DUP.

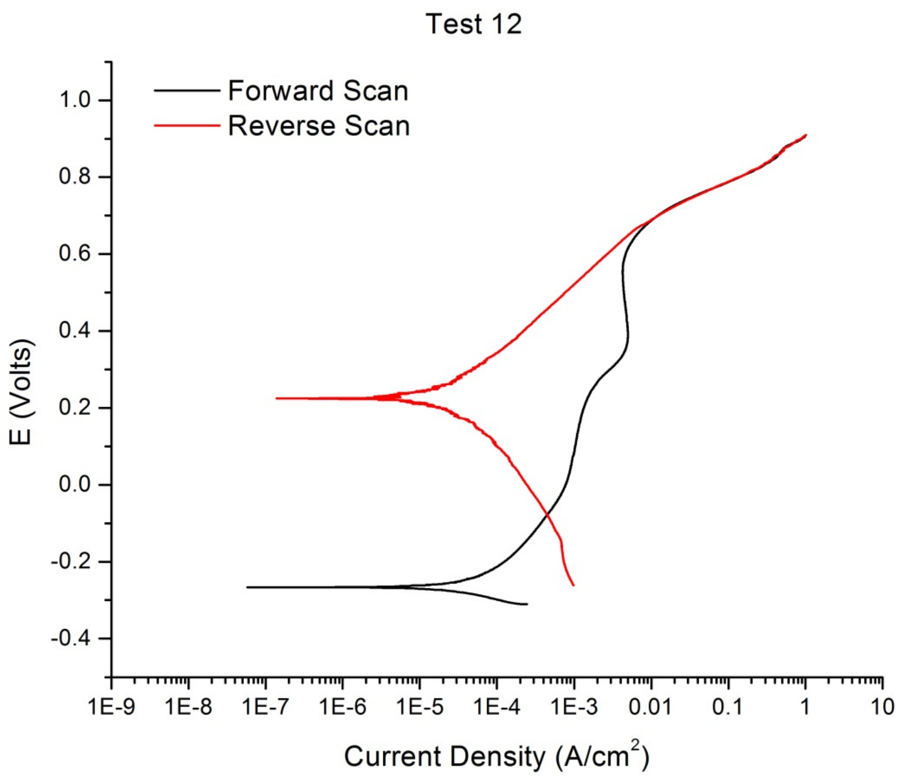


Figure B-23. Cyclic Potentiodynamic Test 12.

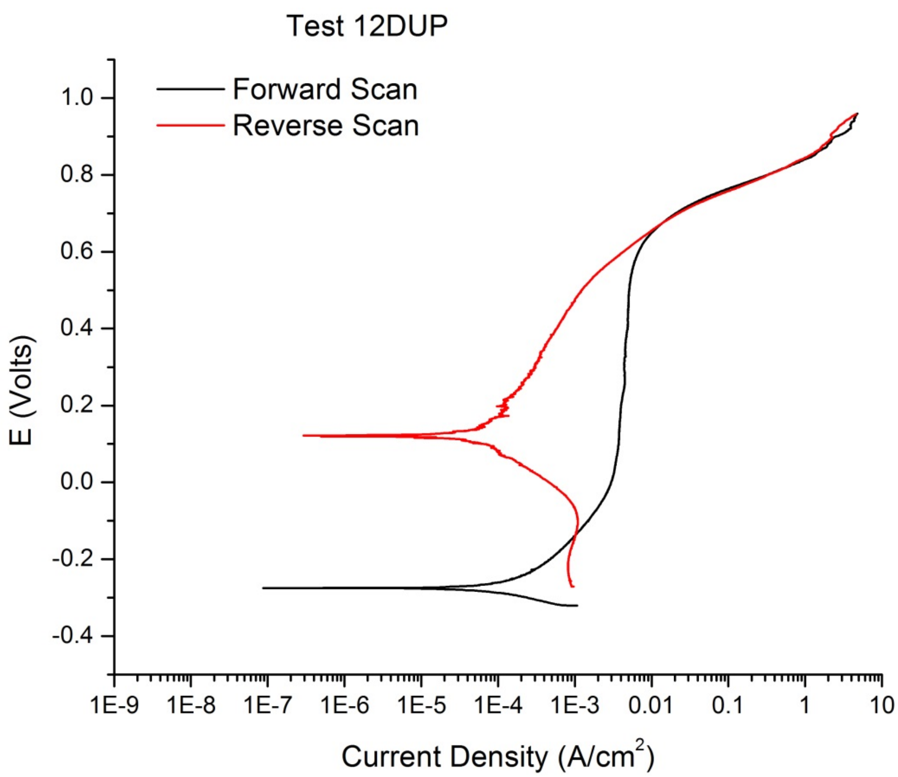


Figure B-24. Cyclic Potentiodynamic Test 12 DUP.

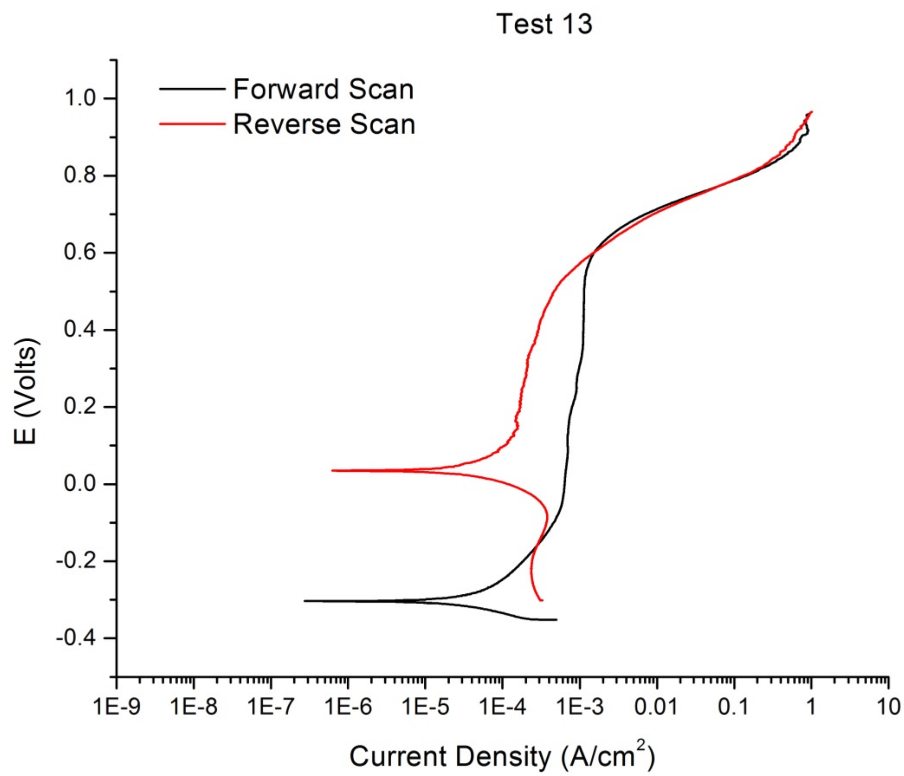


Figure B-25. Cyclic Potentiodynamic Test 13.

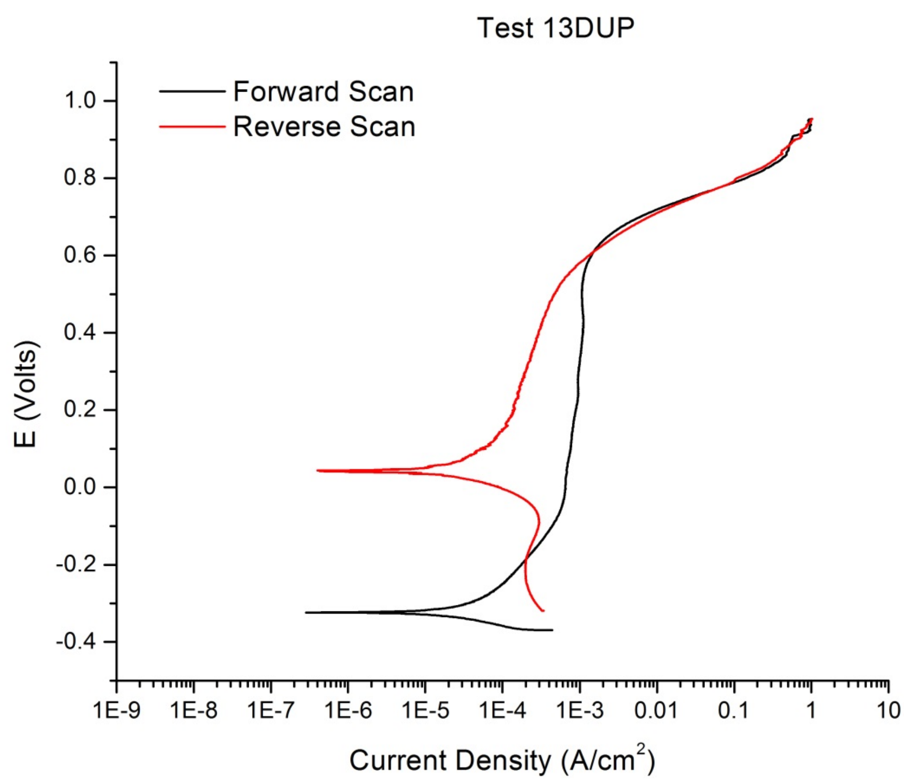


Figure B-26. Cyclic Potentiodynamic Test 13 DUP.

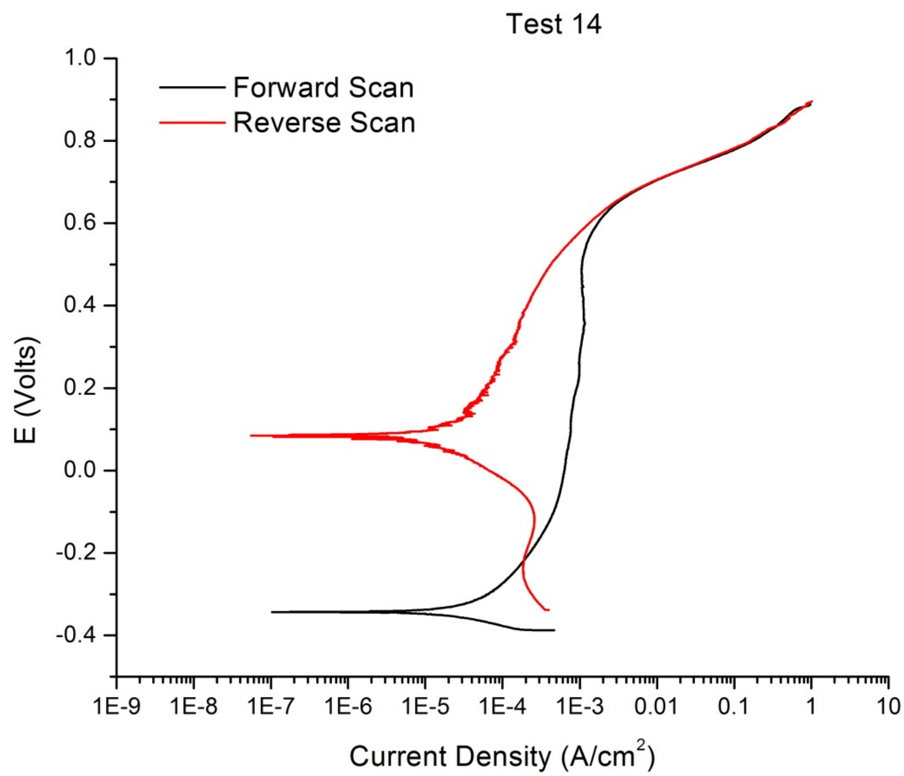


Figure B-27. Cyclic Potentiodynamic Test 14.

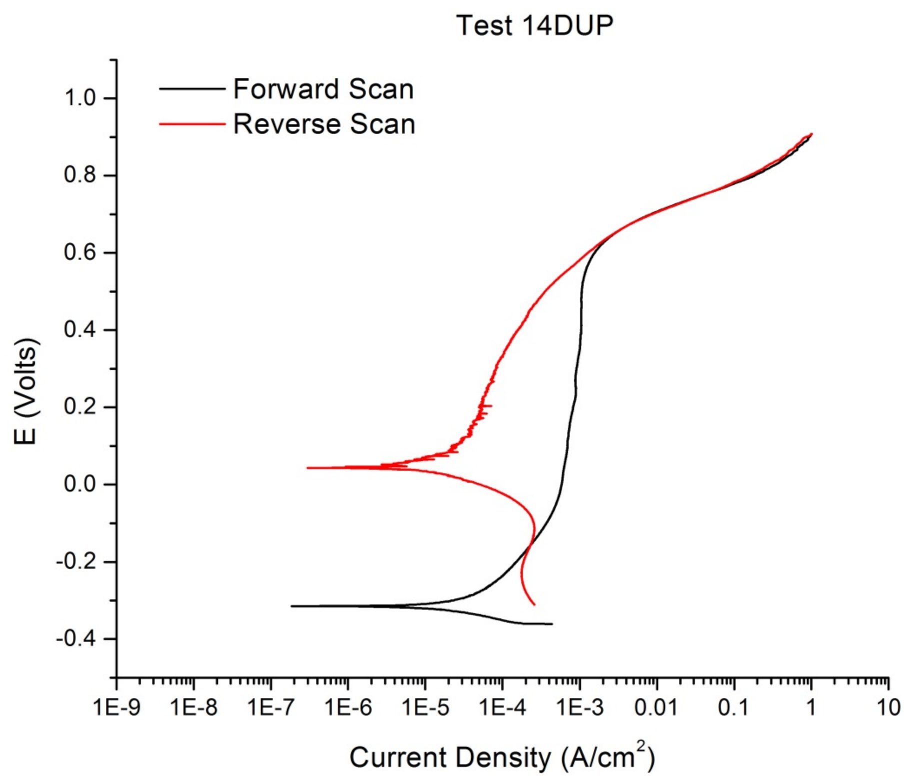


Figure B-28. Cyclic Potentiodynamic Test 14 DUP.

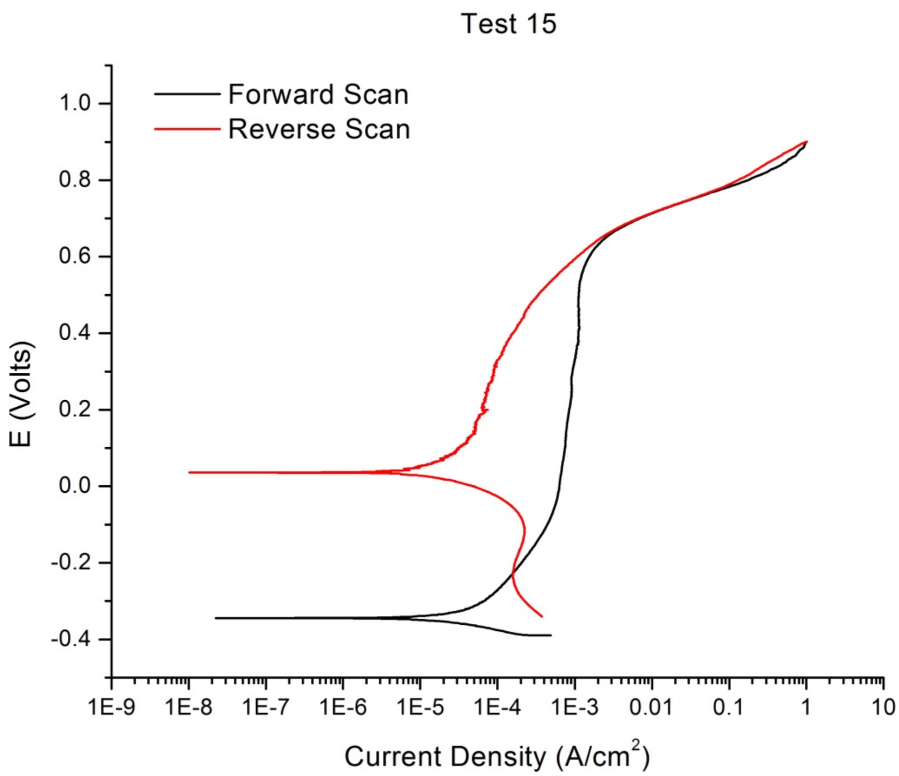


Figure B-29. Cyclic Potentiodynamic Test 15.

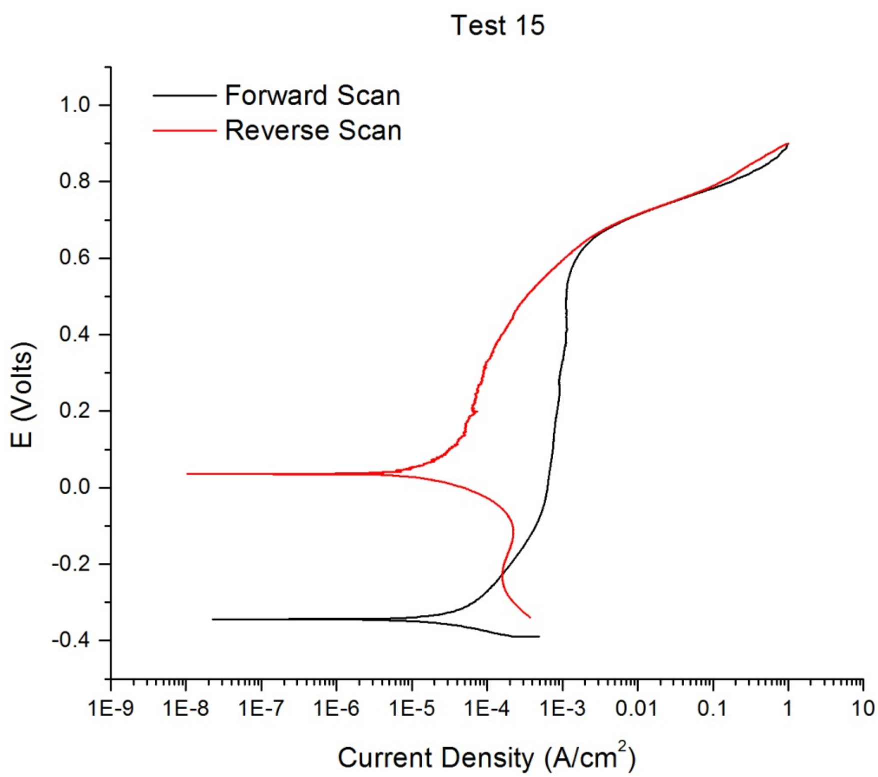


Figure B-30. Cyclic Potentiodynamic Test 15 DUP.

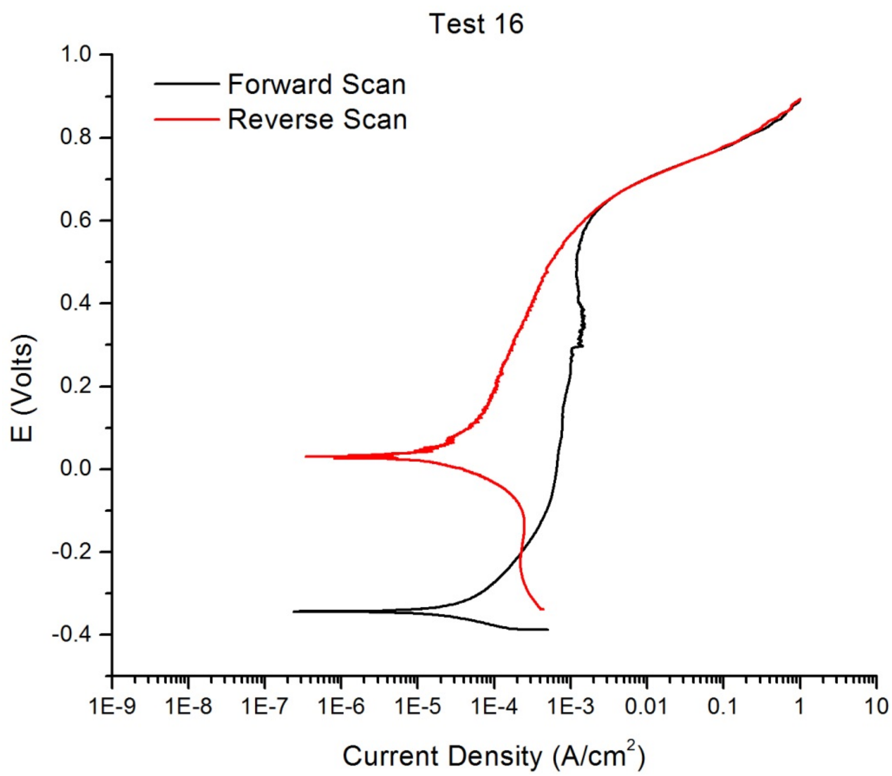


Figure B-31. Cyclic Potentiodynamic Test 16.

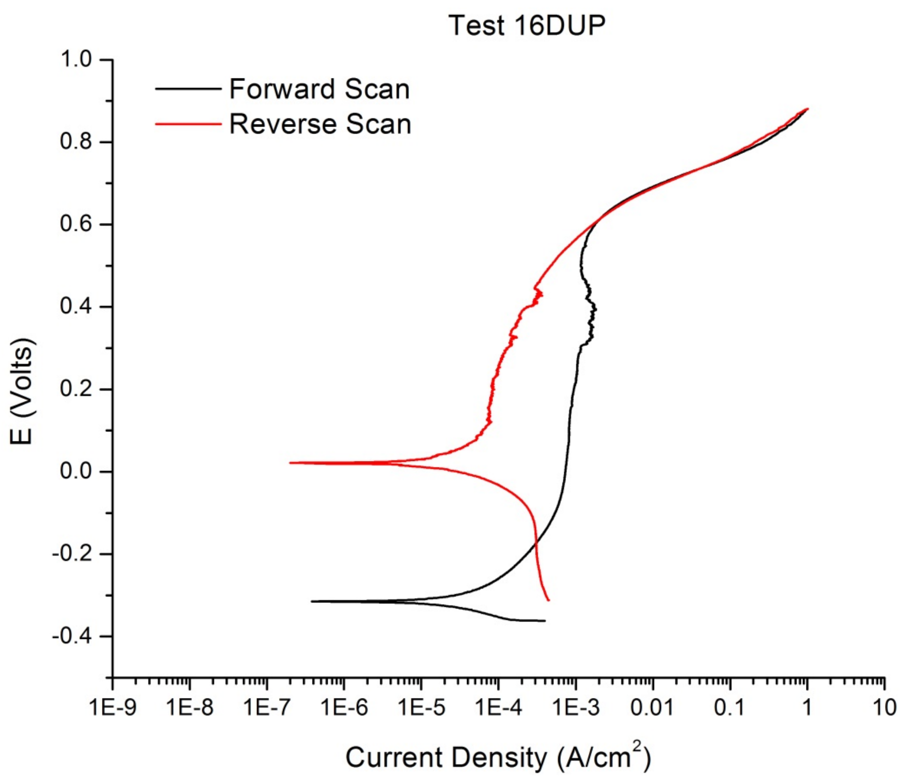


Figure B-32. Cyclic Potentiodynamic Test 16 DUP.

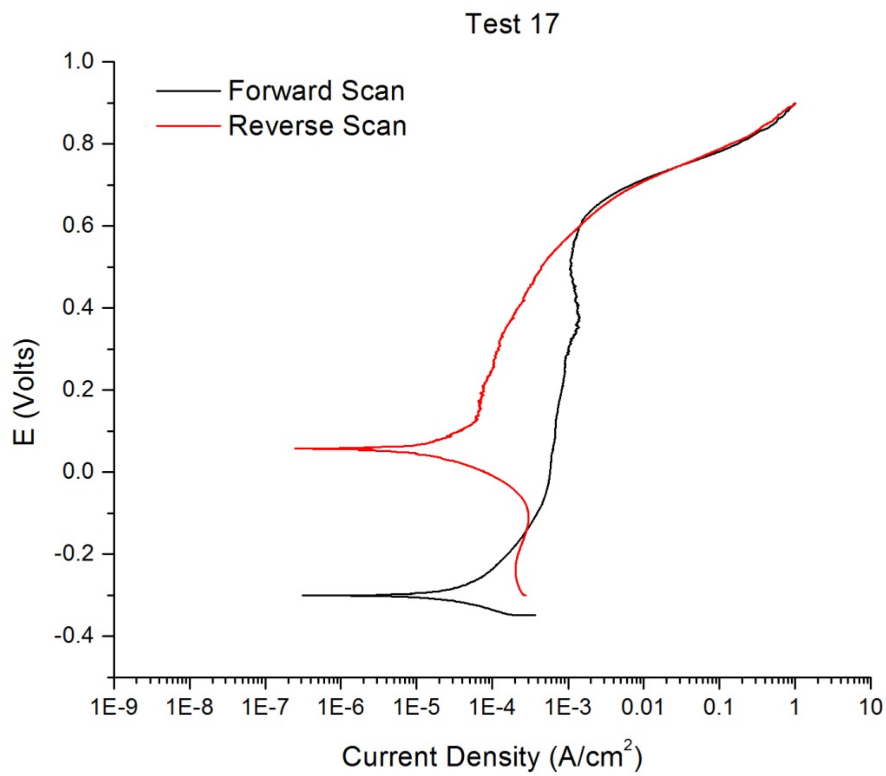


Figure B-33. Cyclic Potentiodynamic Test 17.

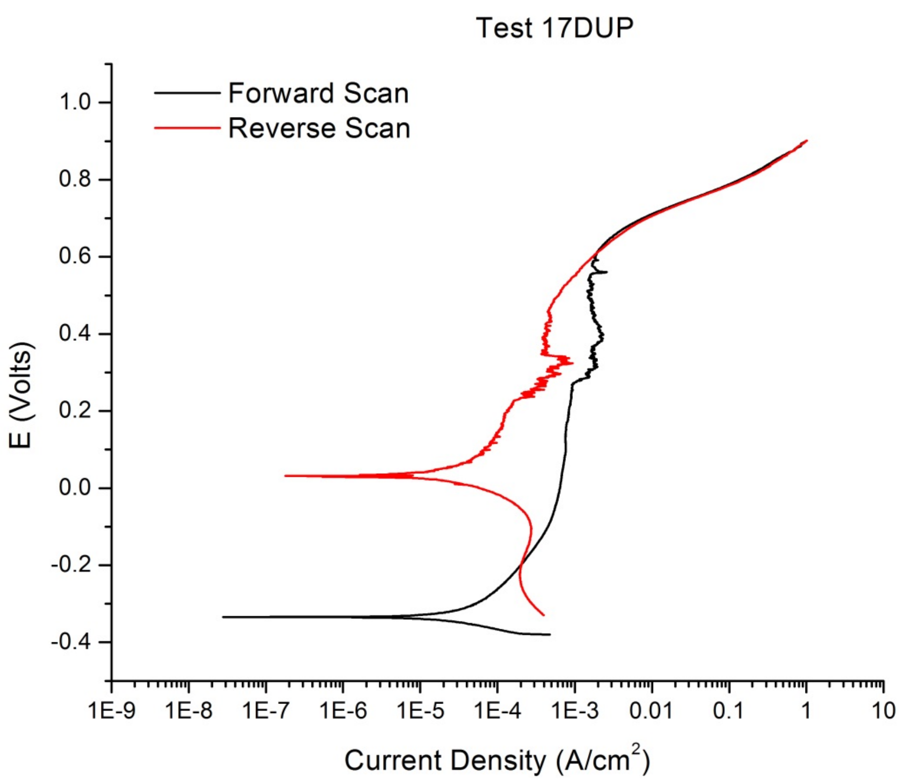


Figure B-34. Cyclic Potentiodynamic Test 17 DUP.

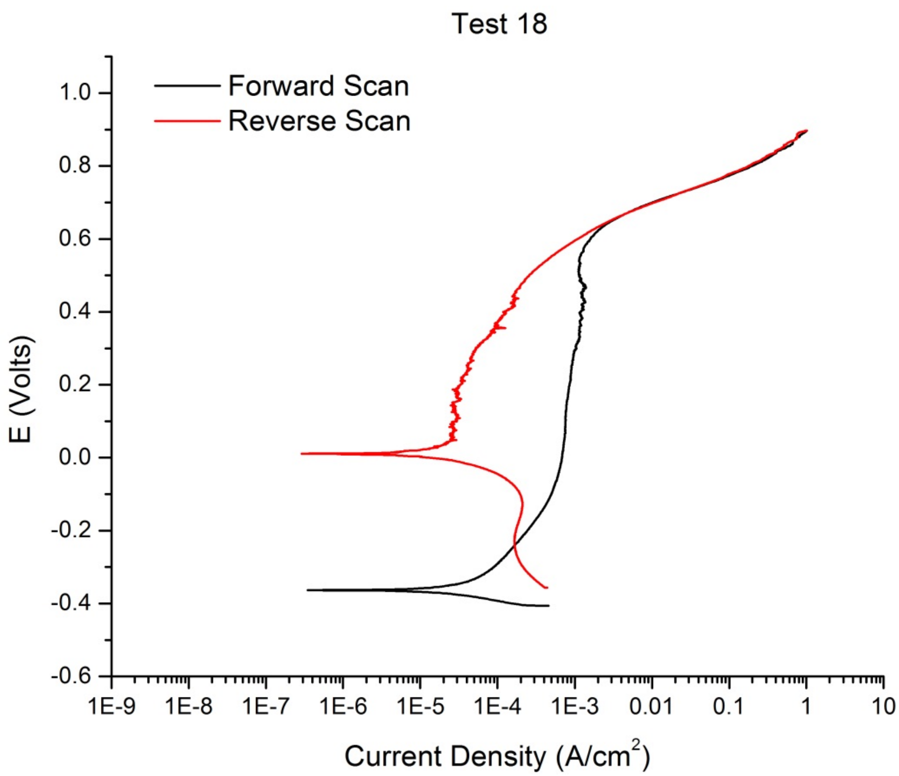


Figure B-35. Cyclic Potentiodynamic Test 18.

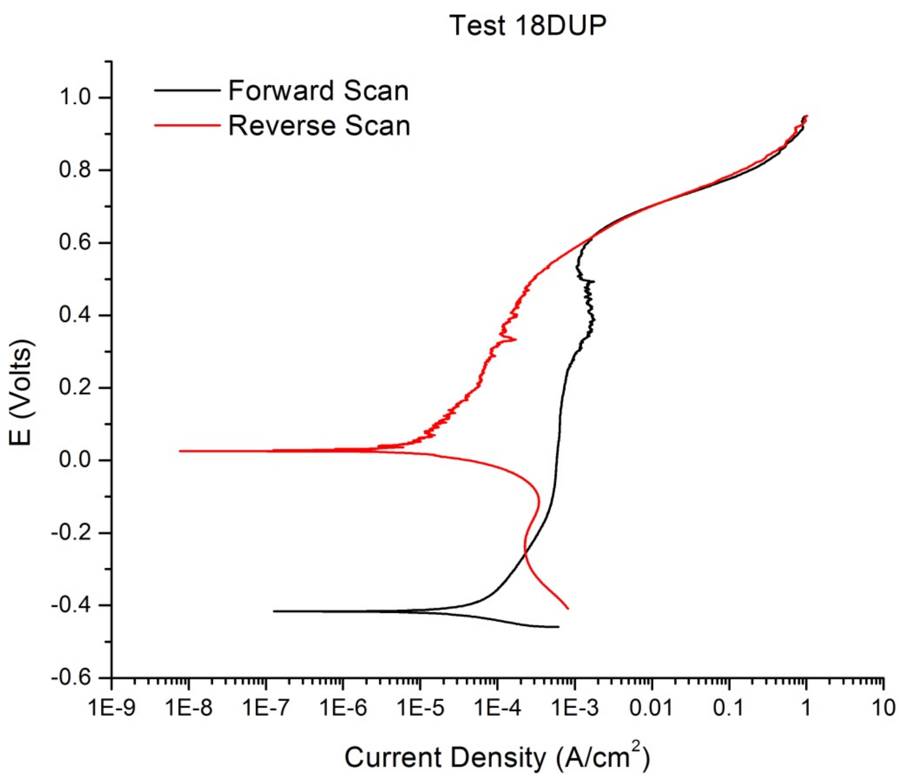


Figure B-36. Cyclic Potentiodynamic Test 18 DUP.

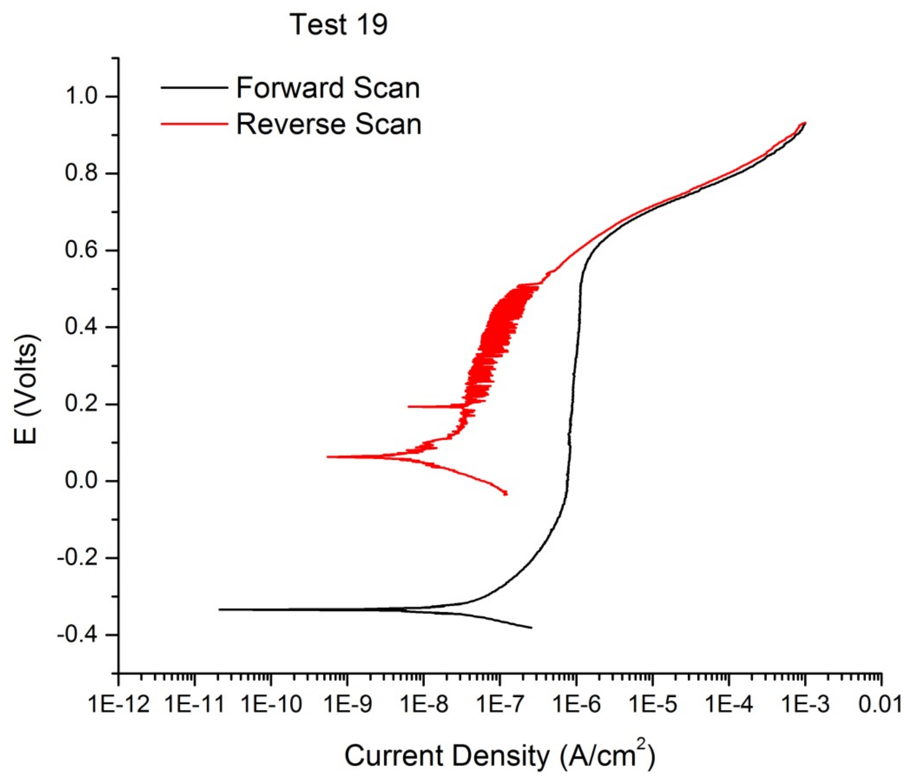


Figure B-37. Cyclic Potentiodynamic Test 19.

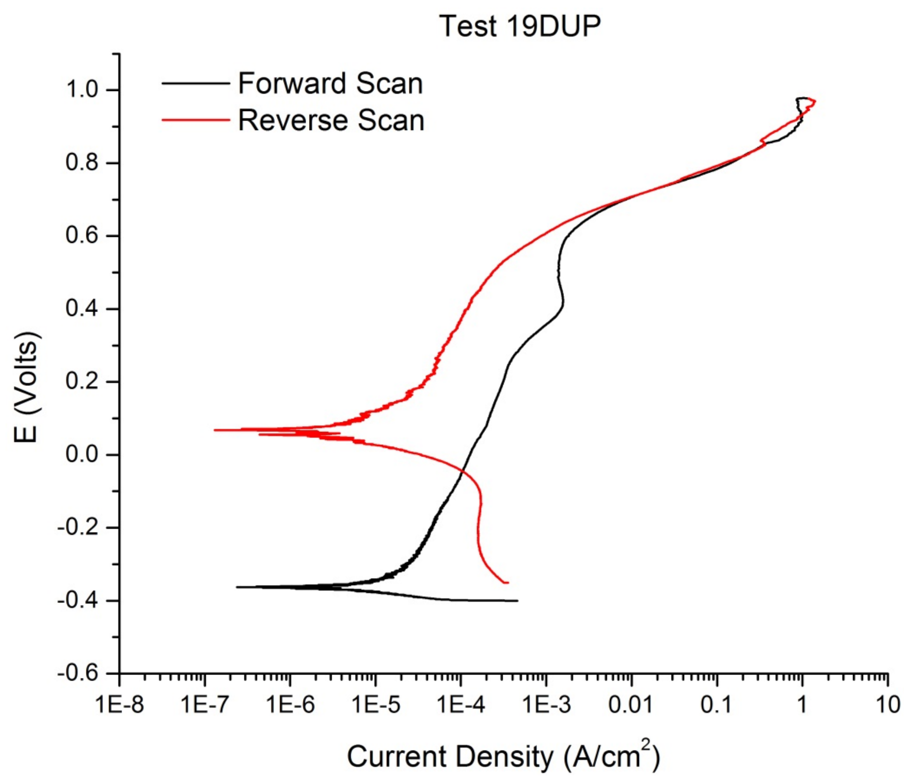


Figure B-38. Cyclic Potentiodynamic Test 19 DUP.

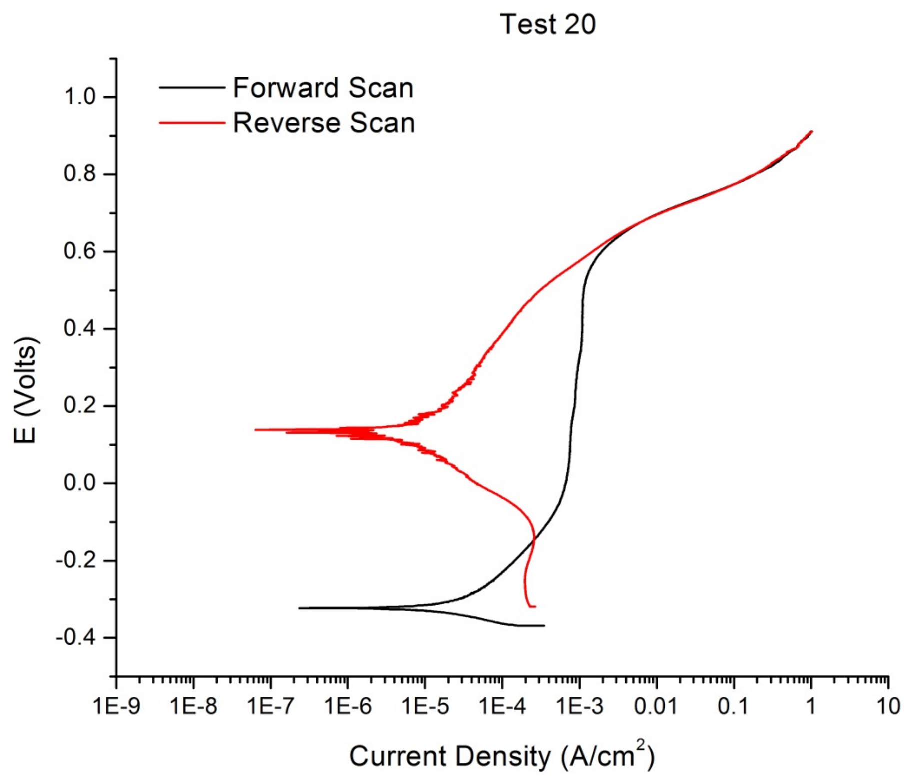


Figure B-39. Cyclic Potentiodynamic Test 20.

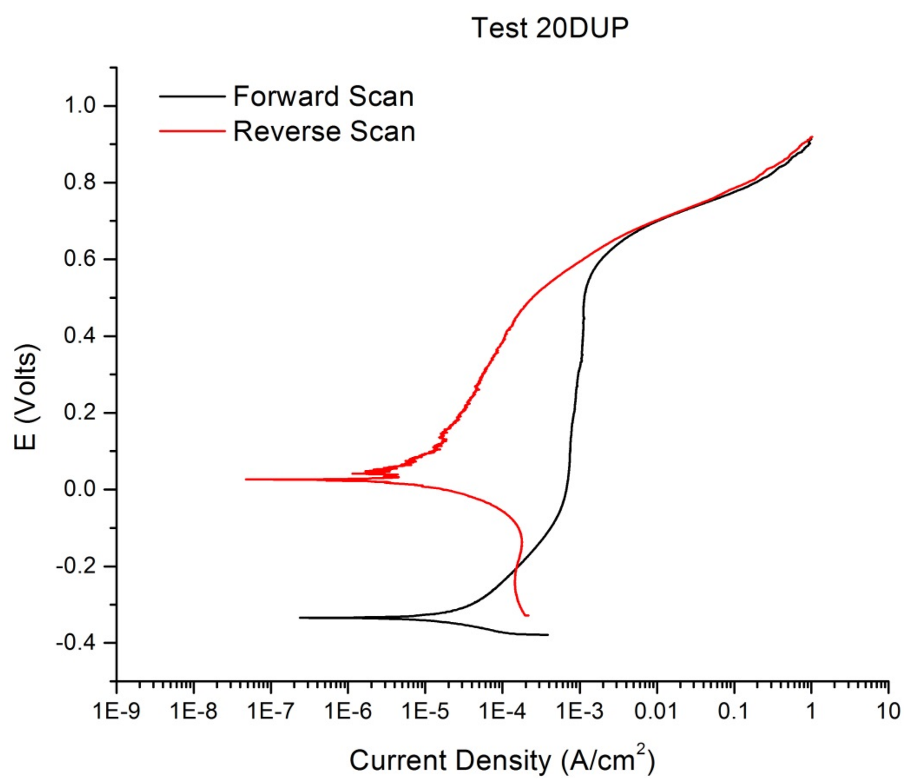


Figure B-40. Cyclic Potentiodynamic Test 20 DUP.

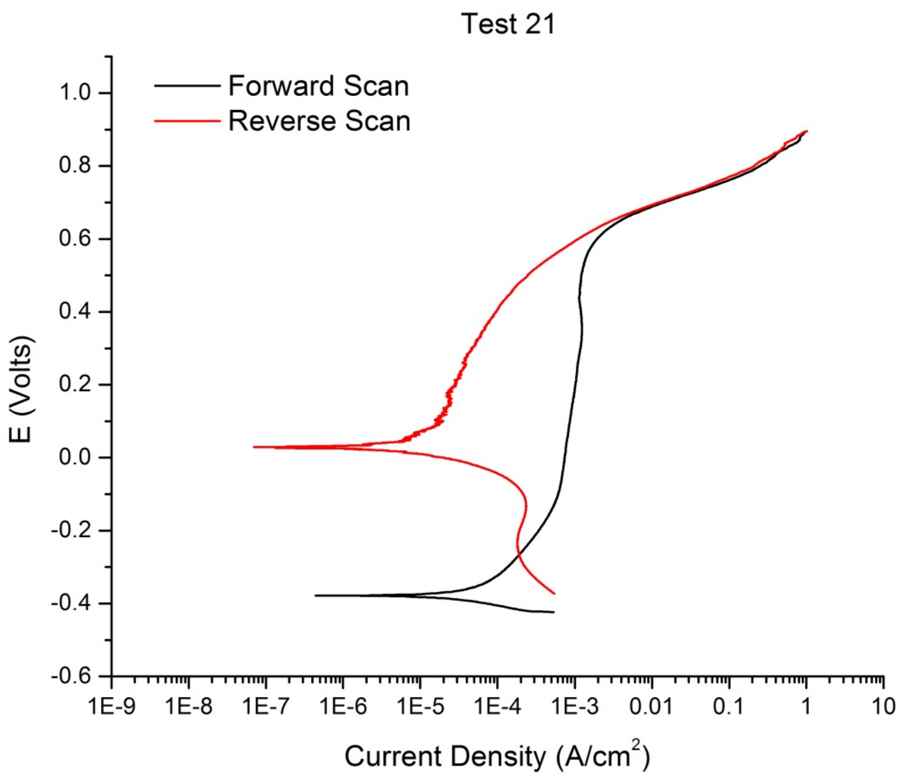


Figure B-41. Cyclic Potentiodynamic Test 21.

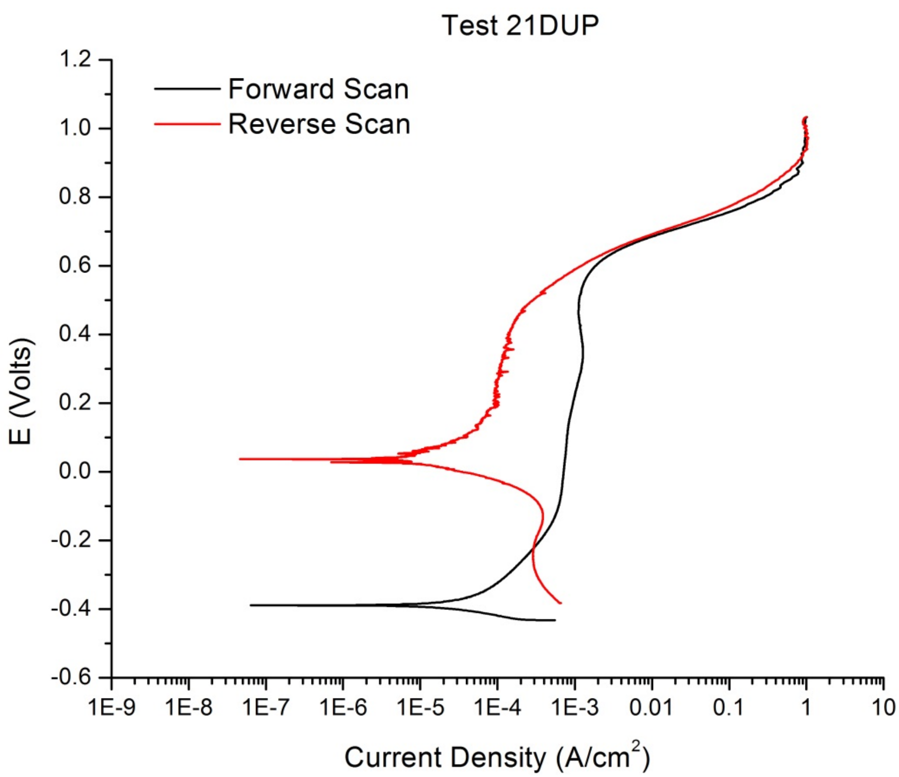


Figure B-42. Cyclic Potentiodynamic Test 21 DUP.

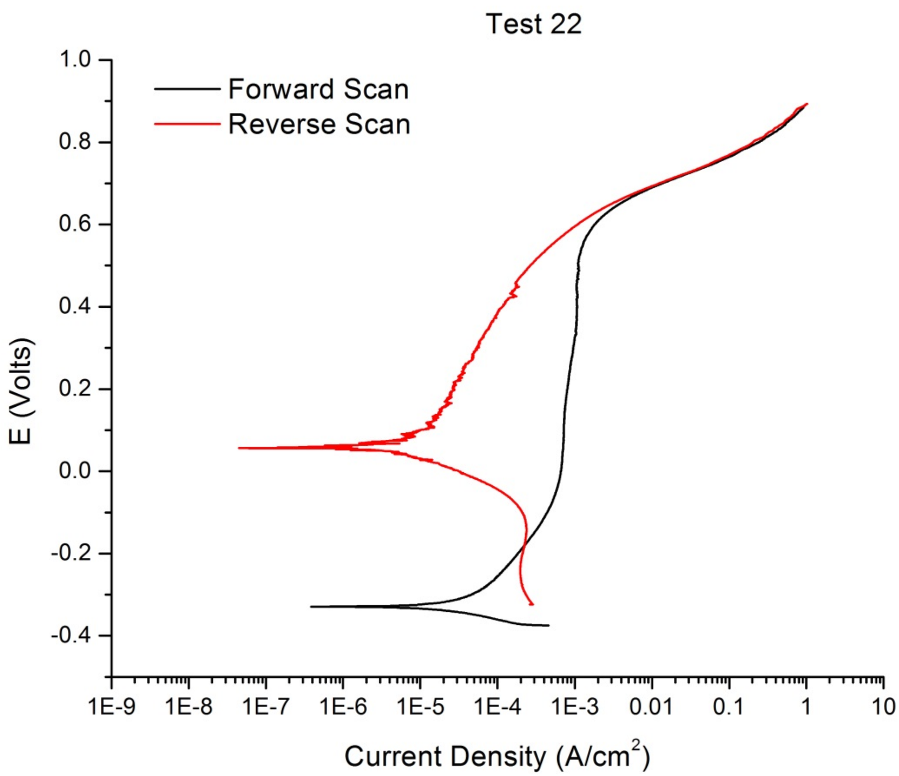


Figure B-43. Cyclic Potentiodynamic Test 22.

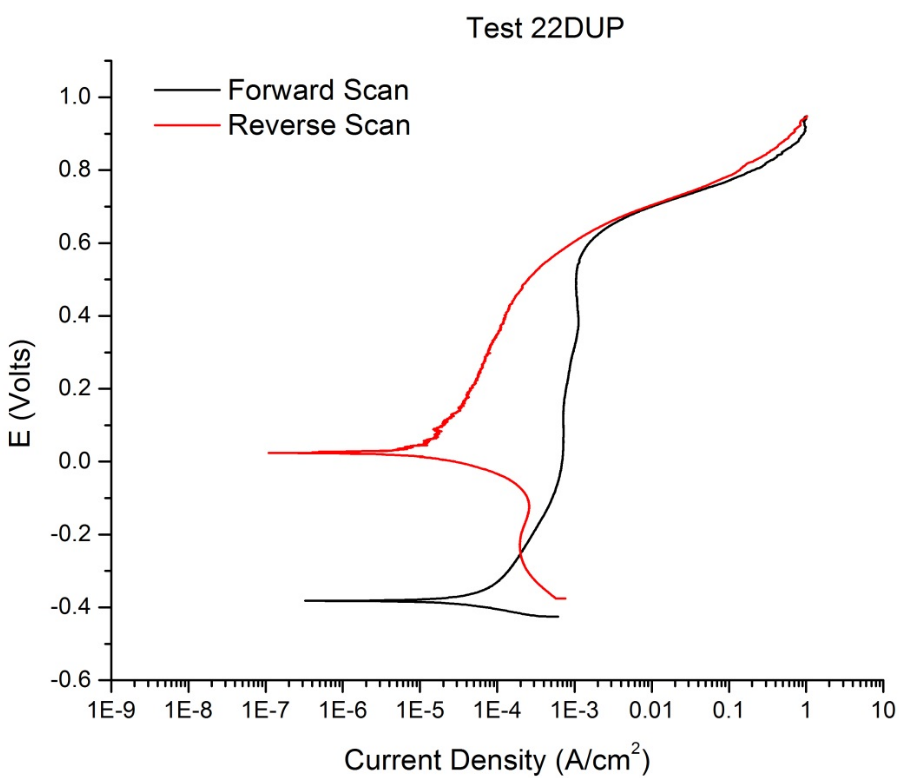


Figure B-44. Cyclic Potentiodynamic Test 22 DUP.

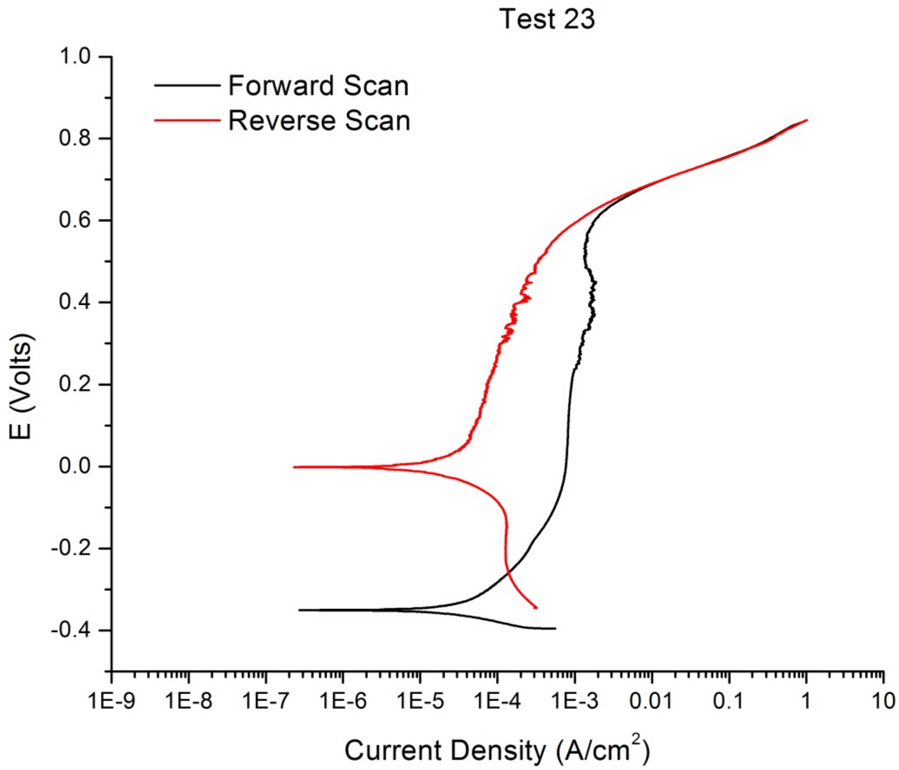


Figure B-45. Cyclic Potentiodynamic Test 23.

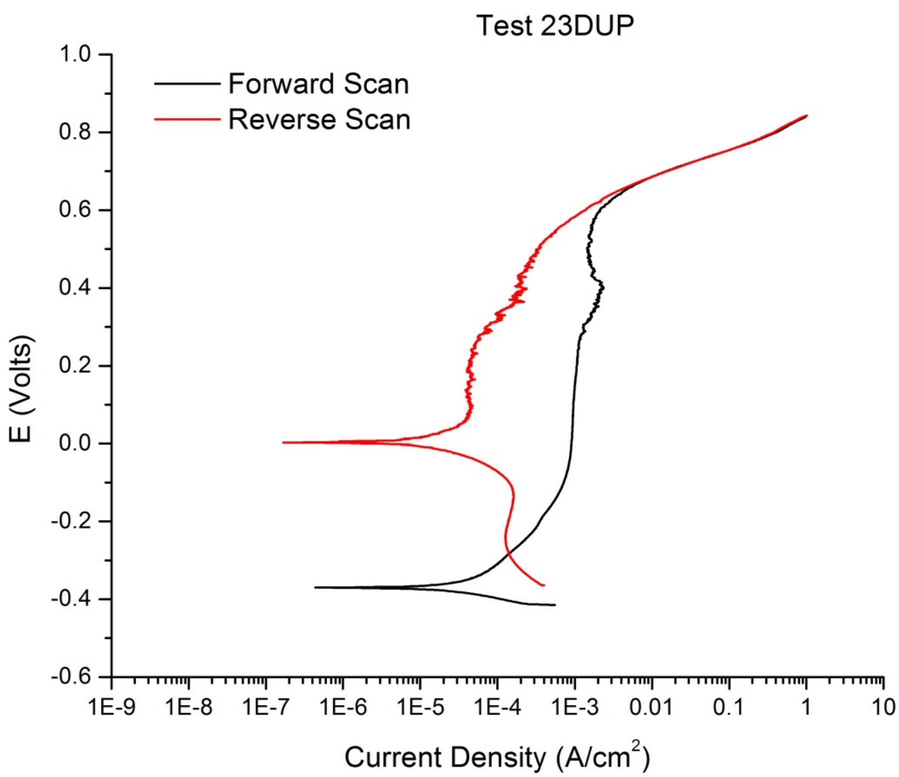


Figure B-46. Cyclic Potentiodynamic Test 23 DUP.

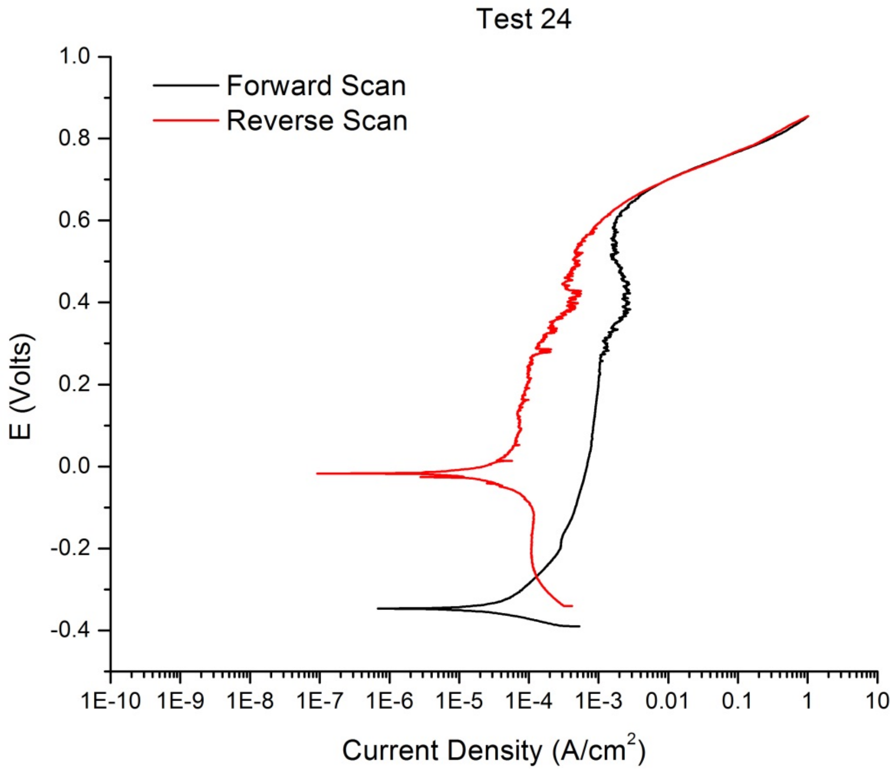


Figure B-47. Cyclic Potentiodynamic Test 24.

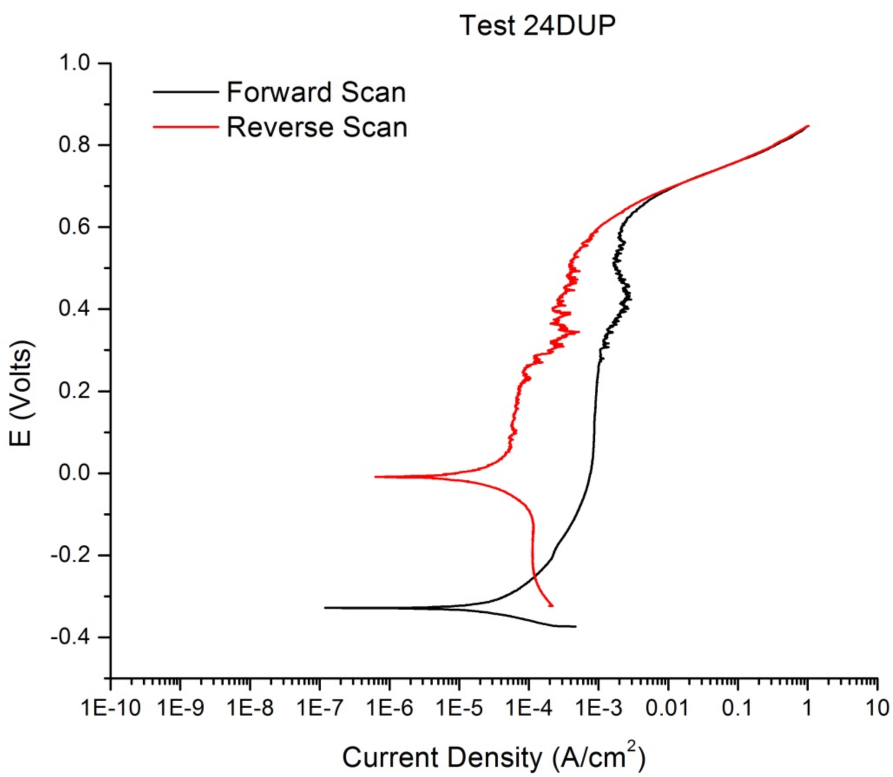


Figure B-48. Cyclic Potentiodynamic Test 24 DUP.

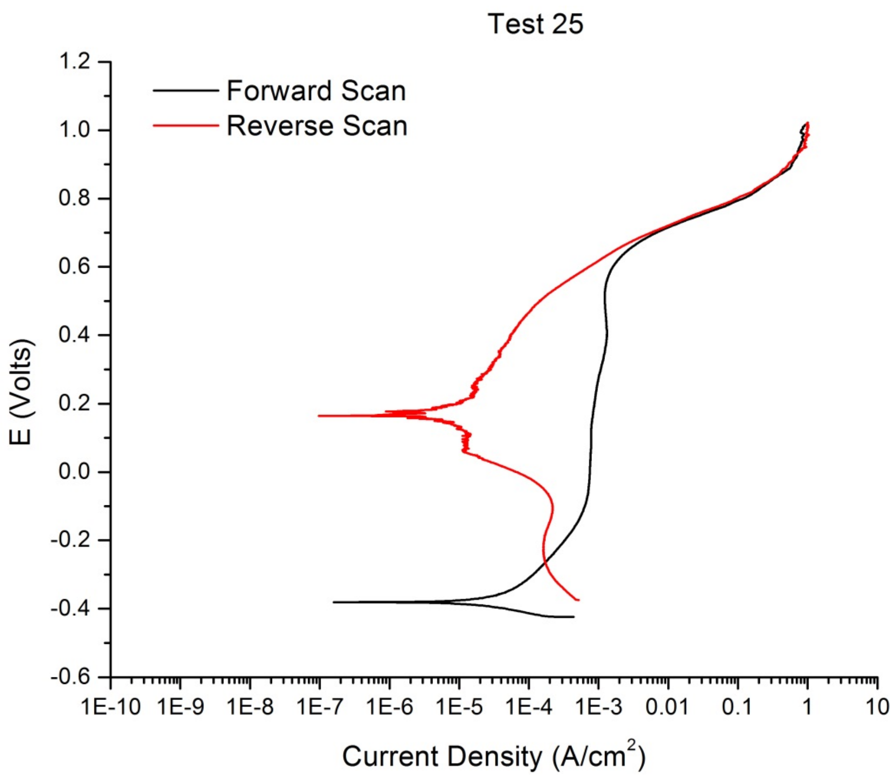


Figure B-49. Cyclic Potentiodynamic Test 25.

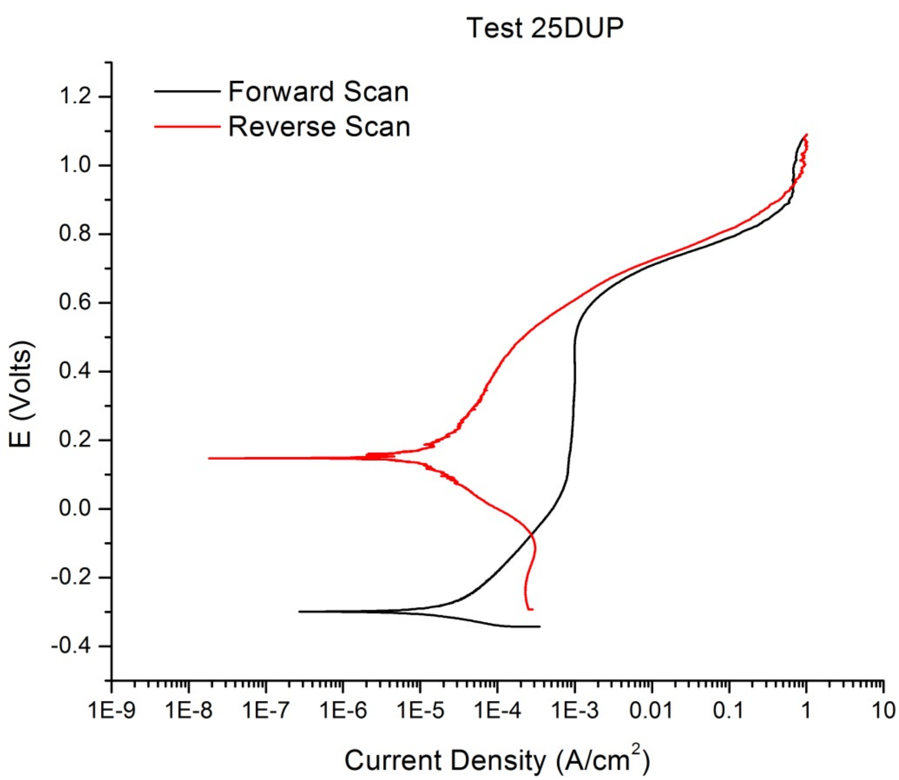


Figure B-50. Cyclic Potentiodynamic Test 25 DUP.

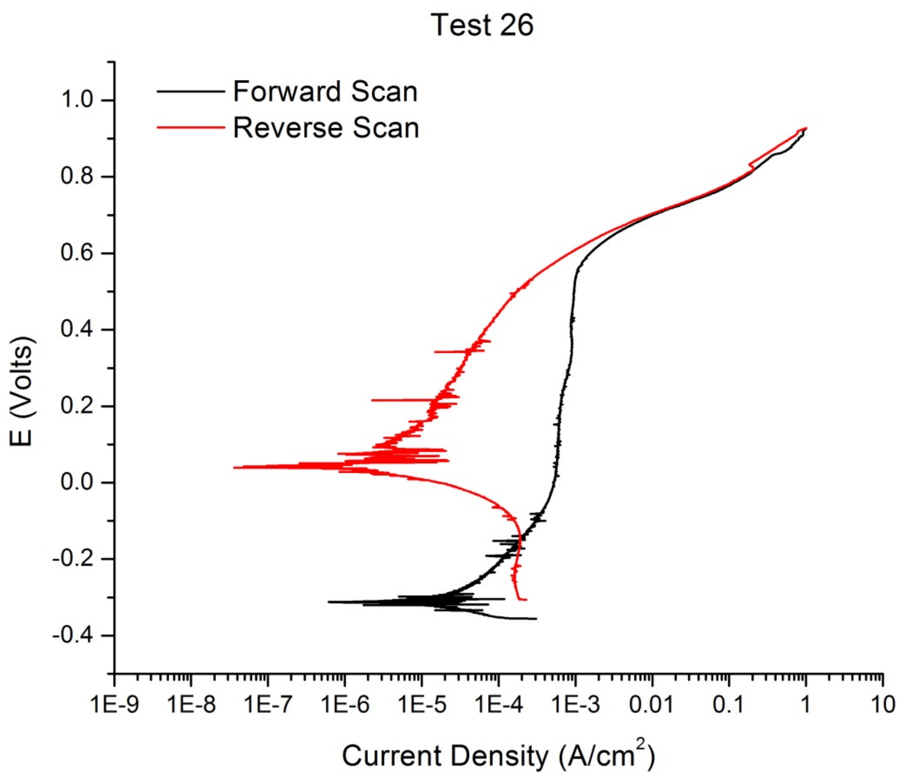


Figure B-51. Cyclic Potentiodynamic Test 26.

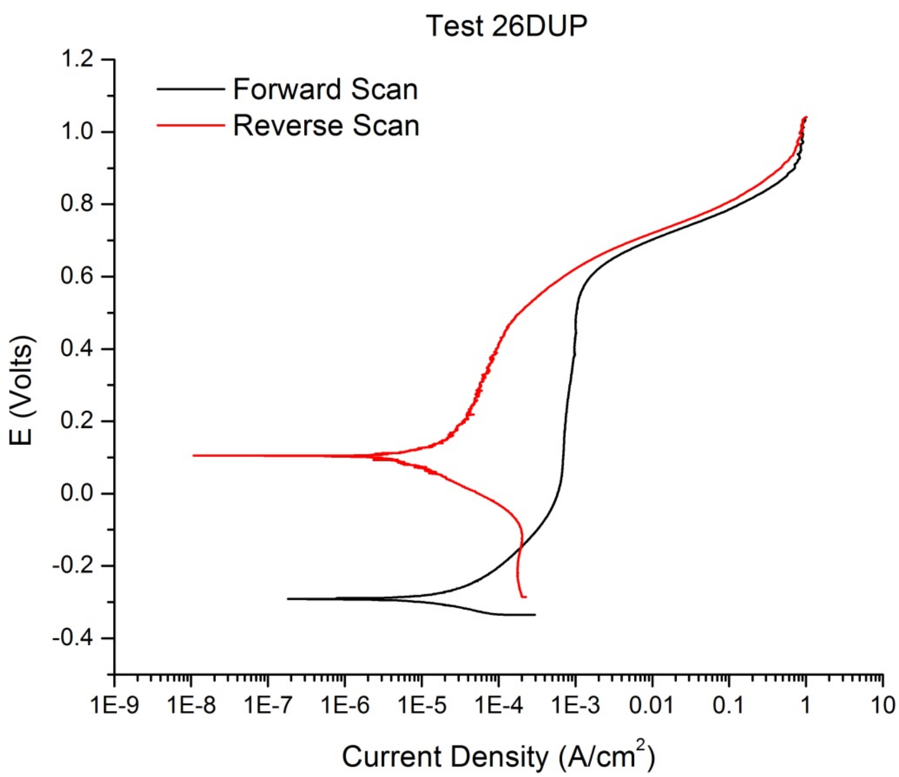


Figure B-52. Cyclic Potentiodynamic Test 26 DUP.

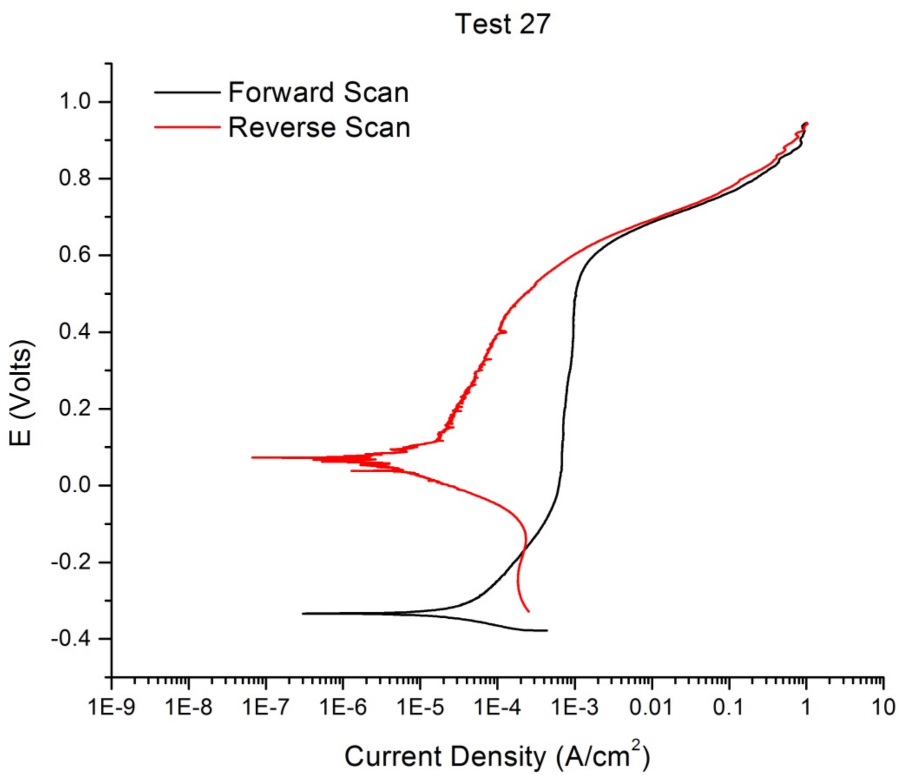


Figure B-53. Cyclic Potentiodynamic Test 27.

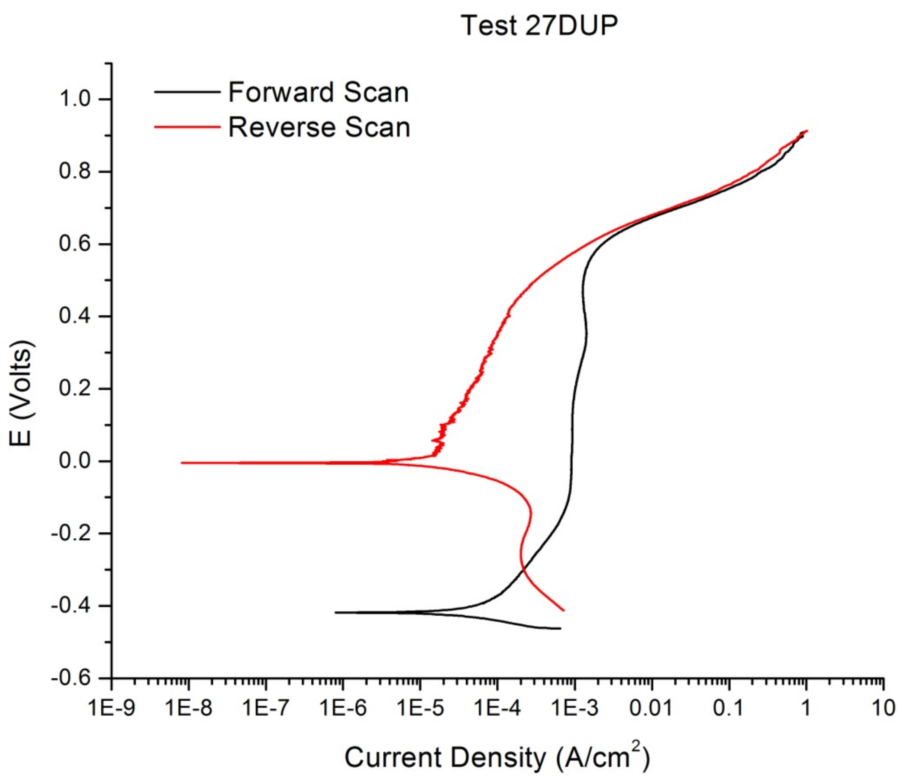


Figure B-54. Cyclic Potentiodynamic Test 27 DUP.

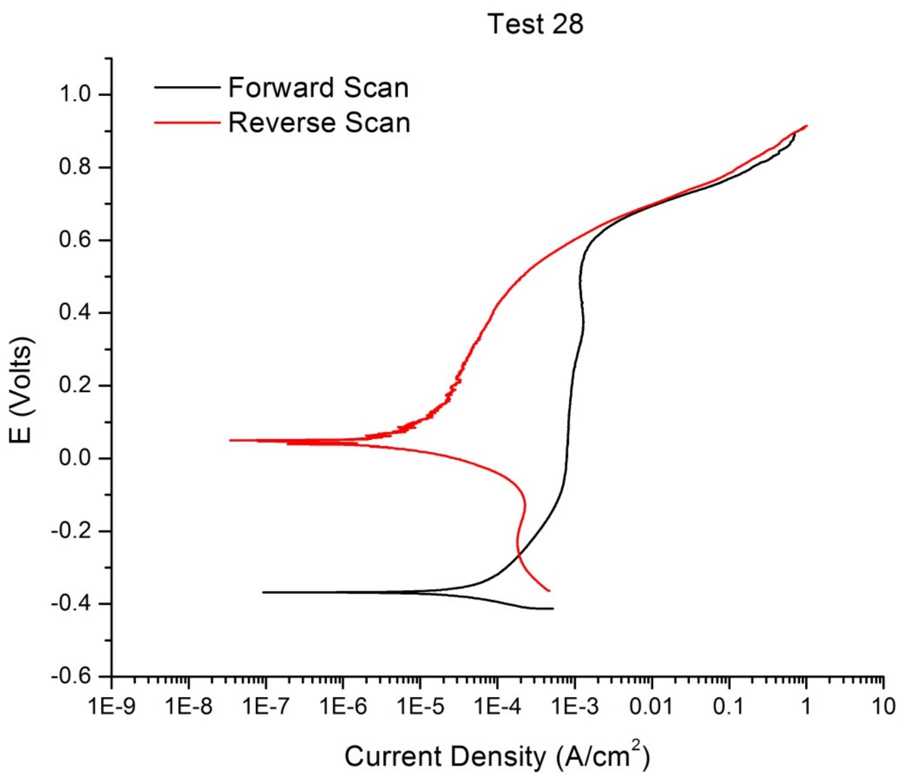


Figure B-55. Cyclic Potentiodynamic Test 28.

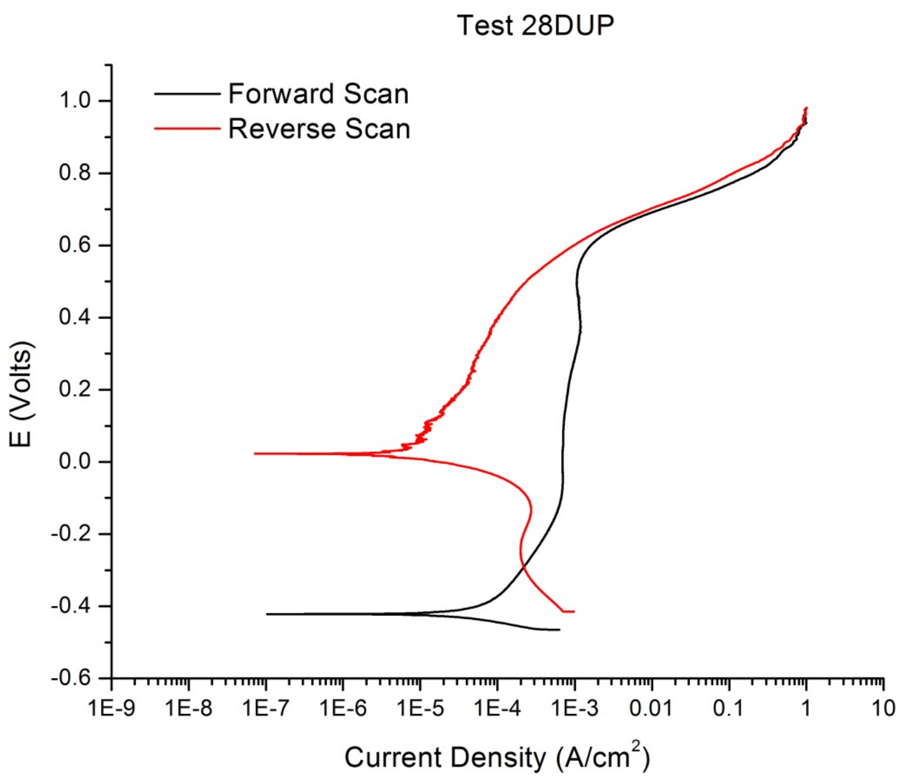


Figure B-56. Cyclic Potentiodynamic Test 28 DUP.

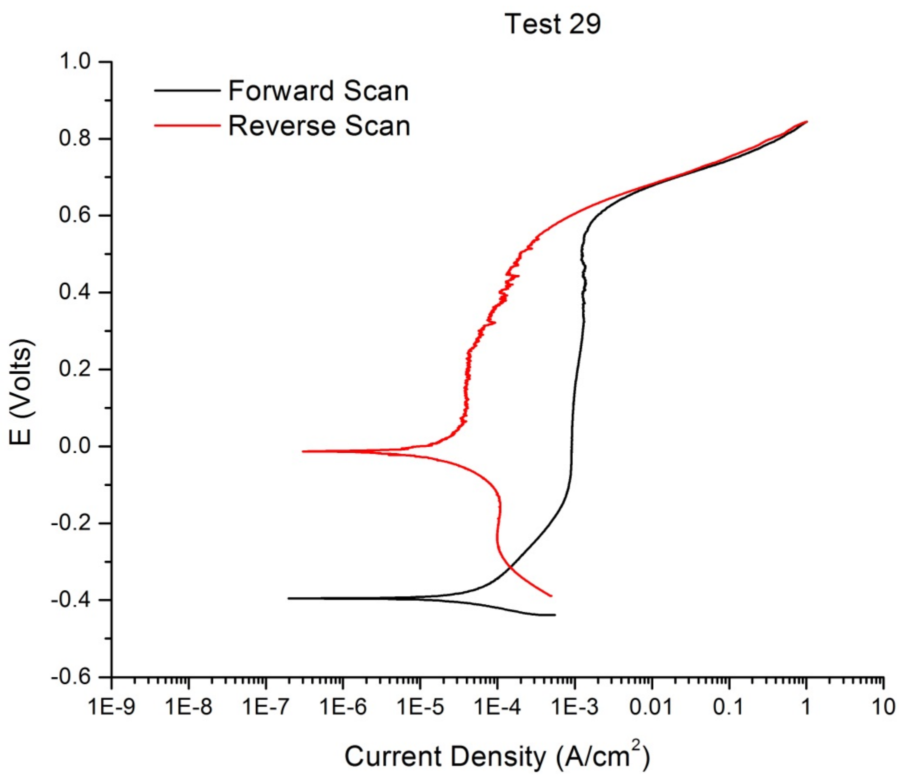


Figure B-57. Cyclic Potentiodynamic Test 29.

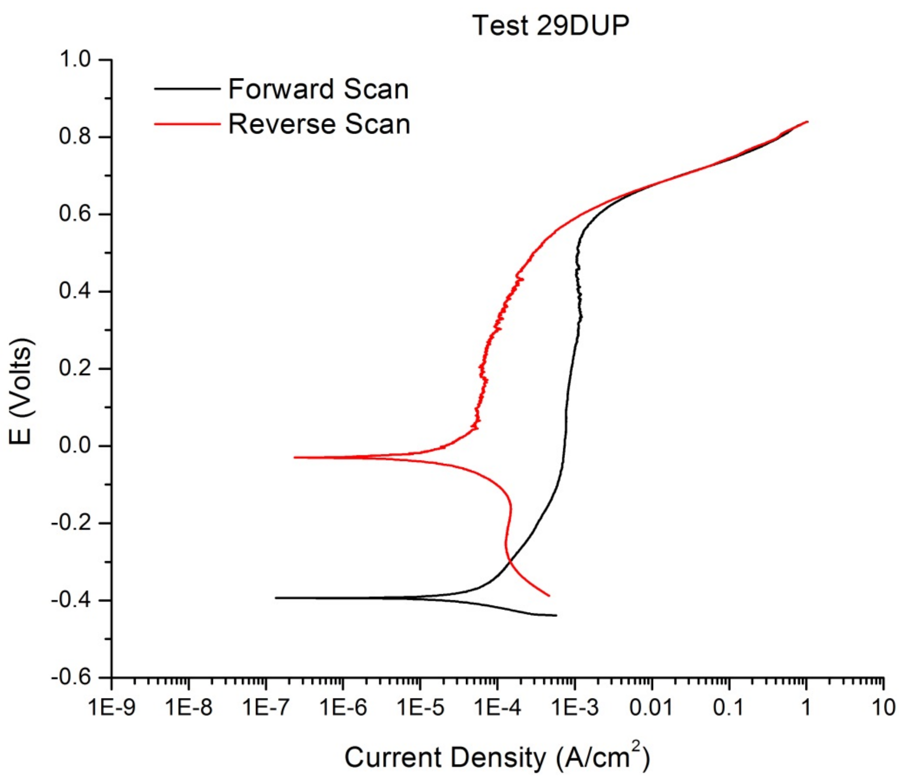


Figure B-58. Cyclic Potentiodynamic Test 29 DUP.

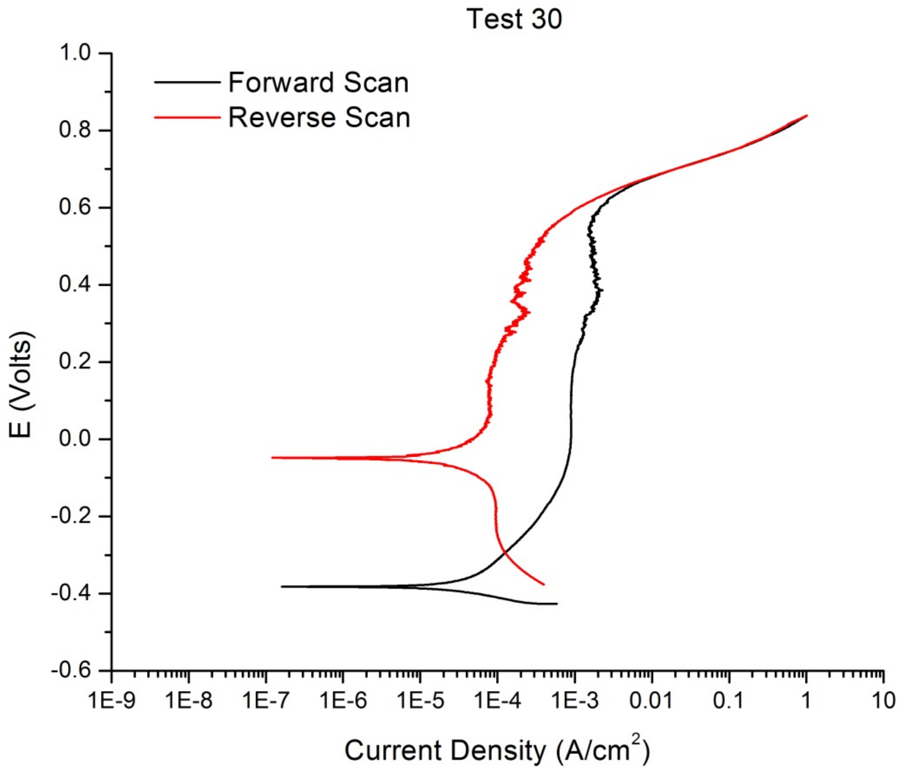


Figure B-59. Cyclic Potentiodynamic Test 30.

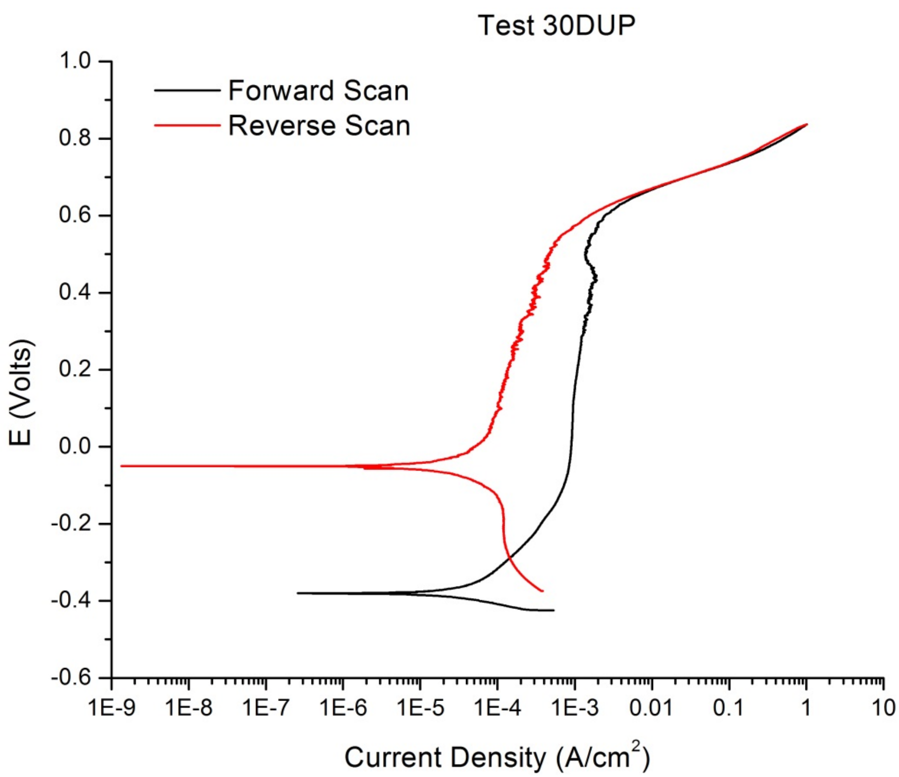


Figure B-60. Cyclic Potentiodynamic Test 30DUP.

Appendix C Solution Preparation and Testing Bench Sheets

SRNL

DFLAW Simulant **Test ID** 1

This formula will make 1 Liters

Date: 6-5-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2207
2	Sodium Fluoride	0.4199	0.4206
3	Sodium Chloride	0.0000	~~0.5853~~
4	Ammonium Chloride	0.5549	0.5553
5	Sodium Nitrite	68.9950	68.9949
6	Sodium Sulfate	14.2130	14.2130
7	Sodium Phosphate, Tribasic 12	1.1404	1.1404
8	Sodium Carbonate	4.3832	~~4.3838~~ 4.3833
9	Sodium Chromate	0.6481	0.6479
10	Potassium Nitrate	0.3033	0.3036
11	Sodium Nitrate	42.2425	42.2422

Heat to 35 ˚C

Measure pH

If pH is great than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.6988	initial	N/A
			Final	
			Total	

Notes/Comments: pH = 10.02 @ 28.5°C ; post Test1 → pH = 9.8 @ 35°C
pH = 9.99 @ 36.5°C ; " Test2 pH = 9.8 @ 35°C

CPP Testing		
Coupon ID	File name	Date
Test-1	\\Data\2015\DFLAW\RIE\Test1-ocp.*	6-8-15
	→ PARSTAT 2734, #00387663	
Test-2	\\ " " \ test2-ocp*	6-8-15

Prepared by: [redacted]

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 2

This formula will make 1.4 Liters

Date: 6-16-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3080
2	Sodium Fluoride	0.5878	0.5874
3	Sodium Chloride	0.9245	0.9240
4	Ammonium Chloride	0.7489	0.7489
5	Sodium Nitrite	96.5930	96.5932
6	Sodium Sulfate	19.8982	19.8983
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5963
8	Sodium Carbonate	6.1365	6.1368
9	Sodium Chromate	0.9074	0.9077
10	Potassium Nitrate	0.4246	0.4244
11	Sodium Nitrate	59.1395	59.1391

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.07
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.9736	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test2 => 9.91 Test2DUP => 9.93
Post-test pH: Test 2 => 10.01 Test2DUP => 10.01
Pitting on both coupons

CPP Testing		
Coupon ID 2232	File name	Date
~~DF~~ Test 2	DFLAW_Test2restart_C01.mpr	6-23-15
Test2DUP	DFLAW_Test2DUP_C03.mpr	6-23-15

Prepared by: [redacted] Date: 6-16-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 3

This formula will make **1.4** Liters

Date: 6-23-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	~~0.3088~~ 0.3086
2	Sodium Fluoride	1.1757	1.1753
3	Sodium Chloride	1.0882	1.0883
4	Ammonium Chloride	0.7489	0.7485
5	Sodium Nitrite	96.5930	96.5935
6	Sodium Sulfate	19.8982	19.8980
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5961
8	Sodium Carbonate	6.1494	6.1491
9	Sodium Chromate	0.9074	0.9079
10	Potassium Nitrate	0.4246	0.4247
11	Sodium Nitrate	59.1395	59.1396

Transfer solution to volumetric flask and fill to mark with distilled water.

Heat to 35 °C ________

Measure pH 10.03

If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test3 => 9.99 Test3DUP => 9.99
Post-test pH: Test3 => 9.98 Test3DUP -> 9.98 Pitting on both

CPP Testing		
Coupon ID 2232	File name	Date
Test3	DFLAW_Test3_C01.mpr	6-24-15
Test3DUP	DFLAW_Test3DUP_C03.mpr	6-24-15

Prepared by [redacted] Date: 6-23-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 4

This formula will make 1 Liters

Date: 6-10-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2200
2	Sodium Fluoride	0.8398	0.8397
3	Sodium Chloride	0.5844	0.5845
4	Ammonium Chloride	0.5549	0.5548
5	Sodium Nitrite	137.9900	137.9898
6	Sodium Sulfate	14.2130	14.2131
7	Sodium Phosphate, Tribasic 12	1.1404	1.1402
8	Sodium Carbonate	4.6990	4.6994
9	Sodium Chromate	0.6481	0.6482
10	Potassium Nitrate	0.3033	0.3030
11	Sodium Nitrate	42.2425	42.2426

Make total Vol. 1000 mL in Vol flask

~~*Heat to 35 ˚C*~~

Measure pH 10.04@35°C

If pH is great than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.6988	initial	
			Final	
			Total	

Notes/Comments: pH - post test4 : 10.11
pH - post test4DUP : 10.18

CPP Testing		
Coupon ID	File name	Date
Test4	:\Data\2015\DFLAW CPP\Test4_OCP.cor	6-16-15
Test4DUP	Test4DUP_OCP.cor	6-16-15

Prepared by: [redacted]

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 5

This formula will make **1.4** Liters

Date: 6-17-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3084
2	Sodium Fluoride	1.1757	1.1757
3	Sodium Chloride	1.3418	1.3421
4	Ammonium Chloride	0.7489	0.7493
5	Sodium Nitrite	193.1860	193.1860
6	Sodium Sulfate	19.8982	19.8982
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5965
8	Sodium Carbonate	6.5826	6.5824
9	Sodium Chromate	0.9074	0.9075
10	Potassium Nitrate	0.4246	0.4243
11	Sodium Nitrate	59.1395	59.1392

Transfer solution to volumetric flask and fill to mark with distilled water.

Heat to 35 ° C —

Measure pH 10.15

If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.9785	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test5 => 9.92 Test5DUP => 9.92
Post-test pH: Test 5 => 10.00 Test5DUP => 9.96
Pitting on both coupons

CPP Testing		
Coupon ID 2232	File name	Date
Test5	DFLAW_Test5_C02.mpr	6-23-15
Test5DUP	DFLAW_Test5DUP_C04.mpr	6-23-15

Prepared by: [redacted] Date: 6-17-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 6

This formula will make 1.4 Liters

Date: 6-25-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3087
2	Sodium Fluoride	1.1757	1.1753
3	Sodium Chloride	1.8000	1.7996
4	Ammonium Chloride	0.7489	0.7488
5	Sodium Nitrite	193.1860	193.1859
6	Sodium Sulfate	19.8982	19.8982
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.5883	6.5884
9	Sodium Chromate	0.9074	0.9074
10	Potassium Nitrate	0.4246	0.4244
11	Sodium Nitrate	59.1395	59.1394

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 9.96
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test6 => 9.97 Test6DUP => 9.96
Post-test pH: Test6 => 9.98 Test6DUP => 9.96 Pitting on both

CPP Testing		
Coupon ID 2232	File name	Date
Test6	DFLAW_Test6_C02.mpr	6-30-15
Test6DUP	DFLAW_Test6DUP_C03.mpr	6-30-15

Prepared by: [redacted] Date: 6-25-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 7

This formula will make **1.4** Liters

Date: 6-26-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	~~0.3081~~ 0.3086
2	Sodium Fluoride	1.7635	1.7635
3	Sodium Chloride	1.4727	1.4725
4	Ammonium Chloride	0.7489	0.7486
5	Sodium Nitrite	193.1860	193.1864
6	Sodium Sulfate	19.8982	19.8984
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.5735	6.5734
9	Sodium Chromate	0.9074	0.9076
10	Potassium Nitrate	0.4246	0.4249
11	Sodium Nitrate	59.1395	59.1399

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C ______
Measure pH 9.97
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test7 => 9.92 Test7DUP => 9.93 Both are pitting
Post-test pH: Test7 => 9.93 Test7DUP => 9.92 Test7 is ~~a~~ scuffed/discolored for about .5 inches at the base. Test7DUP has same discoloration on ≥ ½ the surface.

CPP Testing		
Coupon ID 2232	File name	Date
Test7	DFLAW-Test7_C02.mpr	6-30-15
Test7DUP	DFLAW-Test7DUP_C04.mpr	6-30-15

Prepared by [redacted] Date: 6-26-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 8

This formula will make 1.4 Liters

Date: 6-29-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3080
2	Sodium Fluoride	1.7635	1.7639
3	Sodium Chloride	1.9963	1.9965
4	Ammonium Chloride	0.7489	0.7488
5	Sodium Nitrite	193.1860	193.1856
6	Sodium Sulfate	19.8982	19.8986
7	Sodium Phosphate, Tribasic 12 H_2O	1.5965	1.5960
8	Sodium Carbonate	6.5883	6.5885
9	Sodium Chromate	0.9074	0.9077
10	Potassium Nitrate	0.4246	~~0.4246~~ 0.4245
11	Sodium Nitrate	59.1395	59.1395

Transfer solution to volumetric flask and fill to mark with distilled water.

Heat to 35° C

Measure pH 10.12

If pH is greater than 10, add bicarbonate up to the amount in line 12.

		Target weight, g		Actual Weight, g
12	Sodium Bicarbonate	1.2231	initial	1.2228
			Final	1.0624
			Total	0.1604

pH=>10.04

Notes/Comments: Pre-test pH: Test8=> 10.04 Test8DUP => 10.04
Post-test pH: Test8 => 10.05 Test8DUP=> 10.06 Both have pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test8	DFLAW-Test8_C01.mpr	6-30-15
Test8 DUP	DFLAW-Test8DUP_C03.mpr	6-30-15

Prepared by: [redacted] Date: 6-29-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **9**

This formula will make **1.4** Liters

Date: 6-24-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3085
2	Sodium Fluoride	1.7635	1.7631
3	Sodium Chloride	3.4526	3.4526
4	Ammonium Chloride	0.7489	0.7487
5	Sodium Nitrite	193.1860	193.1865
6	Sodium Sulfate	19.8982	19.8986
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	~~1.5963~~ 1.5964
8	Sodium Carbonate	6.5883	6.5879
9	Sodium Chromate	0.9074	0.9078
10	Potassium Nitrate	0.4246	0.4244
11	Sodium Nitrate	59.1395	59.1391

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.09
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2230
			Final	1.0816
			Total	0.1414 pH => 10.02

Notes/Comments: Pre-test pH: Test 9 => 10.03 Test9DUP => 10.04
Post-test pH: Test9 => 10.06 Test9DUP => 10.07 Pitting on both

CPP Testing		
Coupon ID 2232	File name	Date
Test9	DFLAW_Test9_C01.mpr	6-25-15
Test9DUP	DFLAW_Test9DUP_C03.mpr	6-25-15

Prepared by: [redacted] Date: 6-24-15

We put science to work.™

SRNL

DFLAW Simulant **Test ID** 10

This formula will make **1.4** Liters

Date: 6-24-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3085
2	Sodium Fluoride	1.7635	1.7639
3	Sodium Chloride	4.9090	4.9091
4	Ammonium Chloride	0.7489	0.7491
5	Sodium Nitrite	193.1860	193.1859
6	Sodium Sulfate	19.8982	19.8979
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5963
8	Sodium Carbonate	6.6032	6.6035
9	Sodium Chromate	0.9074	0.9074
10	Potassium Nitrate	0.4246	0.4243
11	Sodium Nitrate	59.1395	59.1393

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.06
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test10 ⇒ 10.06 Test10DUP ⇒ 10.05
Test 10 has a small scratch ¼ in. from base. Post-test pH: Test10 ⇒ 10.11 Test10DUP ⇒ 10.08
Test10DUP has small scratch in middle. Test10 has minute pitting. Test10DUP has slightly larger pits

CPP Testing		
Coupon ID 2232	File name	Date
Test10	DFLAW_Test10_C02.mpr	6-25-15
Test10DUP	DFLAW_Test10DUP_C04.mpr	6-25-15

Prepared by [redacted] Date: 6-24-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 11

This formula will make 1 Liters

Date: 6-9-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2200
2	Sodium Fluoride	1.2597	1.2596
3	Sodium Chloride	1.1045	1.1043
4	Ammonium Chloride	0.5549	0.5546
5	Sodium Nitrite	206.9850	206.9854
6	Sodium Sulfate	14.2130	14.2133
7	Sodium Phosphate, Tribasic 12	1.1404	1.1406
8	Sodium Carbonate	4.6441	4.6442
9	Sodium Chromate	0.6481	0.6481
10	Potassium Nitrate	0.3033	0.3037
11	Sodium Nitrate	42.2425	42.2424
	Heat to 35˚C		
	Measure pH		10.08 @ 35°C

If pH is great than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.6988	initial	N/A
			Final	
			Total	

Notes/Comments:

CPP Testing		
Coupon ID	File name	Date
Test 11B		
Test 11A		

Prepared b[redacted]/15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **11**

This formula will make 1 Liters

Date: 6-12-15 Batch no.: +2

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2205
2	Sodium Fluoride	1.2597	1.2603
3	Sodium Chloride	1.1045	1.1047
4	Ammonium Chloride	0.5349	0.5351
5	Sodium Nitrite	206.9850	206.9845
6	Sodium Sulfate	14.2130	14.2132
7	Sodium dihydrogen Phospate 12 H2O	0.8042	0.80432
8	Sodium Carbonate	4.6441	4.6441
9	Sodium Chromate	0.6481	0.6484
10	Potassium Nitrate	0.3033	0.3034
11	Sodium Nitrate	42.2425	42.2429

Heat to 35° C 9.84@35°C

Measure pH

If pH is great than 10, add bicarbonate up to the amount in line 12.

12

	Target weight, g		Actual Weight, g
Sodium Bicarbonate	0.6988	initial	
		Final	
		Total	

Notes/Comments: pH starts @ 9.93 then drops to 9.84 after ~15 mins ~~after~~

CPP Testing		
Coupon ID 2232	File name	Date
Test 11	Test11_retest_*	6-17-15
Test11 DUP	Test11DUP_retest_*	6-17-15

Prepared b[redacted] Date: 6-12-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** ~~13~~ 12

This formula will make **1.4** Liters

Date: 6-22-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	1.7635	1.7639
3	Sodium Chloride	2.0372	2.0374
4	Ammonium Chloride	0.7489	0.7488
5	Sodium Nitrite	289.7790	289.7792
6	Sodium Sulfate	19.8982	19.8981
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.5035	6.5032
9	Sodium Chromate	0.9074	0.9076
10	Potassium Nitrate	0.4246	0.4247
11	Sodium Nitrate	59.1395	~~59.1390~~ 59.1393

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.13 @ 4:30, 10.20 @ 8:00 a.m.
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2197	initial	1.2192
			Final	1.1325
			Total	0.0867

Notes/Comments: pH after bicarb. => 10.02 Minute pits in test12
pH pre-test: Test12 => 10.03 Test12DUP => 10.03 Slightly larger pitting on Test12DUP
post-test pH: Test12 => 10.12 Test12DUP => 10.12 Test12 Gasket leaked

CPP Testing		
Coupon ID 2232	File name	Date
Test12	DFLAW_Test12_C01.mpr	6-24-15
Test12DUP	DFLAW_Test12DUP_C03.mpr	6-24-15

Prepared by [redacted] Date: 6-22-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 13

This formula will make **1.4** Liters

Date: 6-29-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3082
2	Sodium Fluoride	1.7635	1.7633
3	Sodium Chloride	2.5363	2.5361
4	Ammonium Chloride	0.7489	0.7490
5	Sodium Nitrite	289.7790	289.7786
6	Sodium Sulfate	19.8982	19.8978
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5960
8	Sodium Carbonate	6.4993	6.4994
9	Sodium Chromate	0.9074	0.9073
10	Potassium Nitrate	0.4246	0.4248
11	Sodium Nitrate	59.1395	59.1395

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.13
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2229
			Final	1.0790
			Total	0.1439

pH => 10.03

Notes/Comments: Pre-test pH: Test13 => 10.02 Test13DUP => 10.01
Post-test pH: Test13 => 10.02 Test13DUP => 10.02
Test13 – pitting is only in one small spot. Test13DUP – very small/scarce pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test13	DFLAW_Test13_C02.mpr	6-30-15
Test13DUP	DFLAW_Test13DUP_C04.mpr	6-30-15

Prepared by: [redacted] Date: 6-29-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 14

This formula will make **1.4** Liters

Date: 6-30-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	2.3514	2.3518
3	Sodium Chloride	2.8636	2.8633
4	Ammonium Chloride	0.7489	0.7486
5	Sodium Nitrite	289.7790	289.7792
6	Sodium Sulfate	19.8982	19.8983
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5961
8	Sodium Carbonate	6.4993	6.4990
9	Sodium Chromate	0.9074	0.9074
10	Potassium Nitrate	0.4246	0.4248
11	Sodium Nitrate	59.1395	59.1397

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C ________
Measure pH 10.23
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2224
			Final	0.8700
			Total	0.3524 pH=>10.03

Notes/Comments: Pre-test pH: Test 14=> ~~10.04~~ 10.03 Test14DUP=> 10.04
Post-test pH: Test14=>10.07 Test14DUP=> 10.08 Both have very little pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test14	DFLAW-Test14_C01.mpr	7-1-15
Test14DUP	DFLAW-Test14DUP-C03.mpr	7-1-15

Prepared by: [redacted] Date: 6-30-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **15**

This formula will make **1.4** Liters

Date: 6-30-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3083
2	Sodium Fluoride	2.3514	2.3514
3	Sodium Chloride	4.0908	4.0909
4	Ammonium Chloride	0.7489	0.7486
5	Sodium Nitrite	289.7790	289.7791
6	Sodium Sulfate	19.8982	19.8985
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5968
8	Sodium Carbonate	6.4993	6.4992
9	Sodium Chromate	0.9074	0.9075
10	Potassium Nitrate	0.4246	0.4242
11	Sodium Nitrate	59.1395	~~59.1390~~ 59.1392

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35°C
Measure pH 10.32
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2221
			Final	0.7373
			Total	0.4851 pH => 10.04

Notes/Comments: Pre-test pH: Test15 => 10.04 Test15DUP => 10.02 Test15DUP has scratch 1" from base
Post-test pH: Test15 => 10.05 Test15DUP => 10.04
Test15 – very little pitting. Test15DUP – no pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test15	DFLAW-Test15-C02.mpr	7-1-15
Test15DUP	DFLAW-Test15DUP-C04.mpr	7-1-15

Prepared by: [redacted] Date: 6-30-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 16

This formula will make **1.4** Liters

Date: 7-1-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3082
2	Sodium Fluoride	2.3514	2.3515
3	Sodium Chloride	6.1362	6.1364
4	Ammonium Chloride	0.7489	0.7490
5	Sodium Nitrite	289.7790	289.7791
6	Sodium Sulfate	19.8982	19.8982
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5963
8	Sodium Carbonate	6.5141	6.5145
9	Sodium Chromate	0.9074	0.9072
10	Potassium Nitrate	0.4246	0.4246
11	Sodium Nitrate	59.1395	59.1398

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.24
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2223
			Final	0.8779
			Total	0.3444 pH=>10.03

Notes/Comments: Pre-test pH: Test 16 => ~~9.98~~ 10.00 Test16DUP=> 10.02
Post-test pH: Test16 => 9.93 Test16DUP=> 10.02 No visible pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test16	DFLAW_Test16_C01.mpr	7-6-15
Test16DUP	DFLAW_Test16DUP_C03.mpr	7-6-15

Prepared by: [redacted] Date: 7-1-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID 17**

This formula will make 1.4 Liters

Date: 7-1-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3085
2	Sodium Fluoride	2.3514	2.3515
3	Sodium Chloride	8.1816	8.1816
4	Ammonium Chloride	0.7489	0.7485
5	Sodium Nitrite	289.7790	289.7792
6	Sodium Sulfate	19.8982	19.8983
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.5141	6.5144
9	Sodium Chromate	0.9074	0.9071
10	Potassium Nitrate	0.4246	0.4247
11	Sodium Nitrate	59.1395	59.1394

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.24
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	1.2224
			Final	0.8856
			Total	~~1.0~~ 0.3368 pH => 10.05

Notes/Comments: Pre-test pH: Test17 => 10.09 Test17DUP => 10.02
Post-test pH: Test 17 => 9.94 Test 17DUP => 9.93 No visible pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test17	DFLAW_Test17_C02.mpr	7-6-15
Test17DUP	DFLAW_Test17DUP_C04.mpr	7-6-15

Prepared by: [redacted] Date: 7-1-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **18**

This formula will make **1.4** Liters

Date: 7-6-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	2.3514	2.3517
3	Sodium Chloride	7.3634	7.3634
4	Ammonium Chloride	0.7489	0.7493
5	Sodium Nitrite	386.3720	386.3716
6	Sodium Sulfate	19.8982	19.8980
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.0690	6.0691
9	Sodium Chromate	0.9074	0.9072
10	Potassium Nitrate	0.4246	0.4249
11	Sodium Nitrate	59.1395	59.1390

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.07
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2114	initial	
			Final	
			Total	0.0650 pH=>10.04

Notes/Comments: Pre-test pH: Test18 => 10.03 Test18DUP=> 10.03
Post-test pH: Test18 => 9.98 Test18DUP => ~~9.98~~ 9.97 Some pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test18	DFLAW_Test18_C02.mpr	7-7-15
Test18DUP	DFLAW_Test18DUP_C04.mpr	7-7-15

Prepared by [redacted] Date: 7-6-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY - AIKEN, SC USA 29808 - SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID 19**

This formula will make 1 Liters

Date: 6-10-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2200
2	Sodium Fluoride	1.6796	~~1.6792~~ 1.6798
3	Sodium Chloride	1.8642	1.8644
4	Ammonium Chloride	0.5549	0.5547
5	Sodium Nitrite	275.9800	275.9799
6	Sodium Sulfate	14.2130	14.2132
7	Sodium Phosphate, Tribasic 12	1.1404	1.1403
8	Sodium Carbonate	4.3345	4.3346
9	Sodium Chromate	0.6481	0.6481
10	Potassium Nitrate	0.3033	0.3032
11	Sodium Nitrate	42.2425	42.2427
	Make 1000 mL — *Heat to 35 ° C*		
	Measure pH		9.81@35°C

If pH is great than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.6988	initial	
			Final	
			Total	

Notes/Comments: ~~pH = 10.~~ Pre-test pH: Test19DUP => 9.99 Test19 => 9.99
post-test pH: Test19DUP => 9.91 Test 19 => 9.99

CPP Testing		
Coupon ID 2232	File name	Date
Test19	Test19_*	6-17-15
Test19DUP	Test19DUP_*	6-17-15

Prepared by [redacted]

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID 20**

This formula will make **1.4** Liters

Date: 6-22-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3082
2	Sodium Fluoride	2.3514	2.3512
3	Sodium Chloride	3.4363	3.4361
4	Ammonium Chloride	0.7489	0.7491
5	Sodium Nitrite	386.3720	386.3724
6	Sodium Sulfate	19.8982	19.8979
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	6.0684	6.0682
9	Sodium Chromate	0.9074	0.9072
10	Potassium Nitrate	0.4246	0.4249
11	Sodium Nitrate	59.1395	59.1397

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.19 @ 8:00 a.m.
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2114	initial	1.2110
			Final	1.1388
			Total	0.0722

Notes/Comments: pH after Bicarb. => 10.00 very small pitting started in both
pH Before Test: Test20 => ~~10.04~~ 9.98 Test20DUP => 10.00 Test20 has a scratch about
post test pH: Test20 => 10.14 Test20DUP => 10.15 ¼ inch from tip (after test)

CPP Testing		
Coupon ID 2232	File name	Date
Test20	DFLAW_Test20-C02.mpr	6-24-15
Test20DUP	DFLAW_Test20DUP_C04.mpr	6-24-15

Prepared by: [redacted] Date: 6-22-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY - AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 21

This formula will make **1.4** Liters

Date: 7-7-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	2.9392	2.9391
3	Sodium Chloride	3.2726	3.2729
4	Ammonium Chloride	0.7489	0.7490
5	Sodium Nitrite	386.3720	386.3725
6	Sodium Sulfate	19.8982	19.8980
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5962
8	Sodium Carbonate	6.0596	6.0596
9	Sodium Chromate	0.9074	0.9076
10	Potassium Nitrate	0.4246	0.4243
11	Sodium Nitrate	59.1395	59.1396

Transfer solution to volumetric flask and fill to mark with distilled water.

Heat to 35 ° C

Measure pH 10.12

If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2055	initial	—
			Final	—
			Total	— 0.1308 pH => 10.04

Notes/Comments: Pre-test pH: Test21 => 9.98 Test21DUP => 9.98
Post-test pH: Test21 => 9.97 Test21DUP => 9.97 Test21 => No pitting
Test21DUP => Pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test21	DFLAW_Test21_C01.mpr	7-8-15
Test21DUP	DFLAW_Test21DUP_C03.mpr	7-8-15

Prepared by [redacted] Date: 7-7-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **22**

This formula will make **1.4** Liters

Date: 7-7-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	2.9392	2.9394
3	Sodium Chloride	4.0908	4.0906
4	Ammonium Chloride	0.7489	0.7488
5	Sodium Nitrite	386.3720	386.3719
6	Sodium Sulfate	19.8982	19.8981
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5962
8	Sodium Carbonate	6.0541	6.0540
9	Sodium Chromate	0.9074	0.9076
10	Potassium Nitrate	0.4246	0.4243
11	Sodium Nitrate	59.1395	59.1390

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 10.05
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.2231	initial	—
			Final	—
			Total	~~[illegible]~~

Notes/Comments: Pre-test pH: Test22 => 10.06 Test22DUP => 10.05
Post-test pH: Test22 => 10.03 Test22DUP => 10.02 Test22 => No pitting
Test22 had solution in glass tube Test22DUP => Very little pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test22	DFLAW_Test22_C02.mpr	7-8-15
Test22DUP	DFLAW_Test22DUP_C04.mpr	7-8-15

Prepared by: [redacted] Date: 7-7-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 23

This formula will make **1.4** Liters

Date: 7-8-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3085
2	Sodium Fluoride	2.9392	2.9389
3	Sodium Chloride	8.5907	8.5904
4	Ammonium Chloride	0.7489	0.7485
5	Sodium Nitrite	386.3720	386.3721
6	Sodium Sulfate	19.8982	19.8982
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5962
8	Sodium Carbonate	24.4837	24.4841
9	Sodium Chromate	0.9074	0.9077
10	Potassium Nitrate	0.4246	0.4246
11	Sodium Nitrate	59.1395	59.1391

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35° C
Measure pH 10.23
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1996	initial	–
			Final	–
			Total	~~0.1087~~ 0.9520 pH=> 10.04

Notes/Comments: Pre-test pH: Test 23=> 9.98 Test23DUP=> 10.01
Post-Test pH: Test 23 = 10.02 Test 23 DUP = 9.99

CPP Testing		
Coupon ID 2232	File name	Date
Test 23	DFLAW_Test23_C01.mpr	7-9-15
Test23DUP	DFLAW_Test23DUP-C03.mpr	7-9-15

Prepared [redacted] Date: 7-8-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 24

This formula will make **1.4** Liters

Date: 7-8-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3082
2	Sodium Fluoride	2.9392	2.9390
3	Sodium Chloride	11.4542	11.4546
4	Ammonium Chloride	0.7489	0.7492
5	Sodium Nitrite	386.3720	~~386.3728~~ 386.3723
6	Sodium Sulfate	19.8982	19.8980
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5965
8	Sodium Carbonate	29.6772	29.6774
9	Sodium Chromate	0.9074	0.9078
10	Potassium Nitrate	0.4246	0.4242
11	Sodium Nitrate	59.1395	59.1393

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.30
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12 Sodium Bicarbonate	1.1996	initial	1.1999	
		Final	0.0695	
		Total	1.1304	pH=>10.04

Notes/Comments: Pre-test pH: Test 24=>10.05 Test 24 DUP=> 10.02
Post test pH: Test24 = 10.02, Test 24 Dup = 9.99 9.99

CPP Testing		
Coupon ID 2232	File name	Date
Test24	DFLAW-Test24_C02.mpr	7-9-15
Test24DUP	DFLAW-Test24DUP-C04.mpr	7-9-15

Prepared by: [redacted] Date: 7-8-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY · AIKEN, SC USA 29808 · SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 25

This formula will make **1.4** Liters

Date: 6-23-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3080
2	Sodium Fluoride	2.3514	2.3513
3	Sodium Chloride	3.4363	3.4366
4	Ammonium Chloride	0.7489	0.7488
5	Sodium Nitrite	482.9650	482.9648
6	Sodium Sulfate	19.8982	19.8983
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5967
8	Sodium Carbonate	5.4234	~~5.4232~~ 5.4233
9	Sodium Chromate	0.9074	0.9074
10	Potassium Nitrate	0.4246	0.4244
11	Sodium Nitrate	59.1395	~~59.1395~~ 59.1393

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35° C
Measure pH 9.96
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1879	initial	
			Final	
			Total	

Notes/Comments: pre-test pH: Test 25 => 10.03 Test25DUP => 10.04
post-test pH: Test 25 => 10.06 Test25DUP => 10.05 Pitting on Both

CPP Testing		
Coupon ID 2232	File name	Date
Test25	DFLAW_Test25_C02.mpr	6-24-15
Test25DUP	DFLAW_Test25DUP_C04.mpr	6-24-15

Prepared by: [redacted] Date: 6-23-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID 26**

This formula will make **1** Liters

Date: 6-11-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.2200	0.2204
2	Sodium Fluoride	2.0995	2.0992
3	Sodium Chloride	2.3376	2.3374
4	Ammonium Chloride	0.5549	0.5552
5	Sodium Nitrite	344.9750	344.9753
6	Sodium Sulfate	14.2130	14.2131
7	Sodium Phosphate, Tribasic 12	1.1404	1.1405
8	Sodium Carbonate	3.8781	3.8781
9	Sodium Chromate	0.6481	0.6480
10	Potassium Nitrate	0.3033	0.3034
11	Sodium Nitrate	42.2425	42.2421
	Heat to 35˚ C		
	Measure pH		9.74 @ ~~35°C~~ 42°C

If pH is great than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	0.6988	initial	
			Final	
			Total	

Notes/Comments: 9.82 @ ~~35°C~~ 35°C Pre-test pH: Test26 => 10.02 Test26DUP => 9.91
Post-test pH: Test26 => 9.98 Test26DUP => 9.87

CPP Testing		
Coupon ID 2232	File name	Date
Test26	Test26_*	6-18-15
Test26DUP	Test26DUP_*	6-18-15

Prepared by: [redacted]

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **27**

This formula will make **1.4** Liters

Date: 7-9-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3084
2	Sodium Fluoride	2.9392	2.9390
3	Sodium Chloride	4.0908	4.0910
4	Ammonium Chloride	0.7489	0.7486
5	Sodium Nitrite	482.9650	482.9648
6	Sodium Sulfate	19.8982	19.8983
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5966
8	Sodium Carbonate	5.4309	5.4312
9	Sodium Chromate	0.9074	0.9077
10	Potassium Nitrate	0.4246	0.4245
11	Sodium Nitrate	59.1395	59.1394

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH 9.75
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1890	initial	
			Final	
			Total	

Notes/Comments: Pre-test pH: Test27 => 9.96 Test27DUP => 10.00
Post-test pH: Test27 => 9.99 Test27DUP => 9.97

CPP Testing		
Coupon ID 2232	File name	Date
Test27	DFLAW_Test27_C01.mpr	7-13-15
Test27DUP	DFLAW_Test27DUP_C09.mpr	7-13-15

Prepared by: [redacted] Date: 7-9-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 28

This formula will make 1.4 Liters

Date: 7-13-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3079
2	Sodium Fluoride	2.9392	2.9395
3	Sodium Chloride	4.9090	4.9092
4	Ammonium Chloride	0.7489	0.7485
5	Sodium Nitrite	482.9650	482.9656
6	Sodium Sulfate	19.8982	19.8979
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5967
8	Sodium Carbonate	5.4309	5.4313
9	Sodium Chromate	0.9074	0.9074
10	Potassium Nitrate	0.4246	0.4244
11	Sodium Nitrate	59.1395	59.1395

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 °C
Measure pH ~~9.96~~ 9.98
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1890	initial	
			Final	
			Total	

Notes/Comments: pre-test pH: Test28 => 9.96 Test28DUP => 9.96
Post-test pH: Test 28 => 9.99 Test 28DUP => 10.00 Test 28DUP has very little pitting

CPP Testing		
Coupon ID 2232	File name	Date
Test28	DFLAW_Test28_C01.mpr	7-14-15
Test28DUP	DFLAW_Test28DUP_C03.mpr	7-14-15

Prepared by: [redacted] Date: 7-13-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** **29**

This formula will make **1.4** Liters

Date: 7-13-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3081
2	Sodium Fluoride	2.9392	2.9393
3	Sodium Chloride	8.5907	8.5907
4	Ammonium Chloride	0.7489	0.7485
5	Sodium Nitrite	482.9650	482.9648
6	Sodium Sulfate	19.8982	19.8984
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5968
8	Sodium Carbonate	24.4837	24.4832
9	Sodium Chromate	0.9074	~~0.9078~~ 0.9070
10	Potassium Nitrate	0.4246	0.4248
11	Sodium Nitrate	59.1395	59.1396

Transfer solution to volumetric flask and fill to mark with distilled water.
Heat to 35 ° C
Measure pH 10.19
If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1890	initial	1.1155
			Final	0.6073
			Total	0.5082 pH => 10.04

Notes/Comments: Pre-test pH: Test29 => 10.03 Test29DUP => 10.03
Post-test pH: Test29 => 10.06 Test29DUP => 10.07
Test29DUP & Test29 have a scratch 1/4" from tip

CPP Testing		
Coupon ID 2232	File name	Date
Test29	DFLAW_Test29_C02.mpr	7-14-15
Test29DUP	DFLAW_Test29DUP_C04.mpr	7-14-15

Prepared by [redacted] Date: 7-13-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY • AIKEN, SC USA 29808 • SRNL.DOE.GOV

SRNL

DFLAW Simulant **Test ID** 30

This formula will make 1.4 Liters

Date: 7-14-15 Batch no.: 1

Start with about 50% of the total volume of distilled water.

		Target weight, g	Actual Weight, g
1	Sodium Aluminate	0.3080	0.3084
2	Sodium Fluoride	2.9392	2.9392
3	Sodium Chloride	11.4542	11.4540
4	Ammonium Chloride	0.7489	0.7489
5	Sodium Nitrite	482.9650	482.9653
6	Sodium Sulfate	19.8982	19.8982
7	Sodium Phosphate, Tribasic 12 H2O	1.5965	1.5967
8	Sodium Carbonate	29.6772	29.6769
9	Sodium Chromate	0.9074	0.9073
10	Potassium Nitrate	0.4246	0.4250
11	Sodium Nitrate	59.1395	59.1390

Transfer solution to volumetric flask and fill to mark with distilled water.

Heat to 35 °C

Measure pH 10.22

If pH is greater than 10, add bicarbonate up to the amount in line 12.

12	Sodium Bicarbonate	1.1879	initial	1.1140
			Final	0.4611
			Total	0.6529 pH=> 10.06

Notes/Comments: Pre-test pH: Test 30=> ~~10.08~~ 10.06 Test30DUP=> 10.05
Post-test pH: Test30=> 10.11 Test30DUP=> 10.06

CPP Testing		
Coupon ID 2232	File name	Date
Test30	DFLAW-Test30_C01.mpr	7-15-15
Test30DUP	DFLAW_Test30DUP_C02.mpr	7-15-15

Prepared by: [redacted] Date: 7-14-15

We put science to work.™

SAVANNAH RIVER NATIONAL LABORATORY - AIKEN, SC USA 29808 - SRNL.DOE.GOV

Appendix D Distribution List

Name:	Organization:
K. E. Zeigler	SRNL
B. J. Wiersma	SRNL
R. E. Fuentes	SRNL
R. B. Wyrwas	SRNL
W. A. Drown	SRNL
M. E. Stone	SRNL
J. R. Pelfrey	SRNL
C. C. Herman	SRNL
H. H. Burns	SRNL
R. B. Wyrwas	SRNL
K. D. Boomer	WRPS
J. L. Castleberry	WRPS
S. T. Arm	WRPS
R. H. Spires	WRPS