

Application Note T-3



Subject: Model 303A SMDE Maintenance and Troubleshooting Guide

The Model 303A is a versatile, reliable, useful electrode for voltammetry. Like all sophisticated instrumentation, it must be treated with tender loving care. This TLC must be carried through the capillary installation, storage and maintenance of the Model 303A. If the following guidelines are adhered to, you will experience few or no problems with you Model 303A.

CAPILLARY INSTALLATION

You will find variations of the set-up procedure among users but all will agree that clean mercury is a must. Mercury should be at least triple-distilled. It should never be reused unless it has been redistilled. The installation should follow these steps.

1. Do Not Install Capillary. Turn the Model 303A off; slowly pour approximately 20 mL of mercury into the reservoir.
2. Place a small (50 or 100 mL) beaker directly under the valve body.
3. Turn the Model 303A on.
4. Press DISPENSE 5 times.
5. Turn the Model 303A off.
6. Return the mercury in beaker to the reservoir.
7. Repeat steps 2-7 3 times.
8. Carefully add mercury until at least the entire bottom of the reservoir is covered. You may add up to 5 lbs.
9. Install the capillary and tighten the retaining nut finger tight. Do not use any tools to tighten.
10. Turn the Model 303A.
11. Place the beaker under the capillary.
12. Turn the Model 303A Mode Switch to DME.
13. Draw 5 mL of mercury into the syringe (with needle attached) supplied with the Model 303A.
14. Fit the needle into the filling hole at the top of the valve body (in the bottom of the reservoir directly in front of the threaded knob).
15. Keeping pressure on the syringe body, force mercury through the valve body. Mercury should be observed streaming from the capillary.
16. Repeat steps 15-17 until mercury is dropping freely from the capillary when the syringe is removed.
17. If flow can not be established, return to step 10 and install a new capillary.
18. When flow is established, switch the Model 303A to SMDE Mode.
19. Hold DISPENSE button down and verify the mercury is dropping smoothly from the capillary.
20. Place a solution in a cell and position under capillary.

21. Hold DISPENSE button down and verify that mercury is dropping smoothly from the capillary.
22. Alternate between pressing DISPENSE and DISLODGE. Check to make sure a drop forms and hangs with DISPENSE and is knocked off with DISLODGE. Adjust "DISLODGE ADJ" on rear panel if required. The drop knocker should dislodge the drop with minimum movement of the capillary.
23. Run a voltammogram on 5 ppm Cd or Pb in a 0.1M Acetate Buffer, pH 4.5, scanning from -0.2 to -0.8 V.
24. If an acceptable voltammogram is not obtained, return to step 10.

STORAGE

The capillary of the Model 303A will perform best if the tip is stored in mercury. The reference electrode should be stored in saturated KCl. If it is not possible to store the capillary in mercury, the next best option is to dispense a drop of mercury and store the capillary in air. Again, the reference electrode should be stored in KCl. We do not recommend the storage of the capillary in solution. This will shorten capillary life and can lead to electrode problems.

MAINTENANCE

Problems with the electrode can usually be traced to four causes:

1. Mercury
2. Capillary
3. Valve Body
4. Reference Electrode

1. Mercury

Be sure there is enough mercury in the reservoir to completely cover the bottom. Be sure the mercury is triple distilled. Add additional mercury only if there is no visible surface film on the mercury in the reservoir. If a film is present, carefully vacuum it off the surface.

2. Capillary

If flow cannot be established or maintained, the capillary may be dirty or defective. It can be cleaned as follows:

- a. Aspirate any residual mercury from the capillary.
- b. Aspirate 1:1 HNO₃ through the capillary.
- c. Aspirate deionized water through the capillary.
- d. Aspirate methanol through the capillary.
- e. Continue to draw air through the capillary until dry.
- f. Siliconize if desired following the procedure in the Model 303A manual.

In the majority of problems with the 303A, the capillary is at fault. If a change to a new capillary solves your problems, the "bad" capillary should be cleaned and tried again. If that "bad" capillary still malfunctions, it should be discarded.

3. Valve Body

If a new capillary does not resolve the problem, the valve body may be dirty or defective. It can be cleaned as follows:

- a. Plug the top of the valve body by inserting a yellow micropipette tip in the filling hole at the top of the valve body.
- b. Remove the capillary.
- c. Place a small beaker directly under the valve body.
- d. Dispense the mercury in the valve body into the beaker and discard.
- e. Remove the valve seat. **Caution:** there is some residual mercury in the valve body.
- f. Use a lint free wipe to clean the interior of the valve body, plunger and valve seat.
- g. Inspect plunger for particulates or other dirt.
- h. Reassemble the valve body.
- i. Remove micropipette tip.
- j. Follow capillary installation procedure.

4. Reference Electrode

The reference electrode is generally the source of most noise in an electrochemical experiment. It should be kept wet after it has been exposed to solution. The filling solution should be replenished as needed. Any bubbles in or around the electrode can lead to noise. If the condition of the electrode is doubtful, the Vycor frit should be replaced. Follow the procedure in the Model 303A manual.