

Non-aqueous Reference Electrode

Kit Contents:

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|------------------------------|-------|
| Glass body | 1 |
| Teflon cap w/ silver wire | 1 |
| AgNO ₃ | 1 g |
| 1/8" Vycor plug | 5 pcs |
| 1/2" Teflon heat shrink tube | 6 pcs |
| 600 grit carbide pad | 1 |
| Information sheet | 1 |

The Silver/Silver Ion Electrode

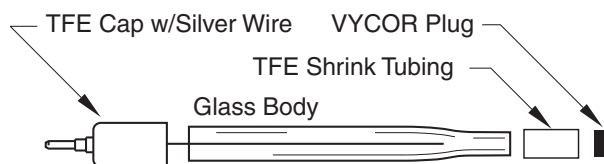
The Ag/Ag⁺ reference electrode is commonly employed in non-aqueous electrochemical studies. Aqueous reference electrodes, e.g., calomel and Ag/AgCl, have several drawbacks. These include liquid junction potentials, contamination of solvent solution by water, and precipitates at the reference electrode frit which cause noise and even failure of the reference electrode.

The Ag/Ag⁺ reference electrode is made by placing a clean silver wire into an electrolyte containing silver ion. Often the electrolyte in the reference compartment is the same as the solution electrolyte. Typical salts are tetrabutylammonium perchlorate (TBAP), tetraethylammonium perchlorate (TEAP), tetrabutylammonium hexafluorophosphate and tetrabutylammonium fluoroborate at approximately 0.1 M concentration. Typical polar or dipolar aprotic solvents are acetonitrile (ACN), dimethylformamide (DMF), dimethylsulfoxide (DMSO) and propylene carbonate (PC). In cases where a salt is not sufficiently soluble in the solvent being used, e.g., methylene chloride, tetrahydrofuran, etc., the recommended reference electrode filling solution uses a more polar, yet miscible solvent like acetonitrile.

Silver ion is provided by dissolving 0.001 to 0.01 M AgNO₃, AgClO₄ or other silver salt in the chosen electrolyte. The silver ion electrolyte is usually changed daily although the frequency depends on the experiments performed.

For additional information see:

Sawyer, D.T., Sobkowiak, A., Roberts J.L. Jr., *Experimental Electrochemistry for Chemists*, 2nd Ed. Wiley Interscience, New York, 1995, pp 201-203.



Assembly Instructions

Fill the glass body with electrolyte (e.g., 0.1 M TBAP/ACN). Place glass body/vycor assembly into electrolyte. This will wet the vycor after an hour or so. Note, gas bubbles will form on top of the vycor as it wets. These are removed by tapping the glass body with your finger. Covering the whole container will prevent or slow evaporation of the solvent.

A clean silver surface is obtained by a light polish on 600 grit sandpaper. The wire is then rinsed with distilled water followed by an acetone rinse. The wire should be allowed to dry before use.

When the vycor is wetted, remove the solvent electrolyte from the glass body assembly. Add a known concentration of silver ion solution (e.g., 0.01 M AgNO₃, 0.1 M TBAP/CH₃CN) to ~1/2" from top of tube. Place the cap and silver wire assembly into the glass body assembly. Air bubbles in the narrow part of the glass should be removed by tapping the electrode body. The reference electrode is ready to use.

For storage, the internal silver ion electrolyte solution is removed and replaced with electrolyte alone. This electrolyte filled, fritted glass body is then placed in a container of electrolyte and covered. Doing this keeps the vycor frit material wetted and also cleans it of foreign material, e.g., precipitates, that may have been deposited during use. **Caution:** Do not allow the vycor tip to become completely dry. The salt crystallizing inside the vycor will cause it to break and start leaking.

Replacement of Vycor Tip

Heat shrink 1/2" length of teflon tubing onto glass body tip and over a 1/8" length vycor plug. Trim off excess teflon tubing.