GAMRY INSTRUMENTS

Dr. Bob's Cell

for Small-scale Electrochemistry

Operator's Manual

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Internet	www.gamry.com/service-support/
Telephone	(215) 682-9330 9:00 AM - 5:00 PM US Eastern Standard Time
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Replacement parts for this kit are available from Gamry Instruments, Inc. Optional parts may be available from Gamry Instruments, or from third-party vendors. Contact your local Gamry sales representative to discuss any special requirements or accessories that you need.

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Introduction

Gamry Instruments designed Dr. Bob's Cell kit for electrochemical testing. Enzyme and catalysis studies, development of electrochemical sensors, basic research into battery mechanisms, and determination of redox potentials of inorganic complexes represent a few of its many applications.

Dr. Bob's Cell can be used with electrolyte volumes as small as 2 mL and as large as 50 mL. It works well with conventional-sized electrodes, such as 1−3 mm diameter disks. It also can be used with micro-electrodes (typically 1 to 10 μm diameter disks). This cell provides simple, flexible, and reliable operation in a wide variety of testing situations.

Gamry Instruments provides platinum, gold, and glassy carbon working electrodes designed for use with this cell. These materials are the most popular for studying heterogeneous and homogeneous chemical reactions. Chemical modification of the surface of these materials is a common technique to gain specificity in a chemical sensor.

Most of the pieces required to run routine experiments are included in Dr. Bob's Cell kit.

Neither a reference electrode nor a working electrode is included in the cell kit. Requirements for these electrodes vary too much user to user to make their inclusion in the standard kit practical. Gamry Instruments sells both reference electrodes (SCE, Ag|AgCl, and $Hg|Hg_2SO_4$) and macro- and micro-disk electrodes (in platinum, gold, and carbon) that are suitable for use with Dr. Bob's Cell kit. Contact Gamry Instruments directly or contact your local sales representative if you need working or reference electrodes.

Dr. Bob's Cell uses five standard ports to implement its required functions. You can customize the cell by rearranging some of Gamry's standard fittings or making or buying additional fittings, electrodes, sensors or adapters.

Too often, temperature-control is neglected in designing electrochemical experiments. Temperature is an important variable in the rate of chemical reactions. Comparing test results recorded at different temperatures can be vital in gaining a full understanding of a chemical system. For these reasons, Gamry offers a special jacketed version of Dr. Bob's Cell. When this cell is connected to a circulating water bath, accurate temperature control of your experiments becomes possible.

Chemical Compatibility of Dr. Bob's Cell

The components in Dr. Bob's Cell were selected to be as chemically inert as possible. In normal use, the only materials in contact with the test solution are:

- The working electrode material and the insulator surrounding the working electrode
- Borosilicate glass (Pyrex® or equivalent),
- Unfired glass frit,
- PTFE,
- Platinum,
- Polyethylene,
- ACE Glass's FETFE O-ring material

Chemical resistance tables for most of these materials are available. One exception is FETFE, which is a elastomer proprietary to ACE Glass, which consists of PTFE particles in a fluorinated-rubber base similar to Viton®. According to ACE Glass, it offers ***slightly*** better chemical resistance than Viton®.

The black ACE-Thred™ fittings supplied with the cell do not normally come in contact with the cell electrolyte. These are nylon fittings, so you can use nylon's properties (which are generally available) as an indication of these fitting's suitability for use in any specific chemical environment.

Caution: The nylon bushings in the ACE Thred™ fittings and the FETFE O-rings may not be suitable for use in some electrolytes (particularly non-aqueous media). If you need better chemical resistance than that offered using the standard ACE-Thred™ components, Ace Glass (www.aceglass.com) can provide replacement fittings made from PTFE and Kalrez®, which are extremely resistant to chemical attack. Contact Gamry Instruments if you need help selecting the proper replacement fittings.

Dr. Bob's cell was not designed for use in electrolytes that dissolve glass (extremely basic solutions or HF containing solutions).

Caution: The glass components in the cell and the glass frits used in the reference bridge tube are not suitable for use with extremely basic solutions or solutions containing hydrofluoric acid. We do not know of any substitute cell components that will completely overcome this limitation. If you need to work with solutions that will damage the Dr. Bob's cell, we recommend that you design and build an all-plastic cell.

The simple construction of the polyethylene stoppers used to seal the unused port makes them easily adaptable as a vent for purge gas: you simply poke a hole in the flat surface of the stopper. While they provide chemical resistance to most aqueous electrolytes, they may not be a good choice for use with some aggressive solvents. If polyethylene is not suitable for your application, glass and PTFE stoppers are available from most laboratory supply houses. If you use these "more solid" stoppers, do not forget the need to vent purge gas from the cell.

Unpacking and Checking a Cell Kit

This section is primarily intended for the user who has just received a new Dr. Bob's Cell Kit.

Checking for Shipping Damage

Your new Dr. Bob's Cell kit was shipped disassembled to minimize shipping damage. All of the pieces have been carefully packaged in anticipation of rough handling in shipment. Unfortunately, no matter how carefully glass pieces are packaged, damage will sometimes occur.

When you first receive your Dr. Bob's Cell kit, please check it for any signs of shipping damage. Be especially careful if the shipping container shows signs of rough handling.

Obviously, the glass pieces are the most susceptible to damage. Check the glass pieces for chipping and small cracks as well as for major damage.

Warning: Do not use any glass parts that are chipped or cracked. Any damage to glass increases the probability of additional damage. Broken glass can have extremely sharp edges that represent a significant safety hazard. Injuries from broken glass can be quite severe.

If any parts were broken in shipment, please contact our US facility or your local Gamry representative as soon as possible. In most cases, Gamry should have replacement parts in stock. Please retain the shipment's packaging material for a possible claim against the shipping company.

Parts List

Please check the contents of your kit versus the Dr. Bob's Cell kit packing list in Table 1. When shipped, all of the Dr. Bob's Cell kit components should be labeled with their Gamry Instruments, Inc. part number.

If you are checking the completeness of an older kit, most of the components are shown in photographs throughout this manual.

Table 1
Dr. Bob's Cell kit Packing List

Quantity	Part Number	Description
1	900-00055	Manual, Dr. Bob's Cell
1	930-00031*	Dr. Bob's Cell Kit Cell body
1	930-00037**	Dr Bob's Cell Kit Jacketed Cell body
1	930-00044	Dr. Bob's Cell Bridge Tube with glass frit
1	930-00033	Dr. Bob's Cell gas flow adapter
1	930-00034	Dr. Bob's Cell bubbler tube
1	930-00035	Fritted counter iolation tube
5	955-00003	Frit, Unfired glass, 3.5 mm diameter, with tubing
3	935-00052	#7 ACE-Thred™ bushing, nylon, with FETFE O-ring
1	935-00053	#11 ACE-Thred™ bushing, nylon
1	935-00054	Adapter, #7 ACE-Thred™ to ¼" hose
150 mm	935-00056	Platinum wire, 0.4 mm diameter
1	935-00057*	Support stand for Dr. Bob's Cell
5	935-00059	Polyethylene stoppers for 14/20 joint
1	935-00065	Stir bar, micro, 6.35 mm × 2 mm

Contact us as soon as possible if any of the essential parts are missing.

The contact information for our US facility can be found immediately following the title page of this manual. If you are outside the US, you may want to contact your local Gamry representative.

*Only included in the Standard Dr Bob's Cell configuration, 990-00193.

**Only included in the Jacketed Dr. Bob's Cell configuration, 990-00200.

Assembly of and Use of Your Cell Kit

This section of the manual tells you how to assemble and use a Dr. Bob's Cell kit in its standard configuration.

This "standard" cell configuration has:

- A standard cell body for use at room temperature
- a working electrode in the center port (#7 ACE-Thred™) of the cell,
- a three-way gas purge/blanket/vent adapter in one 14/20 standard taper port
- a frit isolated platinum counter electrode in one #7 ACE-Thred™ port
- a reference electrode and bridge tube in one #7 ACE-Thred™ port
- one unused 14/20 standard taper port (may be closed with a stopper)

This is one of many possible cell configurations. Feel free to customize your Dr. Bob's Cell. You are only limited by your imagination and the number and size of the ports available on the top of the cell.

Cell Assembly: General Information

A picture of an assembled cell is shown in Figure 1.

If you are assembling your Dr. Bob's Cell kit for the first time, try to assemble the entire cell dry first. After you are comfortable with the assembly process, you can fill the cell with a test solution and run real tests.

We recommend that you start the assembly of your cell by placing the cell body in the epoxy-coated cell stand supplied with your cell kit. The cell body is not stable when laid on a flat surface such as a laboratory bench, and can easily roll off a bench onto the floor with cell-shattering results. Gamry Instruments can always use additional sales, but we hate to have them come in the form of replacement cell bodies! Always keep the cell in the cell stand to avoid problems.

Caution: Always assume that the cell body will roll a substantial distance if laid down on a flat surface. Guard against it rolling off your test bench. Gamry's cell stand can be very useful in preventing this type of accident.

In many cases, you can even run your experiments with the cell placed in the cell stand. If you feel you need a more stable test setup, the cell can be mounted in a ring-stand.

Two of the ports on Dr. Bob's cell are 14/20 standard taper ground glass joints. Do not grease these joints.

Caution: In vacuum work in a chemical laboratory, ground glass joints are often greased. This is unnecessary with Dr. Bob's Cell, and may even cause problems if the grease gets into the test solution or an electrode. **Never grease any of the ground glass joints on your Dr. Bob's Cell.**

Figure 1
The Standard Configuration of Dr. Bob's Cell

Pay careful attention to cell cleanliness. In most electrochemical testing situations, contaminants in the cell and test solution can lead to poorly reproducible results. If you touch the cell components with your fingers, you can inadvertently add salts and organic compounds to your cell solution. We recommend that you carefully clean the cell components using good laboratory practice. After the components are clean, avoid touching their wetted surfaces.

Dr. Bob's Cell includes a number of ACE-Thred™ connectors used for a wide variety of functions. #7 ACE-Thred™ connectors are particularly common on Dr. Bob's Cell. ACE-Thred™ fittings are designed to seal cylindrical objects into the cell. These objects can include glass tubes, glass plugs, thermometers, and plastic electrode bodies. ACE-Thred™ connections are designed to be tightened with finger-pressure only.

Caution: ACE-Thred™ fittings should always be tightened "finger tight." Do not use tools such as a wrench or pliers to tighten an ACE-Thred™ fitting. Over-tightening a fitting can break the cell. In extreme cases this can cause personal danger because broken glass can have very sharp edges.

A given ACE-Thred™ size can only accommodate objects of a specific diameter. For example, a #7 ACE-Thred™ is specified to work with objects of a diameter between 6.5 mm and 7.5 mm. If you need to add non-standard options to your Dr. Bob's Cell kit, make sure you are aware of this restriction.

Figure 2
Picture of Jacketed Dr Bob's Cell Configuration

Gas-Flow Overview and Terminology

The Gas Bubbler Assembly may or may not be required for your experiment. Most of the cases in which you use it involve the removal of atmospheric oxygen from the test solution.

Oxygen is an electrochemically active gas. An aqueous solution in contact with the atmosphere usually contains millimolar concentrations of oxygen. If oxygen can be reduced in the potential region you wish to study, it can interfere with your electrochemical measurements.

You can remove oxygen from the test solution by bubbling nitrogen, or another electrochemically inert gas, through the solution. This process is often (imprecisely) called *de-aeration*. It is more correctly called *de-oxygenation*. Other commonly used terms for the process of oxygen removal are *gas purging* or *sparging*. At least 15 minutes of vigorous bubbling with nitrogen is required to remove most of the oxygen from a test solution.

Bubbling gas through your test solution can cause noise while you are running your experiment. To avoid this noise, you can stop gas-purging during the data-acquisition phase of your experiment. Instead you can flow the inert gas over the top of the test solution, often referred to as "blanketing" the cell. In general, blanketing is used after solution purging, where blanketing prevents dissolution of oxygen from the gas above the solution.

Many modern electrochemical test systems include automatic control of gas-flow in their experimental sequencing. This is true of Gamry Instruments' Pulse Voltammetry and Physical Electrochemistry systems. These systems generate a digital signal that is intended to control a solenoid valve, which in turn routes gas flow to the cell. Gamry's VistaShield™ Faraday Cage, when equipped with its Purge and Stir option, provides a complete solution for purge gas control.

Pre-Saturation of the Purge Gas

Bubbling dry purge gas through your cell electrolyte can cause significant evaporation of the electrolyte's solvent during the purge process. This can be a measurable source of error in some experiments. This problem can often be avoided by pre-saturation of the purge gas with the electrolyte prior to it entering the cell. This is commonly done using a "gas washing bottle," which can be obtained at most laboratory supply companies.

Dr. Bob's Cell Kit does not include a gas washing bottle, for they are already available in many laboratories.

Dr. Bob's Cell Gas Bubbler Assembly

The standard gas bubbler assembly consists of four pieces: the Gas Flow Adapter, the Gas Bubbler Tube, a #7 ACE-Thred™ nut, and an ACE-Thred™-To-Hose adapter. A photograph of these pieces can be seen in Figure 2. Note the O-rings, which are required.

Figure 3. Gas Bubbler Assembly

Figure 3 is representative of the Gas Bubbler used for purge only (no blanketing). Connect one end of a plastic hose (not shown) to the Gas Bubbler Tube using the ACE-Thred™-To-Hose adapter. Connect the other end of this hose to a source of purge gas.

At Gamry, we often refer to the gas bubbler assembly as a three-way adapter, because it can be used to implement three functions: purge, blanket and vent.

The vent function is critical. Regardless of whether gas is flowing through or over the test solution, you must provide a way for it leave the cell. If you, do not, the gas may not flow, or worse, the cell may burst apart unexpectedly. Not providing a vent for the escape of purge gas is a very common and often dangerous "mistake" made when setting up an electrochemical cell.

Warning: If you use purge or blanket gas, you **must** provide a vent for the gas to escape the cell. Dr. Bob's Cell was not designed to withstand gas pressure! Failure to vent the cell so can cause damage to the cell, uncontrolled loss of electrolyte from the cell, and risk of personal injury to the cell's operator.

In normal use, the Gas Flow Adapter is installed in one of the 14/20 ground glass ports on your Dr. Bob's Cell. The Gas Bubbler Tube, with an O-ring installed as shown in Figure 3, slides into a #7 ACE-Thred™ port on top of the Gas Flow Adapter. When you are satisfied with position of the Gas Bubbler Tube, tighten the ACE-Thred™ nut.

Attaching Gas Tubing to the Cell

The position of the Gas Bubbler Tube is normally adjusted so its orifice end sits in the cell's electrolyte. The hose barb on the side of the Gas Flow Adapter can be used as a vent or as an inlet for blanket gas.

Your gas flow system should include a needle valve to control the gas flow rate. Make all gas-tubing connections to the cell with this valve ***turned all the way off***. Making connection with a cell filled with electrolyte or adding electrolyte to a system when the gas flow is on can lead to horrible accidents.

Warning: Excessive gas flow can damage the cell and result in a loss of electrolyte. In extreme cases, this can represent a significant safety hazard.

Connect the gas flow system and add the cell electrolyte **before** you turn the needle valve on. Open the valve slowly, while you watch the bubbles in the cell. Bubbling should not be vigorous enough to splash large quantities of electrolyte on the cell walls.

In addition to the needle valve, a three-way valve is very useful in purge and blanket gas control. Three-way valves are available in both electrically switched and manual versions. A three-way valve switches one gas stream so it flows from a single inlet to one of two outlets.

If your system includes a three-way valve for switched purge **and** blanket gas control, we recommend that you:

- Connect the purge gas to the ACE-Thred™-to-Hose adapter on top of the Gas Bubbler Tube,
- Connect the blanket gas to the hose barb on the side of the Gas Flow Adapter,
- Use the spare port to provide a vent. A small hole poked into one of the polyethylene stoppers in the cell kit is generally a sufficient vent.

If you do not have a three-way gas control valve, you can switch from purge mode to blanket mode manually. Connect the gas flow to the ACE-Thred™-To-Hose adapter. When you want to purge, loosen the ACE-Thred™ nut holding the Gas Bubbler tube in place, slide the Gas Bubbler Tube deeply into the cell so its orifice is in the cell solution, then retighten the ACE-Thred™ fitting. When you want to blanket, slide the Gas Bubbler Tube far out of the cell, so its orifice is above the solution. When you use the cell in this way, the hose barb port on the Gas Flow Adapter provides a convenient vent.

Counter Electrode Assembly

Counter Electrode Overview and Terminology

The counter electrode in a three-electrode electrochemical experiment is generally an inert metal or carbon electrode that provides a source or sink of electrons to the cell. Another term used for this electrode in the *auxiliary electrode*.

The electrochemist is generally not interested in the reactions that occur at the counter electrode. One exception is when the reactions at the counter electrode produce a soluble product that can diffuse to the working electrode and interfere with the working electrode's operation. In this case, the electrochemist may want a diffusion barrier to isolate the counter electrode compartment from the rest of the cell. A glass frit is commonly used as an isolation barrier.

Dr. Bob's Cell Counter Electrode: Standard Isolated Assembly

In the standard configuration of Dr. Bob's Cell, the counter electrode is a length of platinum wire, isolated from the bulk solution by a glass frit. There are three pieces in the counter electrode assembly:

- A fritted isolation tube
- A #7 ACE-Thred™ nut,
- 150 mm of platinum wire.

A complete counter electrode assembly is shown in Figure 4. Note the placement of the required O-ring between the ACE Thred™ nut and the fritted end of the tube.

Figure 4. Counter Electrode Assembly

The fritted tube is slid into a #7 ACE-Thred™ port on the cell. The top end of the frit must be below the electrolyte level. If you are working with very small volumes of electrolyte, you may need to abandon the isolation tube; see the section below.

Slide the platinum wire into the isolation tube. The lower end of the platinum wire must be in contact with electrolyte. A sharp bend in the platinum wire at the upper rim of the isolation tube can help prevent loss of contact between the electrolyte and the platinum. When the counter-electrode wire is clipped to the platinum wire below this bend, gravity helps keep the counter electrode in place.

Direct Immersion of the Platinum in the Electrolyte

When you are working with electrolyte volumes of less than 5 mL, the fritted isolation tube can interfere with the other cell components, or can fail to fill with solution. In this case, you must place the platinum counter wire directly in the electrolyte.

Excessive resistance in the frit can also cause problems, especially when the cell electrolyte is poorly conductive and the cell currents are high. Typically this causes the cell current to limit at a value smaller than the compliance current of the potentiostat you are using. It can also cause slower than expected potentiostat response in fast experiments. As in the case above, when this happens, you can place the platinum wire directly in the electrolyte.

There are some approaches to placement of the counter electrode when you do not have an isolation tube:

- You can insert the platinum wire through the #7 ACE-Thred™ port normally used for the isolation tube. You can do this with or without an ACE-Thred™ nut in the port.

- You have more control of the wire placement if you put a piece of 7 mm glass tube into the port and put the wire into this tube. You can even melt the end of a 7 mm tube until it almost collapses, leaving a ½ to 2 mm hole. You can slide this tube into the #7 ACE-Thred™ port, with the small end down, and then slide the wire into the tube.

Regardless of how the platinum wire is placed in the cell, make sure that the exposed platinum wire does not touch the conductive portion of the working electrode or any other metal components present in the cell.

High-density Graphite Counter Electrodes

You can also use high-density graphite as a counter-electrode material. Graphite is less expensive than platinum, offers a larger surface area, and can often provide cell current via surface reactions that do not involve soluble products.

Gamry sells a 150 mm-long high-density graphite rod (part # 935-00014) that can be used in a #7 ACE-Thred™ port. The rod diameter is approximately 6.5 mm.

To use this graphite rod as the counter electrode in Dr. Bob's Cell, slide the O-ring from the ACE-Thred™ port directly onto the graphite rod, and place the rod into the port until the desired length of the rod is immersed in the electrolyte.

Reference Electrode and Bridge Tube

Overview and Terminology

Most modern electrochemical measurements are performed using a three-electrode potentiostat. The cell includes a reference electrode that has a constant electrochemical potential. All working-electrode potentials are measured with respect to this reference electrode.

In many cases, placing the reference electrode directly in the test electrolyte can be a problem. Examples include:

- When the reference electrode is large, making it inconvenient to use in a small volume,
- When the reference electrode's filling solution will contaminate the electrolyte,
- When the cell is heated and you do not want the reference potential to change with cell temperature.

To avoid these problems, Dr. Bob's Cell always uses a bridge tube to keep the reference electrode above the cell. The bridge tube is a glass tube filled with electrolyte that provides a conductive path from the cell electrolyte to the tip of the reference electrode. The electrolyte in the bridge tube is usually identical to the cell electrolyte, but in some cases a different electrolyte may be necessary.

A bridge tube is similar—but not identical—to a Luggin capillary. A Luggin capillary attempts to move the "sensing point" of the reference electrode close to the working electrode surface. The tip of the bridge tube in Dr. Bob's Cell is too large to qualify as a true Luggin capillary.

The bridge tube in Dr. Bob's Cell is particularly convenient, because it terminates in an unfired glass frit, which makes filling the bridge tube easy yet doesn't add a lot of resistance to the reference electrode circuit. Most other designs for bridge tubes or Luggin capillaries either have excessive resistance (causing noisy data or potentiostat oscillation) or are difficult to fill with electrolyte.

Bridge Tube and Reference Electrode: Assembly

Figure 5 shows the Dr. Bob's Cell Bridge Tube and Reference Electrode. Note the placement of the O-rings. These O-rings are critical in sealing the ACE-Thred™ fittings holding this assembly together.

Dr. Bob's Cell does not include a reference electrode, because different tests may call for different types of reference electrodes. The reference electrode shown in Figure 5 is Gamry's standard saturated calomel reference electrode (abbreviated as SCE), part #930-00003. Gamry also offers an Ag|AgCl Reference (part #930-00015) and a $Hg|Hg_2SO_4$ reference (part #930-00029). Contact your local Gamry sales representative if you need a new or a replacement electrode.

Figure 5. Bridge Tube and Reference Electrode Assembly

Dr. Bob's Cell can be used with third-party reference electrodes, as long as the reference electrode's diameter is between 9 and 10.5 mm.

To fill, assemble, and install the bridge tube and reference electrode:

1) Place an O-ring and #7 ACE-Thred™ fitting on the middle section of the bridge tube (see Figure 5).
2) Place an O-ring and #11 ACE-Thred™ fitting on the reference electrode (see Figure 5).
3) Pour electrolyte into the larger diameter end of the bridge tube, leaving about 1.5 cm between the bottom of the ACE-Thred™ and the top of the electrolyte. Make sure there are no air bubbles in the electrolyte between the frit and the top of the electrolyte.
4) Place the reference electrode, tip first, into the larger end of the bridge tube and tighten the #11 ACE-Thred™ fitting until it is finger-tight and the reference electrode is fixed in place.
5) Place the smaller end of the bridge tube into one of ACE-Thred™ ports on your Dr. Bob's Cell.
6) Adjust the height of the bridge tube so that its tip is in the electrolyte and close to the working electrode.
7) Tighten the bridge tube's #7 ACE-Thred™ fitting until it is finger-tight and the bridge tube is fixed in place.

Bridge Tube and Reference Electrode: Maintenance and Care

The unfired glass tips used on both the reference electrode and the bridge tube are subject to cracking when allowed to go from a wetted to a dry state.

Caution: Do not allow the glass frits on the reference electrode or the bridge tube to dry out. They are likely to crack, greatly increasing the flow of bridge tube or reference filling solution into your cell. If the frit does dry out, we recommend that you replace it. Your Dr. Bob's Cell kit contains three replacement frits with PTFE heat-shrinkable sleeves. Use this kit whenever you need to replace a damaged frit.

Whenever possible the bridge tube tip should be stored in the electrolyte used in your system. Place the bridge tube tip down in a flask containing the electrolyte you use in your tests.

If you cannot keep the frit wetted with electrolyte, you can store the bridge tube tip in the solvent to which it will be exposed during testing. In aqueous testing, always use high quality de-ionized or distilled water. The glass frits have a very large surface area which absorb contaminants from impure solvents.

Periodically, it may be necessary to replace the glass frits on the reference electrode and bridge tube. Dr. Bob's Cell comes with two spare glass frits with PTFE heat-shrink tubing. To replace the frit of the bridge tube, make sure the bridge tube is clean and dry. Cut the old frit off with a sharp blade, taking care not to cut yourself. Turn the tube upside down and place the PTFE sleeve over the end of the tube. Insert the piece of glass frit in the sleeve and then heat the sleeve with a heat gun to shrink the PTFE sleeve around the glass frit. Lastly, trim the sleeve to be flush with the exposed end of the glass frit. You may perform a similar procedure with the reference electrode, however it may be difficult to empty. Instead just turn it upside down and keep the solution away from the tip.

Warning: Be careful when using a sharp knife because of the danger of cutting oneself. Never cut towards your body. Take care when using a heat gun to avoid burning oneself.

The reference electrode is also supplied with a clear plastic sleeve covering a fill hole in its glass body. This fill hole serves two purposes:

- It allows you to refill the electrode with saturated KCl if the level of liquid in the electrode drops.
- It also provides a vent, so the filling solution flows slowly out of the electrode.

In normal use, we recommend that the fill hole be kept slightly open. When storing the electrode, the fill hole should be closed.

If the electrode or the bridge tube has been stored in a pure, poorly-conductive solvent, the glass-fritted tip must refill with ions prior to use. In the case of the reference electrode, half an hour with the vent hole open should suffice. In the case of the Bridge Tube, half an hour with a vented reference in place should also be sufficient.

Gamry's reference electrodes are shipped with a plastic sleeve covering the electrode tip. This tip is effective in keeping the reference electrode's glass-fritted tip wet. A plastic sleeve on one side of Bridge Tube's glass-fritted tip ***will not*** keep the bridge tube tip wet, unless the tube is filled with solvent or electrolyte.

Caution: Do not use a plastic sleeve to cover the Bridge Tube's glass-fritted tip, unless the other side of the tip is covered with solvent or electrolyte. If the tip dries out, it is likely to crack, causing irreproducible results in your electrochemical tests.

Working Electrode Installation

Working Electrode: Overview

An electrochemist is interested in the reactions that occur at or near the working electrode. In some cases, the working electrode is treated as an "inert metal" that functions as a simple source or sink of electrons. In other cases, the properties of the electrode are critical to the tests being performed.

Dr. Bob's Cell can be used with standard electrodes supplied by Gamry Instruments, or custom electrodes made by the cell's user or a third-party vendor. In the latter case, the working electrode must be compatible with either a #7 ACE-Thred™ or a 14/20 ground glass joint. The discussion that follows assumes a 7 mm diameter working electrode mounted in the center port of the cell.

Working electrodes sold by Gamry Instruments include:

Part Number	Description
932-00022	Disk electrode, 3 mm dia., glassy carbon
932-00023	Disk electrode, 3 mm dia., gold
932-00024	Disk electrode, 3 mm dia., platinum
932-00009	Microdisk electrode, 10 μm dia., platinum
932-00010	Microdisk electrode, 10 μm dia., gold
932-00011	Microdisk electrode, 11 μm dia., carbon fiber

All of these electrodes include a 7 mm section suitable for mounting in a #7 ACE-Thred™ fitting. Figure 6 shows Gamry's macro-electrodes.

Figure 6. Macro Working Electrode Assembly

Note that the actual electrode may differ from that depicted above.

Working Electrode: Surface Preparation

Solid electrodes are notorious for problems with surface reproducibility. With use, the electrode surface gets covered with contaminants that can include reaction products, trace impurities in the electrolyte, trace impurities from laboratory air and purge gas, and organic compounds from a chemist's handling of the cell.

A lot of care is often required to prepare a clean electrode surface. One of the most common techniques to clean an electrode is polishing the electrode surface with a very fine abrasive. This removes a thin layer of electrode material and insulator from the electrode face. Usually you do not polish your electrode to remove scratches from the surface—you polish to remove accumulated surface contamination.

Gamry provides a electrode-polishing kit to use to resurface your electrodes. The part number is 990-00195. It contains a flat glass plate, polishing cloths, and a bottle of alumina suspension.

Polishing can leaving polishing abrasive on the electrode surface. Many electrochemists recommend ultrasonic cleaning to remove this residue. Ultrasonic cleaning is generally done in pure solvent (for example ultra-high-purity water).

You can also clean electrodes chemically and electrochemically. The specific cleaning regimen required before a given test is dependent on many factors, so this manual cannot give specific information.

Working Electrode: Installation

The Working Electrode is generally the last part inserted into the cell. If you are deoxygenating your test solution, you might want to purge the cell before you place the Working Electrode into the solution.

Gamry's standard electrodes all mount in the central #7 ACE-Thred™ port on top of the cell. Custom electrodes with an electrode diameter between 6.5 and 7.5 mm can mount in the same port. If your electrode is smaller than 6.5 mm, we recommend that you add plastic heat-shrink tubing or a machined plastic bushing to increase the diameter in the area where the ACE-Thred™ fitting will grip the electrode body.

You must slide a FETFE O-ring and an ACE-Thred™ fitting onto the electrode body before the electrode can be installed into the cell. The electrode in Figure 6 has this piece properly installed.

Slide the electrode into the cell until the tip is immersed in the electrolyte, then tighten the ACE-Thred™ nut. If your working electrode has both a lower glass and an upper plastic region, we recommend that only the glass end contact the solution.

Spare Port

The normal cell configuration includes a spare 14/20 port. If this port is truly not used, you can cap it with one of the 14/20 plastic stoppers included in your cell kit. Remember to poke a vent hole in this stopper if you will have gas flowing into your cell and there is no other gas vent available.

You can also use this spare port for customizing your system. A few examples are listed below.

Addition of reactants

In many experiments, you record a baseline curve before you add a vital reactant to the cell. You then add that reactant, stir the cell, then record another curve. Many of Gamry's analysis packages allow you to subtract the baseline curve from the data curve. The resulting curve shows only electrochemistry related to this reactant.

Another common use for added reactant is current-versus-concentration studies.

To add reactant, remove the stopper, add the reactant, then replace the stopper.

Temperature Sensing and Control

The rate of almost all chemical reactions is strongly temperature-dependent. For this reason, you might want to either measure or control the temperature of your cell.

Ace Glass Inc. has thermometer adapters that are designed to mount a standard lab thermometer to a 14/20 ground glass joint. One example is Ace Glass Inc. part number 5028-26**.** This is an convenient way to add temperature measurement to your system.

In many cases, temperature must be controlled, not measured. One way to do this is:

1. Purchase the jacketed option for Dr. Bob's Cell. This is a special cell body (Gamry part number 930-00037) that allows a flowing temperature-transfer fluid to encase the cell.
2. Plumb the jacket on the cell to a re-circulating constant-temperature bath
3. If the bath offers remote temperature sensing, place a sensor in the spare port of the cell. This may require purchase of another option (for example, an RTD-to-14/20 port adapter).

In some cases, Gamry's software supports automatic temperature setting. The controlled temperature is added to the experiment's **Setup** window, and the software controls the temperature bath via an RS-232 port.

Addition of a pH electrode

Another possible use for the port is addition of a pH electrode. An adapter is required.

Getting a Stir Bar into the Cell

If you want to magnetically stir, and you forget to add a stir bar to your cell, you can add it through the spare port.

Moving the Pieces About

There are several circumstance in which the standard cell configuration may not work. One is a larger-than-normal working or reference electrode.

There is no requirement that any electrode or adapter mount in any specific port. If your working electrode diameter is larger than 7.5 mm, you may be able to insert it into the cell through the spare port.

Electrode Connections

If you are using your Dr. Bob's Cell with a Gamry Instruments Potentiostat, make the following connections to the electrodes.

The Reference Electrode lead plugs into the white pin jack on the cell cable.

The green and blue leads from the cell cable are attached to the Working Electrode. Cell currents in a Dr. Bob's Cell experiment are normally small enough that the blue and green leads can be clipped together, then clipped to the working electrode.

Some potentiostats may not include a blue clip lead. In this case, you only connect the green lead to the Working Electrode.

The red lead on the cell cable clips to the Counter Electrode. A sharp bend in the platinum wire just at the rim of the counter-electrode tube will help keep the platinum wire in place.

Make sure that the long black lead on the cell cable cannot touch any other cell connection. You may find that connection of this lead to a source of earth ground, such as a water pipe, reduces noise in your experimental results.

If you are measuring very small currents, you may find that a metal enclosure completely surrounding your cell further reduces noise. In this case connect the shield, known as a Faraday Cage, to an earth ground. The black lead from the cell cable is then connected to the Faraday Cage.

Gamry's VistaShield™ is a versatile, easy-to-use Faraday Cage that was designed to work well with Dr. Bob's Cell. When equipped with a Purge and Stir option, it provides a complete electrochemistry test stand, well-integrated with Gamry's cells, potentiostats, and software systems.

Always double-check your cell connections. Even an experienced experimenter will occasionally leave one of the cell cable leads lying on the desktop.

Troubleshooting

By far, the most common source of problems is lack of a connection between a cell lead and the cell electrolyte. The lack of connection can be between the potentiostat and the electrode or between the electrode and the electrolyte.

One very common and often embarrassing error is to forget to connect one of the cell leads! Always double-check your cell connections. Even an experienced experimenter can leave one of the cell cable leads lying on the desktop.

A more subtle problem is a gas bubble or air gap blocking an electrode's access to the electrolyte. Places where this can happen include:

- The face of the working electrode, where purge gas or gaseous reaction products collect.
- The counter electrode, if the wire is too high and has pulled out of the solution.
- The counter isolation tube, if it does not fill with electrolyte.
- The Reference Bridge tube, if it contains a bubble between the reference electrode and the electrolyte.
- The glass frit at the end of the reference electrode, where a bubble may collect.

Another common problem is two electrodes shorting together (coming into mutual electrical contact). This can occur within the cell (especially with a bare-wire counter electrode). It can also occur between the cell cable connections.

The following section of the manual is organized as a list of problems that you may encounter. Following each problem is a list of some possible causes for that problem. Neither the list of problems nor the list of their causes is comprehensive.

This troubleshooting guide only applies if you are running a potentiostatic experiment on the cell. Galvanostatic experiments show different symptoms.

Very small current or no current when you run an experiment but no overload indication

- The working electrode (green) lead in the cell cable is not connected to the cell properly.
- There is a gas bubble on the face of the working electrode.

  Stop the experiment, fix the error, and restart. The working electrode is not damaged.

Very small current or no current when you run an experiment, with a control amplifier overloaded

- The counter electrode (red) lead in the cell cable is not connected to the cell properly.
- The counter electrode is partially pulled out of the cell.
- The counter electrode isolation tube has not filled with electrolyte.

  Stop the experiment, fix the error, and restart. The working electrode is not damaged.

Full-scale current and voltage when you run an experiment, overload indications seen

- The reference electrode (white) lead in the cell cable is not connected to the cell properly.
- The working sense (blue) lead in the cell cable is not connected to the cell properly.
- You have incorrect experimental settings (e.g., wrong potential).
- You have a high-impedance frit (dirty or filled with water)
- There is a gas bubble in the Luggin capillary or blocking the face of the glass frit.

  Large currents have passed through the working electrode. It may need to be resurfaced.

Noisy Cell Current, overloads may be present

- Your de-oxygenation gas is still bubbling through the solution.
- You have a high impedance in the reference electrode path.
- There is a gas bubble in the Luggin capillary.
  You are picking up noise; try a Faraday Cage.

Excess back pressure required to bubble deoxygenation gas

- No vent is available for the gas to escape.

Specifications

Cell

Volume	50 mL maximum	
	2 mL minimum	
Port Types	Two 14/20 standard-taper ground-glass Three #7 ACE-Thred™	

Reference Electrode

Size	9 to 11 mm diameter	(using the standard bridge tube)

Working Electrode

Diameter	6.5 to 7.5 mm	(using central #7 ACE-Thred™ port)
Length	50 mm minimum	(using large cell volumes)
	120 mm preferred	(to use electrolyte volumes below 5 mL)

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