ParaCell

Electrochemical Cell Kit

Operator's Manual

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988-00017

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If You Have Problems

Please visit our service and support page at www.gamry.com/service-support/. This page contains information on installation, software updates, and training. It also contains links to the latest available documentation. If you are unable to locate the information you need from our website, you can contact us via email using the link provided on our website. Alternatively, you can contact us one of the following ways:

Internet	www.gamry.com/service-support/
Telephone	(215) 682-9330 9:00 AM - 5:00 PM US Eastern Standard Time
	(877) 367-4267 Toll Free US & Canada Only

Replacement parts for this kit are available from Gamry Instruments, Inc. Do not ask us to supply metal samples in materials other than C1018 Mild Steel. See the Introduction section of this manual for a source of metal samples.

Disclaimer

The information in this manual has been carefully checked and is believed to be accurate as of the time of printing. However, Gamry Instruments, Inc. assumes no responsibility for errors that might appear.

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Introduction

The Gamry Instruments ParaCell™ was designed for simple, reliable operation. The cell is normally used to run electrochemical tests on flat (conductive) specimens. You can also customize the cell for use with other sample types.

> **NOTE: A reference electrode is not included in the cell kit. Requirements for this electrode vary too much user-to-user to make its inclusion in the standard kit practical. Gamry Instruments sells three types of reference electrodes (SCE, Ag/AgCl, and Hg/Hg_2SO_4) that are suitable for use with your ParaCell kit. Your reference electrode should be ordered separately.**

The ParaCell uses the two end plates and one of the four 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 both heterogeneous and homogeneous 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 sells a special jacketed version of the ParaCell cell body. When this cell is connected to a circulating water bath, accurate temperature control of your experiments becomes possible.

Chemical Compatibility of the ParaCell

The components in the ParaCell were selected to be sufficiently chemically inert to handle typical experimental conditions. In normal use the materials in contact with the test solution are:

- the Working Electrode,
- borosilicate glass (Pyrex® or equivalent),
- unfired Vycor®,
- PTFE (Teflon®),
- polycarbonate
- ACE Glass's FETFE o-ring material
- spectroscopic grade graphite

Chemical resistance tables for most of these materials are available (try searching the Internet). 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 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.**

Gamry's Paracell was not designed for use in electrolytes that dissolve glass (extremely basic solutions or HF containing solutions) or organic solvents which could damage the polycarbonate body. Be aware that some biological molecules will "stick" to the cell body.

> **CAUTION: The glass components in the cell and the Vycor™ frits used in the reference bridge tube are not suitable for use with extremely basic solutions or solutions containing hydrofluoric acid.**

Unpacking and Checking Your ParaCell

This section is primarily intended for the user who has just received a new ParaCell.

Checking for Shipping Damage

Your ParaCell is shipped partially disassembled to prevent 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 ParaCell, please check it for any signs of shipping damage. Be especially careful if the shipping container shows signs of rough handling.

Obviously, the glass is the most susceptible to damage. Check the glass 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 have been broken in shipment, please contact us as soon as possible for replacement. Our phone number and address are located just inside the Title page of this manual. 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 ParaCell packing list in Table 1. When shipped, all of the ParaCell components should be labeled with their Gamry Instruments, Inc. part number.

If you are checking the completeness of an older kit, you can identify the components by name using the illustrations in Figures 1 and 2 later in this manual.

Table 1
ParaCell Packing List

Quantity	Gamry Part Number	Description	Figure 1 Label
1	988-00017	Manual, ParaCell	-
2	820-00053	End Plates (polycarbonate)	A
2	820-00054	Foot Bracket (stainless steel)	B
1	820-00055	Cell Body (polycarbonate)	C
2	820-00056	Sample Bracket (stainless steel)	D
2	935-00079	Teflon encapsulated silicone O-ring (body)	-
2	935-00080	Teflon encapsulated silicone O-ring (sample)	-
1	930-00057	Reference Electrode Bridge tube w/ Vycor Tip(incl. #11 bushing w/ o-ring)	E
1	935-00078	Graphite plate counter electrode	F
1	935-00053	#11 Bushing, w/ o-Ring	G
4	935-00052	#7 Bushing, w/ o-Ring	H
2	935-00074	#7 Plug	I
3	930-00042	Spare Vycor Frit w/ Teflon sleeve	

Contact us as soon as possible if any of the parts are missing. Our address and phone numbers can be found immediately following the title page of this manual.

Assembly

This section of the manual tells you how to assemble the kit's components into a complete ParaCell. The descriptions are based on a "standard" cell configuration consisting of a flat metal sample working electrode, a flat graphite pad counter, and a single junction reference electrode in a reference bridge tube. A gas dispersion line can be added but is not included in the standard cell kit.

Feel free to customize your cell configuration. You are only limited by your imagination, the number and size of the ports available, and your willingness to drill some holes.

Cell Assembly- General Information

An assembled cell can be seen in Figure 1. The basic setup shown makes use of the two end holes and one of the four available ports. The cell must be assembled prior to filling. Filling can be done through the threaded holes or by clamping shut one end, holding the cell upright and filling through the other hole. Working (and counter) electrodes will need to be readied if not in place prior to filling.

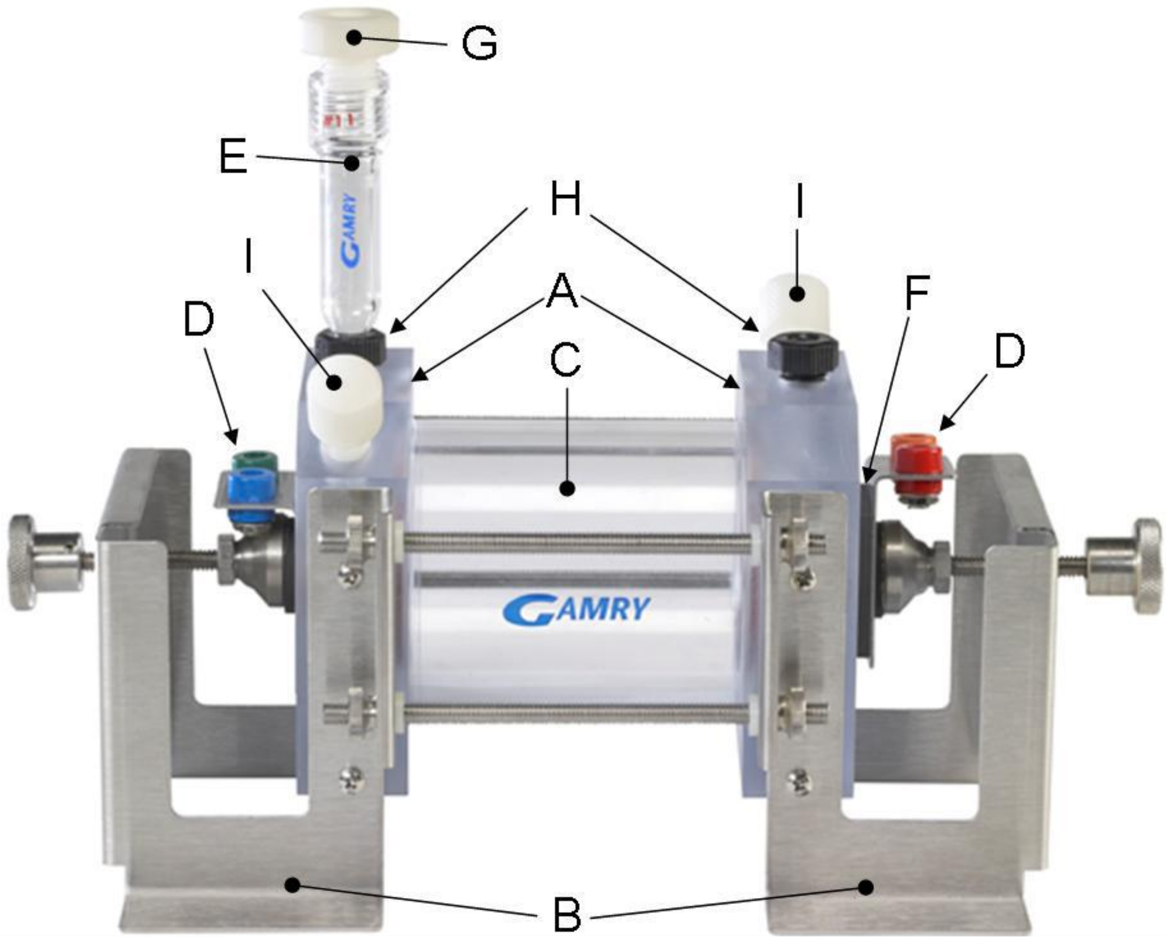
If you are assembling your ParaCell for the first time, you may want to do a leak check after assembly. Because this cell is not designed to be pressurized, this is most easily accomplished by adding a small amount of dye to water, filling the cell and setting it on some paper towels. A slow leak may not leave the paper towels damp but will leave behind the color.

Along with the two electrode/sample holes at on each cell end, there are four threaded joints. These are a #7 internal thread and the standard #7 bushings and o-rings will fit them. Working and counter (or second working) electrodes are designed to be clamped in the end plates. A reference electrode in the included bridge tube should go through the top #7 port on the same end plate as the working electrode. Gas dispersion can be accomplished by running a bubbling line into one of the other threaded ports; make sure that there is an open vent.

You may need to pay attention to cell cleanliness. In many corrosion testing situations, contaminants in the cell and test solution are not a problem if you take minimal precautions. In other cases, trace contaminants can lead to poorly reproducible results. One example is a study of corrosion in tap water.

If you touch the cell components with your fingers, you can inadvertently add chlorides and organic compounds to your cell solution. We recommend that you carefully clean the cell components using good laboratory practices. Once the components are clean avoid touching their wetted surfaces.

Figure 1
Assembled Cell (See Table 1 for Labels)



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

> **CAUTION: ACE-Thred fittings should always be tightened "finger tight". Over-tightening a fitting can get it stuck.**

A given ACE-Thred size can only accommodate specific diameter objects. A #7 ACE-Thred is specified to work with object with a diameter between 6.5 mm and 7.5 mm. If you need to add non-standard options to your ParaCell kit, make sure you keep this restriction in mind.

Main Cell Assembly

Assembly of the cell involves placing the cell body o-rings into the groves on the end plates. Then slotting the body (cylinder) into those groves and drawing the assembly tight with the four threaded rods. Make sure that the plastic "hats" are properly seated so that the metal rods nor nuts are in contact with the metal of the end bracket.

Finger tightening is generally sufficient to prevent leaks. Once the cell is drawn together final tightening should be done on diagonal pairs to produce a good seal against the orings.

> **CAUTION: The cell requires modest, even clamping force. Do not tighten beyond what is necessary to prevent leaks. Overtightening can result in damage to the cell.**

Assembly for the jacketed version is the same but with the jacketed cell body replacing the standard. Make sure that the hose barbs are angled so that the ParaCell will rest flat with the outlet oriented as near to the top as possible.

Stirring With a Magnetic Stirrer

If you want to magnetically stir, it is best to add the stir bar during main cell assembly or prior to adding the working/counter electrodes. Due to the shape of the cell bottom, egg shaped or cross type stir bars work best. Small bar stirrers will turn but will not provide much stirring. If you forget to add a stir bar to your cell, you can add a small one using a spare port.

Bridge Tube and Reference Electrode

The bridge tube will fit any of the #7 ports on the top, but normal use is via the top port on the working electrode side. It can be adjusted in its depth and should generally be positioned with the tip in line with the working electrode exposed area.

The bridge tube allows the reference electrode to be located outside the test solution, isolating it from thermal gradient experiments and more caustic test solutions. Ensure that the brdge tube is filled all the way to the tip with a conductive solution (the test solution when possible) for ideal performance of the reference electrode.

Insert the reference electrode into the #11 thread at the upper end of the bridge tube. It must contact the test solution inside the tube. Various reference electrodes that work with this system are available. Contact us for details.

Figure 3
Reference Bridge Tube

Many experiments do not require a "true" reference electrode to be run. If a pseudo- or quasi-reference electrode is sufficient you do not need to use the reference bridge tube.

Counter Electrode

The standard Counter Electrode is a plate of high density graphite. To fit the counter electrode to the cell, simply clamp it over one of the end plate holes (normally the red/orange side).

The graphite plate that is shipped with your ParaCell is spectroscopic grade. It is very pure and is therefore unlikely to be a significant source of contamination in your initial experiment. However, it is somewhat porous and can adsorb substances present in your test solution. Reuse of a graphite counter can contaminate your test solution. The effect is small, and you are unlikely to see it unless the test solution changes drastically between tests, the effect can also be ameliorated by sanding the surface with a fine grit sandpaper.

If this is a concern to you, consider a platinum counter: platinum foil (or a PT coated metal sample) can be substituted in the same way. If you have a a platinum wire or mesh this can be introduced to the cell body through one of the threaded holes. Mesh in particular may require this be added prior to main cell assembly. If you are not using a (counter) electrode on the end plate opposite the working then make sure to block that hole with a non-conductive material such as a small square of glass.

Gas-flow Overview and Terminology

Gas dispersion 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. Its reduction can act as the cathodic half reaction in a corrosion reaction. You will probably want to remove oxygen from the solution whenever the real world system that you are modeling is oxygen free.

You remove oxygen from the test solution by bubbling nitrogen, or another electrochemically inert gas through the solution. This process is often (imprecisely) called deaeration. It is more correctly called deoxygenation. At least 1/2 hour of vigorous bubbling with nitrogen will be 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. Depending on how you have the cell setup it may be possible to flow gas over the top of the solution while doing your electrochemistry; this is often referred to as "blanketing" the cell. In general, blanketing is used after solution purging, where blanketing prevents acquiring new 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' PV220 and PHE200 systems. These systems output 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 a 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 significant source of error in some experiments. This problem can often be avoided by pre-saturation of the purge gas with the solvent prior to it entering the cell. This is commonly done using a "gas washing bottle", which can be obtained at most laboratory supply companies.

Gas Dispersion Apparatus

In the ParaCell gas flow control apparatus is not included. Outlined is one way to handle gas flow. Use a thin flexible tubing (Teflon or other) for the gas feed line. With the cell assembled run the feed line in through the angled threaded access port on the counter side. Feed through to the bottom of the cell, as near the center as possible. Leave the top threaded port on the counter side open for venting. A fritted 6mm glass tube can also be used for the feed gas but will be less efficient due to the cell geometry. Note that the cell geometry will mean that extra time should be allotted to purging in all cases but particularly if the purging is being done to the side (as with a fritted gass tube).

When purging, the vent function is critical. Whenever gas is flowing into the cell, you must provide a way for it leave the cell. If you, do not, the gas may not flow or worse, the cell may pressurize, which could cause leaking. Not providing a vent for the escape of purge gas is a very common and potentially hazardous "mistake" made when setting up an electrochemical cell. There are vent points set high into the end plates which feed into the top threaded ports. So long as one of those is cleared the cell can vent properly.

> **CAUTION: If you use purge and/or blanket gas, you <u>must</u> provide a vent for the gas to escape the cell. The ParaCellwas not designed to withstand gas pressure! Failure to vent the cell can cause damage to the cell, uncontrolled loss of electrolyte from the cell, and risk of personal injury to the cell's operator.**

Attaching Gas Tubing to the Cell

Your gas flow system should include a needle valve to control the gas flow rate. All gas tubing connections to the cell should be made with this valve <u>turned all the way off</u>. Making connection with a cell filled with electrolyte or adding electrolyte to a system when the gas flow is on can lead to accidents.

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

The gas flow system should be connected and the cell electrolyte added before the needle valve is turned on. The valve should be opened slowly, while you watch the bubbles in the cell. In addition to the needle valve, a 3-way valve is very useful in purge and blanket gas control. 3-way valves are available in both electrically switched and manual versions. A 3-way valve switches one gas stream so it flows from a single inlet to one of two outlets.

If your system includes a 3-way valve for switched purge and blanket gas control, you can create a setup with both purge and blanketing options depending on solution fill level and your available adapters/tubing/glassware. Check to make sure that venting is available to both purge and blanket gas flow.

Unused Ports

More ports than are strictly necessary are available. These may be used for customization by the user (see later). In most experiments, however, one or mor port will be unused. It is necessary to keep one (top) port open for venting when bubbling. Otherwise it is recommended that spare ports be kept plugged to prevent solvent evaporation.

Working Electrode (Sample)

Because the working (and counter) electrodes form the end seals for the ParaCell, they must be in place prior to filling the cell with test solution.

You should take great care to insure that the surface of your Test Sample is not altered prior to the test. Avoid contacting the sample with your fingers. You may want to clean/degrease the electrodes just prior to starting your test.

Sample surface finish and other sample preparation are critical if you want to obtain reproducible results. Consult the corrosion measurement literature for details about the handling of corrosion test specimens. Most of the surface preparation techniques used for weight loss coupons are also applicable to electrochemical test specimens.

To mount the electrodes to the ParaCell simply position them over the opening and tighten the leveling sample bracket down until the sample is well sealed against the o-ring. Hand tight should be sufficient to seal the electrode. It may be easiest to center the working electrode(s) with the ParaCell disassembled.

The ParaCell is designed for use with flat samples that are conductive through the sample—from the solution interface to the back wall in contact with the leveling sample bracket. When this is the case the banana jacks in the brackets can be used for electrical connection to the potentiostat. The colors are coded for Gamry potentiostats with the working side having green and blue banana jacks, and counter side having red and orange, but the coloring is only a visual aid; the cell can be connected in other manners.

Flat samples that are not conductive through to the back side can be used but an alternate electrical connection method must be employed.

Sample Masking

While the opening sealed with the included o-ring does define an electroactive area, in many cases it is advised to mask off a somewhat smaller area on the sample surface. When sealing to a flat sample with an o-ring or gasket, a crevice region is formed. This area will have unusually high activity for corrosion and will also affect physical electrochemistry experiments.

Masking minimizes crevice effects that can skew results. Masking an electrode can be done in different ways. PortHole sample masks are available from Gamry Instruments to provide a fixed, known area. It is not necessary to mask the counter electrode in three electrode potentiostatic/galvanostatic experiments. If doing Galvanic Corrosion then both metal samples should be masked in the same way. When masking, make sure the revealed area is centered in the hole when mounting the electrode.

Electrode Connections - Corrosion

If you are using your ParaCell with a Gamry Instruments Potentiostat, the following connections are normally made to the electrodes.

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

The Green, Blue, Red and Orange leads are plugged into their respective colored jacks (when using thru-conductive samples setup with the working electrode on the green/blue side). For most experiments the counter sense lead (orange) is not necessary, but it is nice to have some place to put it. Some potentiostats do not have a separate work sense lead. While this does make for less accurate voltage measurement/control, it doesn't affect the use of this cell. You will simply have only one lead to attach to the working electrode side.

Make sure that the Black lead on the cell cable cannot touch any other cell connection, or other metal of the cell body/sample(s). You may find that connection of this lead to a source of earth ground, such as a water pipe, will reduce noise in your experimental results. If you are measuring very small currents, you may find that a metal enclosure completely surrounding your cell will further reduce noise. In this case, the shield, known as a Faraday cage, must be connected to the potentiostat's ground lead and may be connected to earth ground as well. The ParaCell works with Gamry's VistaShield™ Faraday Cage, which allows the user to see/watch the cell without breaking the shielding.

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

If you are using the ParaCell with a potentiostat sold by a different manufacturer, consult that potentiostat's documentation for electrode connection information.

Electrochemical Noise and Galvanic Corrosion

The ParaCell was designed to work for a wide variety of experiments but it is ideal for Galvanic Corrosion and Noise experiments. These are ZRA (Zero Resistance Ammeter) mode corrosion experiments that measure the current passed between two electrodes of equal size. In Galvanic Corrosion the metals will be different, whereas in Noise they are the same.

ZRA mode makes use of the counter sense lead (orange) from a Gamry potentiostat. For galvanic corrosion and other ZRA-mode experiments with non-Gamry ZRA's the reference replaces the counter sense lead (Work-Work Sense on one electrode, Reference-Counter on the other).

Customizing Your Cell

The ParaCell was designed to have options available even for a user who uses two references and two "working" electrodes. There are six total ports and only three or four necessary for most experiments. With 2 to 3 spare access ports and full access with the cell dissassembled, customization options are myriad. Some common modifications to experiments are mentioned below.

Addition of Corrosive Agents

Spare ports can be used to add reagents. 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.

Temperature Sensing and Control

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

Many thermometers will fit the standard bushing for a #7 thread. 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:

- you use the jacketed option for the ParaCell. This is a special cell body that allows a flowing temperature transfer fluid to encase the cell.
- you plumb the jacket on the cell to a re-circulating constant temperature bath
- if the bath offers remote temperature sensing, you place a sensor in a spare port of the cell. This may require purchas of a different adapter.

In some cases, Gamry's software supports automatic temperature setting. The controlled temperature is simply added to the experiment's Setup window and the software controls the temperature bath via an RS232 port. In other cases, a modified script is required.

Addition of a pH Electrode

Another possible use for the space port is addition of a pH electrode. Again, an adapter may be required.

Selected Specifications

Cell

Volume	300 ml standard operating volume	
Port Type	End Plate Holes	#7 thread
Number of Ports	2 (o-ring sealed)	4 (2 vented)

Working Electrode(s)

Exposed area 2.85 cm^2 (0.44 in^2) nominal

Troubleshooting

This 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.

> NOTE: This troubleshooting guide only applies if you are running a potentiostatic experiment on the cell. Galvanostatic experiments will 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 completely blocking 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 amp overload

- 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.

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

Full scale current and voltage when you run an experiment, many overloads

- The reference electrode (white) lead in the cell cable is not connected to the cell.
- The working sense (blue) lead in the cell cable is not connected to the cell.
- You have incorrect experimental settings (e.g. wrong potential).
- Two of your electrodes are shorted together.
- There is a gas bubble in the Reference Bridge Tube.

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

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 Reference Bridge Tube.
- You are picking up noise – try a Faraday cage.

Excess back pressure required to bubble deoxygenation gas

- No path is available for the gas to escape.

Poor Experimental Reproducibility

- A poor working electrode seal is allowing some leaking of the test solution.
- Your cell, solution, or working electrode surface has a contamination problem. Carefully clean the cell and components. Avoid touching the wetted surfaces of these parts.
- Contaminants are entering the cell from the graphite counter electrode.
- Your electrochemical system is inherently irreproducible. Often true of localized corrosion phenomena.

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