CENTER FOR NUCLEAR WASTE REGULATORY ANALYSES

TECHNICAL OPERATING PROCEDURE

Proc. TOP-022
Revision 0

Title Procedure for Verification of the Performance of a Potentiostat and the Associated Data Acquisition Software

EFFECTIVITY

Revision 0 of this procedure became effective on 3/30/94. This procedure consists of the pages and changes listed below.

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1-6	0	3/30/94

Supercedes Procedure No. NONE

Approvals

Written by N. Eisenberg	Date 3/25/94	Technical Review	Date 3/30/94
Quality Assurance	Date 3/30/94	Cognizant Director	Date 3/30/94

TOP-022
PROCEDURE FOR VERIFICATION OF THE PERFORMANCE OF A POTENTIOSTAT AND THE ASSOCIATED DATA ACQUISITION SOFTWARE

1. PURPOSE AND APPLICABILITY

Potentiostats and galvanostats are used to maintain the potential and current respectively at a given electrode at constant, predetermined values. The same instrument can be used in either mode. Hence, reference is made in this document only to potentiostats, although the same procedures are applicable to a galvanostats also. Generally, the potentiostats are not calibrated directly, but the measurements of potential and current made by a potentiostat are verified independently by calibrated voltmeters and ammeters in accordance with Center Quality Assurance Manual (CQAM), Section 12. However, for some potentiostats that are 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 whole system of potentiostat and associated data acquisition software can be used to measure known parameters such as the resistance of a precision, pre-calibrated resistor. The purpose of the document is to provide such a procedure for the verification of the performance of a potentiostat and the associated data acquisition software. This procedure establishes controls required by CQAM, Section 12, "Control of Measuring and Test Equipment."

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 does not need to be followed, if provision is made to measure potential and current independently by calibrated voltmeters and ammeters. If calibrated voltmeters and ammeters are used to verify the measurements, these readings shall be recorded in the Scientific Laboratory Notebook in accordance with Quality Assurance Procedure QAP-001.

Other procedures for verifying the performance of the potentiostat and the data acquisition system for specific electrochemical tests have been described in TOP-008 and TOP-009. These procedures may be followed in addition to the procedure outlined in the present TOP. The procedures described in TOP-008 and TOP-009 are useful to verify systems including the potentiostat, electrochemical cell, and the electrodes. However, because the electrochemical response of an alloy to a given environment is dependent on a number of factors, the results of tests described in TOP-008 and TOP-009 can only be used to verify the system performance in an approximate manner. In contrast, the current procedure enables a precise, quantitative verification of the performance of the system.

2. RESPONSIBILITY

2.1 The cognizant Principal Investigators are responsible for the implementation of this procedure.

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

3. ABBREVIATIONS

The following abbreviations are used throughout this document:

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 $\pm 1\%$) 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 1μA to 1mA, then a 1kΩ can be used. The applied potentials in this case would range from 1mV to 1V which is within the capabilities of most potentiostats to apply and measure. If higher currents are anticipated, then a lower resistance must be used and vice versa.

4.2 It is preferred that the precision resistor 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 Section 12.4.1 of the CQAM and the appropriate calibration label shall be placed on the resistor box. The calibration interval will depend upon the laboratory conditions and can be determined initially by calibration in 6-month intervals. The calibration interval may be extended if the provisions of CQAM Section 12.4.1(2) are met.

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 increased or scanned automatically and the current-potential data can be collected. Care must be exercised not to exceed the wattage of the resistor.

4.5 The current-potential data can then be exported or manually input in appropriate analysis software to calculate the resistance (R = V/I) and various statistical measures associated with it. A sample measurement is shown in Figure 2. The measured resistance (along with its 95% 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 is considered to be significantly different from the specified tolerance of the resistor, then the resistor must be checked independently for possible damage. If damage of the resistor is found, step 4.4 shall be repeated with another calibrated resistor. If, on the other hand, no damage to the resistor is found, the system (either the data acquisition system or the potentiostat) shall be considered to be defective, and further corrective actions shall be undertaken before using that system.

4.6 The procedure outlined in steps 4.4 and 4.5 shall be performed prior to a series of potentiostatic experiments and the results shall be recorded in the appropriate Scientific Notebook.

4.7 In the case of 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, 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. However, it is preferable, but not necessary, to maintain this scan rate to be as close to the scan rate that will be used in subsequent electrochemical experiments. In electrochemical experiments, potential scan rate can be an important factor and hence will need an independent verification. Since the potential sweep is linear in these programs, the scan rate is calculated by dividing the total potential range by the time interval between the start and end of potential scan. The time at which the potential scan is started and the time at which the scan is stopped must be noted in the appropriate Scientific Notebook.

5. RECORDS

The files and the Scientific Notebooks containing the test data and the analyses are controlled as records in-process in accordance with CQAM Section 17.

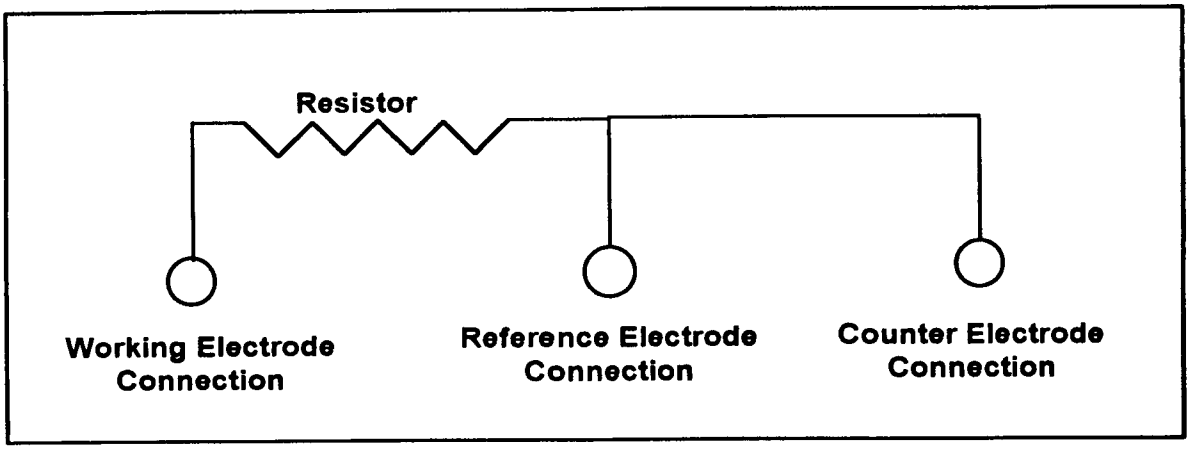

Figure 1. Schematic diagram of the resistor box with the precision resistor. The appropriate connections to the potentiostat are indicated.

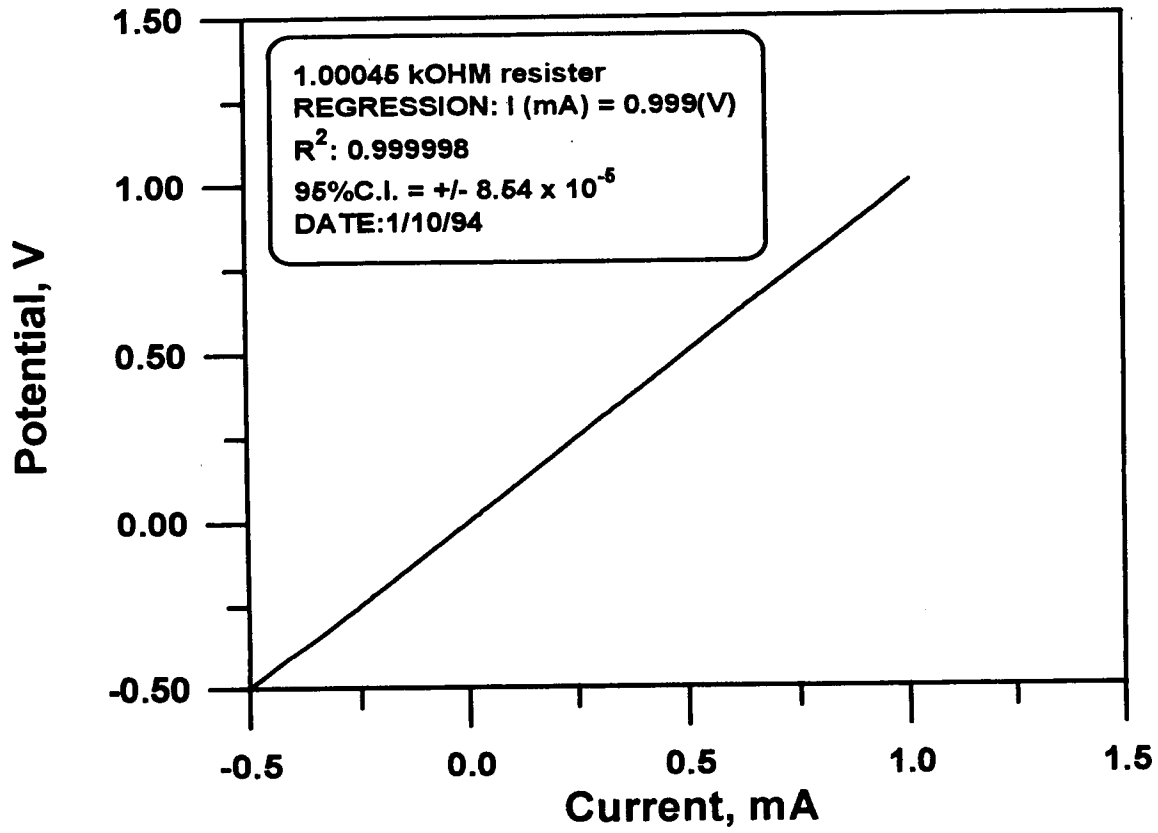

Figure 2. A sample potential-current plot for a 1.0044 KΩ resistor using the EG&G model VersaStat Potentiostat and the EG&G model M352 data acquisition software