GEOSCIENCES AND ENGINEERING DIVISION

TECHNICAL OPERATING PROCEDURE

Proc. TOP–022

Rev. 1

Title: **PROCEDURE FOR VERIFICATION OF THE PERFORMANCE OF A POTENTIOSTAT AND THE ASSOCIATED DATA ACQUISITION SOFTWARE**

EFFECTIVITY AND APPROVAL

Revision 1 of this procedure became effective on September 16, 2005. This procedure consists of the pages and changes listed below.

Page No.	Change	Date Effective
All	0	09/15/2005

Supersedes Procedure No. TOP–022, Rev 0 Chg 0, dated 3/30/1994

Prepared by	Date	Approved by	Date
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PROCEDURE FOR VERIFICATION OF THE PERFORMANCE OF A POTENTIOSTAT AND THE ASSOCIATED DATA ACQUISITION SOFTWARE

1. PURPOSE AND APPLICABILITY

1.1 The purpose of this procedure is to provide a method for the verification of the performance of a potentiostat and the associated data acquisition software. This procedure establishes controls required by Quality Assurance Manual (QAM), Section 12, "Control of Measuring and Test Equipment."

1.2 Potentiostats and galvanostats are used to control the potential or current, respectively, of electrodes or test specimens. The same instrument can be used in either potential or current mode. Hence, reference is made in this document only to potentiostats, although the same procedures are applicable to a galvanostats. Generally, potentiostats are not calibrated directly, but the measurements of potential and current made by a potentiostat are verified independently with calibrated voltmeters and ammeters.

1.3 For some types of potentiostats designed to operate only with automated data acquisition systems (i.e., no front panel controls), it is not always convenient to measure the potentials independently by other instruments. In such cases, the potentiostat/data acquisition system can be used to measure known parameters such as the resistance of a precision, pre-calibrated resistor.

1.4 This procedure applies to those cases where it is difficult to verify the performance of a potentiostat independently by calibrated voltmeter and ammeter. This procedure need not be applied if potential and current are verified by calibrated voltmeters and ammeters.

2. RESPONSIBILITIES

2.1 Principal investigators are responsible for the implementation of this procedure.

2.2 Personnel performing electrochemical tests are responsible for complying with the requirements of this procedure.

3. ABBREVIATIONS

The following abbreviations are used throughout this procedure.

- I = Current
- V = Potential
- R = Resistance
- A, mA, µA = Amps, milliamps, microamps
- V, mV = Volts, millivolts

- Ω, kΩ = Ohms, kiloohms

4. PROCEDURE

4.1 A precision resistor (with a tolerance of ±1 percent) of appropriate resistance shall be used in the verification tests. The value of the resistance can be chosen depending on the current and voltage ranges anticipated in the actual electrochemical tests. For example, if the anticipated currents in the electrochemical experiments range from 1 μA to 1 mA, then a 1 kΩ resistor should be used. The applied potentials in this case would range from 1 mV to 1 V, which is within functional range of most potentiostats. If higher currents are anticipated, then a lower resistance must be used and vice versa.

4.2 The precision resistor should be placed inside a box with appropriate three terminal connections as shown in Figure 1.

4.3 The resistor shall be calibrated periodically as specified in QAP–019, Control of Measuring and Test Equipment. The initial calibration interval shall be six months, but shall be adjusted as needed depending on laboratory conditions and stability of the resistor.

4.4 The connections to the potentiostat are shown in Figure 1. The ground connection of the potentiostat need not be connected since the working electrode provides the ground. Once the connections are made, the applied potential from the potentiostat can be either manually changed or scanned automatically, and the current-potential data can be collected.

4.5 The current-potential data can then be exported or manually input to appropriate analysis software to calculate the resistance ($R = V/I$) and various statistical measures associated with it. An example measurement is shown in Figure 2. The measured resistance (along with its 95 percent confidence interval) should fall within the tolerance of the resistor if the potentiostat and the associated data acquisition system are functioning properly. If the measured resistance and 95 percent confidence interval are significantly different from the specified tolerance of the resistor, then the resistor must be checked independently for possible damage. A calibrated electrometer with resistance measuring capability (with a tolerance of ± 0.2 percent) should be used to determine if the resistor is damaged. If resistor damage is found, Section 4.4 shall be repeated with another calibrated resistor. If, on the other hand, no damage resistor is found, the system (either the data acquisition system or the potentiostat) shall be considered defective, and further corrective actions shall be undertaken before using that system. Measurements taken since the last acceptable verification shall be evaluated to determine if the potentiostat error invalidates the results as required by QAP–019, Control of Measuring and Test Equipment, and QAP–009, Nonconformance Control.

4.6 The procedure outlined in Sections 4.4 and 4.5 shall be performed and documented prior to conducting potentiostatic measurement. When the performance of the potentiostat has been verified, a performance verification label shall be placed on the instrument. An example performance verification label is shown in Figure 3. The label should include the verification date and the date the next scheduled verification should be performed. Performance verification of potentiostats should be performed twice per year or whenever the instrument performance is suspicious.

4.7 For potentiostat systems in which the potential is scanned automatically, the scan rate is a parameter that is usually specified and controlled. For the case of a pure resistor shown in Figure 1, the scan rate has no effect on the measured resistance. Therefore, any convenient scan rate (typically 1 mV/sec) can be used in step 4.4 to conduct the verification test.

In electrochemical experiments, the potential scan rate can be an important factor and hence, will need an independent verification. Since the potential sweep is linear in these experiments, the scan rate is calculated by dividing the total range of the potential by the time interval between the start and end of potential scan. The selected scan rate should be compared to the calculated scan rate and must be noted in the appropriate scientific notebook.

5. RECORDS

Documents containing verification data, including scientific notebooks, shall be controlled as QA records in accordance with QAP–012, Quality Assurance Records Control.

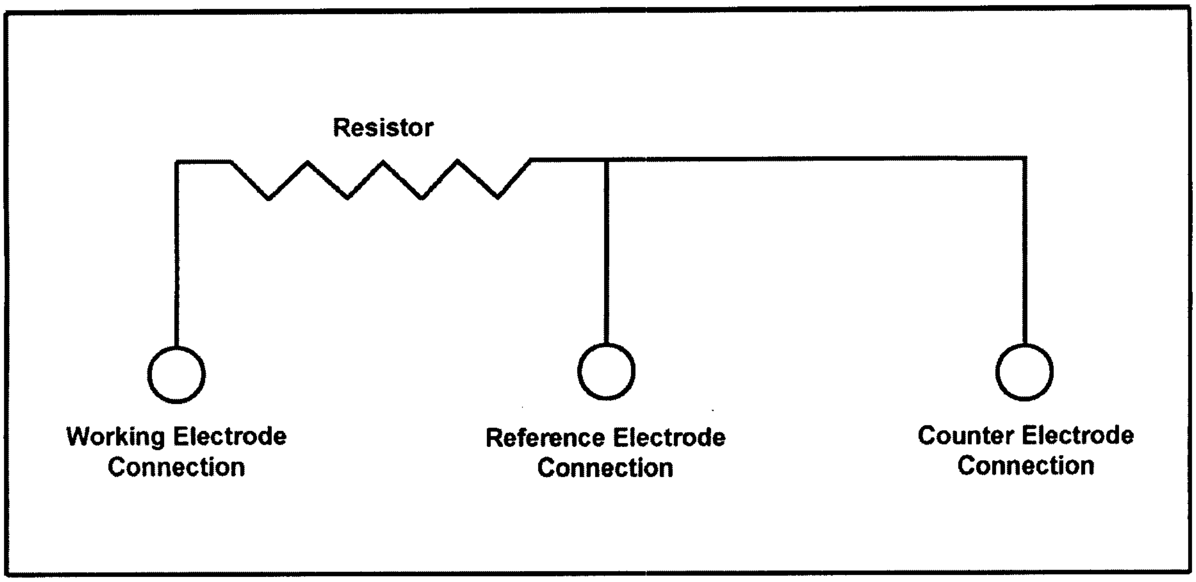

Figure 1. Schematic Diagram of the Resistor Box with the Precision Resistor. The Appropriate Connections to the Potentiostat Are Indicated.

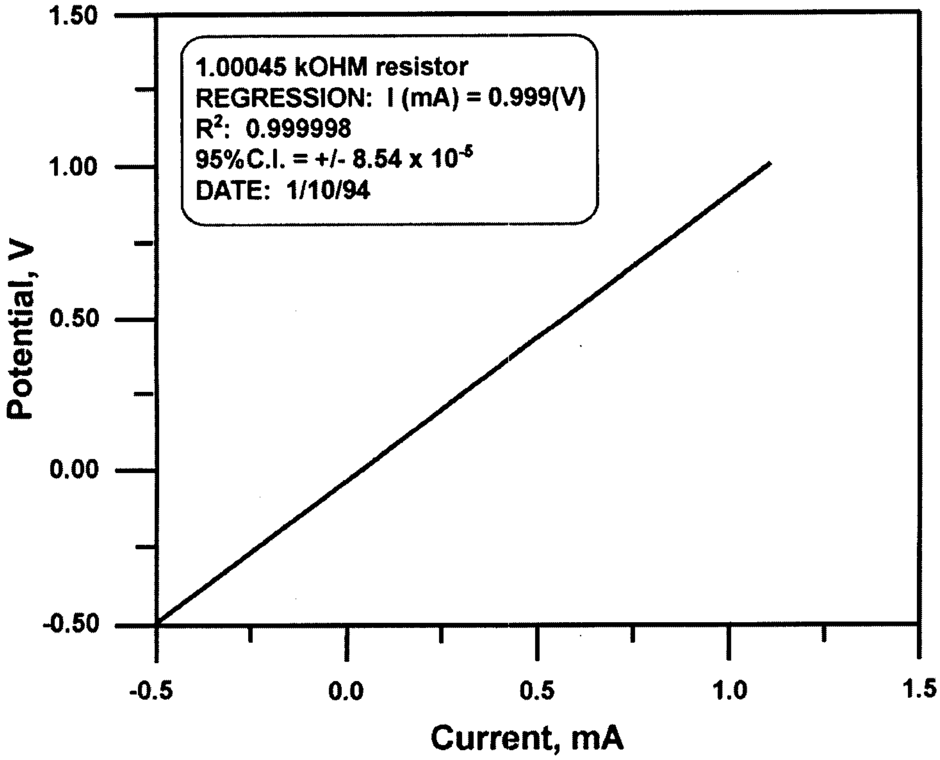

Figure 2. A Sample Potential-Current Plot for a 1.00045 KΩ Resistor Using the EG&G Model VersaStat Potentiostat and the EG&G Model M352 Data Acquisition Software

PERFORMANCE VERIFICATION	
VERIFIED BY:	Xihua He
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Figure 3. An Example of Performance Verification Label Showing the Verification Date and the Date the Next Scheduled Verification Should be Performed