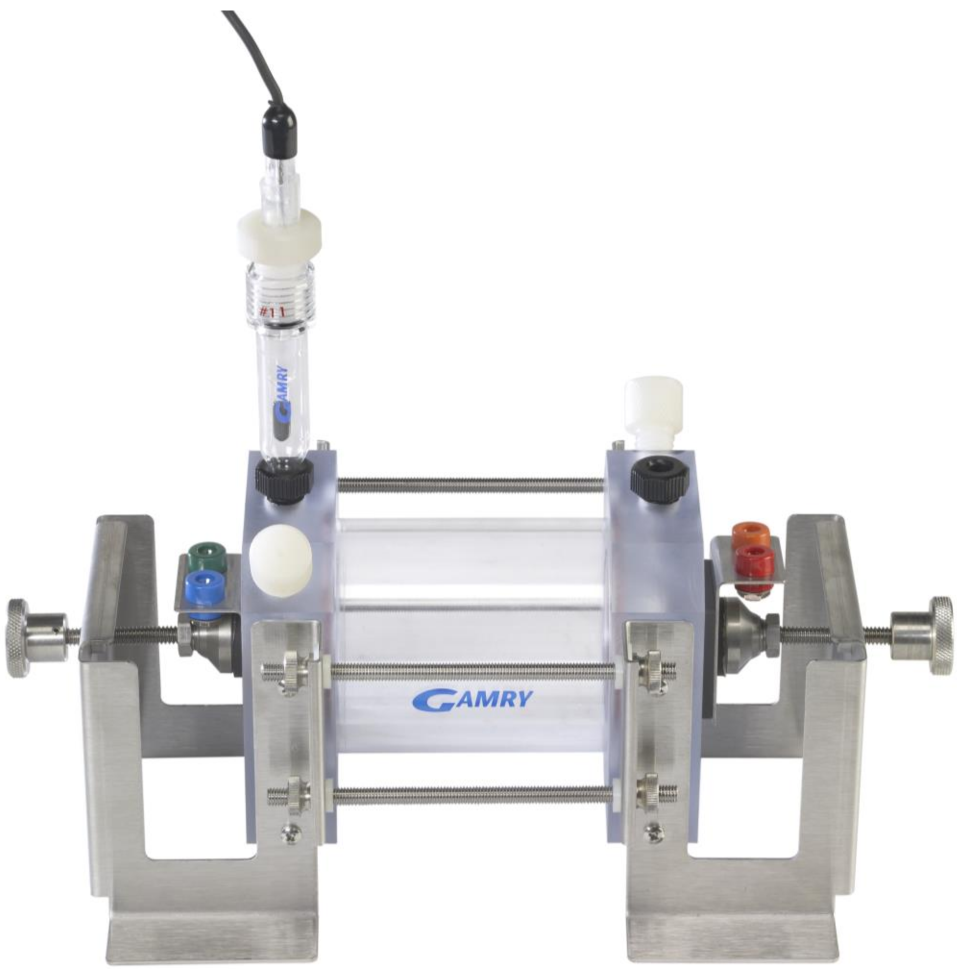
ParaCell™

Electrochemical Cell Kit

Operator's Manual



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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. 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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ParaCell

Operator's Manual

Revision 1.41
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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.

A reference electrode is ***not*** included in the cell kit. Requirements for this electrode vary too much from 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. Order your reference electrode 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.

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 glass frit,
- PTFE,
- 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 glass 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 new ParaCell 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 ParaCell 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.

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.

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 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 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	PTFE encapsulated silicone O-ring (body)	-
2	935-00080	PTFE encapsulated silicone O-ring (sample)	-
1	930-00057	Reference Electrode Bridge tube with glass-frit tip (incl. #11 bushing with o-ring)	E
1	935-00078	Graphite plate counter electrode	F
1	935-00053	#11 Bushing, with O-Ring	G
4	935-00052	#7 Bushing, with O-Ring	H
2	935-00074	#7 Plug	I
5	955-00003	Spare glass frit with PTFE 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.

General Information

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

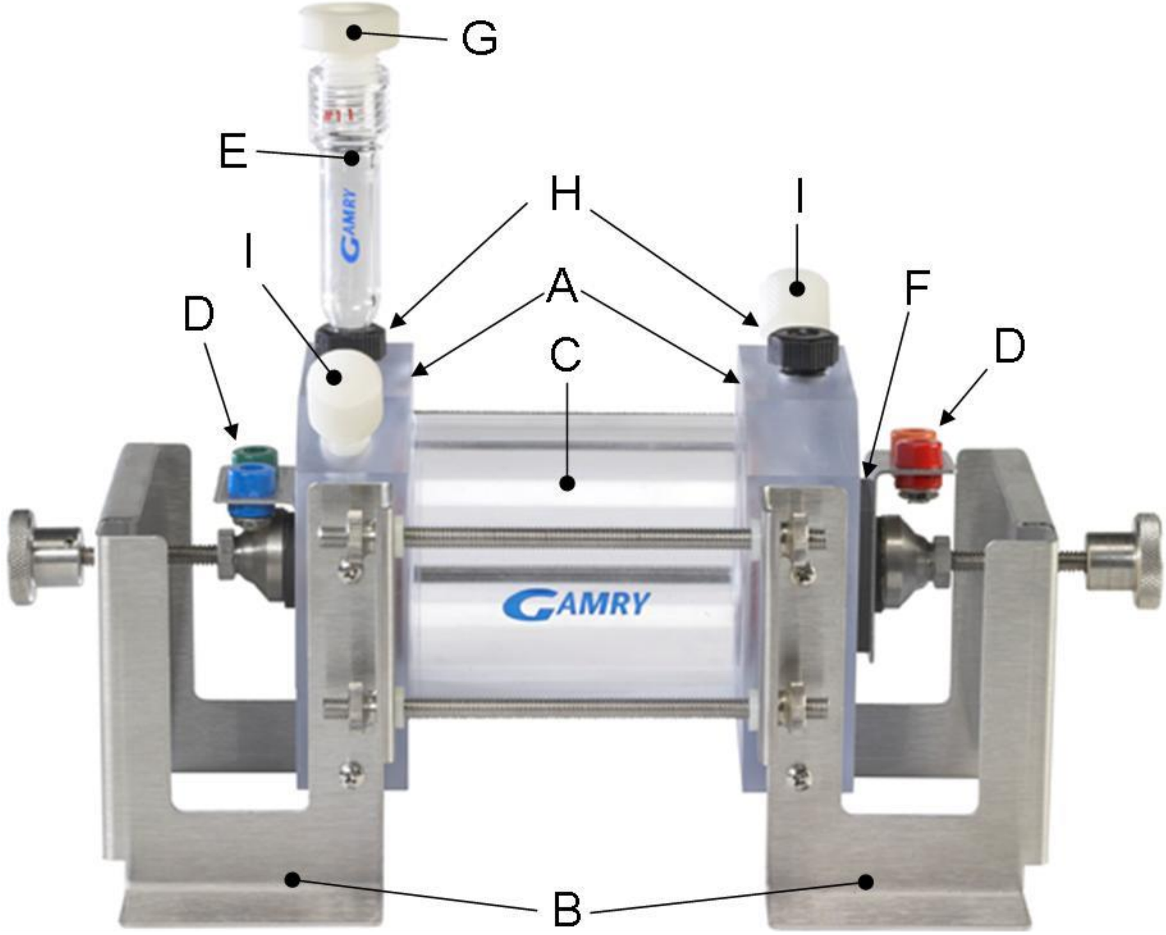
If you are assembling your ParaCell for the first time, you may want to check for leaks after assembly. Because this cell is not designed to be pressurized, this is most easily accomplished by adding a small amount of dye to water, filling the cell, and setting it on some paper towels. A slow leak may 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 have a #7 internal thread; the standard #7 bushings and O-rings 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.

Pay careful 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 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.

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 bodies of plastic electrodes. ACE-Thred fittings™ are designed to be tightened with finger-pressure only.

> **Caution:** ACE-Thred™ fittings should always be tightened "finger-tight". Never use tools such as a wrench or pliers to tighten an ACE-Thred™ fitting. Over-tightening a fitting can break the cell.

A given ACE-Thred™ size can only accommodate specific diameter objects. A #7 ACE-Thred™ is specified to work with objects 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

1. Place the cell body O-rings into the grooves on the end plates.

2. Slide the body (cylinder) into those grooves.

3. Draw 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. After the cell is drawn together, perform final tightening 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**.**

Stirring With a Magnetic Stirrer

If you want to magnetically stir, it is best to add the stir bar during main cell assembly or before adding the working/counter electrodes. Because of 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 fits 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. Position it generally with the tip in line with the working electrode's exposed area.

The bridge tube allows the reference electrode to be placed outside the test solution, isolating it from more caustic test solutions. Ensure that the bridge 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, 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. Any 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 electrode: 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 to be added before assembly of the main cell. If you are not using a (counter) electrode on the end plate opposite the working electrode, 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.

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 half an hour 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. Depending on how you have the cell set up, 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' PV and PHE software. 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 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 (PTFE 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. You can also use a fritted 6 mm glass tube for the feed gas, but this is less efficient because of the cell geometry. The cell geometry means that you must allow extra time for 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. As long as one of those vents is cleared, the cell can vent properly.

Warning: If you use purge of blanket gas, you ***must*** provide a vent for the gas to escape the cell. The ParaCell was 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

Warning: 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 severe accidents. 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 on the needle valve. Open the valve slowly while you watch the bubbles in the cell. In addition to the needle valve, a three-way valve is 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, you can create a setup with both purge and blanketing options depending on solution-fill level and your available adapters/tubing/glassware. Check that venting is available to both purge and blanket-gas flow.

Unused Ports

More ports than are strictly necessary are available. You may use these ports to customize your system (see later). In most experiments, however, one or more port are unused. You must keep one (top) port open for venting when bubbling. Otherwise we recommend that you keep spare ports 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.

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 degrease the metal sample mounted on the Sample Holder 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, 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.

You may use flat samples that are not conductive through to the back side, but you must find an alternate method for electrical connection.

Sample Masking

While the opening sealed with the included O-ring does define an electroactive area, in many cases we advise to mask off a somewhat smaller area on the sample surface. When sealed 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 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 you are 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, make the following connections 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 through-conductive samples set up 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 give less-accurate voltage measurement and control, it does not affect the use of this cell. You just 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). Connecting this lead to a source of earth ground, such as a water pipe, may reduce noise in your experimental results. If you are measuring very small currents, a metal enclosure completely surrounding your cell may further reduce noise. In this case, connect the shield, known as a Faraday cage, to the potentiostat's ground lead and perhaps also to earth ground. The ParaCell works with Gamry's VistaShield™ Faraday cage, which allows you to see 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 instructions for information on how to connect the electrodes.

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 are 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 ZRAs, 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 a total of six ports, but only three or four are necessary for most experiments. With two to three 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

You can add reagents through spare ports. 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

The rate of almost all chemical reactions is strongly temperature-dependent. For this reason, you might want to measure 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.

Addition of a pH Electrode

Another possible use for the space port is addition of a pH electrode. 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 (two 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.

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 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 amplifier 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 leaks 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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Redefining Electrochemical Measurement

Part #: 988-00017

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