Application Area: Batteries

Simple CV and EIS test measurements carried out with electrochemical cells for air or moisture sensitive measurements

Keywords

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

Introduction

The TSC SW and TSC battery cells are compact systems designed for measurement of air or moisture sensitive materials, such as those materials used in rechargeable batteries. These cells offer well-controlled environment for the in-temperature measurement of solid and gel like materials in contact with metal electrodes in planar geometry. For example, battery active materials, ionically conductive solid-state electrolytes and battery separators can be tested using these cells. In this experiment, standard resistors of 100 Ω are used in both cells to understand the cell effects, if any, on the measurements. It is recommended to test the experiment with a standard resistor before starting with a real sample in order to optimize the test methodology.

This application note details two testing procedures:

1. Potentiostatic cyclic voltammetry (CV)
2) Electrochemical impedance spectroscopy (EIS).

Experimental Setup

An Autolab Microcell HC and an Autolab PGSTAT204 (Figure 1) were used. Both TSC SW and TSC battery cells (Figure 2) were used in this experiment

Figure 1 – Autolab Microcell HC and the Autolab PGSTAT204

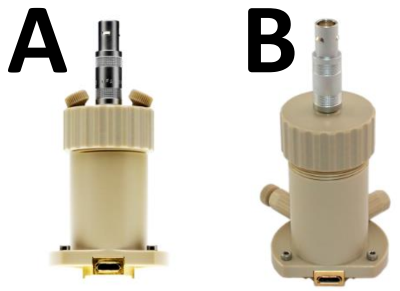

Figure 2 – (A) TSC SW; (B) TSC Battery.

The resistors used in this experiment were circular in shape with a diameter of 1 cm and a thickness of 4 mm (Figure 3).

Figure 3 - Standard resistor used in this study. The number 100 refers to the approximate resistance of 100 Ω.

The resistors were supplied with their accurate resistance values (Table 1).

Table 1 - Resistance values of the four resistors.

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

During the measurement the temperature was maintained at 25 °C.

The procedures

The TSC SW and TSC battery cells were both tested by cyclic voltammetry and EIS technique in a two electrode setup.

The CV consists of 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 as the step potential. Figure 4 shows an example of a CV result. As the CV test is conducted on a simple resistor, the response of the CV measurement is linear.

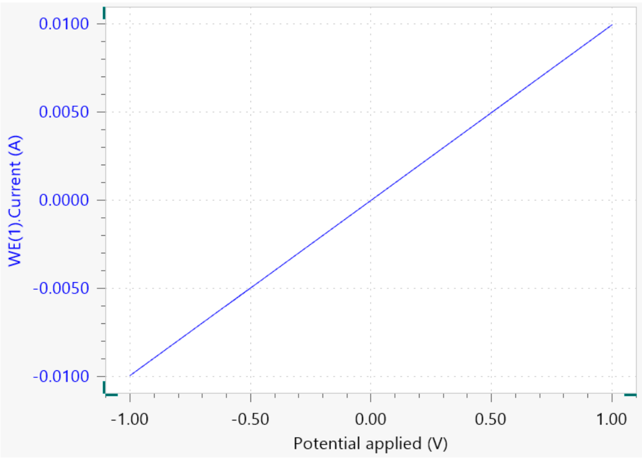

Figure 4 - An example of the CV plot for the 100 Ω resistor.

The relationship between current (I), resistance (R) and potential (V) is shown in Equation [1]:

$$R = V/I \quad [1]$$

The current vs. potential plot (Figure 4) is fitted with a linear regression line *y*, (Equation [2]). The resistance is calculated as the inverse of the slope, (Equation [3]).

$$y = a + bx \quad [2]$$

$$R_{CV} = \frac{1}{b} \quad [3]$$

The potentiostatic EIS was 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. Figure 5 shows the Nyquist plot for the 100 Ω resistor. Again, since a standard resistor was used for this measurement the Nyquist plot of the EIS measurement shows the similar response at different frequencies resulting scattered points around 100 Ω.

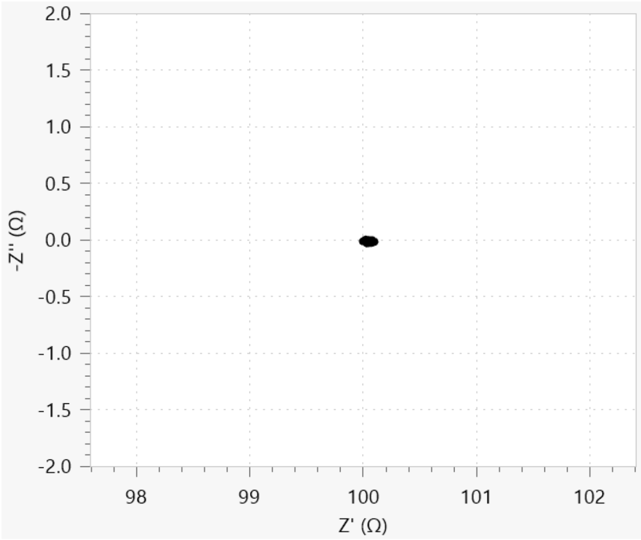

Figure 5 - An example of a Nyquist plot of a 100 Ω resistor.

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

The results of CV and EIS experiments for all four resistors 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 determine precisely the internal resistance of the measurement cell, if necessary.

Conclusions

This application note details 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**.