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Model 400
Electrochemical Detector
Technical Reference

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Troubleshooting

Overview

Although the Model 400 has proven to be a reliable instrument, you may, at some point during the life of the instrument, have a problem that requires analysis and corrective action. Generally, if you have a problem, there's no need to worry; problems in LCEC operation are easily identified and fixed. This technical reference will help you develop a sound probem-solving approach and identify and fix some of the more common problems. It will also describe the most reliable way of polishing the Model 400 working electrode, one of the key techniques for ensuring problem-free operation.

Problem-Solving Approach

Generally, LCEC problems fall into three main areas--electronic, mechanical, and chromatographic. Although electronic problems are rare, it is relatively easy to test the Model 400 circuitry. Thus the first step in problem solving is to test the Model 400 electronics. If the circuitry tests OK, you can then try to isolate the mechanical or chromatographic problem by scanning down a list of common symptoms and possible causes. Follow this two-step approach:

Step 1

The Model 400 contains an internal dummy cell that allows you to simulate a measurement without using a live cell. To begin isolating the problem, run the dummy-cell test described on page 15 of the manual. If the instrument fails this test, you can be reasonably sure you have an electronic problem within the Model 400.

To confirm and further isolate the electronic problem, run the Self-Test described on page 37 of the manual. This test checks out virtually all of the digital circuitry and much of the analog circuitry in the Model 400. Record the results of all the tests and contact the factory about how to proceed.

Step 2

If the instrument passes the dummy cell test, you can be reasonably sure that the Model 400 electronics are working correctly. The problem is either mechanical (that is, in the plumbing or external electronic connections) or chromatographic (that is, in the design or setup of your experiment).

There is no general way to differentiate between a mechanical and chromatographic problem. However, given the specific symptom, you can check through a list of problem causes until you find the fix. Produce a representative recorder tracing of the problem experiment and use the following troubleshooting guide to isolate and correct the problem.

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Troubleshooting Guide

To use the troubleshooting guide, scan down the list of symptoms on the left until you come to the symptom you are experiencing. Then read the list of possible causes in the middle column and perform the action described in the right column. Continue this procedure until the problem is fixed.

Symptom	Cause	Action
Noisy baseline, Constant frequency	▸ Pump pulsations	Use a better pump or a pulse dampening system.
	▸ Detector waste line dripping	Place the waste line below the fluid level of the waste container.
	▸ No electrical isolation	Make sure the grounding plug is in the A (top) position (see p. 61 of the manual).
		Install an isolation union between the column and the detector (see p. 10 of the manual).
	▸ System improperly grounded	Plug all system components into a single power strip (use surge suppression, if possible).
Noisy baseline, Random frequency	▸ Electronic malfunction	Test with a dummy cell if you haven't aleady (see p. 15 of the manual).
	▸ Recorder voltage incorrect	Set the recorder to the proper output voltage (see pp. 17, 58, 59 of the manual).
	▸ Output cabling incorrect	Choose the proper output (see pp. 58, 59 of the manual).
		Check the wiring to the recorder.

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Symptom	Cause	Action
Noisy baseline, Random frequency (Cont'd)	▸ Insufficient filter time constant	DC Mode: Increase the time constant.
		Pulse Mode: Increase the T1 time.
	▸ Current range too sensitive	Adjust to a higher current range.
	▸ Reference electrode fouling	Renew the reference electrode filling solution and replace the Vycor frit (see the procedure later in this technical reference).
	▸ Working electrode contaminated	Clean or polish the working electrode (see the procedure later in this technical reference).
	▸ Scratches on working electrode	Polish the working electrode (see the procedure later in this technical reference).
	▸ Air in cell	With pump flow on, loosen the black nylon bushing for 10 seconds and then retighten it (see p. 8 of the manual).
	▸ Leaky cell	Tighten the cell seal (see p. 7, Fig. 4 of the manual).
		Change the gasket.
		Remove any imperfections from the electrode surface by polishing (see the procedure later in this technical reference).
	▸ Leak at reference electrode	Change the Kalrez O-ring.
	▸ Column contamination	Clean or replace the column.

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Symptom	Cause	Action
Noisy baseline, Random frequency (Cont'd)	▸ Mobile phase contamination (high background currents)	Use superior quality ingredients in your mobile phase preparation.
	▸ Metal corrosion from stainless steel components	Passivate the stainless steel components of the HPLC sytem with 6N. nitric acid.
Loss in sensitivity	▸ Dummy cell connected	Change to a real cell using the System utility (See p. 31 of the manual).
	▸ Current range incorrect	Adjust to a lower current range.
	▸ Applied potential incorrect	Increase the applied potential until you reach maximum current.
	▸ Leaky cell	Tighten the cell seal (p. 7 Fig. 4 of the manual).
		Change the gasket.
		Remove any imperfections from the electrode surface by polishing (See the procedure later in this technical reference).
	▸ Leaky sample injector	Tighten the sample injector connections or replace the sample injector seal.
	▸ Fouling of reference electrode resulting in a potential shift	Renew the reference electrode filling solution and replace the Vycor frit (see the procedure later in this technical reference).
	▸ Air in reference electrode	Remove the reference electrode and shake out any air.
	▸ Contaminated working electrode	Clean or polish the working electrode (see the procedure later in this technical reference).

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Symptom	Cause	Action
Loss in sensitivity (Cont'd)	▸ Sample auto oxidation	Prepare a fresh standard or add an antioxidant.
	▸ Change in mobile phase composition	Correct the mobile phase pH, the ionic strength, or the ionic composition.
	▸ Pump seal material shredding	Replace the pump piston seals.
Baseline drift	▸ Electronic malfunction	Test the electronics using the dummy cell if you haven't already (see p. 15 of the manual).
	▸ Electrode equilibration	Allow the electrode to stabilize.
		Electrochemically condition the electrode before using it (see the procedure later in this technical reference).
	▸ Fouling of reference electrode	Renew the reference electrode filling solution and replace the Vycor frit (see the procedure later in this technical reference).
	▸ Leaky cell	Tighten the cell seal (see p. 7, Fig. 4 of the manual).
		Replace the gasket.
		Remove any imperfections from the electrode surface by polishing it (see the procedure later in this technical reference).
	▸ Leaks in LC system	Tighten the fittings or replace the ferrules.
	▸ Working electrode contamination	Clean or polish the working electrode (see the procedure later in this technical reference).

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Symptom	Cause	Action
Baseline drift (Cont'd)	• Column bleed	Clean or replace the column.
	▸ Mobile phase contamination	Use superior quality ingredients in your mobile phase preparation.
	▸ Metal corrosion from stainless steel components	Passivate the stainless steel components of the HPLC sytem with 6N. nitric acid.
	▸ Temperature fluctuations	Use a column heater to stabilize the temperature.
	▸ Flow fluctuations	Replace the pump seals or use a different pump.
Noise spikes	▸ Air in cell	With the pump flow on, loosen the black nylon bushing for 10 seconds and then retighten it (see p. 8 of the manual).
	• Air in reference electrode	Remove the reference electrode and shake out any trapped air.
	• Air in sample	Reinject the sample without air.
	▸ Instrument grounding problem	Plug all system components into a single power strip (use surge suppression, if possible).
	▸ Voltage spikes	Plug the system power strip into another circuit or use a voltage surge suppressor.
	▸ Loose electrode connections	Disconnect the electrode, inspect it for damage, and reconnect it.

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Symptom	Cause	Action
Noise spikes (Cont'd)	▸ Erratic pump performance causing pressure fluctuations	Inspect the pump seals, check the valves, remove any air from the pump, and replace any defective parts.
		Use a pulse dampening system.
Stairstepping baseline	▸ Recorder or integrator signal is highly amplified	Adjust the recorder input voltage, increase the attenuation (that is, reduce the signal amplification), or use the analog output.
Flat-top peaks	▸ Recorder input voltage is incorrect	Adjust the recorder input voltage or adjust the detector output cable to the proper position (see pp. 58, 59 of manual).
Current overload light remains lit	▸ Electronic malfunction	Test with the dummy cell if you haven't already (see p. 15 of manual).
	▸ Current range too low	Select a higher current range.
	▸ Insufficient offset	Press the auto offset key (see p. 54 of manual).
	▸ Mobile phase contamination (high background current)	Use superior quality ingredients in your mobile phase preparation.
	▸ Metal corrosion from stainless steel components	Passivate the stainless steel components of the HPLC sytem with 6N. nitric acid.
	▸ Sample concentration too high	Dilute the sample.
	▸ System grounded improperly	Plug all system components into a single power strip (use surge suppression, if possible).

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Symptom	Cause	Action
Potential overload light remains lit	▸ Electronic malfuntion	Test the electronics with the dummy cell if you haven't already (see p. 15 of the manual).
	▸ Reference electrode malfunction	Renew the reference electrode filling solution and replace the Vycor frit (see the procedure later in this technical reference).
		Disconnect the reference electrode, inspect it for damage, and reconnect it.
		Remove the reference electrode and shake out any trapped air.

Note: If you still cannot isolate the problem after performing the procedures listed in this troubleshooting guide, call the factory at (800) 274-7272 (U.S.A. only) or (609) 895-0100.

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Working Electrode Care

Overview

The cleaning and polishing procedures described in this section are designed to help you keep your Model 400 working electrode in the best possible condition. The procedures apply to the two classes of electrodes used with the Model 400--glassy carbon and metal.

To ensure consistent productivity during cleaning, we recommend that you stock an extra working electrode. Also, we recommend that you install a new gasket every other time you change your electrode. The following sections describe some important considerations for both electrode types.

Glassy Carbon Electrode Care

Unlike most electrode metals, glassy carbon is a porous material vulnerable to chemical penetration of up to a few microns into the electrode surface. This penetration can alter the electrode surface, making it necessary to equilibrate for long periods before the electrode can be used for analytical work. Thus, for glassy carbon electrodes, you should try to avoid aggressive cleaning techniques like mechanical polishing, which can penetrate the surface.

Fortunately, there are effective cleaning techniques available for glassy carbon electrodes. At EG&G PARC, we've had success with three techniques in particular--two involving chemical procedures and one involving an electrochemical procedure. These procedures are described below.

We suggest that you take a conservative approach to cleaning your electrodes. First use the procedure which causes the least amount of physical damage to the surface of the working electrode. If you don't get the required results from one procedure, then try a more aggessive one. The procedures are presented in order of aggressiveness, beginning with the least aggressive.

Metal Electrode Care

Mechanical polishing procedures, which can penetrate the surface of glassy carbon electrodes, are safe to use on metal electrode materials. The chemical procedure described below is useless for metal electrodes, but you can use the electrochemical procedure. You can also use pulse amperometric techniques to clean metal electrodes, although we don't describe the procedures here.

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Chemical Procedures (Glassy Carbon Only)

We have successfully used these chemical procedures to clean single or dual glassy carbon working electrodes. The procedures are presented in order of increasing aggressiveness. Remember, more aggressive procedures make it necessary to equilibrate for a longer period before doing analytical work.

Methanol Cleaning Procedure (Less Aggressive)

1. Remove the glassy carbon electrode from the cell compartment.
2. Rinse it with distilled water. This will remove any encrusted buffer 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 a Kimwipe or lense tissue. This procedure may clean the electrode enough to restore a suitable response.

Chromic Acid Cleaning Procedure (More Aggressive)

1. Remove the electrode from the cell compartment.
2. Rinse it off with distilled water.
3. Dry the electrode.
4. Apply one or two drops of chromic acid cleaning solution to the surface of the electrode.
5. Let the solution stand for 30 seconds.
6. Rinse the surface off with distilled water.
7. Re-insert the electrode into the cell compartment.

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Electrochemical Cleaning Procedure (Glassy Carbon and Metal)

When you use potentials over 800 mv in an analysis, some organic compounds can deposit onto the electrode surface. Applying a high negative and high positive potential in sequence can often desorb these compounds from the electrode surface. Use this procedure to remove the compounds from the electrode surface:

1. Apply a -600 mv potential to the electrode for 1 minute.
2. Apply a +1200 mv potential for 10 minutes.
3. Apply the potential appropriate for the analysis of your compound.

Mechanical Cleaning Procedures (Glassy Carbon and Metal)

If the above procedures do not sufficiently clean the electrode, you must mechanically polish the electrode surface. We recommend the following procedure:

Caution! The Model 400 working electrodes are precision devices, machined to be flat within 0.001 inch. Unnecessary polishing of the surface may cause electrode defects.

Polishing an EG&G PARC electrode with materials not supplied in our standard kit (K0252) voids your warranty. You can purchase the following standard kit items separately:

- K0253 Polishing Compounds (6 micron diamond paste and 0.05 micron alumina slurry)
- K0254 Polishing Cloth (Texmet and Microcloth)

Fine Polishing Procedure (For Minor Imperfections)

Kit materials needed:

- 1 glass polishing plate
- 1 Microcloth disc-shaped polishing pad (the soft, felt-like polishing 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.

   Note: To avoid mixing different polishing compounds on one pad, it's a good idea to identify the polishing compound you're using. Simply write the name of the compound on the side with the Microcloth pad.

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3. Wet the pad with several drops of distilled water

4. Shake the bottle of alumina suspension before applying it (the large bottle containing white solution).

5. Add 2 to 4 drops of alumina slurry to the pad.

6. Remove the working electrode from the cell compartment.

7. Rinse the electrode with distilled water to remove any traces of buffer.

8. Press the working electrode face down onto the Microcloth pad impregnated with alumina.

9. Use a smooth medium pressure to polish the electrode. Move the electrode in a figure-8 pattern to ensure an even grinding action. Do this for two to three minutes.

10. When you're finished polishing, remove the electrode and clean it with large amounts of distilled water. Place the electrode face up in distilled water in a sonic cleaner for three minutes to make sure you remove any alumina grit. Dry the electrode and re-insert it into the cell compartment.

Coarse Polishing Procedure (For Major Imperfections)

Kit materials needed:

- 1 glass polishing plate
- 1 Texmet disc-shaped polishing pad (the paper-like polishing pad)
- 1 syringe containing 6 micron yellow diamond paste

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.

   Note: To avoid mixing different polishing compounds on one pad, it's a good idea to identify the polishing compound you're using. Simply write the name of the compound on the side with the Texmet pad.

3. Wet the pad with several drops of distilled water

4. Place a ¼-inch dab of yellow diamond paste onto the pad.

5. Remove the working electrode from the cell compartment.

6. Rinse the electrode with distilled water to remove any traces of buffer.

7. Press the working electrode face down onto the Texmet pad impregnated with diamond paste.

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8. Use a smooth medium pressure to polish the electrode. Move the electrode in a figure-8 pattern to ensure an even grinding action. Do this for two to three minutes.

9. When you're finished polishing, remove the electrode and clean it with large amounts of distilled water. Place the electrode face up in distilled water in a sonic cleaner for three minutes to make sure you remove any alumina grit. Dry the electrode.

10. Perform the Fine Polishing Procedure described in the previous subsection.

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Reference Electrode Care

Overview

The function of the reference electrode is to maintain the correct applied potential at the working electrode. If you don't regularly maintain the reference electrode, the applied potential can drift several millivolts from the reference point, resulting in an inaccurate measurement.

When properly maintained and used with a constant mobile phase composition, a reference electrode can operate properly for several weeks. However, because the glass frit at the electrode junction is porous, the reference electrode solution will change in composition over time. Osmotic gradients between the mobile phase and the reference electrode filling solution will develop, causing chloride ions to leave the electrode and ions from the mobile phase to enter the electrode. Thus, you must renew the reference electrode solution and replace the Vycor frit regularly.

The recommended regular maintenance frequency depends on the ionic composition and ionic strength of your mobile phase. We recommend that you renew the reference electrode solution whenever you significantly alter the ionic composition or strength.

Another way to evaluate your reference electrode solution is to regularly monitor chromatograms of standards. If the peak heights in the chromatogram change with time under constant mobile phase and operating conditions, you should renew the filling solution and replace the Vycor frit.

Reference Electrode Renewal Kit

The following list shows the parts in the accessory kit used to renew the reference electrode:

Reference Electrode Kit	K0250
3M Potassium Chloride/ Saturated Silver Chloride Solution, 1 oz.	SL0070
Vycor Tips, 4 mm	G0100

Note: The porous vycor frit may dissolve when you use a strong basic eluent (pH > 10). An alternate polyethylene frit, which can withstand this type of mobile phase, is available. Order EG&G PARC Part Number G0194.

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Reference Electrode Solution Testing Procedure

You can easily test a reference electrode to determine whether the reference potential is correct. Use this procedure to make the determination:

1. Place a freshly filled reference electrode in a small beaker containing 3M potassium chloride/saturated silver chloride solution. You must assume that the fresh electrode is at the correct potential.

2. Remove the reference electrode to be tested from the Model 400 cell compartment and place it in the same beaker as the fresh reference electrode.

3. With a 3½ digit voltmeter, measure the potential difference between the two reference electrodes.

4. If you measure more than 20mv potential difference between the two reference electrodes, we recommend that you renew the filling solution and replace the Vycor frit.

Refilling Procedure

1. Remove the reference electrode from the Model 400 cell.

2. Remove the old Vycor tip from the glass electrode body by pulling it off or cutting it out with a single-edge razor blade.

3. Empty the reference electrode fluid by shaking it out.

4. Take some fresh 3M potassium chloride/saturated silver chloride filling solution and refill the electrode to about ¼" from the top.

5. With minimal handling, place a fresh Vycor frit into a piece of Teflon heat shrink tubing.

   Note: Vycor is a porous glass material vulnerable to contamination from handling or airborne contaminants.

6. Place the Teflon heat shrink tubing with Vycor frit onto the tip of the reference electrode. Make sure there are no gaps between the glass tip and the Vycor frit.

7. Using a heat gun at moderate temperature, seal the frit onto the glass reference electrode. Rotate the electrode body in front of the heat gun to maintain an even temperature around the circumference of the seal.

Caution! Excessive heating can vaporize the filling solution and push the frit away from the electrode tip.

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8. Using a sharp single-edge razor blade, trim the shrink tubing so that it is flush with the Vycor tip. Make sure no edges project beyond the tip of the frit. Air bubbles can easily become trapped in the recesses created by these projections and cause detection problems that are difficult to diagnose.

9. To eliminate air bubbles or trapped gases at the frit end of the electrode, shake the electrode a few times (use the same action you use when shaking a thermometer). You can also flick the tip of the electrode with your finger.

10. Allow any remaining trapped air to escape by placing the electrode frit-down in filling solution for a few minutes.

11. Reinstall the reference electrode into the Model 400. Make sure the O-ring is intact. If you observe a leak, change the O-ring.

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