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ACTIVITY/PROJECT SPECIFIC PROCEDURE

SP 12-40
CALIBRATION AND USE OF THE GAMRY INTERFACE 1010E POTENTIOSTAT/GALVANOSTAT
Revision 1

Effective Date: March 3, 2021

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1.0 Purpose and Scope

This procedure prescribes the Sandia National Laboratories (SNL) Waste Isolation Pilot Plant (WIPP) process for the calibration and use of the Gamry Interface 1010E Potentiostat/Galvanostat Electrochemical system.

The Gamry Interface 1010E Potentiostat/Galvanostat consists of a main electrical unit and accompanying cables. The purpose of this unit is to use electrical potentials and currents to perform various electroanalytical experiments, in this case specifically Electrical Impedance Spectroscopy and Potentiodynamic Polarization.

This document is not meant to substitute for the manufacturer's instruction manuals for the Gamry Interface 1010E Potentiostat/Galvanostat or the manufacturer's recommendations for the potentiostat calibration. The user is responsible for reading and understanding the appropriate manuals for the items being calibrated and the equipment used.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

2.0 Implementation Actions

2.1 Safety

The activities described in this Activity/Project Specific Procedure (SP) shall conform to SNL Environmental Health and Safety Programs (ES&H). All activities described in this SP are subject to ES&H requirements governed by the WIPP Industrial Safety Program and the WIPP Industrial Hygiene Program.

As defined in the SNL ES&H manual, personnel will not be exposed to hazardous voltages. The voltage levels expected in the performance of this procedure should be 24 VDC or less.

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2.2 Responsibilities

The Principal Investigator (PI) or designee, whose activities warrant the use of this procedure, shall be responsible for implementing the requirements of this procedure.

The technical staff is responsible for performing the measurements following the requirements of this procedure, and documenting all of the requiring information as described in this SP. Technical staff should familiarize themselves with the manufacturer's manuals for the Gamry Interface 1010E Potentiostat/Galvanostat for an understanding of the assembly parts and the nomenclature, controls, operation, and measurement methods. The Potentiostat/Galvanostat is controlled by the included software via the host computer (see Section 2.8.6).

If this procedure cannot be worked as written, the user has the responsibility to stop work and resolve all concerns with either the PI, Safety or QA representative, as appropriate, prior to proceeding with the work.

2.3 Standards

The Gamry Interface 1010E Potentiostat/Galvanostat uses a 2 kΩ calibration cell included with the unit. During the calibration procedure, cables are inserted into the proper ports of the calibration cell. Upon completion of the calibration program, the software will determine if the calibration is successful. If unsuccessful, a calibration warning will appear and the appropriate corrective action can be taken.

2.4 Frequency

The Gamry Interface 1010E Potentiostat/Galvanostat will be calibrated in accordance to the manufacturer recommendations. Gamry recommends running a calibration under the following conditions:

1. It has been at least one year since the last calibration.
2. The potentiostat has been serviced.
3. Breaks or discontinuations in data curves are observed.
4. The system is being operated in an environment that is significantly different from that of the previous calibration.

The PI or technician may elect to length or shorten the calibration interval based on the results of previous calibrations and the stability of the equipment. Any deviations from the established calibration interval will be justified in the applicable scientific notebook/supplement.

2.5 Acceptance Criteria

The accuracy of the potentiostat/galvanostat will be based on manufacturer's specifications included in the calibration check or the requirements of the PI or technician. If the calibration of the Potentiostat/Galvanostat fails the manufacturer's or PI's specifications, the calibration will be identified as "failed" and tests should not be run until any issues are resolved.

2.6 Corrective Action

If a calibration warning is received, it should be investigated in order to determine the necessary corrective action. A warning does not necessarily indicate a critical failure, and Gamry can use calibration information to determine the source of the warning. If a warning is received, the following steps should be implemented:

1. Double check the following:
   - Regular Connections are to the Calibration side of the 2 kΩ calibration cell.
   - Floating-ground cable is connected to the calibration shield.
   - If possible, the chassis ground is connected to a known, good earth ground.
2. Click the retry button and restart the calibration.
3. After a failed calibration, locate the MyGamry Data folder and select the calibration results. This file can be emailed to Gamry at techsupport@gamry.com to resolve any issues.

2.7 Calibration / Verification Procedure

The calibration process includes the following general steps:

2.7.1 Turn on the equipment to be used in the performance of this procedure, allow the equipment to warm-up a minimum of 10 minutes before starting the calibration.

2.7.2 While the equipment is warming-up, inspect and ensure the main cable connection is securely connected to the potentiostat/galvanostat.

2.7.3 Locate the chassis ground port on the back of the potentiostat/galvanostat and if possible, connect it to a known earth ground.

2.7.4 Locate the 2 kΩ calibration cell included with the unit. Plug the cell cables into their respective ports (Figure 1). The cables and labels on the calibration cell are color coded.

Figure 1. Connection of Color-Coded Cables for Calibration

2.7.5 Place the connected calibration cell inside of the included calibration shield box (Figure 2a). Close the lid of the box (Figure 2b) and connect the floating ground lead of the cell cable to the shield box grounding post.

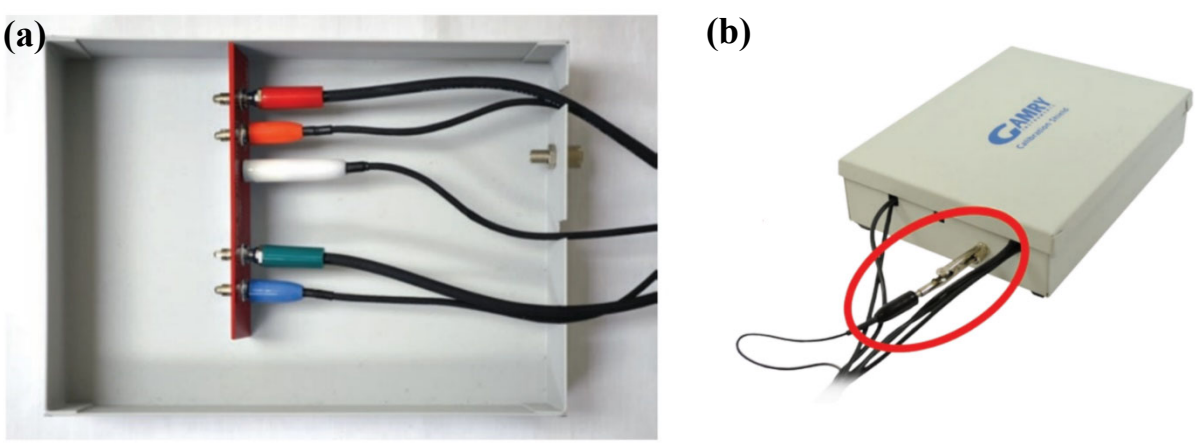

Figure 2. Connected Calibration Cell Inside the Shield Box: (A) Opened Box, (B) Closed Box

2.7.6 Open the Gamry Framework software and select Experiment → Utilities → Calibrate Instrument (Figure 3).

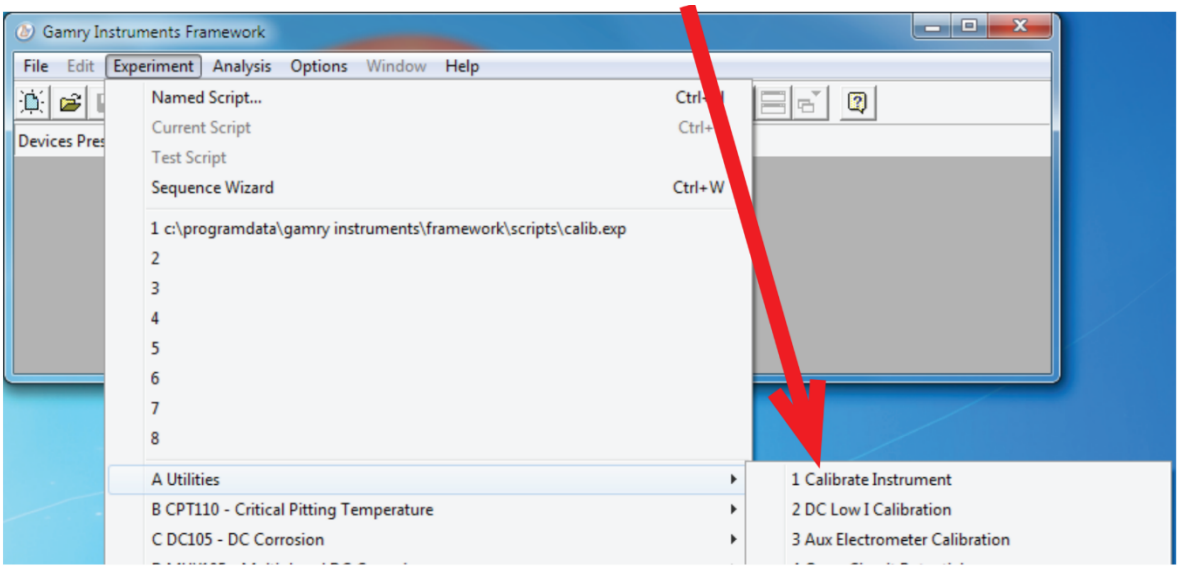

Figure 3. Snapshot of Gamry Framework

2.7.7 In the pop-up menu, ensure that calibration type "both" is selected (Figure 4.). This will perform both a DC and AC calibration.

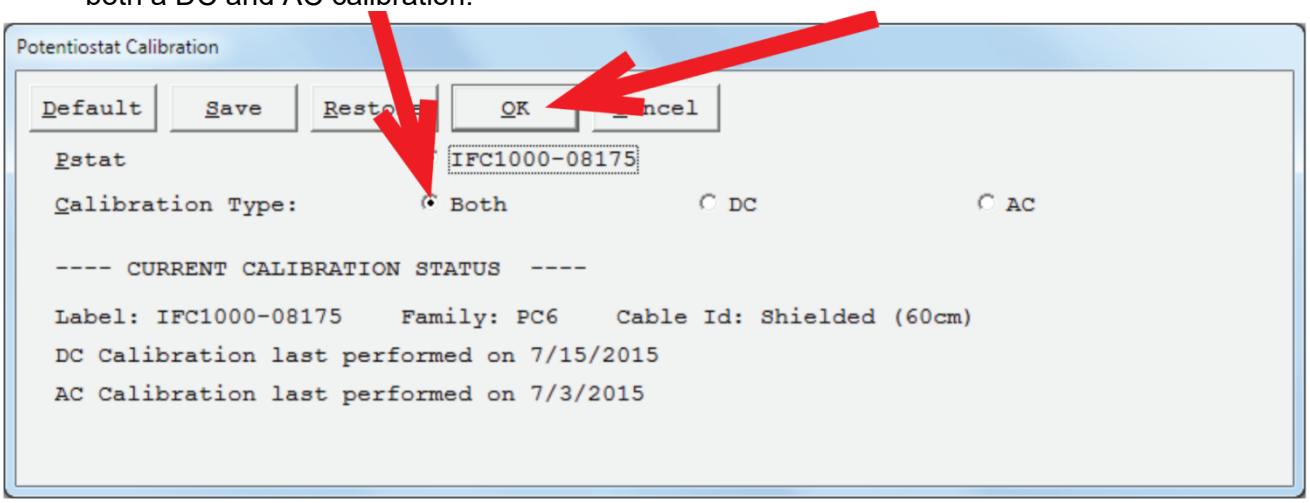

Figure 4. Snapshot of Calibration Process from Gamry Framework

2.7.8 Confirm any instructional messages that appear on screen (Figure 4). Calibration process will then initiate.

2.7.9 After calibration is complete, a calibration successful message should appear. If a calibration warning appears, follow the troubleshooting method discussed in Section 2.6. Calibrations will be documented in the scientific notebook or supplement.

2.8 Operation of The Gamry Interface 1010E Potentiostat/Galvanostat

Figure 5 shows the photo of the Gamry interface 1010E Potentiostat/Galvanostat. The operation process of this instrument includes the following steps:

Figure 5. Gamry Interface 1010E

2.8.1 Turn on the equipment to be used in the performance of this procedure, allow the equipment to warm-up a minimum of 10 minutes before starting a test.

2.8.2 While the equipment is warming-up, inspect and ensure the main cable connection is securely connected to the potentiostat/galvanostat.

2.8.3 Locate the chassis ground port on the back of the potentiostat/galvanostat and if possible, connect it to a known earth ground.

2.8.4 Connect the cell cables to the proper ports on the Gamry Electrochemical Cell. For a typical three electrode set-up, they are as follows:

- **Working Electrode**: For corrosion experiments, the working electrode is typically the sample of interest. In this set-up it includes the steel sample emplaced in the flat sample holder. Connect both the Work Cable (Green) and the Work Sense Cable (Blue) to the matching ports on the top of the sample holder (Figure 6).

Figure 6. Connection of the Working Electrode Attached to the Flat Sample Holder

- **Reference Electrode:** Reference electrode type can vary but they are often Saturated Calomel or Silver/Silver Chloride types. Connect the Reference Cable (White) to this electrode (Figure 7).

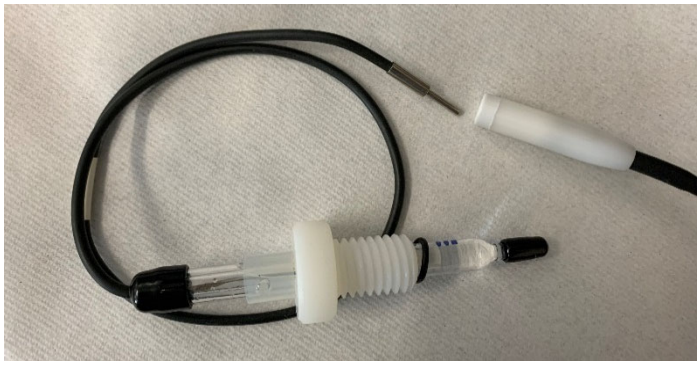

Figure 7. A Photo of a Silver/Silver Chloride Type Reference Electrode

- **Counter Electrode:** The counter electrode is often either a graphite rod or platinum wire. Connect the Counter Cable (Red) and Counter Sense Cable (Orange) to this electrode (Figure 8).

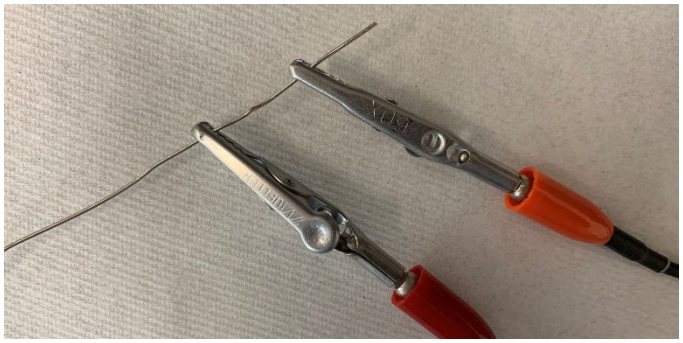

Figure 8. Connection of a Platinum Wire Counter Electrode

2.8.5 Ensure the rest of the conditions in the electrochemical cell are as desired and proceed to run experiment (Figure 9).

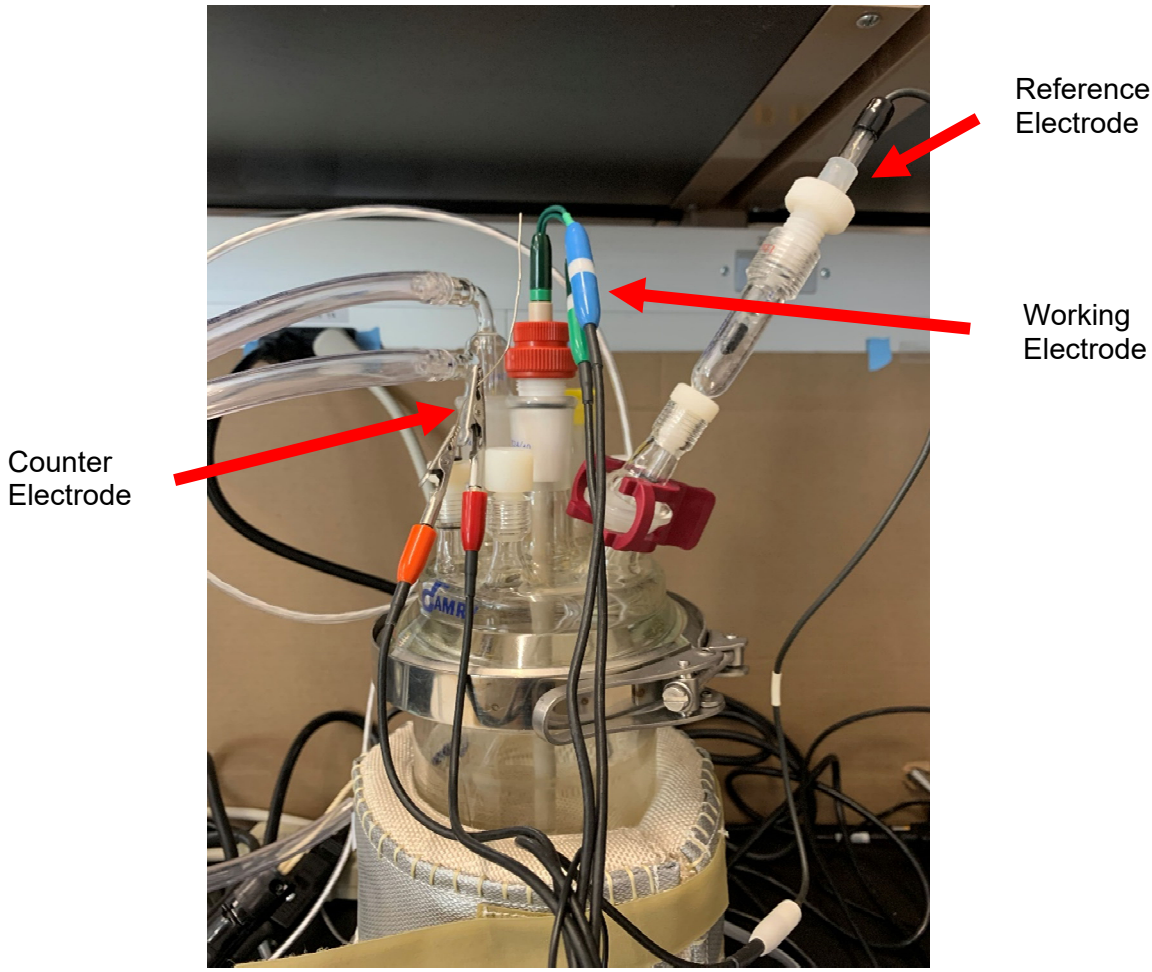

Figure 9. A Typical Experimental Setup of the Electrochemical Cell with All Connections

2.8.6 Open the Gamry Framework software and select Experiment → desired experiment type (Figure 10).

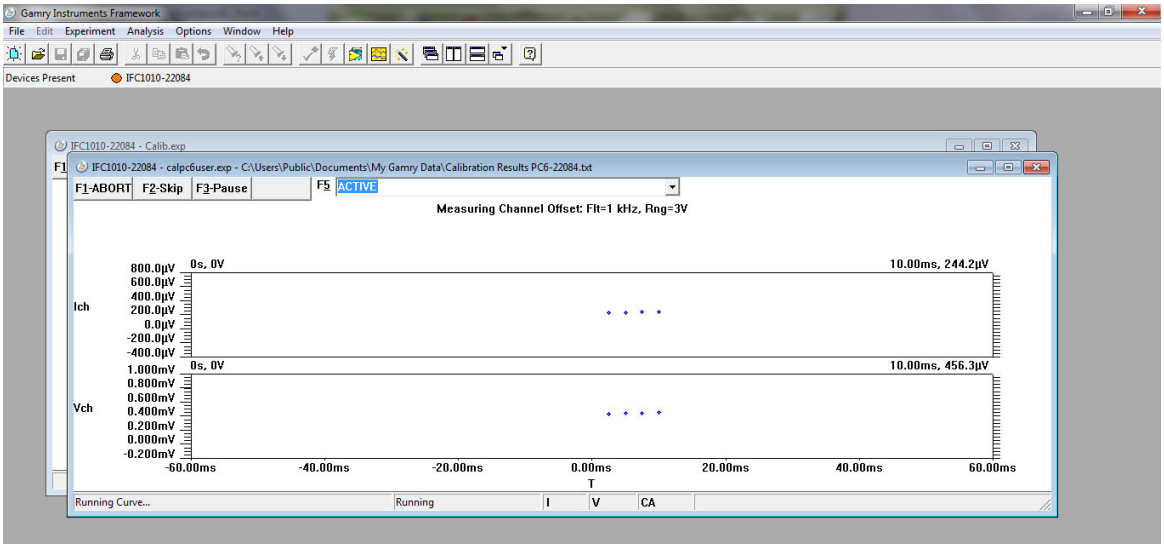

Figure 10. Snapshot of the Gamry Framework Software at Acquiring Data

2.8.7 In the menu enter any notes relevant to the test as well as the desired specifications and hit 'OK' (see Figure 11a) to acquire all data as shown in Figure 10. The following subsections list a few testing examples that are most often used in corrosion experiments.

2.8.7.1 Electrochemical Impedance Spectra (EIS) measurements in a single run:

- From Gamry Framework menu, select Experiment→G Electrochemical Impedance→5 Potentiostatic EIS. It brings up the 'Potentiostatic EIS' window (Figure 11a).
- Input Test Identifier, Output File, Notes, and other parameters in the window. The parameters shown in Figure 11a are the default values for an EIS measurement of iron corrosion. Depending on testing materials and/or systems, the parameters could be different. See the Gamry Interface 1010E Manual for details.
- Hit 'OK' to acquire all data.

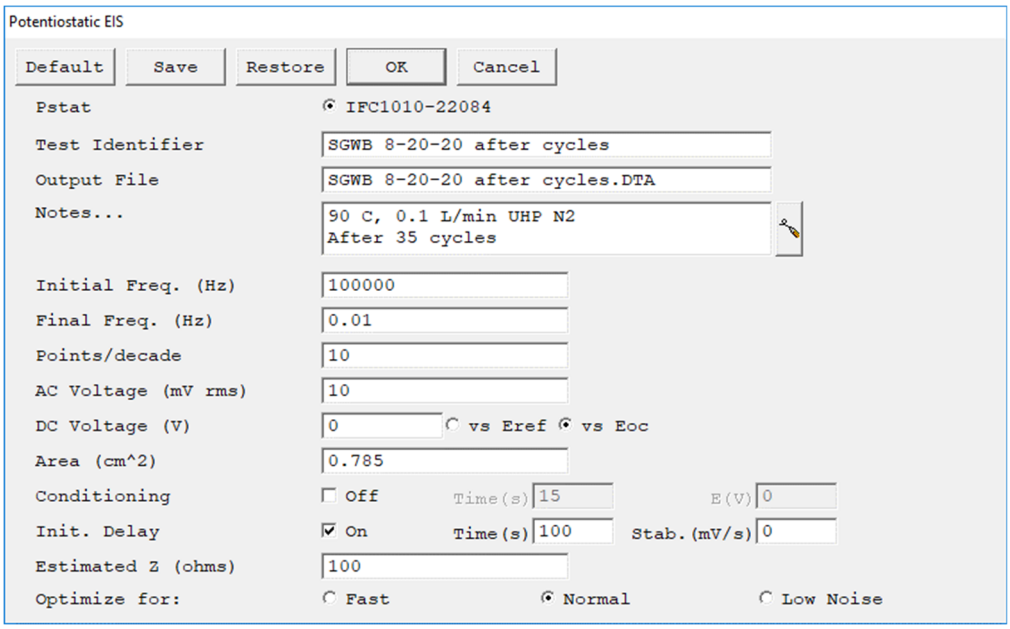

Figure 11a. Snapshot of the EIS measurement window for parameter inputs

2.8.7.2 Electrochemical Impedance Spectra (EIS) measurements in a sequence of runs: Gamry Framework has a sequence feature in which a number of runs at different time periods can be set up so that a sequence of EIS measurements can be obtained at a desired time period. This feature is particularly useful when EIS data are necessary to acquire at different time periods. The sequence runs can be performed automatically by creating a sequence file.

- From Gamry Framework menu, select Experiment→Sequence Wizard. It brings up a window (Figure 11b)
- Pick the desired steps from the Available Steps panel (left panel in Figure 11b) and move them to the right (User Defined Sequence panel). For EIS measurement, the following steps are suggested:
  (1) Loop (cycle): This step creates a sequence runs at a desired time period.
  (2) Open Circuit Potential: An 'Open Circuit Potential' MUST be included before each individual EIS is run in a sequence of measurements since the open circuit potential is required prior to each EIS measurement.
  (3) Potentiostatic EIS: This step is similar to the one described in Section 2.8.7.1. User needs to enter Test Identifier, Output File, Notes, and other parameters in the window.

(4) Delay: This step specifies how long (hours/minutes/seconds) is the delay before next EIS measurements. No standard interval is required, but 1-2 hours is suggested for corrosion experiments,

(5) User Prompt: This step prompts a message (e.g., 'EIS Cycles are Done! Press OK to continue...') when a sequence of EIS runs are completed.

- Hit 'Run Sequence' to start the sequence runs until the 'User Prompt' message pops up. All acquired data in sequence runs should be saved automatically in the file location specified above.

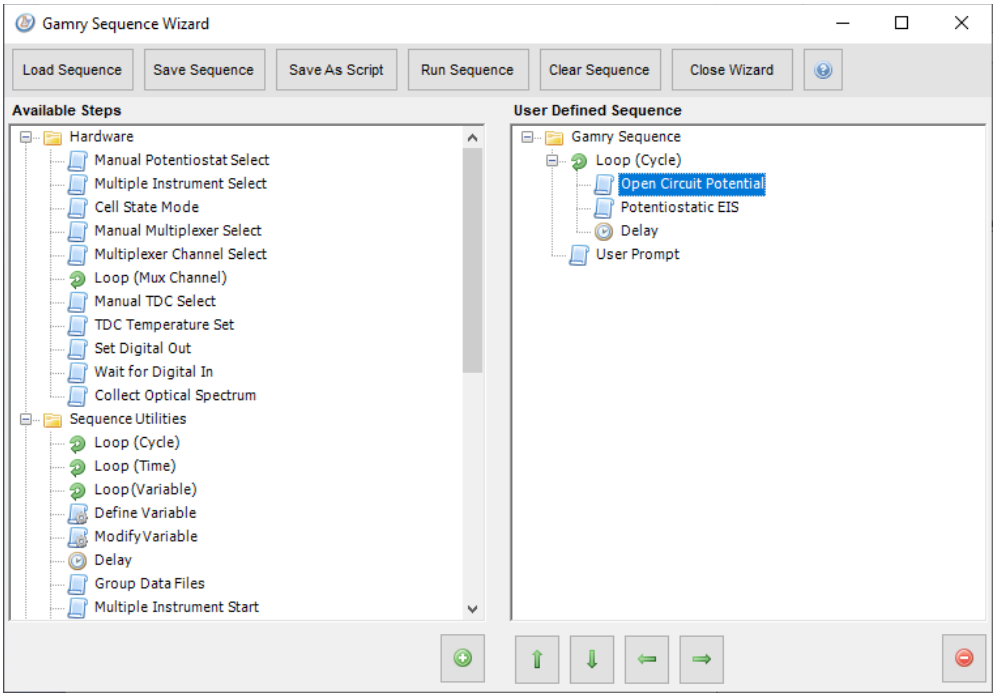

Figure 11b. Gamry Sequence Wizard window

2.8.7.3 Potentiodynamic Polarization (PDP) measurements in a single run:

- From Gamry Framework menu, select Experiment→DC Corrosion→A Potentiodynamic and bring up a window (Figure 11c).
- Input parameters on the window. CAUTION: The DC applied to the coupon material in corrosion studies should be specified from low to high (e.g., 'Initial E=-0.5 V' to 'Final E=+0.5 V' as shown in Figure 11c), so that the DC is scanning from cathodic to anodic. The E range (from Initial to Final) is dependent on open circuit potential (OCP). Generally, a small potential vs. OCP is applied to the coupon for a PDP test so that a good Tafel fitting could be obtained. The parameters shown in Figure 11c are the default values for a PDP measurement of iron corrosion. Depending on testing materials and/or systems, the parameters could be different. See the Gamry Interface 1010E Manual for details.
- Hit 'OK' to acquire all data.

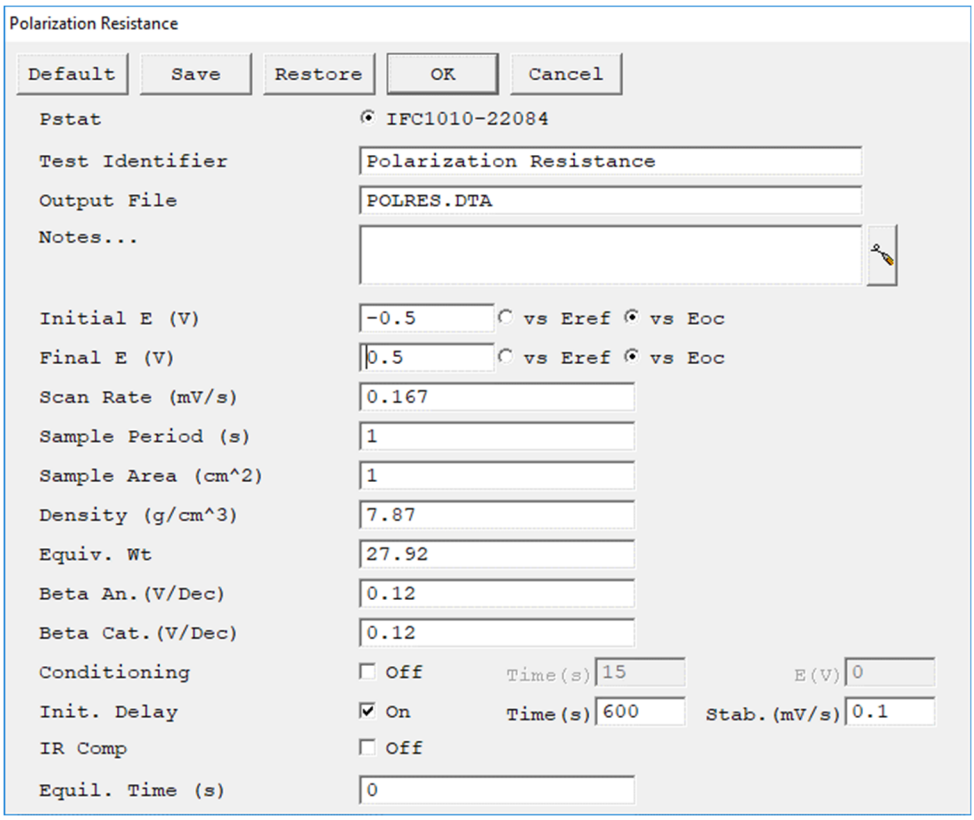

Figure 11c. Snapshot of the PDP measurement window for parameter inputs

2.8.8 Save the test in the desired folder. The acquired data will be recorded as a .DTA file which is readable by the Gamry Echem Analyst software.

2.8.9 From the Gamry Echem Analyst software (Figure 12), data can be exported by pressing the "copy to clipboard" button in the software and pasting into an excel sheet.

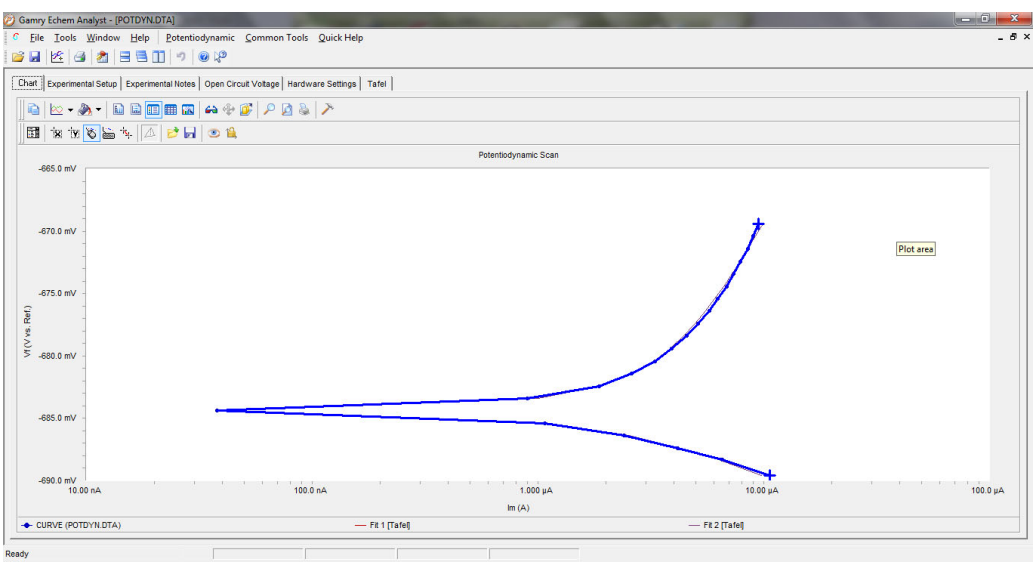

Figure 12. Snapshot of the Gamry Echem Analyst software

2.8.10 Raw data excel files will be printed off and recorded in an associated supplemental binder and analyzed data will be written up in the scientific notebook. Sample set up will also be documented in the scientific notebook.

2.9 Turning off/Showdown the Gamry Interface 1010E Potentiostat/Galvanostat

2.9.1 Once desired testing is complete and the potentiostat/galvanostat is no longer in use, close and exit the Gamry Framework software.

2.9.2 Press the power button on the front of the potentiostat/galvanostat and ensure the light switches off (Figure 5).

2.9.3 Disconnect all electrode cables from the electrochemical cell.

2.10 Maintenance of the Gamry Interface 1010E Potentiostat/Galvanostat

There is no maintenance associated with this instrument.

2.11 References

The Gamry Interface 1010E Potentiostat/Galvanostat Operator Manual. A PDF version of the user manual is located on the 'desktop' of the computer.

3.0 Records

The following records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

QA Record

- Scientific Notebook (SN)
- Scientific Notebook Supplement

4.0 Appendices

Not Applicable. There are no appendices associated with this document.

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