

LCM Chemical Test Procedure

Purpose

The purpose of this test is to perform a final examination of the LCM Low Current Module before it is shipped to the customer, and to provide the customer with typical data and output. From these outputs, the customer can verify that the instrument is working properly on arrival and can gain some experience with its operation.

The BAS 100B/W Electrochemical Workstation is used in this test; the CV-50W and CV-27 can also be used, and the modifications required are discussed at the end.

Test Solution

1 mM ferrocene with 0.1 M tetrabutylammonium hexafluorophosphate (TBAH) in acetonitrile.

Preparation of the Test Solution

1. Weigh 4.8 mg ferrocene and place in a 25 mL volumetric flask.
2. Weigh 0.57 g TEAP and add to the flask.
3. Add 12 mL acetonitrile and stir to dissolve. Add more acetonitrile to bring to volume.

Procedure

1. Remove the back panel of the C2 Cell Stand (which is a Faraday cage) and replace with the metal plate attached to the current-voltage transducer for the LCM.
2. Connect the 15 pin ribbon cable (EW-7522) to the transducer and the preamplifier (PA-1).
3. Connect the LEMO-LEMO cell lead (EW-7524) to the transducer and the BAS 100B/W.
4. Connect the 35 pin ribbon control cable (EW-7526) to the LCM and the BAS 100B/W.
5. Add the ferrocene solution to a cell vial and place inside the Faraday cage (a Faraday cage is need in order to exclude interference from external electric noise).
6. Inside the Faraday cage, attach the black lead to a platinum 10 μm microelectrode (MF-2005), the white lead to a Ag/AgCl reference electrode (MF-2063) and the red lead to a platinum wire auxiliary electrode (MW-1032). Ensure that these electrodes are placed in solution with no gas bubbles on their surfaces and close the door of the Faraday cage.

7. The LCM can be used in 2 modes - **Local** and **Remote** (use the switch to toggle between these modes). In the **Remote** mode, the **Gain**, **Filter**, **Multiplier** values and **Cell On/Off** are software-controlled, whereas in the **Local** mode, these functions are set using the switches on the front panel of the PA-1. The **Remote** mode can only be used with the BAS 100B/W, whereas the **Local** mode can be used with the BAS 100B/W, the CV-50W, and the CV-27.
8. The switches on the PA-1 should be set as follows: **Gain** = 1 nA/V, **Multiplier** = x1, **Filter** = 5.0 Hz, Toggle = **Remote** and **Cell Off**.
9. Open the BAS 100W software and switch on the BAS 100B potentiostat.
10. Open **Select Mode** dialog box from the **File** menu and select **Sweep Techniques - CV**.
11. Enter the **General Parameters** shown below, then click **Specific**.

12. Enter the **Specific Parameters** shown below, then click **OK**.

13. Select **Start Run** from the **Control** menu to start the experiment. The experimental data will be automatically displayed at the end of the experiment. The data should show the sigmoidal shape characteristic of steady state behavior. Print the data

14. Change the operation mode to OSWV (Square Wave Techniques - OSWV), and enter the **General Parameters** shown below. Click **Specific** to access the **Specific Parameters**, and enter the **Specific Parameters** shown below.

The image shows two dialog boxes for OSWV (Square Wave Techniques - OSWV) parameters. The first dialog box is titled "OSWV General Parameters" and contains the following fields: "Initial E:" with a value of 200 mV, "Final E:" with a value of 800 mV, and "Sensitivity" set to 1 nA/U. The second dialog box is titled "OSWV Specific Parameters" and contains the following fields: "Step E:" with a value of 4 mV, "S.W. Amplitude:" with a value of 25 mV, "S.W. Frequency:" with a value of 15 Hz, "Samples per Point" with radio buttons for 1, 16, and 256 (where 256 is selected), and "Quiet Time:" with a value of 12 sec. Both dialog boxes have "Cancel", "OK", and "Specific >>" or "General >>" buttons.

15. Run the experiment.
16. Select **Graph Options** from the **Graphics** menu, and activate **X Invert** and **Y Invert**. Print the data.
17. Change the operation mode to CV (see above), and enter the **General** and **Specific Parameters** shown below.

The image shows two dialog boxes for CV (Cyclic Voltammetry) parameters. The first dialog box is titled "CV General Parameters" and contains the following fields: "Initial E:" with a value of 200 mV, "High E:" with a value of 800 mV, "Low E:" with a value of 200 mV, "Scan Rate:" with a value of 20 mV/s, "Initial Direction" with radio buttons for Negative and Positive (where Positive is selected), "Number of Segments:" with a value of 2, and "Sensitivity" set to 100 uA/U. The second dialog box is titled "CV Specific Parameters" and contains the following fields: "Sample Interval:" with a value of 1 mV and "Quiet Time:" with a value of 12 sec. Both dialog boxes have "Cancel", "OK", and "Specific >>" or "General >>" buttons.

18. Change the toggle switches on the PA-1 to **LOCAL** and **ON**.
19. Run the experiment and print the data.

Modifications for using the LCM with the CV-50W and CV-27.

The CV-50W and CV-27 are not capable of remote control of the LCM (hence, the 35 pin ribbon accessories cable is not used), and can only be used with the LCM in the **LOCAL** mode.

When the LCM is connected to the CV-50W, the current-to-voltage transducer of the CV-50W becomes a gain stage of 1, 10 or 100 (depending on whether the **Sensitivity** is 100, 10 or 1 $\mu\text{A/V}$). The sensitivity of the connected system is determined by three factors; the **Gain** of the LCM, the **Multiplier** of the LCM and the **Sensitivity** of the CV-50W. The values to be used in this test are given above (#s 8, 17 and 18); for values appropriate under other conditions, see the User's Manual for the LCM. The current scale displayed by the CV-50W must be multiplied by a conversion factor to give the correct values:

$$\text{Actual Current} = \frac{(\text{current from CV-50W})(\text{LCM Gain})(\text{LCM Multiplier})}{100 \mu\text{A}}$$

When the LCM is connected to the CV-27, the CV-27 **Gain** should be to 0.1 mA/V for a gain of unity. The current output of the CV-27 is then determined by the **Gain** and **Multiplier** values set on the LCM.