EQCM-I

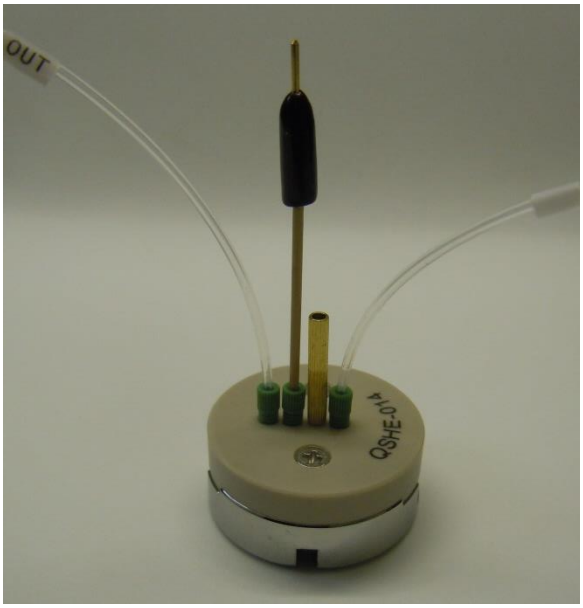
ELECTROCHEMICAL MODULE FOR

QUARTZ CRYSTAL MICROBALANCE WITH IMPEDANCE MEASUREMENT

&

BIOSENSE 3 SOFTWARE

Quick Start Guide



This guide will help you get started quickly, using the electrochemical module with the QCM-I and QCM-I Mini. It covers the additional software features relevant for electrochemical experiments using compatible Gamry potentiostats and the hardware setup. It should be used in conjunction with the *QCM-I Mini Quick Start Guide*. It is intended as a brief overview for new users or as a reminder for occasional users. More detailed information can be found in the *QCM-I Mini* and *QCM-I Operator's Manuals*.

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Disclaimer

The manufacturer accepts no liability arising from the use of this document. Please see the safety and warranty notices that are given at the start of the “QCM-I Operator’s Manual”. This document is a brief guide and is not intended to replace the Operator’s Manual.

1. Introduction

The QCM-I instruments can be used as an electrochemical QCM for combined electrochemical and microbalance measurements. These measurements enable the mass changes at the QCM sensing electrode surface to be monitored as a function of the electrochemical potential and the current and charge passed. This is shown schematically in Figure 1.

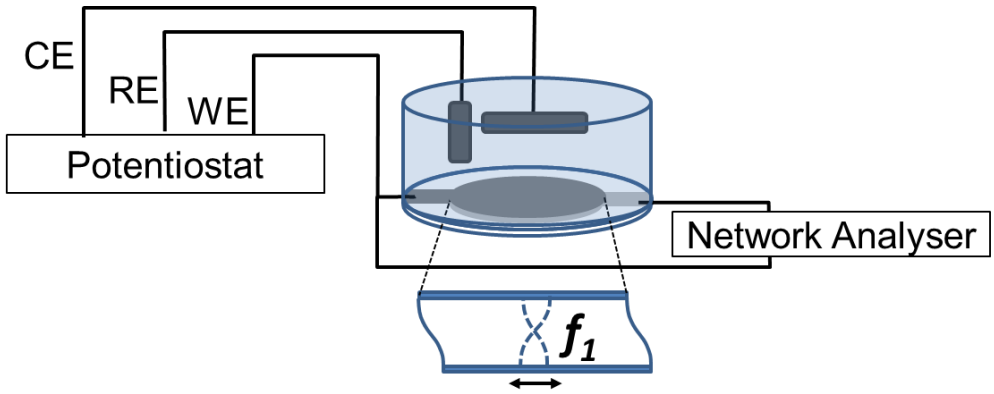

Figure 1: Schematic eQCM-I

This guide summarises the main features of the electrochemical module for the QCM-I and QCM-I Mini instruments with a Gamry or other compatible Potentiostat and the electrochemical flow cell or cuvette. It details the additional software components for electrochemistry in the BioSense EC 3 software, and should be used together with the *QCM-I Quick Start Guide*, or *Operator's manual*.

The QCM-I instruments can be used with other unsupported potentiostats. In this case the QCM-I measurement can either trigger or be triggered by the potentiostat to help synchronise the measurements. This is done through the Aux port (if fitted), see *Technical Note: Auxiliary In/Out Port.*

2. Installation

The BioSense 3 software and QCM-I instrument can be upgraded to include an electrochemical module, BioSense EC 3. This can be done during a factory service or remotely by a service engineer with support from the Instrument Administrator using TeamViewer. Please see the QCM-I Software Manual for information about TeamViewer.

First install the QCM-I or QCM-I Mini (see the User Manual or QCM-I Mini Quick Installation Guide). Next install the potentiostat. Connect it to the power supply and via USB to a USB port on the QCM-I computer, instrument or a USB Hub provided. If not preinstalled, the Manufacturer's service engineer will configure the BioSense software and USB ports for the potentiostat. The USB port used by the potentiostat is fixed and is labelled if preinstalled or should be labelled by the Administrator during remote installation.

After installation, when a compatible potentiostat is connected to the QCM-I computer, the BioSense 3 software will recognize it and the EC options will be displayed in the BioSense EC Software.

2.1 EARTH ISOLATION

The working sensor electrode (WE) is connected to earth through the QCM-I instrument. The potentiostat used should have a floating earth, or there may be issues caused by earth loops if the WE is connected to earth via both QCM-I and potentiostat. For potentiostats which are earthed/connected via a USB connection, isolation can be achieved by using a Galvanic Isolation Dongle.

3. Electrochemical Cell Connections

The electrical connections from the potentiostat to the eQCM-I Mini (are the same for the QCM-I) are shown in Figure 2. Connect the working electrode directly via a 2 mm banana plug connector on the underside of the Channel A thermal chamber, shown in more detail in Figure 3. Connect the counter and reference electrode cables with the linking wires to the 1 mm pins of the RE and CE on top of the electrochemical flow cell sensor holder.

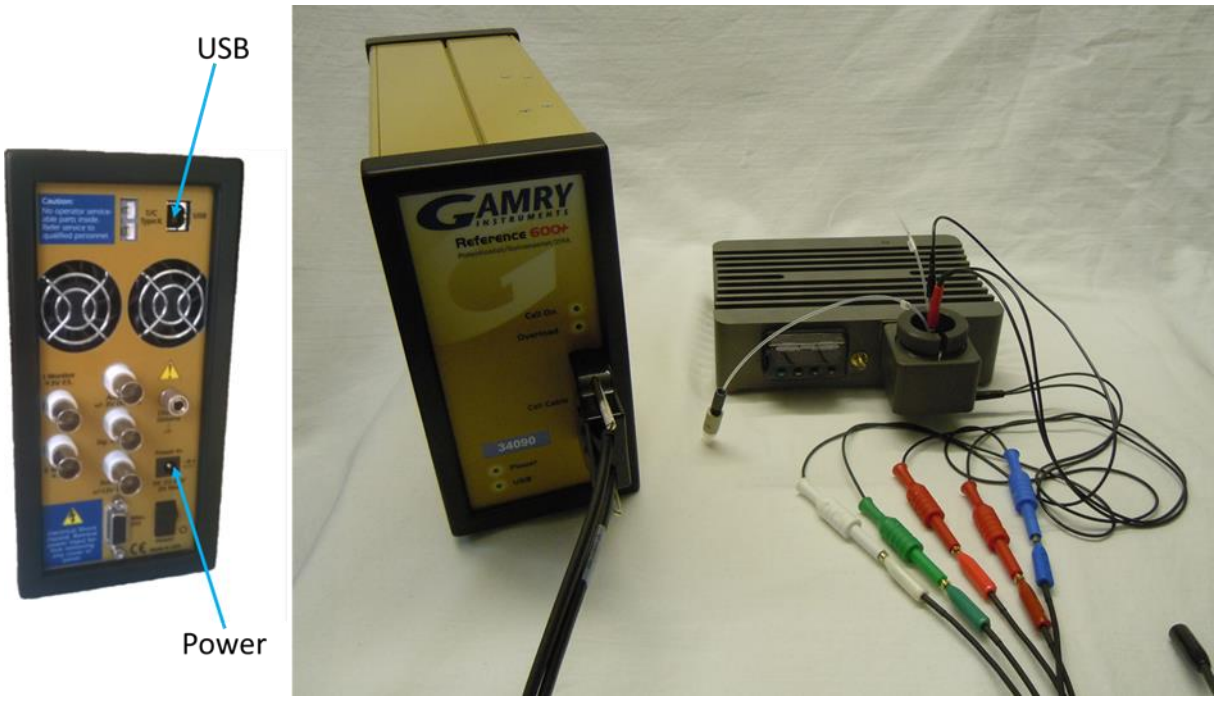

Figure 2: Gamry Potentiostat and QCM-I Mini, showing the Reference (white), counter (red), counter sense (orange) and working electrode (green) and working sense (blue) connections.

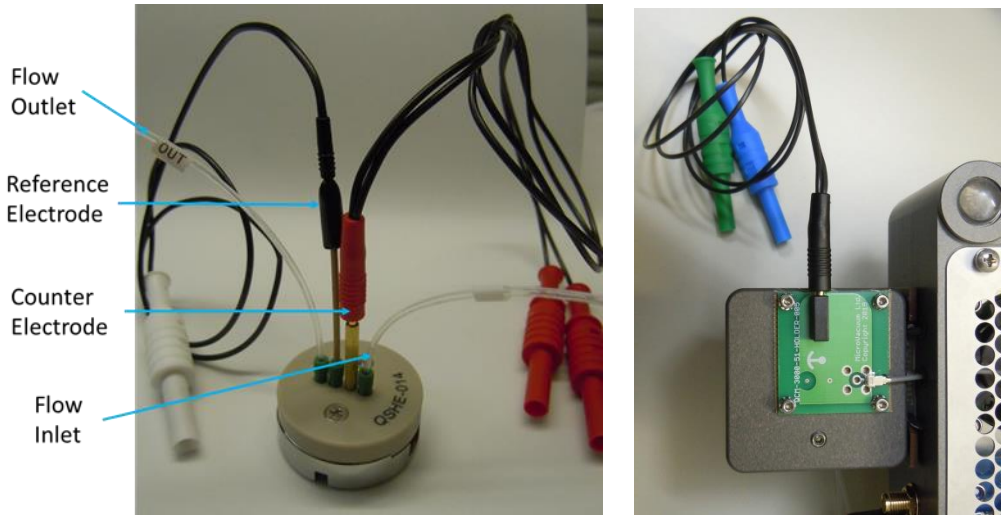

Figure 3: Electrochemical Flow-cell Sensor Holder, QSHE-014 (left) and underside of QCM-I Mini thermal chamber showing the working electrode banana connector from the potentiostat (right).

4. Electrochemical Flow-cell Sensor Holder

The electrochemical flow-cell is shown schematically in Figure 4. The Counter Electrode (CE) is a Pt disc[1] above the QCM sensor Working Electrode (WE). The Reference Electrode (RE) is a Ag/AgCl microelectrode situated in the exit flow path.

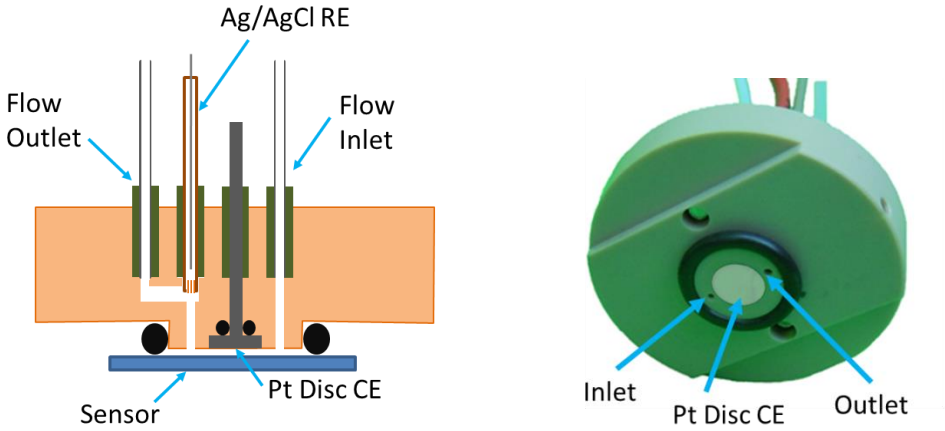

Figure 4: Schematic Diagram of the Electrochemical Flow Cell (left) and underside of flow cell lid (right)

The flow cell is assembled in the same way as the standard flow-cell, summarised below:

- (!) Ensure that the gold contact wires on the O-ring are aligned with the contact pads on the sensor holder base.
- (*) Orient the QCM Sensor so that the keyhole pattern of the back electrode matches the pattern on the PCB base.
- Tighten the screws alternately until fully closed.

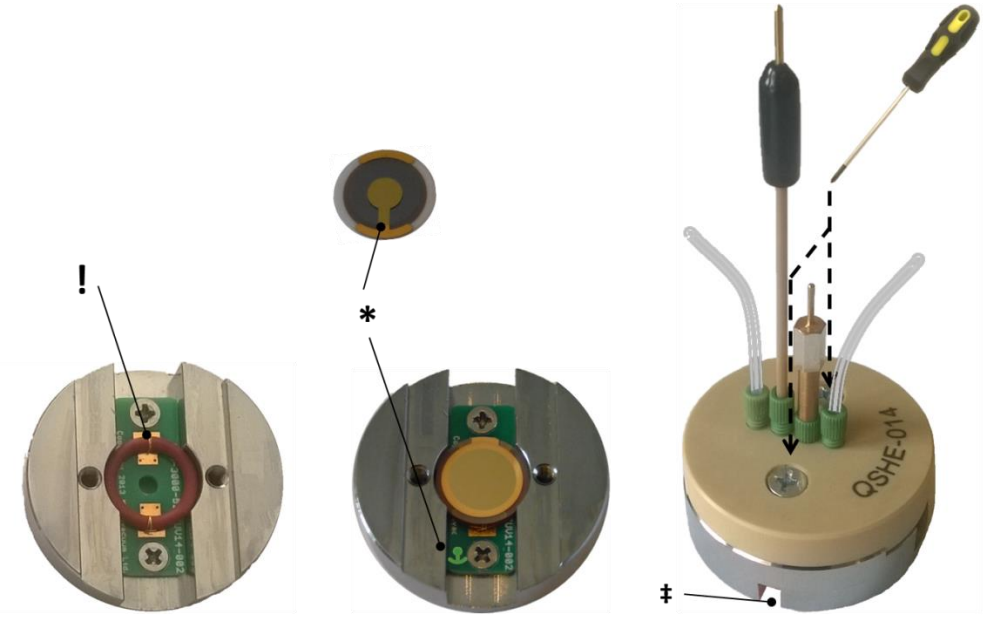

Figure 5: Electrochemistry Flow-Cell Assembly

[1] Other metals are available for the counter electrode, such as Cu or stainless steel.

The flow cell is also loaded into the QCM-I thermal chamber in the same way as for the standard flow cell. The notch on the base (‡) locates onto the pin in the base of the chamber and, as for the standard flow-cell, is held in place with the screw-in fixing ring. See Figure 2.

After fixing in place into the QCM-I thermal chamber, the flow cell can be filled with liquid and should be allowed to stabilise for ~ 10 minutes.

When filling the flow cell, take care to flush the cell completely to ensure that there are no bubbles trapped in the outlet, so that there is a good electrical contact to the reference electrode.

4.1 Ag/AgCl REFERENCE ELECTRODE

The reference electrode is shipped in the same vial as we receive it form the manufacturer. According to the manufacturer the vial contains "…diluted sulfuric acid, 0.05-0.1 M (exact concentration not needed) with a few drops of KCl solution…". The ferrules at the base of the reference electrode are tightened in the correct position so they fit into the downstream fluidic path of the EC flow cell. However, the ferrules should not be tightened too firmly, because it can damage the electrode tubing. This means that the distance of the ferrules to the tip of the electrode should be watched carefully, and care should be made not to pull the electrode out through the rubber seal of the vial cap.

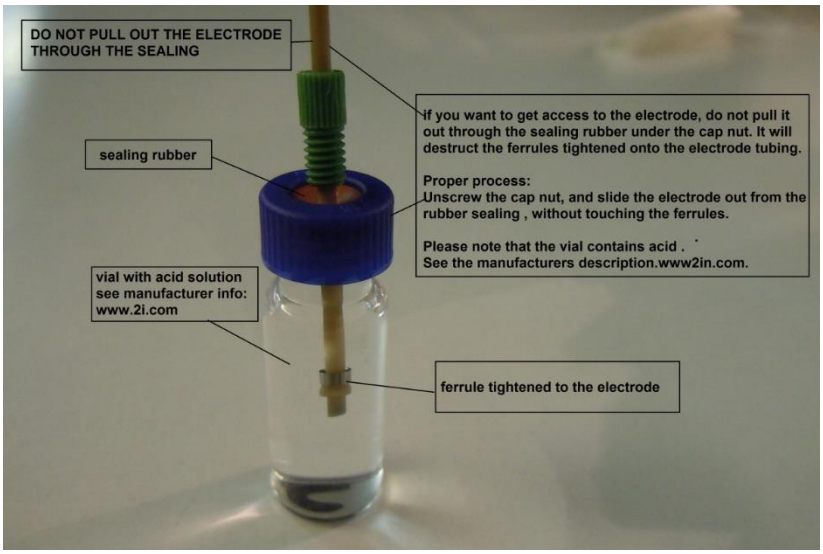

Figure 6: Reference electrode in original vial shipping container

In Figure 7 you can see a cut in the rubber seal, so when the reference electrode is removed from the vial, this can be done by first unscrewing the cap and then sliding the rubber seal off perpendicular to the electrode, without disturbing the position of the ferrules.

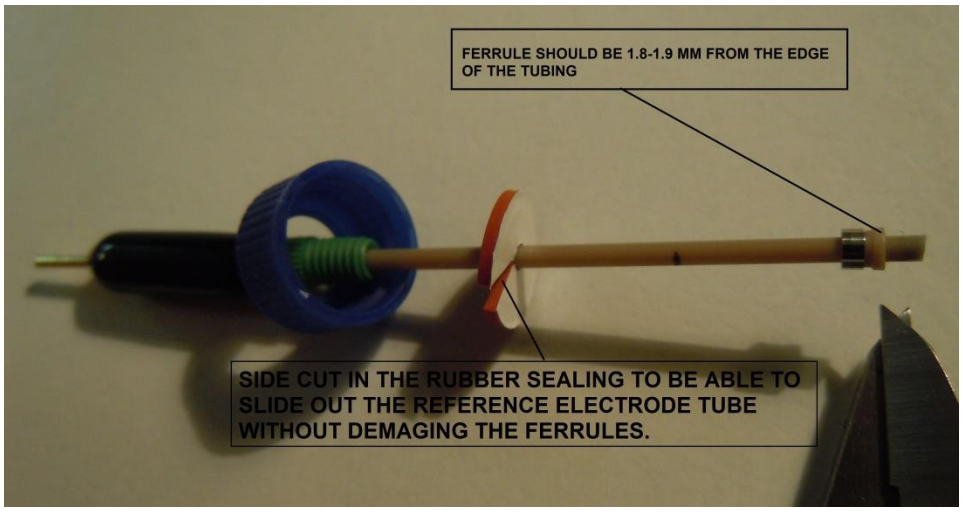

Figure 7: Reference electrode removed from the vial shipping container

5. Electrochemical Cuvette (QSHE-Open)

The electrochemical cuvette is shown in Figure 6. The cuvette has a volume of 5 ml. The Counter Electrode (CE) is a coiled Pt wire inserted in the cuvette lid above the QCM sensor Working Electrode (WE). The Reference Electrode (RE) is a Ag/AgCl electrode also inserted through the cuvette lid. The lid also has in and outlet ports for bubbling gas if required.

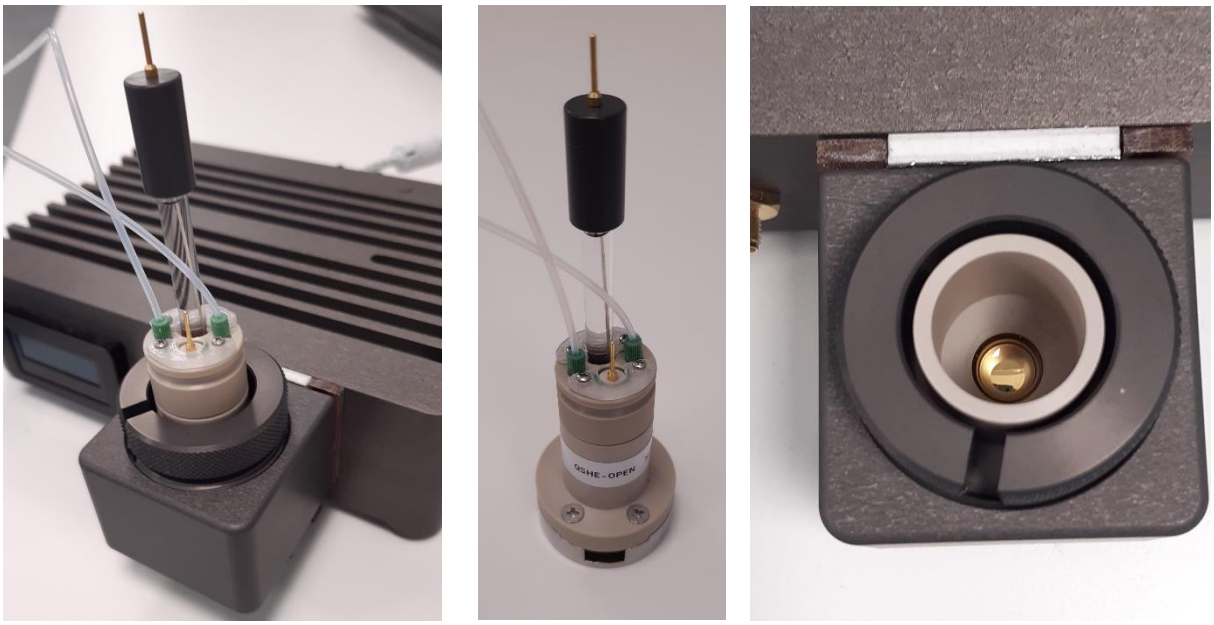

Figure 6: Open Electrochemical Cuvette Cell

The counter and reference electrodes are fixed in place with an O-ring and a small plastic insert, which is screwed in place, as shown in Figure 7. The reference electrode can be positioned just above the edge of the working electrode on the sensor surface. The height of the reference electrode should be adjusted when the cuvette is outside of the QCM-I instrument and detached from the metal base (i.e. without a sensor loaded) to ensure that it does not press onto the sensor surface when assembled.

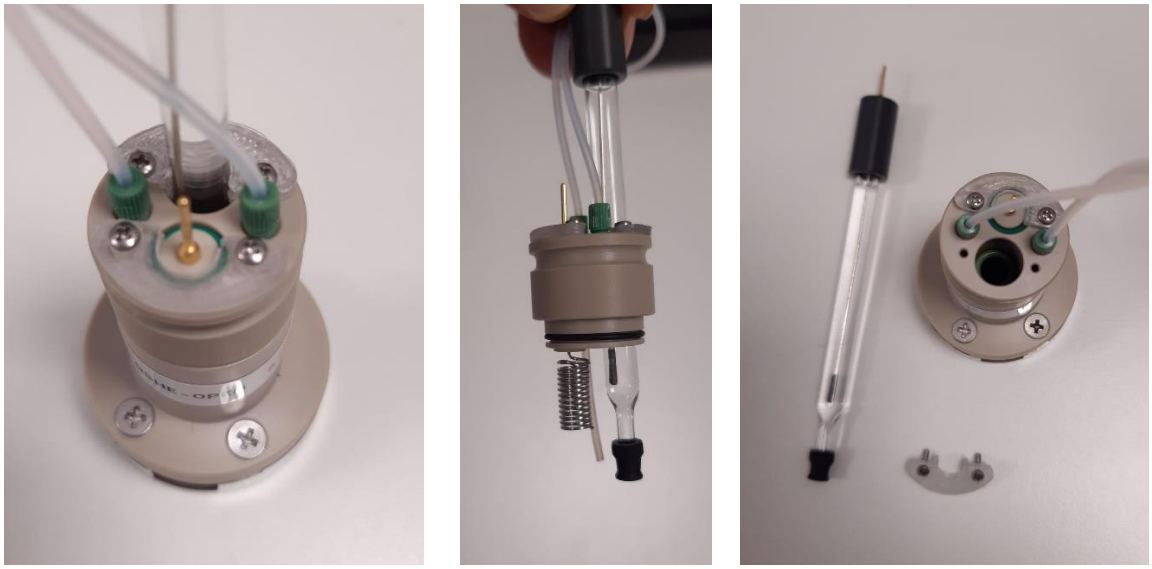

Figure 7: Fixing and removal of electrodes from cuvette lid.

6. BioSense EC Software

The BioSense EC software includes electrochemical measurement types as well as those in the standard BioSense for QCM-I software. The software can control a Gamry potentiostat for simple Cyclic Voltammetry, Chronoamperometry and Chronopotentiometry experiments either alone, or at the same time as QCM-I measurements. The EC setup and display windows are summarised here.

6.1 MEASUREMENT SETUP

To perform an EC measurement, click on the ( ) icon in the toolbar or select the “Setup and Start Measurement” option in the “Measurement Control” menu. This opens the Measurement Setup window shown in Figure 8. The additional QCM(t)-EC and EC measurement types are now available.

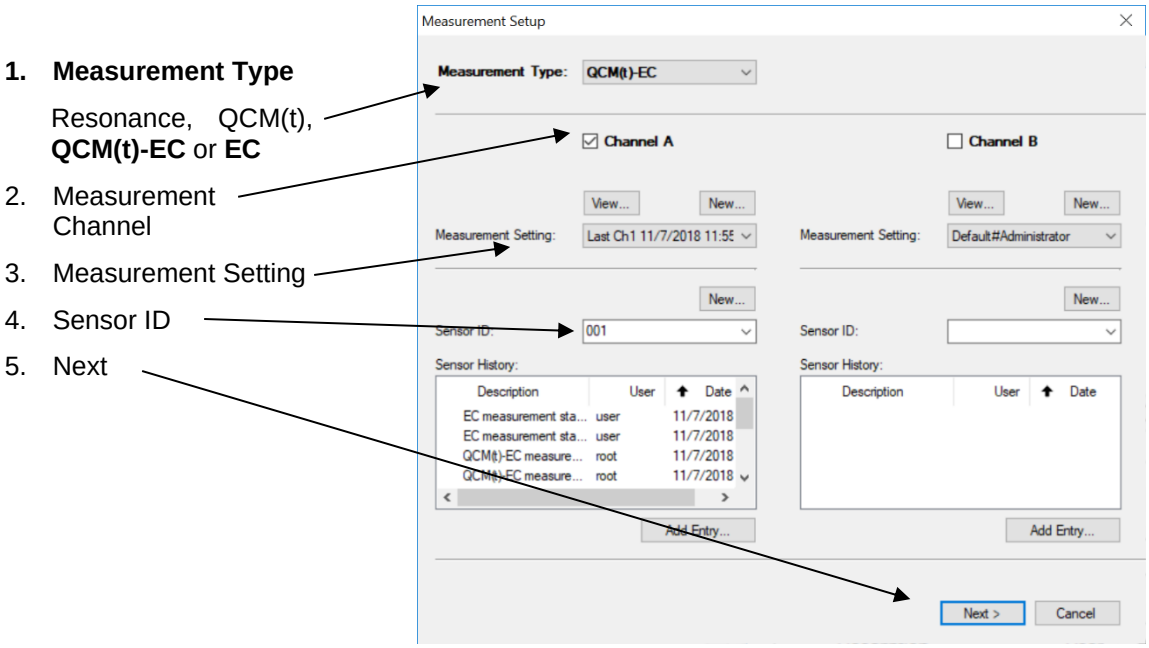

Figure 8: Measurement Setup Window

Measurement Type

- EC measurement: Controls the potentiostat for an electrochemical measurement. This supports a single electrochemical cell. This is assumed to be on Channel A.
- QCM(t)-EC measurement: Controls both the potentiostat and the QCM-I instrument. The EC measurement is made on one channel, but the QCM-I measurement can be made on multiple channels.

After completing the Measurement setup window, and the QCM(t) Setup window (if QCM(t)-EC was selected), the EC Setup window is shown.

6.2 EC SETUP WINDOW

The EC Setup window is used to set the parameters and EC mode for the measurement. As well as from the “Setup and Start Measurement” tab, this can also be accessed from the Settings in the main BioSense menu.

The settings section changes according to the EC Mode selected. Figure 9 shows the versions where Cyclic Voltammetry or Chronoamperometry were selected.

For Chronoamperometry or potentiometry, the EC cell can either be switched on at the start of the experiment, or left at open circuit at the start of the measurement so it can be changed later in the course of the experiment.

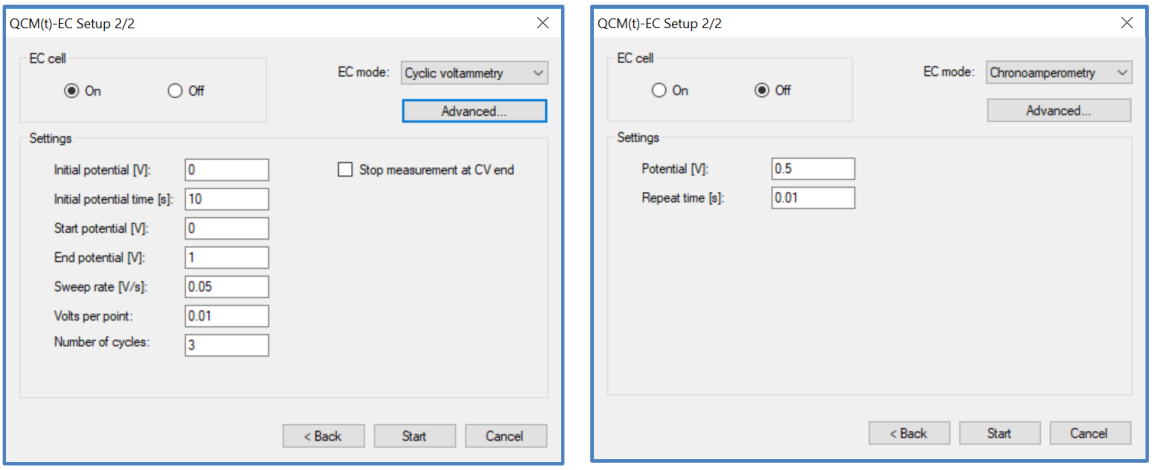

Figure 9: QCM-I Setup Window: Cyclic Voltammetry (left) and Chronoamperometry (right)

Electrochemical Measurement Modes

- Cyclic Voltammetry: In the Cyclic Voltammetry setup screen the CV parameters are chosen. The QCM measurement can also be automatically stopped at the end of the CV. When the CV measurement is started the settings can no longer be modified.
- Chronoamperometry: In this setup screen the initial measurement parameters are setup, but these can also be modified during the measurement.
- Chronopotentiometry: In this setup screen the initial measurement parameters are setup, but these can also be modified during the measurement.

A range of more advanced settings can be selected with the “Advanced…” button. This is shown in Figure 10 for Cyclic voltammetry and Chronoamperometry. The electrochemical setup is described in more detail in the QCM-I Software Manual.

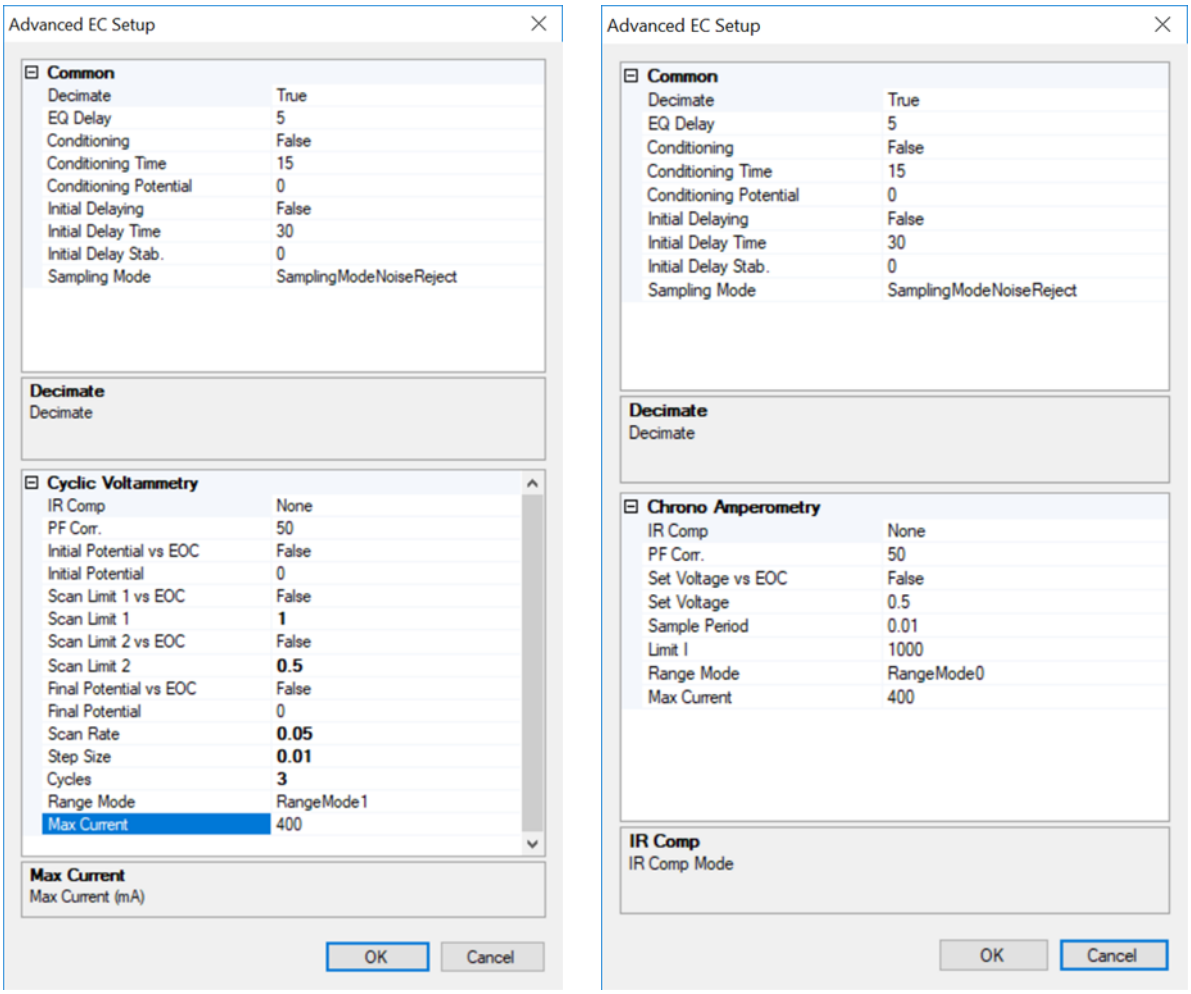

Figure 10: Advanced settings for Cyclic Voltammetry and Chronoamperometry

Once the start button is pressed the measurement starts. For EC measurements this occurs immediately, for QCM(t)-EC measurements it starts after the QCM frequency measurement has been initialised and the QCM-I has also started measuring.

6.3 QCM(t)-EC AND EC MEASUREMENT DISPLAY

Once the measurement is started the measurement display window is shown, Figure 11. For EC type measurements the graph displays the Current and the Potential vs. time on the two Y- axes. For QCM(t)-EC measurements, by default, Graph 1 shows the Frequency data from channel A and Graph 2 shows the Current and the Potential vs. time on the two Y- axes.

For both EC measurement types the Navigator Panel features an additional *EC Settings* tab. For QCM(t)-EC measurements a *QCM(t)-EC View Settings* tab is also present to select the displayed parameters.

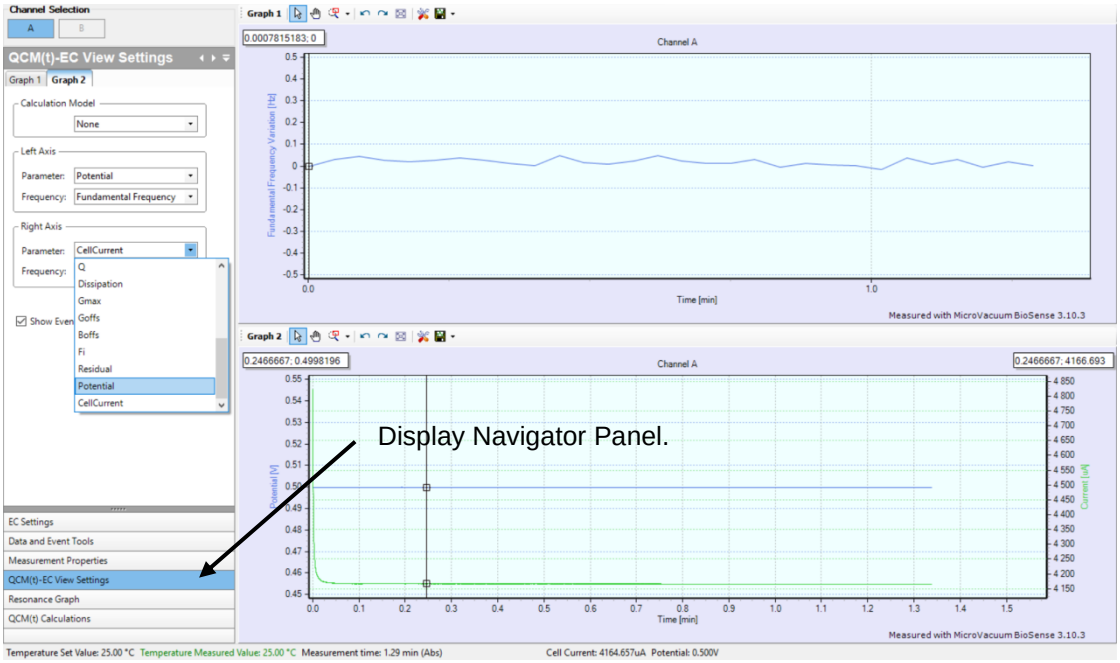

Figure 11: Measurement Display Window

6.4 DISPLAY NAVIGATOR PANEL

EC Settings

When an EC measurement has been chosen (EC or QCM(t)-EC measurement types), the EC settings panel is visible, see Figure 12. It shows the measurement mode. During an EC Chronopotentiometry or Chronoamperometry measurement, it also allows the Settings to be modified. So the cell can be switched on or off, and the potential and repeat time can be changed. Clicking on the Modify EC Settings…. Button opens the QCM(t)-EC Setup window. During a Cyclic Voltammetry measurement however, the settings cannot be changed.

QCM(t)-EC View Settings

This allows the different data to be selected and plotted on the graphs, see Figure 11. In addition to the QCM parameters the EC data that can be selected is: Current, Potential, CV and Saved CV options. *CV* and *Saved CV* plot a conventional cyclic voltammogram (i.e. Current vs. Potential).

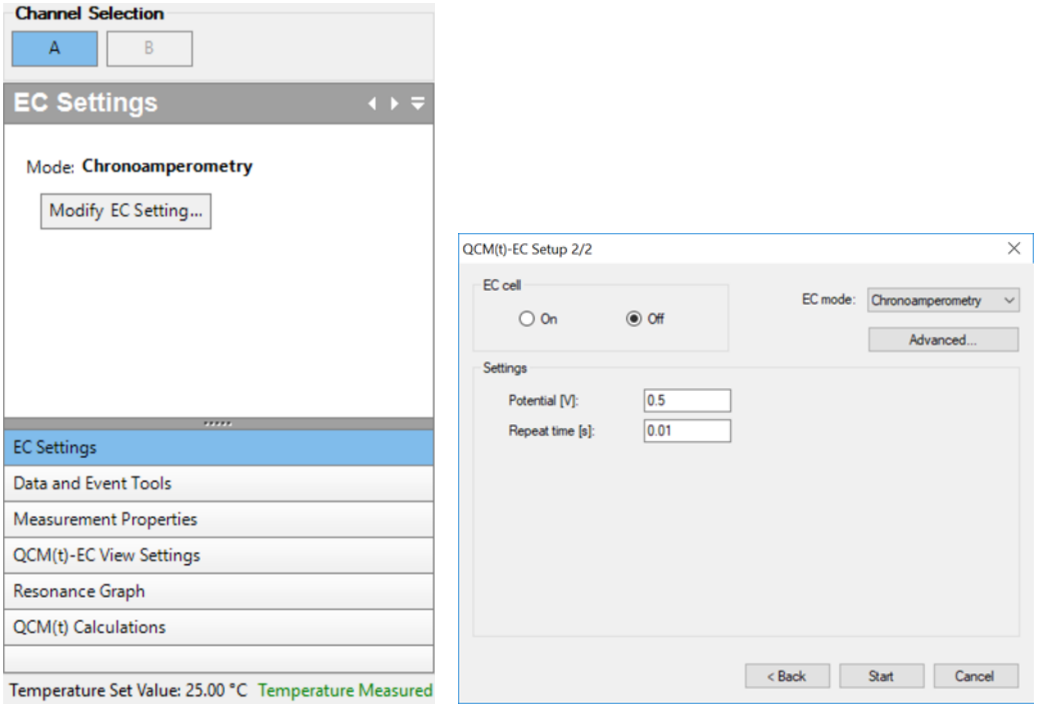

Figure 12: EC Settings Panel (left) and QCM(t)-EC setup panel (right)

6.5 DATA EXPORT

After stopping and saving a measurement, in addition to the different ways of exporting or outputting data described in the *Quick Start Guide* and *QCM-I Software Manual*, EC and QCM(t)-EC data can also be exported for analysis in Gamry software:

File - Gamry export

The open EC or QCM(t)-EC Measurement is exported in the *.DTA format for analysis with Gamry Echem Analyst™ software.

7. Further Information

A more detailed description of the experimental procedures and software is provided in the QCM-I Operator's Manual.

Spare parts and consumables for the Electrochemistry Module can be ordered, please contact your supplier or Semilab.

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