Metrohm Autolab

Application Area: Fundamental

Cyclic Voltammetry and Electrochemical Impedance Spectroscopy measurements carried out with the Microcell HC setup, the TSC SW Closed and the TSC Battery cells

Keywords

Autolab Microcell HC, 100 Ω Resistor, TSC SW Closed, TSC Battery, cyclic voltammetry, CV, electrochemical impedance spectroscopy, EIS.

Introduction

The TSC SW closed and TSC battery cells are compact systems designed for measurements on air or moisture sensitive materials, such as those used in batteries. They offer well-controlled environments for the in-temperature testing of planar substrates like active materials, separators, or solid ion conducting materials. When getting started with the cells it may be helpful to first work with a model sample in order to optimize experimental methodologies before moving on to sensitive, and often precious, sample materials. To this end, reliable and easy to handle test resistors are available.

In this document, two testing procedures are explained. The first procedure is a potentiostatic cyclic voltammetry (CV), while the second is an electrochemical impedance spectroscopy (EIS).

Experimental Setup

In these measurements an Autolab Microcell HC and an Autolab PGSTAT204 have been used, Figure 1.

Figure 1 – The Autolab Microcell HC, on the left, and the Autolab PGSTAT204, on the right.

The TSC SW and TSC Battery measurement cells are shown in Figure 2-A and Figure 2-B, respectively.

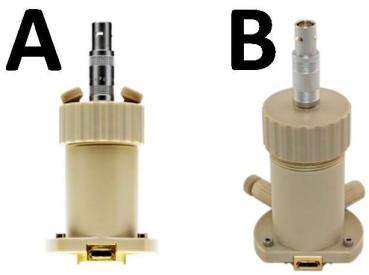

Figure 2 - The TSC SW (A) and the TSC Battery (B) cells.

In this study, four resistors, like the one shown in Figure 3, were used. Each resistor measures 1 cm in diameter and 4 mm in thickness. For more info on the resistor used, please contact Metrohm Autolab.

Figure 3 - one of the four test resistors. The number 100 refers to the approximated resistance, 100 Ω.

The resistance values of the four resistors are listed in Table 1. Each sample resistor is supplied with this information.

Table 1 - Resistance values of the four resistors.

Resistor number	R / Ω
1	99.947
2	99.979
3	99.968
4	99.947

The temperature has been kept constant during the measurement at 25 °C.

The procedures

Both the cells have been tested with CV and EIS, in a two electrode setup.

The CV consists in a potentiostatic staircase scan from -1 V to 1 V, starting from 0 V. The scan rate is set at 0.1 V/s, with 0.00244 V step potential. An example of a CV result is shown in Figure 4.

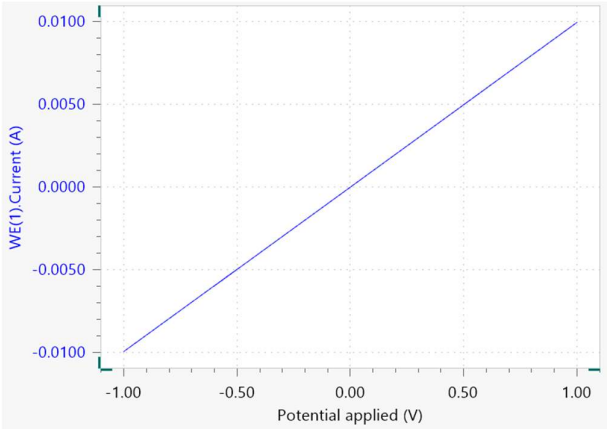

Figure 4 - An example of the CV plot for the 100 Ω resistor.

The current vs. potential plot (Figure 4) is fitted with a linear regression line *y*, Equation 1. The resistance is calculated as the inverse of the slope, Equation 2.

$$y = a + bx \quad (1)$$

$$R_{CV} = \frac{1}{b} \quad (2)$$

The potentiostatic EIS has been performed by applying a 100 mV RMS sinusoidal signal with a 0 V DC offset. The frequency range was from 1 kHz to 1 Hz with 10 frequencies per decade. An example of a result for the EIS measurement, in the form of a Nyquist plot, is shown in Figure 5.

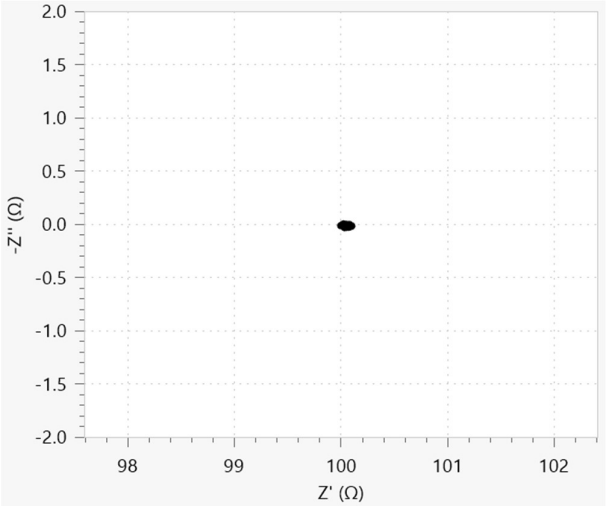

Figure 5 - An example of a Nyquist plot of a 100 Ω resistor.

The resistance has been calculated as the mean value of the real part of the impedance.

The results of both experiments are shown in Table 2.

Table 2 – Resistance values for the four resistors, measured with CV and EIS using the TSC SW and the TSC Battery cells.

Resistor number – test procedure	$R_{TSC\ SW}$ / Ω	$R_{TSC\ Battery}$ / Ω
1 – CV	101.24	100.49
1 – EIS	100.78	100.04
2 – CV	101.05	100.37
2 – EIS	100.58	99.37
3 – CV	100.61	101.62
3 – EIS	100.17	101.08
4 – CV	100.19	102.14
4 – EIS	100.63	101.69

In all the cases, the measured resistance values are slightly higher than the data sheet values tabulated in Table 1. The difference is attributed to the internal resistance of the TSC measurement cells, which is on the order of 1-5 Ω. Thus, the methods described herein can also be used to precisely determine the internal resistance of the measurement cell, if necessary.

Conclusions

This document outline two simple test measurements that can be carried out with the TSC SW and the TSC Battery cells using ~ 100 Ω test resistors. Cyclic voltammetry and electrochemical impedance spectroscopy procedures have been used. The resulting data shows very good agreement with the expected 100 Ω value. The difference between the measured and the tabulated values corresponds to the internal resistance of the cells.

Date

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For more information

Additional information about this application note and the associated NOVA software procedure is available from your local **Metrohm distributor**. Additional instrument specification information can be found at **www.metrohm.com/en/products/electrochemistry**.