

How to Guide



Subject: RDE Tip, Micro, and Milli Electrode Care

OVERVIEW

The cleaning and polishing procedures described in this section are designed to help you keep your PAR RDE Electrodes RDE004, RDE005, RDE008, RDE0071, RDE0072, and RDE0073; Microelectrodes G0224, G0225 and G0226; and Millielectrodes G0227, G0228, and G0229 in the best possible condition. The procedures apply to the two classes of electrodes we supply, glassy carbon and solid metal. To ensure that you have the ability to continue your work while cleaning the electrode, we recommend that you stock an extra working electrode of the same type.

Glassy Carbon Electrode Care

Unlike many metal electrode materials, glassy carbon is porous and vulnerable to chemical penetration of up to a few microns into the electrode surface. When the surface becomes fouled, lower currents are observed for a given analyte concentration. This decreases sensitivity and gives rise to poor reproducibility. Mechanical polishing removes the effects of this penetration by creating a fresh carbon surface. However polishing also makes it necessary to equilibrate for long periods before the electrode can be used for analytical work. Therefore, although polishing is often used to renew glassy carbon electrodes, you should first try to use a less aggressive technique.

There are several effective cleaning techniques available for glassy carbon. Three techniques are described, two involving chemical procedures and one involving an electrochemical procedure. The procedures are outlined below. For glassy carbon, cleaning frequency depends on the type of sample being analyzed, the potential applied and the electrolyte composition. We suggest you take a conservative approach to cleaning the electrodes and choose the procedure that causes the least amount of physical damage to the surface of the working electrode. If the results are not as expected from one procedure, then try a more aggressive one. The procedures are presented in order of aggressiveness, beginning with the least aggressive.

Metal Electrode Care

Mechanical polishing procedures, which should be used conservatively on glassy carbon electrodes, can be used more freely on solid metal electrodes therefore we do not recommend the chemical procedures described below for metal electrodes. To maintain a high degree of sensitivity and repeatability a microelectrode must be polished periodically. The interval between polishing depends upon the experiments being run and the accuracy required. For best results the

surface should be repolished after each run and it should be polished using a consistent, repeatable technique.

Cleaning Procedures

The procedures are presented in order of aggressiveness, beginning with the least aggressive. Remember the more aggressive procedures make it necessary to equilibrate for a longer period before doing analytical work.

Methanol (Methyl alcohol) Cleaning Procedure (Least Aggressive)

CAUTION! Methanol is toxic by ingestion and in contact with the skin. It can also be damaging if splashed into the eyes. Use caution in handling it and wear the appropriate protective goggles, gloves and clothing.

1. Remove the glassy carbon electrode from the cell.
2. Rinse it in distilled water. This will remove any electrolyte salts from the electrode surface.
3. Apply several drops of reagent grade methanol to the surface of the electrode.
4. Dry the surface off with an absorbent wipe or lens tissue. This procedure may clean the electrode enough to restore a suitable response.
5. Re-insert the electrode into the cell.

Electrochemical Cleaning Procedure (Glassy Carbon)

When potentials over 800mV are used in an analysis some organic compounds can deposit onto the electrode surface. Applying a high negative potential can often desorb these compounds from the electrode surface and reduce any oxides. Applying a subsequent high positive potential will then recondition the electrode. Use this procedure to remove the compounds from the electrode surface.

1. Place the electrode in the cell and fill the cell with electrolyte (see Note below).
2. Connect the Counter, Reference and Working (as well as Sense, if available) lead clips to their appropriate electrodes.
3. Apply a -500 mV potential to the electrode for 1 minute (desorption/reduction).
4. Apply a positive +1200mV potential for 10 minutes (reconditioning).
5. Run your normal experiment, applying the potential appropriate for your analysis.

Note: Suitable electrolytes are:

- Ammonium citrate buffer (made by dissolving 42.5 mg of citric acid in 750 mL of deionized water and adjusting to pH 3 with Ammonium Hydroxide. Dilute to 1 L with deionized water).
- Acetate buffer pH 4.5 (made by dissolving 8.2vg of anhydrous Sodium Acetate in 800 mL of deionized water and adjusting the pH to 4.5 with glacial acetic acid and diluting to 1 L).

Mechanical Cleaning Procedure (Glassy Carbon and Metal)

If the above procedures do not sufficiently clean the electrode you must mechanically polish the electrode surface.

CAUTION! Millielectrodes and Microelectrodes are precision devices. Unnecessary polishing of the surface may cause electrode defects. This procedure removes electrode material and therefore reduces the life expectancy of your electrode.

Note: Polishing a PAR electrode with materials not supplied in our standard polishing kit (K0252) voids the warranty. The kit comes with diamond paste and alumina slurry polishing compounds, Texmet and Microcloth polishing pads and a glass plate.

Fine Polishing Procedure (for Minor Imperfections)

K0252 Kit materials required:

- 1 glass polishing plate
- 1 Microcloth polishing pad (disc-shaped, soft, felt-like pad)
- 1 bottle of 0.05 micron white alumina slurry

Use the following procedure to polish your electrode:

1. Rest the glass plate on a flat surface.
2. Remove the adhesive backing from the Microcloth pad and press it onto the glass plate so that it adheres.
3. Wet the pad with several drops of distilled water.
4. Shake the bottle of alumina suspension before applying it (the large bottle containing the white suspension).
5. Add 2 to 4 drops of alumina slurry to the pad.
6. Remove the working electrode from the cell.
7. Rinse the electrode with distilled water to remove any traces of buffer.
8. Hold the working electrode face down onto the Microcloth impregnated with alumina. Be sure to keep the electrode as vertical as possible.
9. Using a smooth medium pressure begin to polish the electrode. Move the electrode in a figure-8 pattern to ensure even grinding action. Do this for two to three minutes.
10. When you have finished polishing, remove the electrode and clean it with copious amounts of distilled water.
11. To further clean the electrode, hold it in a bath of distilled water in a sonicator to make sure all alumina grit has been removed.
12. Dry the electrode and re-insert into the cell.

Coarse Polishing Procedure (for Major Imperfections)

K0252 Kit materials required:

- 1 glass polishing plate.
- 1 Texmet polishing pad (disc-shaped, paper like polishing pad).
- 1 syringe containing 1 micron blue diamond paste.

Use the following procedure to polish your electrode:

1. Rest the glass plate on a flat surface
2. Remove the adhesive backing from the Texmet pad and press it onto the glass plate so that it adheres.
3. Wet the pad with several drops of distilled water.
4. Place a 0.25 inch dab of blue diamond paste onto the pad.
5. Remove the working electrode from the cell.
6. Rinse the electrode with distilled water to remove any traces of buffer.
7. Hold the working electrode face down onto the Texmet pad impregnated with diamond paste. Be sure to keep the electrode as vertical as possible.
8. Using a smooth medium pressure, begin to polish the electrode. Move the electrode in a figure-8 pattern to ensure even grinding action. Do this for two to three minutes.

9. When you have finished polishing, remove the electrode and clean it with copious amounts of distilled water.
10. To further clean the electrode, hold it in a bath of distilled water in a sonicator to make sure all the diamond paste particles have been removed.
11. **IMPORTANT:** Perform the Fine Polishing Procedure described in the previous section until all visible scratches have been removed.

If none of the above procedures produce sufficient results, you have likely either reached the end of the usable life of the electrode or have damaged the electrode beyond the scope of what these procedures are designed to handle. Please contact your local sales representative to order a replacement if this is the case.

Working Electrode Potential Ranges in Aqueous Electrolyte

(Approximate values vs. Saturated Calomel Electrode)

Glassy Carbon	Acidic	-0.50 V	to	+1.50 V
	Neutral	-0.80 V	to	+0.95 V
	Basic	-0.90 V	to	+0.75 V
Mercury	Acidic	-1.10 V	to	+0.40 V
	Neutral	-1.90 V	to	+0.10 V
	Basic	-0.10 V	to	- 2.00 V
Gold	Acidic	-0.50 V	to	+1.00 V
	Neutral	-0.70 V	to	+0.90 V
	Basic	-0.80 V	to	+0.80 V
Platinum	Acidic	-0.40 V	to	+1.40 V
	Neutral	-0.80 V	to	+0.95 V
	Basic	-1.00 V	to	+0.50 V