

Reference Electrode Overview and Care

Gamry sells the Saturated Calomel (930-00003) and Silver-Silver Chloride (930-00015) Reference Electrodes. Proper care must be taken to ensure that your electrode continues to take accurate measurements. The tip of the electrode is vycor frit or electro porous KT glass (opaque white). The frit allows ionic transport into the electrode. In order to operate properly the frit must always be kept wet with electrolyte.

Filling Solution

Both electrodes are filled with Saturated KCl. To fill the electrode, slide the rubber grommet located at the top of the electrode down. This will reveal a small filling port where saturated KCl can be added.

Storage

Both Ag/AgCl and SCE electrodes should be stored in just slightly less than saturated KCl solution. Keeping the solution just below saturation allows enough ionic flow to prevent salt crystals from forming in the glass pores. The frit must be kept wet at all times to ensure the impedance of the electrode remains low.

Checking the Impedance of the Reference Electrode

It's very important for optimum potentiostat performance that the impedance of the Reference Electrode in your cell is low! A high impedance Reference Electrode will cause problems that range from simple overloads to potentiostat oscillation.

The impedance of your Reference Electrode should be less than 1 kohm. Impedance higher than 1 kohm is not good and impedance higher than 5 kohm is unacceptable and must be corrected.

Testing Procedure

1. Partially fill a beaker with electrolyte. If you normally use a Luggin capillary, the concentration of this electrolyte should be approximately the same as that of your test solution.
2. Immerse the tip of your Reference Electrode into the solution. If you will be using a Luggin capillary, place the tip of the Luggin capillary in the solution and place your reference in the Luggin. Make sure you have an unbroken electrolyte path from the tip of the Reference to the tip of the Luggin capillary.

3. Add a high surface area platinum wire or graphite rod counter electrode to the solution.
4. Connect the Reference Electrode to the Working (green) and Working Sense (blue) leads of your potentiostat. Connect the graphite rod to the Reference (white) and Counter (red) electrode leads
5. Open Gamry Framework and go to Experiment>Utilities>Measure Reference Electrode Impedance. Framework will now run an EIS curve measuring the impedance of the reference electrode. Once it is finished it will tell you if the impedance is acceptable, and also gives you the measured impedance and phase angle of your electrode.
6. Framework will now run an EIS curve measuring the impedance of the reference electrode. Once it is finished it will tell you if the impedance is acceptable, and also give you the measured impedance and phase angle of your electrode.
7. If the program tells you the impedance is acceptable then you may continue with your experiments. If the impedance is out of range, one possible solution is to replace the frit. Please see the following section for details on this section.

Replacing a Reference Electrode Frit

A Vycor frit should be replaced if the impedance of the reference electrode is out of specification (see above), or if it has been allowed to dry out, has been cracked or chipped, or if it has become discolored. A frit cannot be reused. You will need a new vycor frit and a piece of shrink-wrap teflon tubing before beginning this process.

1. Remove the old frit by cutting the heat-shrink Teflon. A sharp knife or razor blade is ideal
2. Place a single Vycor frit inside the piece of heat-shrink Teflon tubing supplied. See below



3. Slide the Teflon over the end of the reference electrode and be sure that the Vycor touches the end of the glass tube. Warm the Teflon with an electric "heat gun" until the Teflon shrinks tightly around the Vycor and the end of the reference electrode. Do not hold the heat gun too close or the Teflon will melt. Rotate the reference electrode to evenly heat all sides of the Teflon. A suitable

heat gun can be purchased from your local laboratory supply house. A common "paint stripper" heat gun can also be obtained locally. See below.



4. Trim the excess Teflon so that it is even with the end of the Vycor. If you do not, the excess Teflon tube can trap a gas bubble and isolate your reference electrode from the solution.



5. Refill the interior electrode body with saturated KCl. Allow the electrode to soak in saturated KCl for at least 1 hour before use to ensure the new frit is completely wet.
6. Finally, measure the impedance of the reference electrode to make sure the frit replacement and re-wetting process worked correctly. See the above section for details on this.

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